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“EVALUACION DE LAS PROPIEDADES MECÁNICAS DE COMPUESTOS BIODEGRADABLES ELABORADOS CON ÁCIDO POLILÁCTICO (PLA) Y REFORZADOS CON CASCARA DE ARROZ”

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RESUMEN

En la actualidad los polímeros biodegradables están siendo utilizados en algunas industrias como la industria del empaquetamiento, el área médica, la fabricación de carcazas de artefactos electrónicos, entre otros. Sin embargo, estos polímeros normalmente no han incursionado en el diseño de elementos más complejos. El ácido polilactico (PLA) es un polímero termoplástico degradable presenta buenas propiedades físicas, buena resistencia a la tracción, sin embargo su flexibilidad es baja, su costo es alto; por tanto en el presente proyecto se ha estudiado diferentes materiales compuestos a base de un polímero biodegradable como el Ácido Polilactico (PLA) y Cascara de Arroz obtenidos por moldeo por compresión, con el objetivo de producir un nuevo material compuesto con Ácido Poliláctico (PLA) y fibras naturales nacionales como la cascarilla de arroz de manera que el nuevo material compuesto contenga mejores propiedades mecánicas y reológicas que el material base que es el PLA, ademas al usar la cascara de arroz, el cual es un residuos agroindustrial, se aminorarian los costos.

Como resultado de este estudio de obtuvo mejoras de hasta un 30.11 % y 5.47% en la resistencia a la flexión y dureza, respectivamente. Además las mezclas muestran un aumento de la fluidez con la adición de la cascara de arroz lográndose aumentar el índice de fluidez hasta un 28.79 % para una carga de 3.75 %.

Por otro lado existe una disminución de la resistencia a la tracción desde un 43.29% hasta un 66.48% al aumentar la carga (g) de cascara de arroz en 2.5 % y 5 % respectivamente.

ABSTRACT

Today biodegradable polymers are being used in some industries such as packaging industry, the medical field, manufacturing housings of electronic devices, among others. However, these polymers typically have not penetrated in the design of more complex elements. The polylactic acid (PLA) is a biodegradable thermoplastic polymer has good physical properties, good tensile strength, but flexibility is low, the cost is high; therefore in this project it has studied various composite materials based on a biodegradable polymer such as polylactic acid (PLA) and rice husk obtained by compression molding, in order to produce a new composite material with polylactic acid (PLA) and domestic and rice husks so that the new composite material better mechanical and rheological containing the base material is the PLA, in addition to using rice husk, which is an agro-industrial waste properties, natural fibers would lessen the costs.

As a result of this study improvements of up to 30.11% and 5.47% in the flexural strength and hardness, respectively it was obtained. Moreover the mixtures show an increased flowability with the addition of rice husk achieving increase the melt index up to 28.79% for a 3.75% load.

On the other hand there is a decrease in tensile strength from 43.29% to 66.48% with increasing load (g) rice husk 2.5% and 5% respectively.

DEDICATORIAS

Dedico mi tesis a Dios, por darme su bendición y fortaleza para alcanzar mis objetivos y metas. A mis queridos padres que han sabido formarme con buenos hábitos, sentimientos y valores, apoyándome en todo momento para salir adelante. A mi hermana, quien siempre estuvo brindándome su afecto y alegría, A mi querido abuelo Julio Gonzales que con mucho amor, paciencia y dedicación siempre estuvo a mi lado.

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I.- INTRODUCCIÓN

En las últimas décadas la tecnología va avanzando a pasos agigantados y es necesario cuidar el medio ambiente, debido a que nuestros recursos naturales como el petróleo se están agotando alarmantemente. Para lo cual es necesario desarrollar nuevas líneas de investigación en la elaboración de productos en base a recursos renovables o sostenibles, con propiedades biodegradables y compostables que tengan menos impacto en el medio ambiente al final de su vida útil.

En el siglo XX el plástico revoluciono al mundo con los productos derivados del petróleo conquistando el mercado con bajos costos, por presentar estabilidad química, bajo peso y fácil fabricación. Por otro lado algunas de estas propiedades producen daños medioambientales por ser un producto desechable y posteriormente se queda en el medioambiente por su resistencia a la degradación.

En los últimos años el alza de precios en los combustibles fósiles, su escases y el efecto provocado en la atmosfera, los productos renovables permitieron redescubrir los polímeros, a los que hoy en día se les da la debida importancia. Un ejemplo es el caso del PLA.

Se utilizan recursos renovables como la remolacha, el trigo, maíz y otros productos ricos en almidón para obtener ácido láctico y a partir de este fabricar PLA la cual es una sustancia con muchas aplicaciones y es un polímero [1]. Este polímero biodegradable (PLA) posee varias aplicaciones potenciales y además es de fácil transformación. El PLA se utiliza desde hace 25 años en Medicina. [2, 5].

II.- OBJETIVOS

-Objetivo General

Producir un nuevo material compuesto con Ácido Poliláctico (PLA) y fibras naturales nacionales como la cascarilla de arroz de manera que el nuevo material compuesto contenga mejores propiedades mecánicas y reológicas que el material base que es el PLA.

-Objetivos Específicos

1) Comparar las propiedades mecánicas y reológicas entre el PLA y el material compuesto obtenido mediante experimentación.

2) Determinar los efectos principales que afectan a la obtención del material compuestos con fibras naturales como la cascara de arroz.

3) Analizar la posibilidad de producción del compuesto a nivel industrial tomando como base la combinación de dos materiales amigables al ambiente.

III.- JUSTIFICACIÓN

En la Universidad Católica de Santa María en el laboratorio de Ensayo de Materiales se tiene como linea de investigación el estudio de materiales compuestos en base a materiales biodegradables, una experiencia de esta linea a sido PLA combinado con fibra de soga. Tomando como base esta experiencia el objetivo de este proyecto es preparar un material compuesto en base a PLA y fibra de cascara de arroz, ambos componentes son amigables al ambiente. La tesis estará centrada en analizar si existe alguna mejora al incorporar las fibras de cascara de arroz.

CAPÍTULO I

MARCO CONCEPTUAL Y TEÓRICO

1.1 POLÍMEROS

Podemos definir a los polímeros como materiales formados por moléculas o unidades repetitivas, las cuales pueden estar dispuestas de una forma regularmente ordenada. Se le denomina macromoléculas por estar formado por meleculas de gran tamaño. El término “Polímeros” deriva del griego “poli”=mucho y “meros”=partes. Se denomina oligómero a la molécula que contiene pocas unidades repetitivas, ya que “oligo”=poco.

Las unidades repetitivas, en los polímeros, se nombran como la Unidad Constitucional Repetitiva (UCR) [5]. Se denomina grado de polimerización (X) al número de UCR que se repite en la cadena, el peso molecular de un polímero está dado por el peso de la UCR multiplicado por el grado de polimerización. Se llama polimerización al proceso químico mediante el cual se obtiene un polímero. En este proceso, el polímero se forma a partir de la unión entre sí de pequeñas moléculas. A estas pequeñas moléculas se les denomina monómeros. Para que una molécula pueda generar el polímero, es necesario que presente dos o más grupos funcionales que puedan reaccionar, es decir, debe tener una funcionalidad igual o superior a dos [4].

1.1.1 Clasificación de los Polímeros

A) Clasificación según su origen

- **Naturales:** Son sustancias producidas por organismos vivos que se emplean sin modificación. Ejemplos de estos polímeros son: proteínas como las empleadas por las arañas para tejer su tela, polisacáridos, caucho natural.
- **Sintéticos:** Son macromoléculas creados por el hombre y se dividen en dos categorías según su comportamiento al ser calentados: termoplásticos y termorrígidos. Los primeros al calentarse se ablandan o funden, y son solubles en disolventes adecuados. Están formados por moléculas de cadenas largas, a menudo sin ramificaciones. Los termorrígidos, en cambio, se descomponen al ser calentados y no pueden fundirse ni solubilizarse. Tienen estructuras elaboradas tridimensionales con reticulación.

B) Clasificación química de los polímeros

Los polímeros se clasifican químicamente en base a la composición química de la unidad que se repite (Monómero) [4]:

- **Polímeros heterocadena:** Estos polímeros presentan en su cadena principal heteroátomos o átomos diferentes al Hidrógeno (H) y al carbono (C).
- **Polímeros homocadena:** Estos polímeros presentan en su cadena principal solo átomos de Hidrógeno (H) y carbono (C).

C) Clasificación según el número de unidades repetitivas

De acuerdo al número de unidades que se repiten del que están formados los polímeros pueden clasificarse en [4]:

- **Homopolímero:** cuando esta formado por una única unidad repite.
- **Copolímero**: Cuando está formado por dos o más diferentes unidades que se repiten.

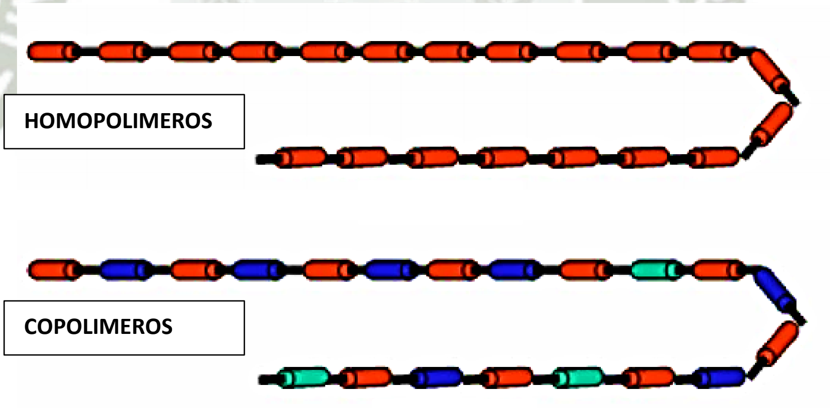
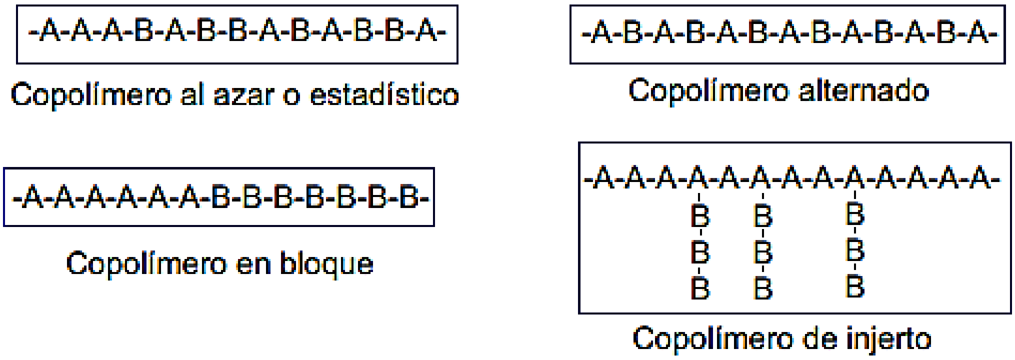


Figura 1.1 *Clasificación según el número de unidades repetitivas*

A su vez la organización, en los copolímeros, de las diferentes unidades que se repiten puede estar dispuesta de varias formas (Figura 1.2):

- **Al azar:** las unidades que se repiten se hallan dispersas de forma aleatoria o estadística.

- **Alternantes:** En este caso las unidades que se repiten se disponen alternativamente.
- **En bloque:** existen secuencias largas de cada una de las unidades repetitivas.
- **De injerto:** a una cadena formada por la misma unidad repetitiva le cuelgan como ramificaciones cadenas formadas por otra unidad repetitiva.



Copolímero al azar o estadístico

Copolímero alternado

Copolímero en bloque

Copolímero de injerto

Figura 1.2: *Tipos de copolímero.*

D) **Clasificación según su comportamiento mecánico**

Atendiendo el comportamiento mecánico del polímero, se clasificación generalmente en tres grandes grupos [4]:

- **Elastómeros:** son los polímeros que poseen una consistencia gomosa y elástica. Son sustancias que poseen la elasticidad que caracteriza al caucho y al igual que este se emplean para fabricar gomas, mangueras o neumáticos.
- **Fibras:** son materiales poliméricos con propiedades optimizadas en una dirección, que presentan una relación longitud/diámetro de como mínimo 100. Son materiales capaces de orientarse para formar filamentos largos y delgados como el hilo. Poseen una gran resistencia a lo largo del eje de orientación, tal como ocurre con el algodón, la lana y la seda. Tienen su principal aplicación en la industria textil [5].
- **Plásticos:** son todos aquellos polímeros de síntesis cuyas propiedades son intermedias entre las de los elastómeros y las de las fibras.

Son polímeros que pueden ser moldeados a presión y transformados en diversos objetos con formas diferentes, o bien, usados como pinturas o recubrimientos de superficies.

E) Clasificación según su comportamiento térmico

Según su comportamiento térmico, los polímeros pueden dividirse en tres grandes grupos [4, 5, 6]:

- **Termoplásticos**: son polímeros que se ablandan y funden, al someterlos a un calentamiento progresivo; es un proceso que puede ser reversible. Como su nombre lo indica, se comportan de manera plástica a elevadas temperaturas, en este tipo de polímeros es posible realizar el moldeo. A su vez, este grupo de polímeros puede dividirse en:
  - **Amorfos:** son polímeros termoplásticos que no cristalizan debido a que sus irregularidades impiden que una cadena se ordene junto a sus vecinas.
  - **Semicristalinos:** Presentan zonas, denominadas cristalitas, en el que las moléculas muestran un ordenamiento. Estos polímeros termoplásticos están constituidos por moléculas lineales ordenadas.
- **Elastómeros:** son polímeros que presentan una consistencia elástica y gomosa que se pierde a temperaturas muy bajas.. No son termoconformables ni soldables.

  Los elastómeros también denominados cauchos o hules tienen un comportamiento térmico que puede variar de termoplástico a termorrígido según su estructura sea lineal o reticulada. La clasificación se realiza en base a su comportamiento mecánico: se trata de materiales poliméricos que tienen la capacidad de deformarse mucho más que el 300% en forma elástica, esto es, cuando se remueve la fuerza aplicada para estirarlos recuperan sus dimensiones originales. Esto se debe a que las largas cadenas poliméricas se encuentran enrolladas e idealmente podemos decir que, cuando se aplica un esfuerzo para estirar la muestra, las cadenas lineales se desenredan. Cuando se libera el esfuerzo las cadenas vuelven a enrollarse y el polímero regresa a su forma y tamaño originales. Sin embargo, esta es sólo una idealización del comportamiento de las macromoléculas ya que las cadenas no sólo se desenrollan sino que también deslizan unas sobre otras, de modo que al eliminar el esfuerzo aplicado desaparece la deformación elástica (instantánea y reversible) pero puede quedar una deformación remanente (deformación plástica, debida al deslizamiento) [6].

- **Termoestables:** son polímeros que al calentarlos no funden, sino que se mantienen rígidos y sólidos a temperatura elevada, hasta que se descomponen por efecto de la energía térmica. Los polímeros termorrígidos también denominados termoestables son polímeros reticulados durante la reacción de polimerización o mediante la introducción de entrecruzamientos químicos (crosslinks). Este reticulado no permite que estos polímeros sean reprocesados después de que han sido conformados. Son moléculas reticuladas de malla muy tupida. No son procesables por fundido.
- **Adhesivos:** Tienen cierto grado de extensibilidad, alta adhesión pero conservando cierto grado de cohesión. Suelen tener baja cristalinidad.
- **Recubrimientos de superficies**: Sus características son similares a las de los adhesivos, pero además tienen gran resistencia a la abrasión. Se usan para proteger y decorar.
- **Fibras:** Pueden tejerse o enlazarse para formar prendas dimensionalmente estables; es necesario que no cedan demasiado. Deben ser resistentes y con tendencia a la cristalización.

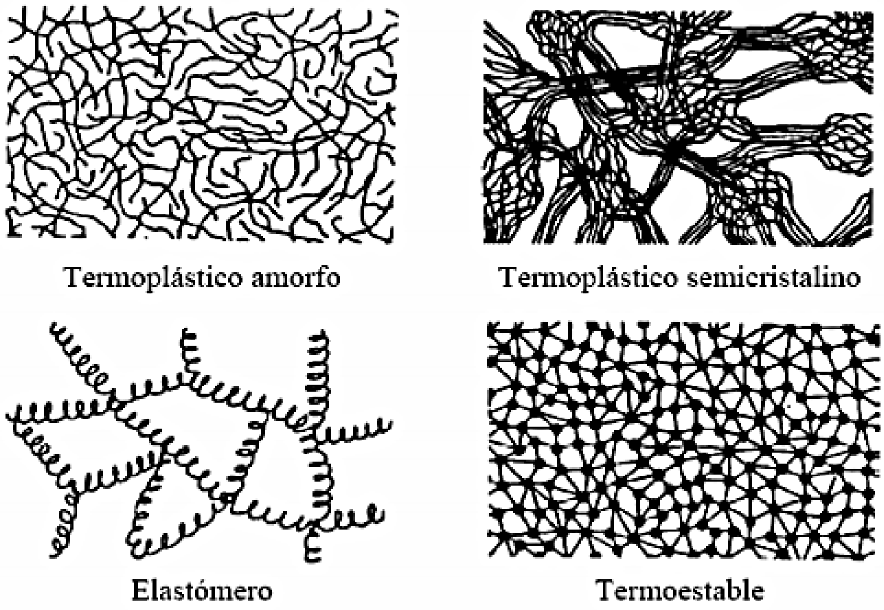

Termoplástico amorfo
Termoplástico semicristalino
Elastómero
Termoestable

Figura 1.3 *Tipos de polímero según su comportamiento térmico*

1.1.2 Mecanismos de Polimerización.

1.1.2.1 Polimerización por condensación.

Los polímeros se producen por la formación de enlaces entre dos tipos de meros reactivos. Una característica de esta reacción es que se condensan (de ahí el nombre) productos derivados por reacción. A este proceso también se le llama polimerización por etapas; ya que la molécula del polímero crece etapa por etapa hasta que se consume uno de los reactivos.

1.1.2.2 Polimerización por adición.

También conocida como polimerización por crecimiento de cadena o por reacción en cadena; debido a la rapidez con que se forman simultáneamente las moléculas largas, por lo general en unos cuantos segundos; el enlace se realiza sin productos derivados de la reacción.

Esta velocidad es mucho mayor que la de polimerización por condensación. A esta reacción se agrega un iniciador para abrir el doble enlace entre dos átomos de carbono, que inicia el proceso de unión agregando muchos más monómeros a la cadena creciente.

1.1.3 Peso molecular

Como consecuencia de los procesos de polimerización empleados para la obtención de los polímeros sintéticos, las cadenas de las moléculas de polímero obtenidas suelen presentar diferentes longitudes, es decir, que la masa molecular no es única para todas las moléculas [4].

Estas distribuciones de masas moleculares se suelen caracterizar por valores promedios mediante el peso molecular medio en número Mn y el peso molecular medio en peso Mw.

A la suma de los pesos moleculares de los meros en una cadena representativa se le llama peso molecular del polímero. Cuanto mayor sea dicho peso, mayor será la longitud promedio de la cadena. La mayoría de los polímeros comerciales tienen un peso molecular entre 10000 y 10000000. Debido a que la polimerización es un evento aleatorio, no todas las cadenas de polímeros que se producen son de igual longitud: esta cae dentro de una curva de distribución tradicional. Mediante el promedio determinamos y expresamos el peso molecular promedio de un polímero en una base estadística. A la distribución de los pesos moleculares en una cadena se le conoce distribución del peso molecular (MWD) por sus siglas en ingles. El peso molecular de un polímero y su MWD tiene una fuerte influencia en sus propiedades.

En general, a medida que aumentan los pesos moleculares, aumentan también las propiedades mecánicas del material plástico, pero por el contrario también aumenta la dificultad de procesar o transformar el material como consecuencia del aumento de su viscosidad.

1.1.4 Comportamiento cristalino y amorfo

Los polímeros como los polimetilmetacrilato, el policarbonato y el poliéster suelen ser amorfos; esto es, las cadenas de los polímeros existen sin un patrón repetitivo de largo alcance. Se dice con frecuencia que el arreglo amorfo de las cadenas de los polímeros es como un plato de espagueti o como lombrices en una cubeta: todos enlazados unos con otros. Sin embargo, en ciertos polímeros es posible impartir algunas cristalinidad y, de esta manera modificar sus características. Este arreglo se puede promover durante la síntesis del polímero, o por deformación durante su procesamiento subsecuente.

A las regiones cristalinas en los polímeros se les llama cristalitas, las cuales se forman cuando las moléculas largas se arreglan de modo ordenado entre ellas, igual que como se dobla una manguera para incendios en su gabinete o pañuelos desechables en una caja. Se puede considerar un polímero parcialmente cristalino (semicristalino) como un material bifásico, con una fase cristalina y otra amorfa.

Mediante el control de la rapidez de solidificación durante el enfriamiento y la estructura de la cadena, es posible aportar diferentes grados de cristalinidad a los polímeros, aunque nunca al 100%. La cristalinidad varía desde un cristal casi completo (de casi 95%en volumen) hasta un polímero ligeramente cristalizado (en su mayoría amorfos). El grado de cristalinidad también se ve afectado por la ramificación. Un polímero lineal puede volverse altamente cristalino, pero ni un polímero muy ramificado, aunque puede desarrollar un nivel bajo de cristalinidad, nunca alcanzara un alto contenido de cristalitas, debido a que las ramas interfieren en la alineación de las cadenas en un arreglo cristalino regular.

1.1.5 Propiedades Térmicas de los Polímeros

Las propiedades térmicas describen el comportamiento de los polímeros frente a la acción del calor.

a. Temperatura de degradación

A muy alta temperatura los enlaces covalentes entre los átomos de la macromolécula pueden destruirse, el polímero se quema o carboniza. La temperatura a partir de la cual se manifiesta este deterioro, conocida como temperatura de degradación Td limita la utilidad del polímero y representa la temperatura superior a la cual el polímero puede ser conformado en forma útil.

b. Temperatura de fusión

Es la temperatura por debajo de la cual las cadenas se organizan para formar regiones cristalinas.

c. Temperatura de transición vítrea (Tg)

Es un parámetro de particular interés en la manufactura de polímeros sintéticos. Se la define como la temperatura a la cual se produce la transición de comportamiento elastomérico (o tipo goma) a comportamiento vítreo. Esta transición presenta cierta correlación con la transición dúctil- frágil que se manifiesta en los metales. Esta temperatura puede modificarse mediante cambios en el grado de ramificación o entrecruzamiento o mediante el agregado de plastificantes.

1.2 BIOPOLÍMEROS

Un polímero biodegradable se define como un polímero con el cual la degradación es en parte asegurada por un sistema biológico como un microorganismo, bacteria o una célula viva.

1.2.1 Ácido Poli láctico

Recientemente los poliésteres alifáticos hidrobiodegradables pueden ser obtenidos, a escala industrial, a partir de recursos renovables. Efectivamente, en este caso, los monómeros son obtenidos por proceso de fermentación a partir de microorganismos capaces de producirlos a partir de azúcares (azúcar del almidón de maíz, azúcar de caña a azúcar o de remolacha…) [7].

El PLA es el más prometedor de los polímeros biodegradables ya que en su preparación se parte de productos agrícolas que no aumentan el contenido de CO2 atmosférico, y sus propiedades y comportamiento son superiores a los de otros polímeros biodegradables (posee las mayores temperaturas de fusión y de transición vítrea) y su precio puede disminuir mucho en el futuro [8]. El PLA se obtiene actualmente de almidón procedente del maíz, pero en el futuro se espera utilizar también celulosa como materia prima.

El PLA pertenece a la familia de los poliésteres alifáticos, es un polímero termoplástico, es biodegradable y compostable. Con equipos estándar, se pueden elaborar fácilmente films, fibras y piezas moldeadas [9, 12].

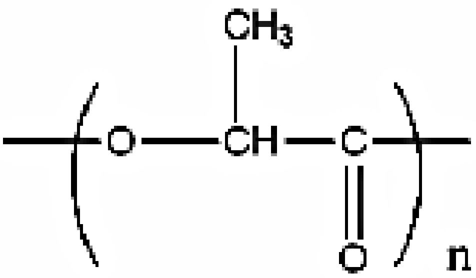


Figura 1.4 *Ácido poliláctico.*

Actualmente el PLA es utilizado para dos principales aplicaciones: las aplicaciones biomédicas (como suturas, tornillos para huesos, etc.) y el embalaje gracias a sus bastante buenas propiedades mecánicas (polímero relativamente rígido y frágil con una baja deformación a la ruptura), su carácter biocompatible.

1.2.1.1 Historia del PLA

El PLA se fabrica desde hace décadas, no es un polímero reciente. En 1932 se sintetizó un producto de bajo peso molecular calentando el ácido láctico en vacío, por el científico Wallace Carothers de la famosa empresa Dupont. Más tarde en 1954, después de otros ajustes, Dupont patentó el proceso de Carothers [9, 10].

Una de las primeras compañías en desarrollar ampliamente el PLA fue Cargill, quienes iniciaron investigando en 1987 la tecnología de la fabricación del Ácido Polilactico, y empezó en 1992 la fabricación en una planta piloto de cantidades importantes de PLA [11].

En 1997, Cargill unificandose con Dow Chemical Company. Inc., crearon Cargill Dow Polymers LLC (CDP) ellos estudiaron la obtención de plásticos a partir de carbohidratos de las plantas. La empresa NatureWorks, se estableció en 1997 operada por Cargill-Teijin Limited. NatureWorks LLC esta fue la primera compañía en brindar una familia de biopolímeros derivados de recursos 100% renovables, cuyos costes y rendimientos quieren competir con los materiales para embalaje y fibras a base de petróleo. NatureWorks LLC, comercializa el PLA bajo la marca Ingeo™ [13].

Se han desarrollado termoplásticos biodegradables de Ácido polilactico (PLLA modificada y PLLA) por compañías japonesas (Kanebo, Shimadzu, Dai-nippon Ink, Mitsuitoatsu) con la denominación de Lactron, Lacty. [14].

Actualmente la capacidad del principal productor mundial, CONSORTIUM CARGILL-DOW, es de 140 000 toneladas por año. Así este polímero se inscribe en una política de desarrollo sostenible [7].

1.2.1.2 Obtención del Ácido Poliláctico (PLA)

En 1780 el químico sueco Scheele descubrió el ácido láctico en la leche agria, el cual es el monómero básico del PLA. El ácido láctico es utilizado como acidulante, inhibidor bacteriano y condimento [15]. Por otra parte es posible obtener el ácido láctico por síntesis química o fermentación, siendo el ultimo la obtención predominante [9, 16, 17, 18].

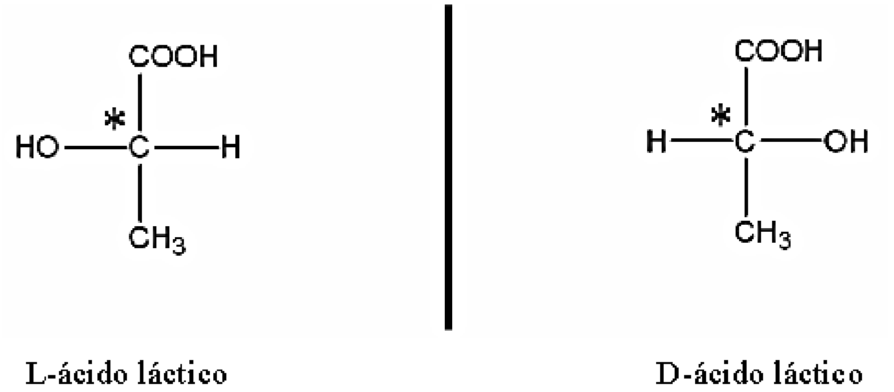


Figura 1.5. Enantiómeros del ácido láctico [19].

El enantiómero que se encuentra más frecuentemente en la naturaleza es el L-ácido láctico [7].

El ácido láctico de fórmula química $CH_3CHOHCOOH$ (ácido 2- hidroxilpropiónico) con masa molecular de 90,08 g/mol es una sustancia líquida incolora, viscosa y no volátil. El ácido láctico contiene un átomo de carbono quiral o asimétrico y existe en dos configuraciones óptimamente activas, levógira y dextrógira, generalmente denominadas L (+) y D(-) ácido láctico (ver figura 1.5) [11].

El PLA puede ser sintetizado con dos métodos distintos:

A) Directamente a partir de los ácidos lácticos gracias a una policondensación

El PLA obtenido es un PLA de masa molar reducida (Mw=2000-10000g/mol) y con propiedades mecánicas insuficientes para las aplicaciones pretendidas.

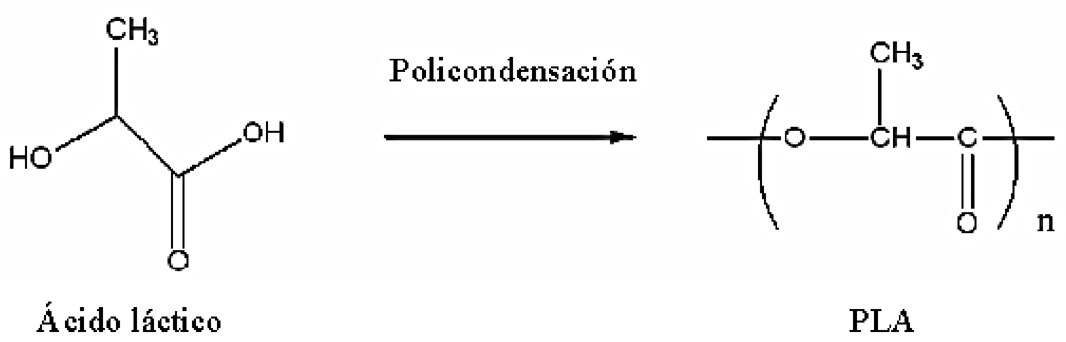
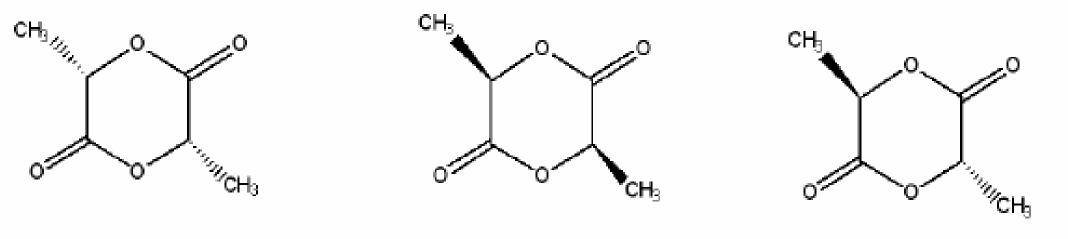


***Figura 1.6.** Policondensación del PLA.*

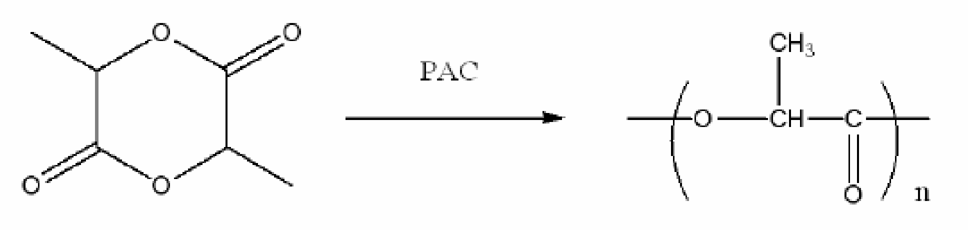
B) A partir de dímero correspondiente: los lactides

Dímeros del PLA (los lactides) son obtenidos a partir de una depolimerización controlada de los PLA de masa reducida. Así los lactides constituyen los nuevos monómeros. Los diferentes estereoisomeros del lactide se presentan en la figura 1.7 Después una polimerización por apertura de ciclo (PAC) permite obtener PLA con fuerte masa molar como lo muestra la figura 1.8.



L-lactide **D-lactide** **meso-lactide**

Figura 1.7. Los diferentes estereoisomeros del lactide.



L-lactide **PLA con M_w>10000 g/mol**

Figura 1.8. Polimerización por apertura de ciclo.

90% de la producción mundial de ácidos lácticos es obtenido por fermentación. El productor más importante de PLA es Cargill-Dow [7]. El principio del procedimiento de fabricación del PLA se presenta en la figura 1.9.

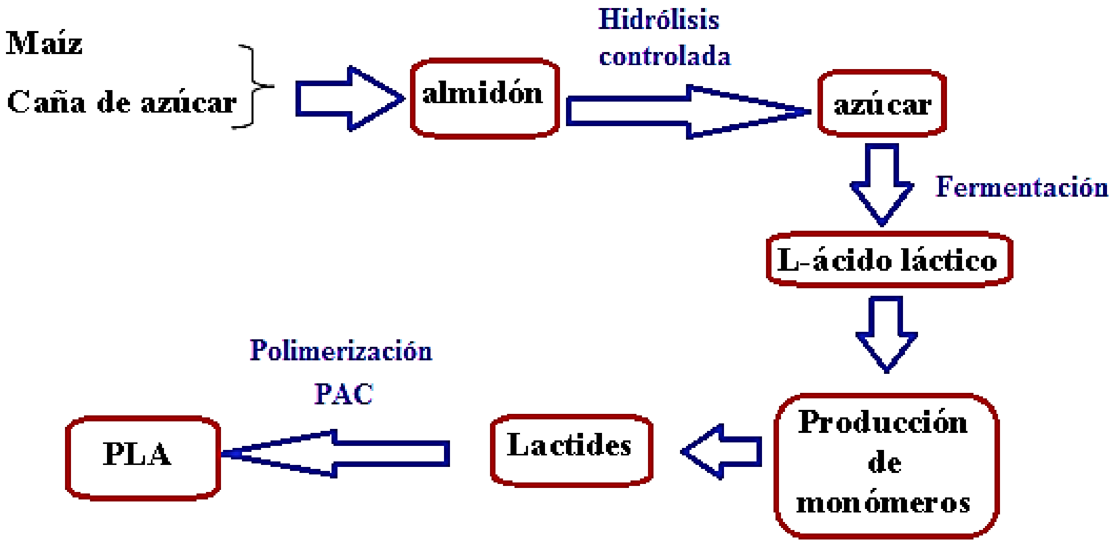


Figura 1.9 *Procesado de fabricación del PLA[19]*

El proceso de fermentación generalmente es un proceso no continuo el cual tiene una duración de 3 a 6 días. Se usa una concentración de azúcar entre el 5-10% para la fermentación y el rendimiento de la obtención es de 2 gramos de ácido por litro de caldo de cultivo/hora. Para la separación del ácido Lactico obtenido se han perfeccionado varios métodos. La adición de hidróxido cálcico ($Ca(OH)_2$) es el más importante método de separación y consiste en obtener disoluciones de lactato cálcico soluble la cual se filtra, después se lleva a ebullición para evaporar el agua y recristalizar luego se adiciona ácido sulfúrico para formar sulfato cálcico sólido con ácido láctico líquido , el cual se filtra y se desecha el sólido quedándonos el ácido láctico en solución; para purificar y eliminar los hidratos de carbono y las proteínas se usa la destilación. Después de la purificación se obtiene un producto que contiene más de un 99,5% de L-isómero y 0,5% de D-isómero [11].

La cantidad de energía acumulada durante el ciclo de vida del PLA es de 82,5 MJ/kg (ver Figura 1.10) [11].

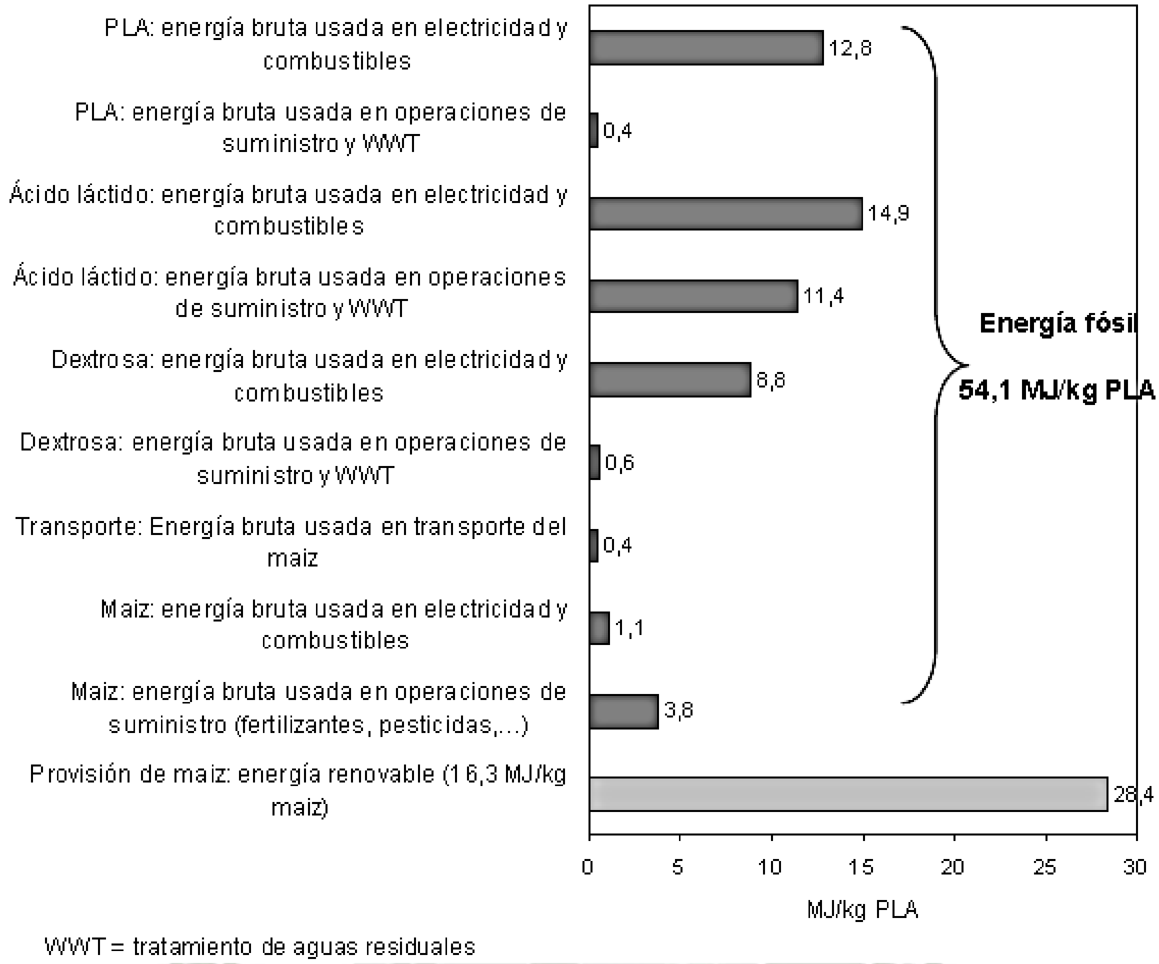


Figura 1.10 Uso de energía en cada parte del proceso de obtención del PLA

1.2.1.3 Propiedades Reologicas del PLA

Para examinar a los polímeros termoplásticos en sus variadas formas durante las operaciones de procesamiento se usan las propiedades reológicas [11]. El PLA se utiliza ampliamente en varios procesos de conformados como en las películas orientadas y la hilatura de fibras, en estos procesos los tiempos de flujo son muy diferentes; el grado y tipo de ramificación, la distribución del peso molecular, distribuciones ópticas de la longitud del bloque estabilidad a la fusión y composición óptica son factores que afectan las características de flujo del PLA [20].

El PLA, es un fluido no Newtoniano y un polímero pseudoplástico. El PLA fundido, es decir por encima del punto de fusión (Tm), posee un comportamiento similar a un polímero clásico de cadena flexible [20, 21].

El comportamiento reológico del PLA puede modificarse con la inclusión de ramificaciones.

1.2.1.4 Propiedades del Ácido Poliláctico (PLA)

Comparando las propiedades del PLA con los polímeros existentes derivados del petróleo, el PLA posee excelentes propiedades físicas, ópticas, de barrera y mecánicas [22, 23]. El Ácido Poliláctico posee buenas propiedades al rizado, excelente resistencia a los aceites y grasas; buena resistencia al arrugado, y es una buena barrera para los sabores y aromas [24].

Las características físicas del PLA de alto peso molecular en gran parte dependen de la temperatura de transición vítrea, por cualidades comunes como la densidad, la capacidad calorífica y propiedades mecánicas y reológicas. En estado sólido, el PLA puede ser amorfo o semicristalino, depende de la estereoquímica e historia térmica [24].

1.2.1.4.1 Propiedades Mecánicas del PLA

El Ácido Polilactico no orientado tiene buena rigidez y resistencia, pero es muy frágil. Sin embargo el Ácido Polilactico orientado es comparable a las propiedades del PET y tiene mejores propiedades que el Poliestireno orientado [11]. El módulo de tensión y flexural del PLA es mayor que la del propileno (PP), polietileno de alta densidad (HDPE) y Poliestireno, pero la elongación a la rotura tiene valores inferiores que los de estos polímeros [25, 26]. Física y mecánicamente las propiedades del PLA dependen mucho de la relación L/D (L y D representan los enantiomeros del PLA, ver figura 1.5), su peso molecular, su orientación, su cristalinidad y el método usado para su obtención.

En cuanto a la resistencia del PLA al impacto, los cuales se obtienen mediante la realización de un ensayo de impacto de Charpy o Izod, los valores se encuentran en el rango de 2 a 3 KJ/m^2 para materiales con baja cristalinidad y probetas sin muesca; sin embargo la resistencia al impacto de una probeta con muesca o entalle reduce el valor.

Por debajo de la Temperatura de transición vítrea (Tg) el PLA posee baja flexibilidad sobre soporte de carga, siendo este rígido y quebradizo y por encima de la Temperatura de transición vítrea el Ácido polilactico tiene un comportamiento de un polímero cristalino.

1.2.1.4.2 Propiedades Térmicas del PLA

El PLA a temperatura ambiente es un plástico rígido [28]. El PLA en estado sólido puede ser semicristalino o amorfo, dependiendo de la historia térmica y estereoquímica [11].

En el PLA amorfo, para la mayoría de usos comerciales, la Tg establece la máxima temperatura de utilización. La Tg se encuentra entre 55-65ºC y la temperatura de fusión (Tm) está entre 160-180ºC del PLA que solamente contiene el isómero D- PLA o L-PLA [9, 28]. Las transiciones térmicas Tm y Tg son afectadas por la historia térmica la estructura primaria, el peso molecular y la composición isomérica (L o D) (Fig. 1.11) [8,11].

A temperaturas debajo de la Tg el PLA se comporta como un polímero frágil. A temperaturas encima de la Tg el PLA tiene un comportamiento como un polímero cristalino con la posibilidad de ser enfriado hasta la temperatura de β-transición (Aprox. -45ºC) [29].

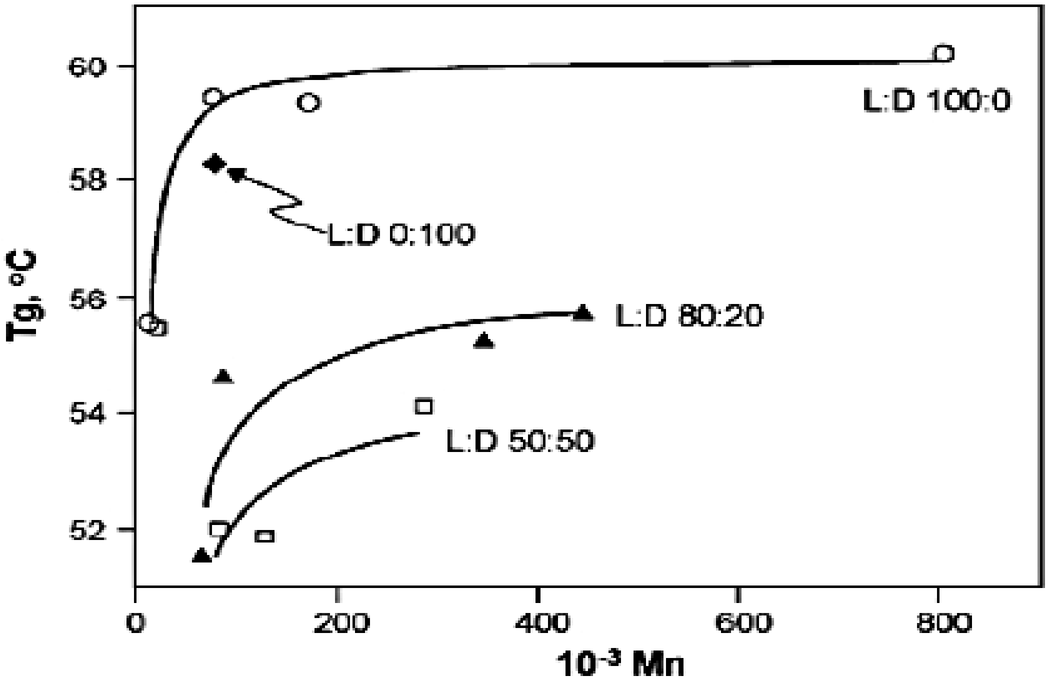


Figura 1.11 Temperaturas de transición vítrea del PLA por diferentes contenidos de L- en función del peso molecular

1.2.1.4.3 Absorción de agua

El Ácido Poliláctico contiene un % de humedad aproximadamente entre 0,4-0,6.

1.2.1.4.4 Resistencia química

El PLA es un polímero termoplástico que tiene afinidad por el agua y su resistencia a la hidrólisis es baja.

1.2.1.4.5 Densidad

Se producen variaciones en la densidad del PLA de acuerdo al grado de cristalización o copolímero (ver tabla 1.1).

Tabla 1.1 *Densidades del PLA*

PLA	***Densidad (g/cm^3)***
L-lactida	1.36
Meso-lactida	1.33
PLA Cristalina	1.36
PLA Amorfa	1.25

Fuente: Montero Palacios A. 2012. Métodos físico-químicos de caracterización de las fibras de PLA. Universidad Politécnica de Catalunya. Terrassa.

1.2.1.4.6 Solubilidad

La mayoría de disolventes orgánicos pueden disolver el PLA amorfo, disolventes como: el benceno, solventes clorados, el tetrahidrofurano (THF), dioxano y acetonitrilo [11, 10]. Por otro lado los disolventes halogenados, específicamente los clorados y el benceno calentado, pueden disolver el PLA cristalino. En agua o en mezclas agua/alcohol, el PLA no se disuelve [11].

1.2.1.5 Degradación del Ácido Poliláctico (PLA)

Debido a la biodegradación del PLA en el ambiente natural y su resistencia a la hidrolisis baja en el cuerpo humano los estudios sobre el PLA, han ido incrementando [12].

Los factores ambientales [30, 31], tales como: calor, luz o radiación, acción biológica, condiciones químicas o humedad producen degradación ya sea química, física o biológica en los polímeros estos factores hacen que los enlaces de la cadena polimérica se rompan (ver Tabla 1.2).

El L-PLA es un polímero biodegradable (compostable), además es semicristalinos es decir en su estructura tiene regiones cristalinas y amorfas, y su Tg está a 50°C.

Tabla 1.2 *Tipos de degradación de los polímeros*

TIPOS DE DEGRADACION	AGENTE
Mecánica	Tensión, fuerzas de cizallamiento y Compresión.
Foto degradación	Luz o Radiación
Térmica	Temperatura
Biodegradación	Hongos, bacterias y Microorganismos
Degradación Química Hidrólisis	Agua
Degradación Química enzimática	Enzimas
Degradación Química Oxidación	Oxígeno

Fuente: Montero Palacios A. 2012. Métodos físico-químicos de caracterización de las fibras de PLA. Universidad Politécnica de Catalunya. Terrassa.

Las condiciones ambientales al que están expuestos los polímeros son: el entierro, el clima, lluvias, vientos, variaciones de temperatura en el día y la noche, la radiación solar, etc. Estas condiciones después de unos meses provocan transformaciones en la estructura polimérica (Hidrólisis abiótica) [33] y contribuyen a debilitar al polímero [34, 35] e iniciar el proceso de biodegradación [36]. Se puede decir que la degradación del PLA se da en dos etapas.

- Primera etapa, El PLA se vuelve quebradizo; ya que se produce el rompimiento no enzimático aleatorio de la cadena polimérica de grupos éster del PLA, esto conlleva obtener cadenas de menor tamaño y por consiguiente se disminuye la masa molecular. Esta etapa se puede catalizar por la presencia de bases o ácidos, aumento de temperatura o la humedad.
- Segunda etapa, Se produce agua (H_2O), dióxido de carbono (CO_2) y humus [61] debido a que el PLA de baja masa molecular obtenido en la primera etapa se desplaza fuera de la masa del PLA y es atacada por los microorganismos.

1.2.1.6 Usos del Ácido Poliláctico.

A partir del PLA se pueden fabricar: films, hilos, fibras, láminas, envases, entre otros.

En la tabla 1.3 se mencionan los sectores en el que se usa el PLA y sus aplicaciones.

- **Fibras**: Una aplicación potencial del PLA es la fabricación de fibras debido a que el PLA se hila fácilmente por fusión y puede ser diseñado para diferentes usos domésticos como: relleno de fibra hueca para Peluches, colchones y almohadas; trapos, cortinas, tapicería de muebles etc. Y para usos industriales como: filtros, Geotextiles, Redes de pesca, redes para contener vegetales en agricultura y en artículos médicos (Hilos de sutura, gasas o tejidos protectores).
- **Filmes:** El PLA posee propiedades como su rigidez que posibilita una disminución de espesor mayor que el PET y que lo convierten en un polímero atractivo y versátil en el sector de los embalajes y envases [11]. El PLA en film se usa, para la fabricación envase y contenedores de alimentos, platos, artículos médicos y de higiene. El PLA es usado también en cartones y papel laminado [8], también como manto aislador de aromas, olores y sabores en el envasado de productos a baja temperatura [11].

- El PLA ha sido aprobada por la FDA (Food and Drug Administration) para ciertas aplicaciones médicas; también ha sido utilizado ampliamente en usos médicos, específicamente para el cultivo y crecimiento de células vivas, provenientes de órganos humanos, sobre un soporte textil degradable y absorbible de fibras de PLA [11]. Una de las características del PLA es su biocompatibilidad con los cuerpos o tejidos vivos ya que se degrada por hidrólisis y es bioabsorbido, siendo lentamente metabolizado y apartado del cuerpo una vez cumplido su función [28].

Tabla 1.3 *Sectores de mercado y aplicaciones del PLA*

	SECTOR	SUBSECTOR	APLICACIONES
1	Textiles	Vestimenta	Moda, Sport
		Textil-hogar	Covertores, Alfombras, Almohadas, Mantas
		No tejidos	Aplicaciones domésticas e Industriales (Relleno para Almohadas, fabricación de fieltros, etc.)
2	Limpieza	Aseo	Higiene femenina (toallas Higiénicas), Pañales.
		Filtración y Separación	Filtros, medios triboelectricos
3	Aplicaciones Médicas	-	Implantes temporales.
4	Embalajes, Filmes y otras aplicaciones	Termo conformados Rígidos	Bandejas y tapas, Envases opacos para leche, Componentes Electrónicos, copas
		Filmes orientados biaxialmente	Envoltorios, Embalajes
		Botellas	Botellas para productos de vida corta como leche y aceite

Fuente: Montero Palacios A. 2012. Métodos físico-químicos de caracterización de las fibras de PLA. Universidad Politécnica de Catalunya. Terrassa.

1.3 Fibras naturales

Se conoce con el nombre genérico de fibras a un amplio conjunto de materiales que tienen en común la característica de poseer un alto valor de la relación longitud/diámetro (l/d) Así, fibras naturales de origen vegetal como el cáñamo y el yute tienen relaciones del orden de 100 a 1000. El algodón y la lana superan ampliamente ese intervalo y se sitúan entre 1000 y 3000. La seda es la fibra natural con mayor relación l/d, ya que se pueden conseguir filamentos de hasta 500 m de longitud y unas pocas micras de diámetro [37].

1.3.1 Clasificación de las fibras naturales

Las fibras naturales comprenden todas las fibras que se han formado por un proceso de la naturaleza. Los tres reinos suministran fibras de factible utilización.

Se podrían clasificar dependiendo de sus características, debido a que son muy diversas, pero quizá la clasificación más general sea la siguiente:

- Fibras animales
- Fibras minerales
- Fibras vegetales

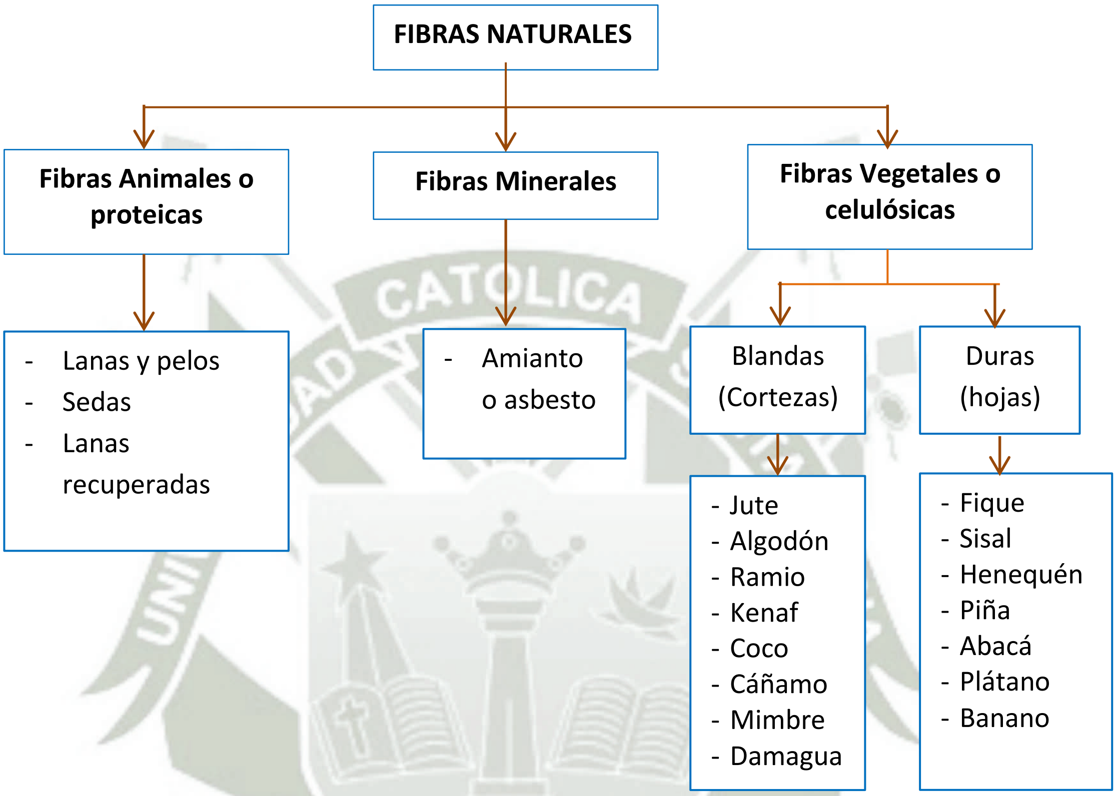


Figura 1.12. *Clasificación general de las fibras naturales*

Fuente: VILLAREAL, Andrés. Fibras Naturales: alternativa para el desarrollo nacional. Quito: SICA. s.f.

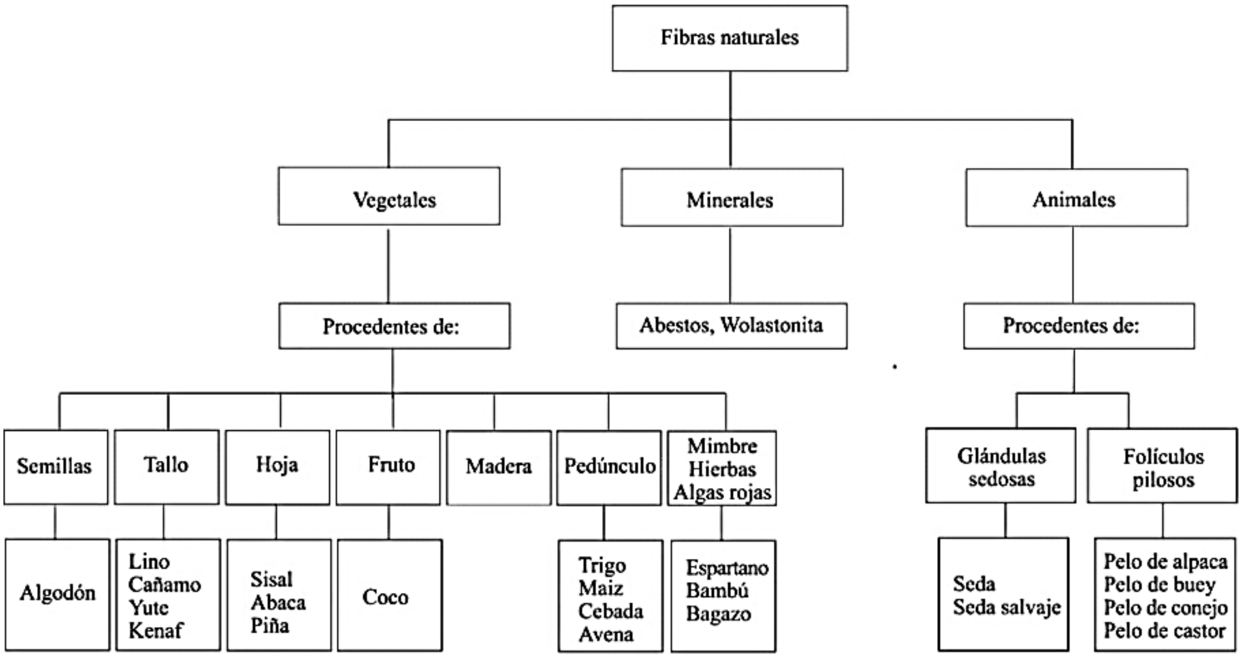


Figura 1.13. *Clasificación de las fibras naturales*

Fuente: Moriana Torró R. Desarrollo y caracterización de biocomposites enfibrados procedentes de recursos renovables. Estudio de su degradación en tierra. Universidad Politécnica de Valencia. Octubre 2010

1.3.2 Importancia

La fibras naturales son de importante uso debido a que no causan ningún efecto negativo al ambiente (tienen un ciclo neutro de CO2)" [38], pueden ser cultivadas en zonas climáticas diferentes, tienen buenas propiedades de aislamiento eléctrico y acústico, sus costos son potencialmente más bajos o baratos, reducen el peso de materiales compuestos (comparados con Compuestos de fibra de vidrio) y, por supuesto, ofrecen un reciclaje más fácil.

1.3.3 Aplicaciones

Los usos de las fibras naturales son reconocidos, y con una alta aplicabilidad dentro del campo de la agricultura, empaques, decoración, artesanías, etc. Algunas fibras como el lino, algodón, yute, sisal, kenaf y fibras de plantas similares, que han sido usadas desde hace más de 6000 años A.C, empiezan a utilizarse como materia prima no solamente para la industria textil, sino también para la fabricación de compuestos modernos eco-amigables usados en diferentes áreas de aplicación, como materiales de construcción (sogas, adobes, puentes, etc.), tableros de partículas, tablas de aislamiento, nutrición, cosméticos, medicina y recursos para bio-polímeros.

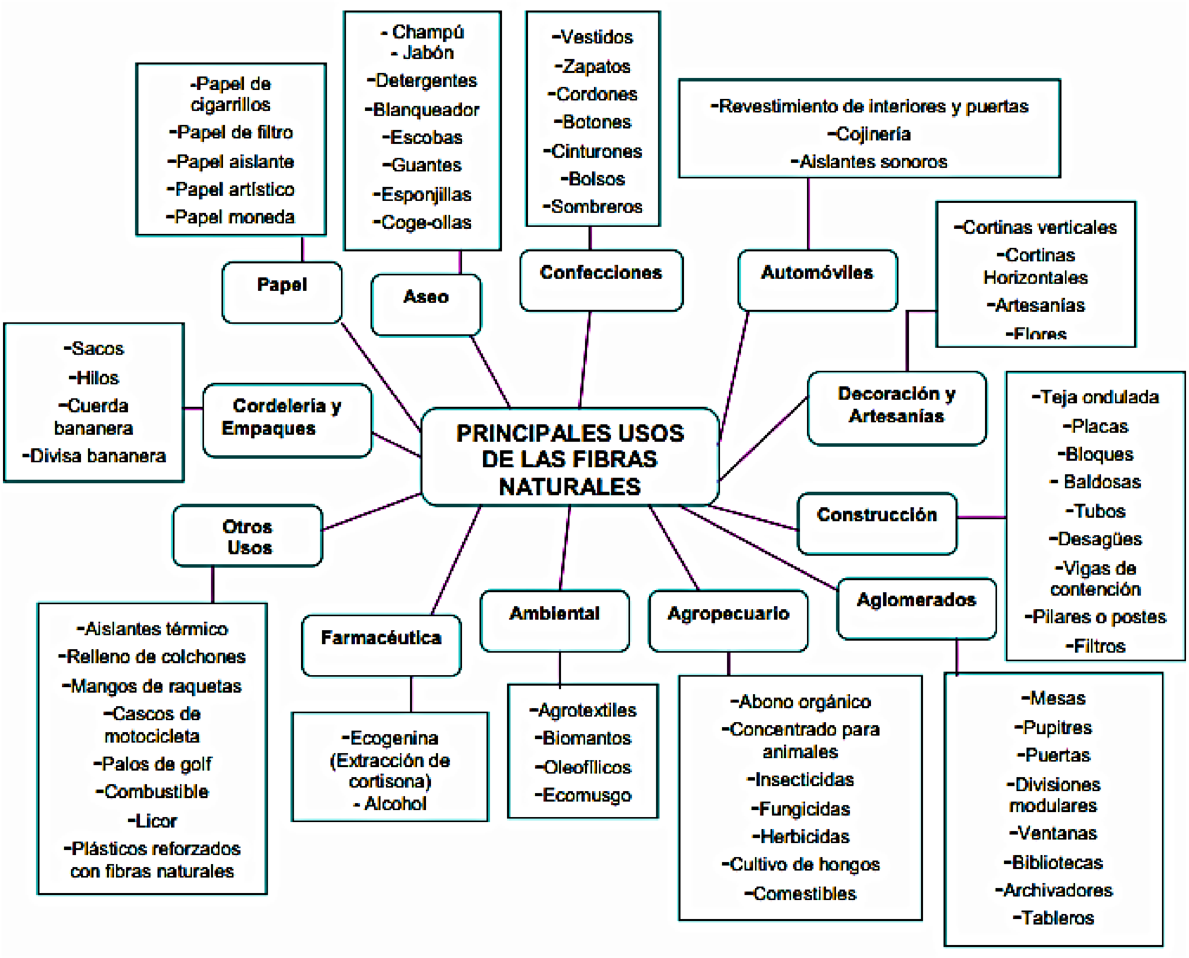


Figura 1.14 *Principales usos de las fibras naturales*

Fuente: VILLAREAL, Andrés, Fibras Naturales. Alternativa para el desarrollo nacional, Coordinación, investigación y Desarrollo ECAA, Ecuador, p. 3-4 *p.5*

1.3.4 Interacción Fibra Natural – Matriz polimérica

Para la producción de materiales compuestos resistentes reforzados con fibras vegetales, es necesario que la superficie de la fibra sea áspera de tal forma que aumente el número de puntos de adhesión, ofreciendo así una buena interacción mecánica fibra- Matriz polimérica, donde la presencia de grupos reactivos en éstas, es esencial para una alta energía interfacial [39].

La presencia de sustancias cerosas en la superficie de la fibra interfiere entre el vínculo de la fibra con la resina, y es causante de la adherencia superficial pobre. También, "la presencia de agua libre y grupos hidroxilo, especialmente en las regiones amorfas, empeora la capacidad de las fibras para desarrollar características adhesivas con la mayoría de los materiales poliméricos [40].

La alta absorción de agua y de humedad causan a las fibras un hinchamiento y efectos plastificantes, dando por resultado inestabilidad dimensional y características mecánicas pobres, esto limita el uso de las fibras naturales como reforzante para los materiales plásticos, y es ahí donde surge la necesidad de realizarle tratamientos superficiales con el objetivo de mejorar su adherencia con las resinas y disminuir su capacidad hidrofílica [41].

1.4 RESIDUOS AGRÍCOLAS: Cascarilla de Arroz

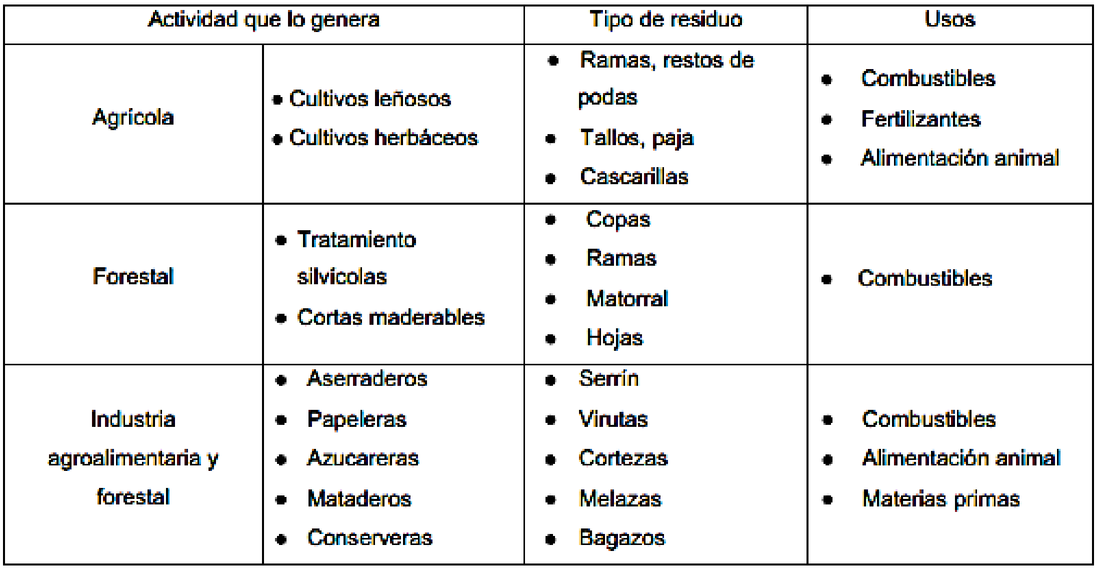
La cáscara de arroz, también denominada "pajilla", se obtiene del proceso de pilado, en el cual la cáscara se separa haciendo pasar el arroz en cáscara entre rodillos de jebe, luego de una operación previa de limpieza.

Fracción o fracciones de un cultivo que no constituyen la cosecha propiamente dicha y aquella parte de la cosecha que no cumple con los requisitos de calidad mínima para ser comercializada como tal. De forma similar, los restos de poda de los cultivos leñosos deben ser considerados asimismo residuos agrícolas estrictos [42].

Los desechos pueden ser definidos como subproductos (de la agricultura, forestales, procesos industriales e incluso domésticos), que esencialmente no tienen nada que ver con la construcción, pero que, con tratamiento y procesamiento especial, o unidos con otros materiales, puede sustituir económicamente, o incluso mejorar la calidad de los materiales de construcción convencionales [42].

En las industrias arroceras el residuo básico de esta industria es la cascarilla de arroz, que puede ser utilizada como combustible o como sustrato de cultivo, sola o mezclada con otros sustratos.

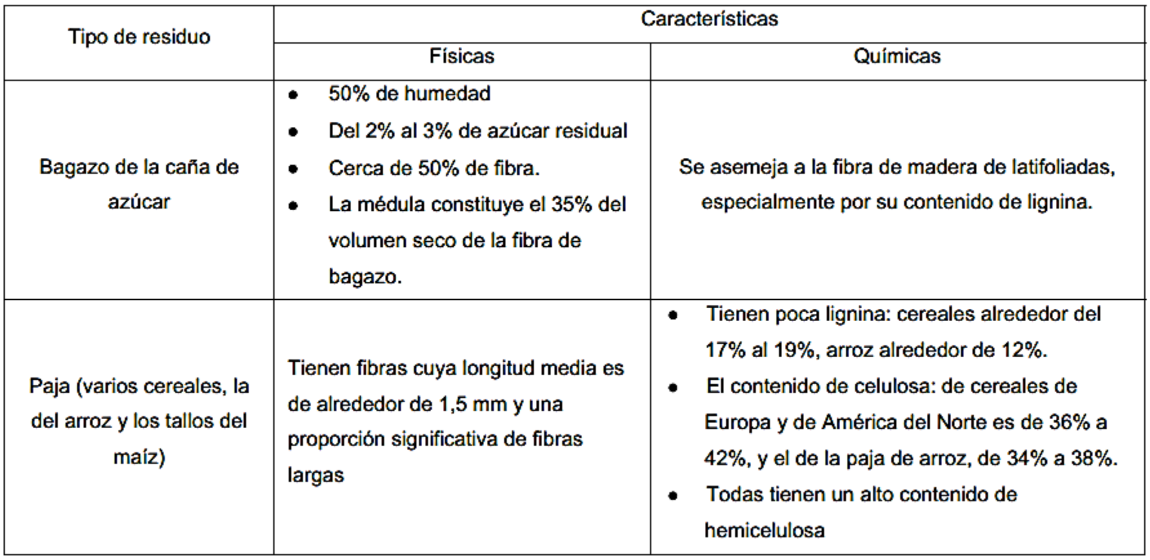
Tabla 1.4 Clasificación y usos de residuos agrícolas



Actividad que lo genera		Tipo de residuo	Usos
Agrícola	• Cultivos leñosos • Cultivos herbáceos	• Ramas, restos de podas • Tallos, paja • Cascarillas	• Combustibles • Fertilizantes • Alimentación animal
Forestal	• Tratamiento silvícolas • Cortas maderables	• Copas • Ramas • Matorral • Hojas	• Combustibles
Industria agroalimentaria y forestal	• Aserraderos • Papeleras • Azucareras • Mataderos • Conserveras	• Serrín • Virutas • Cortezas • Melazas • Bagazos	• Combustibles • Alimentación animal • Materias primas

Fuente: Giancarlo Chur Pérez. EVALUACIÓN DEL USO DE LA CASCARILLA DE ARROZ COMO AGREGADO ORGÁNICO EN MORTEROS DE MAMPOSTERÍA. Universidad de San Carlos de Guatemala. Facultad de Ingeniería. Escuela de Ingeniería Civil. 2010

Tabla 1.5 Características de algunos residuos agrícolas



Tipo de residuo	Características	
	Físicas	Químicas
Bagazo de la caña de azúcar	• 50% de humedad • Del 2% al 3% de azúcar residual • Cerca de 50% de fibra. • La médula constituye el 35% del volumen seco de la fibra de bagazo.	Se asemeja a la fibra de madera de latifoliadas, especialmente por su contenido de lignina.
Paja (varios cereales, la del arroz y los tallos del maíz)	Tienen fibras cuya longitud media es de alrededor de 1,5 mm y una proporción significativa de fibras largas	• Tienen poca lignina: cereales alrededor del 17% al 19%, arroz alrededor de 12%. • El contenido de celulosa: de cereales de Europa y de América del Norte es de 36% a 42%, y el de la paja de arroz, de 34% a 38%. • Todas tienen un alto contenido de hemicelulosa

Fuente: Giancarlo Chur Pérez. EVALUACIÓN DEL USO DE LA CASCARILLA DE ARROZ COMO AGREGADO ORGÁNICO EN MORTEROS DE MAMPOSTERÍA. Universidad de San Carlos de Guatemala. Facultad de Ingeniería. Escuela de Ingeniería Civil. 2010

1.4.1 Descripción de la Cascarilla de Arroz

La cáscara de arroz constituye un subproducto del proceso agroindustrial con aplicaciones reducidas, que en la práctica puede considerarse como un material de desecho (por término medio, por cada tonelada de arroz se generan 200 kg de corteza o cascarilla) [42].

La cascarilla de arroz es un tejido vegetal constituido por celulosa y sílice, propiedades que le dan diferentes usos como combustible alternativos, sustituto de madera, abono de cultivos y es utilizado en el campo agrícola, de la construcción y como aislante térmico [43].

El beneficio del cultivo de arroz genera un residuo, denominado cascarilla o cascara, tan sólo un 5% se está aprovechando. La cáscara de arroz constituye un subproducto del proceso agroindustrial con aplicaciones reducidas, que en la práctica puede considerarse como un material de desecho. La combustión de la corteza de arroz produce una media del 20% de cenizas con un gran porvenir ya que es un claro sustituto o aditivo del cemento en la formulación de concretos o morteros. Por sus características físicas y químicas la cáscara de arroz resulta poco biodegradable y se convierte en un desecho altamente contaminante en especial para las fuentes de agua [42].

Figura 1.15 *Imagen de la cascarilla de Arroz.*

1.4.2 Ventajas en construcción y usos

El peso y volumen de la cáscara de arroz ocasionan elevados costos de almacenamiento y transporte para la industria, además por ser poco digestible su uso en la elaboración de alimentos concentrados para animales es restringido. El contenido de humedad de la cáscara de arroz cuando sale del descascarador varía entre el 5% al 40% después de haber estado a la intemperie (en época no lluviosa por sus características químicas presenta un 10% de humedad).

Entre sus ventajas como material de construcción se pueden mencionar:

- Alto contenido de cenizas (materia sólida no combustible por kg del material ± 20%).
- Elevado contenido de sílice de las cenizas (90%).
- Estructura física de la sílice (estructura alveolar de gran superficie específica).
- Disponibilidad a lo largo del año.
- Retención de humedad.
- Material liviano.
- Material abrasivo.

El cultivo del arroz y su beneficio genera un residuo denominado cascarilla, de este tan solo un 5% se está aprovechando (limpieza de los campos, combustión a campo, disposición del material en rellenos), conduciendo a un problema de carácter ambiental. La cascarilla de arroz genera un gran volumen de cenizas, RHA, del inglés Rice Husk Ash, que tiene una elevada proporción de sílice. Se estima que por cada tonelada de arroz se generan 200 kg de cascarilla y de ésta se pueden producir 40 kg de cenizas con un contenido del orden del 90% en sílice.

a) Combustible

El poder calorífico de la cáscara de arroz es similar al de la madera y al de otros residuos agrícolas (debido a su composición alta en celulosa y sílice), por lo que inclusive se le ha considerado como una alternativa en usos domésticos. Se han desarrollado hornos para cereales que la utilizan como combustible, con lo que se obtienen un mejor rendimiento, el residuo después de quemarla puede ser usado en la construcción.

b) Abono

De acuerdo con sus características físico-químicas en China la han utilizado para regenerar suelos como compost (abono).

c) Adición mineral en mezclas de concreto y morteros

Considerando que la selección natural de materiales es uno de los factores que puede mejorar el desempeño del concreto desde el punto de vista de durabilidad, una posibilidad es la utilización de adiciones minerales tal como la cascarilla de arroz, la cual contribuye a mejorar las características del concreto en estado fresco y endurecido, disminuyendo la permeabilidad del concreto e incrementando sus propiedades mecánicas. Debe mencionarse que el uso de esta adición puede requerir el empleo de reductores de agua, especialmente cuando se elabora concreto con baja relación agua/cemento. En otros casos podrá requerirse del empleo de aditivos modificadores de viscosidad, especialmente para concretos auto consolidables.

d) Agregado orgánico en mezclas de concretos y morteros.

Se consideran específicamente los materiales conglomerados (concretos y morteros) de cemento Portland con cascarilla de arroz en estado natural o con tratamiento previo como agregado granular compuesto con partículas silíceas.

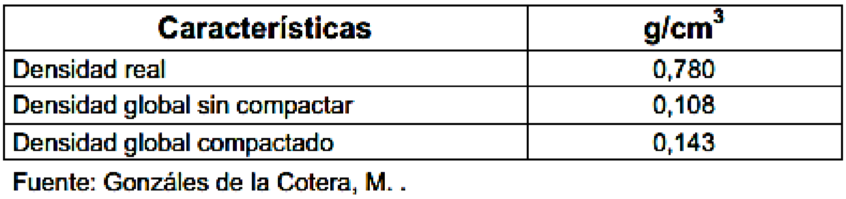
Se han diseñado distintas dosificaciones y determinado las propiedades geológicas en estado fresco y mecánicas en estado endurecido, de las mezclas resultantes. El empleo de cascarilla de arroz (material de desecho común en la zona) como componente granular, y una tecnología que no requiere mano de obra especializada o equipos sofisticados de compactación y colocación, permitirá acercar esta propuesta a los usuarios de menores recursos, y la utilización de estos materiales en la construcción de viviendas de bajo costo.

1.4.3 Propiedades Físicas de la cascarilla de Arroz

Su longitud promedio varía entre 5 a 11 mm según la especie o variedad, es una estructura ondulada y apariencia superficial irregular, es altamente abrasivo, posee el valor de 6 en la escala de dureza de Mohs en estado natural. Su estructura presenta un volumen poroso del 54%, cavidades que permanecerán cerradas en tanto no se someta a un proceso de combustión. Su coeficiente de conductividad térmica (0.0330 W/m*k) le permite ser utilizado para aislante térmico. [44]

Una característica principal de la Cascara de arroz (CA) es su poder calorífico, esta depende directamente de la humedad en el que se encuentre el material. [45]

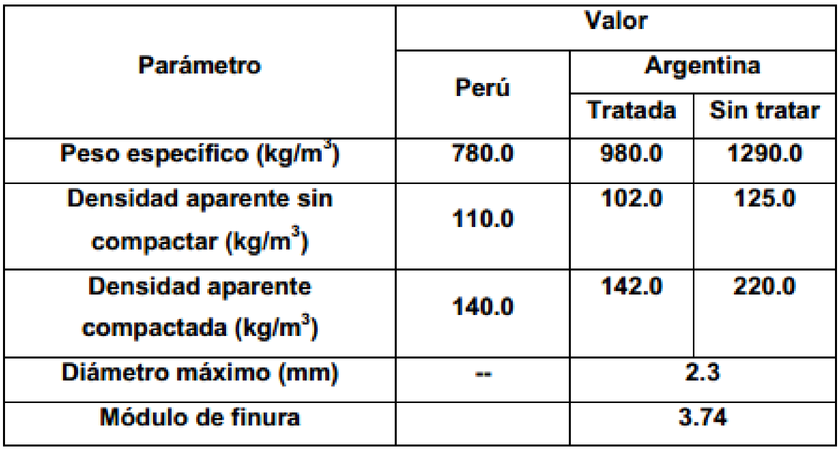
Tabla 1.6 Características físicas de la cáscara de arroz



Características	g/cm^3
Densidad real	0,780
Densidad global sin compactar	0,108
Densidad global compactado	0,143

Fuente: Gonzáles de la Cotera, M. .

Tabla 1.7 Características físicas de la cáscara de arroz



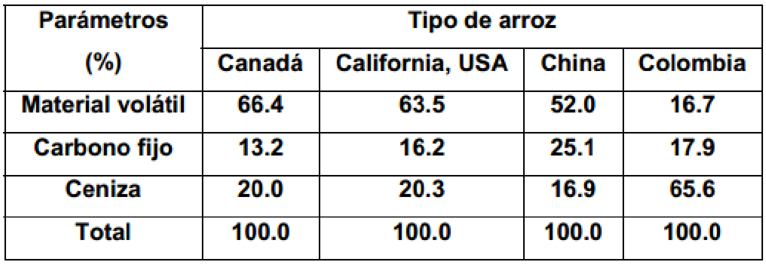
Parámetro	Valor		
	Perú	Argentina	
		Tratada	Sin tratar
Peso específico (kg/m^3)	780.0	980.0	1290.0
Densidad aparente sin compactar (kg/m^3)	110.0	102.0	125.0
Densidad aparente compactada (kg/m^3)	140.0	142.0	220.0
Diámetro máximo (mm)	--	2.3	
Módulo de finura		3.74	

Fuente: Giancarlo Chur Pérez. EVALUACIÓN DEL USO DE LA CASCARILLA DE ARROZ COMO AGREGADO ORGÁNICO EN MORTEROS DE MAMPOSTERÍA. Universidad de San Carlos de Guatemala. Facultad de Ingeniería. Escuela de Ingeniería Civil. 2010

1.4.4 Composición química

Es un tejido vegetal constituido por celulosa (± 40%) y sílice, presenta un alto contenido de dióxido de silicio (SiO2), al fundirse con otros óxidos metálicos genera diferentes variedades de vidrio y se utiliza en la fabricación de cementos y materiales cerámicos. Entre los porcentajes más significativos de la cáscara de arroz se encuentran las cenizas, tiene un elevado contenido de materia volátil en comparación con los carbones.

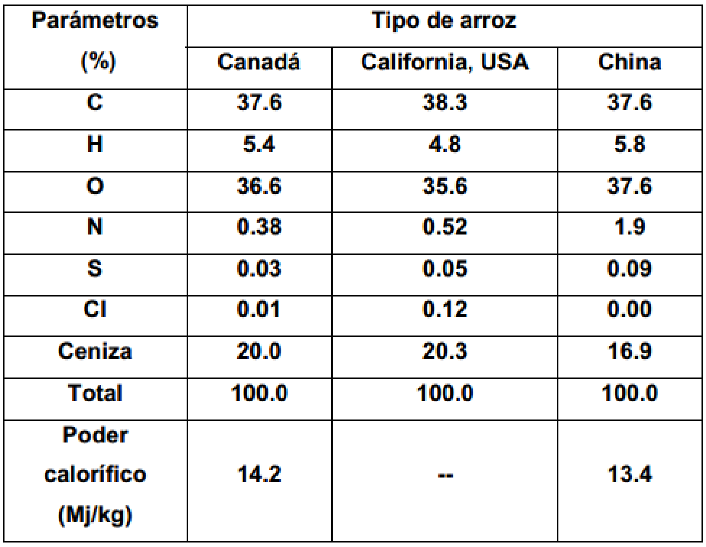
Tabla 1.8 Composición de cáscara de arroz, diferente origen



Parámetros (%)	Tipo de arroz			
	Canadá	California, USA	China	Colombia
Material volátil	66.4	63.5	52.0	16.7
Carbono fijo	13.2	16.2	25.1	17.9
Ceniza	20.0	20.3	16.9	65.6
Total	100.0	100.0	100.0	100.0

Fuente: Giancarlo Chur Pérez. EVALUACIÓN DEL USO DE LA CASCARILLA DE ARROZ COMO AGREGADO ORGÁNICO EN MORTEROS DE MAMPOSTERÍA. Universidad de San Carlos de Guatemala. Facultad de Ingeniería. Escuela de Ingeniería Civil. 2010

Tabla 1.9 Composición elemental de cáscara de arroz, diferente origen



Parámetros (%)	Tipo de arroz		
	Canadá	California, USA	China
C	37.6	38.3	37.6
H	5.4	4.8	5.8
O	36.6	35.6	37.6
N	0.38	0.52	1.9
S	0.03	0.05	0.09
Cl	0.01	0.12	0.00
Ceniza	20.0	20.3	16.9
Total	100.0	100.0	100.0
Poder calorífico (Mj/kg)	14.2	--	13.4

Fuente: Giancarlo Chur Pérez. EVALUACIÓN DEL USO DE LA CASCARILLA DE ARROZ COMO AGREGADO ORGÁNICO EN MORTEROS DE MAMPOSTERÍA. Universidad de San Carlos de Guatemala. Facultad de Ingeniería. Escuela de Ingeniería Civil. 2010

1.4.5 Producción y cultivo del Arroz

El arroz es el segundo cereal de mayor consumo en el mundo, la producción está geográficamente concentrada y más del 85% proviene de Asia. Tan solo siete países asiáticos (China, India, Indonesia, Bangladesh, Vietnam, Myanmar y Tailandia) producen y consumen el 80% del arroz del mundo [46].

La producción mundial de arroz cáscara en el 2005 fue de 628 millones de toneladas. China es el mayor productor con 28% del volumen total seguido de la India con 22%, Indonesia con 9%, Bangladesh y Vietnam con 6%, Tailandia con 5%, Myanmar con 4% y otros países representan el 20% [46].

Las variedades de arroz cultivadas han ido variando en los últimos años, mediante una gradual renovación de las más antiguas, en función de las mejores características; provocando la desaparición de determinadas variedades, pues las nuevas ofrecen mejores rendimientos, una mayor resistencia a enfermedades, altura más baja, mejor calidad de grano o una mayor producción. Los programas de mejora genética se basan en la producción de plantas de arroz dihaploides, mediante el cultivo de anteras de plantas obtenidas a partir de cruzamientos previos [46].
Las principales organizaciones gremiales en el Perú son el Comité Nacional de Productores de Arroz, algunos Comités Regionales de Productores de Arroz y la Asociación Peruana de Molineros de Arroz [46].

En el mercado mundial los precios que se toman como referencia para el comercio de arroz son los de Tailandia y los de Estados Unidos.

El comercio mundial del arroz durante los próximos 15 años (de 18 millones en 1996 a 21 millones en 2010), se estima que incrementará a razón de una tasa anual de 1.11%, tasa significativamente inferior a la actual (8.82%) y refleja el hecho de que el impacto mayor de la liberalización comercial mundial ya surtió efecto.

La devaluación de las monedas asiáticas de mediados del año 1997 provocó una fuerte caída de los precios internacionales del orden del 54% en Estados Unidos y del 40% en Tailandia entre las zafras 1996/1997 y la 2002/2003. Al efecto de la devaluación se sumó un récord en la producción mundial de la zafra 1999/2000 y excedentes en los principales países exportadores, todo lo cual mantuvo los precios internacionales deprimidos.

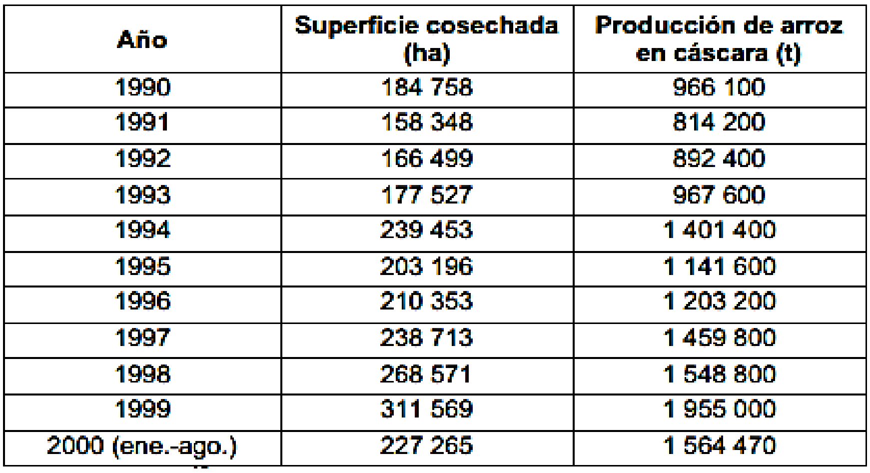
La cáscara de arroz constituye un subproducto del proceso agroindustrial, con aplicaciones tan reducidas en la actualidad, que en la práctica se le puede considerar como material de desecho.

La más significativa implantación de la industria arrocera en el Perú se encuentra en la costa norte, que posee más del 50% de las instalaciones existentes. La cáscara de arroz se comercializa con precios variables según la zona, el molino y la oportunidad [47].

Actualmente en el Perú la cáscara de arroz se utiliza como combustible, como aislante térmico para conservar bloques de hielo y como materia prima para la elaboración del llamado pulitón que es un abrasivo barato para uso doméstico. Excepcionalmente se le utiliza como ingrediente en la fabricación de adobes. La producción de arroz se ha incrementado en los últimos años por el aumento de la superficie dedicada a este cultivo, por la acción de nuevas variedades introducidas y el incremento de la productividad [47].

En la Tabla 1.10 se muestra la producción de arroz en cáscara y la superficie cosechada en los últimos 10 años.

Tabla 1.10 Producción de arroz en cáscara



Año	Superficie cosechada (ha)	Producción de arroz en cáscara (t)
1990	184 758	966 100
1991	158 348	814 200
1992	166 499	892 400
1993	177 527	967 600
1994	239 453	1 401 400
1995	203 196	1 141 600
1996	210 353	1 203 200
1997	238 713	1 459 800
1998	268 571	1 548 800
1999	311 569	1 955 000
2000 (ene.-ago.)	227 265	1 564 470

Fuente: MINAG [47]

1.5 Materiales Compuestos

Se denomina material compuesto a un material que está formado por la combinación de diferentes componentes obtenido a partir de la unión, no química, de ellos (por ejemplo, resina + fibras), de tal manera que se consigue un efecto sinérgico en las propiedades finales, obteniéndose materiales con unas propiedades o características específicas superiores a las de los componentes individuales iniciales. Además, las fases constituyentes deben ser químicamente distintas y separadas por una intercara.

Los compuestos presentan discontinuidad estructural, de tal modo que los componentes son distinguibles físicamente y separables física (Mecánicamente) o químicamente. Se pueden así diseñar materiales que poseen propiedades que no están disponibles en los materiales individuales.

Normalmente, se tiene que las propiedades de la matriz se mejoran al incorporar el refuerzo. En estos materiales se deben considerar las matrices, las cargas y los refuerzos. La matriz es la fase continua que rodea a la otra fase y el refuerzo es la fase discontinua o dispersada. Existe una intercara entre la matriz y el refuerzo, que es el tercer componente del material compuesto. La importancia de la intercara radica en que es la responsable de la interacción refuerzo (fibra)-matriz.

Figura 1.16 *- Formación de un material compuesto usando una resina y fibras*

La definición dada de material compuesto no es suficiente y deben satisfacerse otras tres condiciones antes de que un material pueda considerarse como compuesto.

1.- Ambos constituyentes deben estar presentes en una proporción razonable (> 5 %).

2.- Las fases constituyentes presentes han de tener propiedades diferentes y además las propiedades del material compuesto han de ser notablemente diferentes de las propiedades de los constituyentes

3.- Los materiales compuestos se producen, usualmente, por un mezclado íntimo y combinación de sus constituyentes empleando varios medios

Existen materiales compuestos naturales, como por ejemplo la madera, que consiste en fibras de celulosa flexibles embebidas en un material rígido llamado lignina. El hueso es un material compuesto formado por colágeno, una proteína resistente pero blanda, y por apatito, un mineral frágil.

En cuanto a los materiales compuestos de polímeros y fibras naturales podemos mencionar algunos estudios que se realizaron como por ejemplo en Brasil la multinacional Draimler Benz en conjunto con la UNICEF promovieron la aplicación de biocompuestos en la elaboración de sus vehículos clase E y camiones clase A y después de realizar investigaciones para sustituir las fibras sintéticas con fibras naturales comenzaron un plan piloto con la fabricación de apoyacabezas en los vehículos a base de coco obteniendo excelentes resultados. También han usado yute y algodón en la parte interior de sus vehículos, y han desarrollado biocompuestos con características técnicas, utilizando estos biocompuestos en los paneles de las puertas en los pilares de las cabinas, como parte de asientos, parachoques, entre otros [48].

La aplicación de las fibras en estos compuestos también ha servido para usarlos como aislante acústico y además poseen la virtud de no astillarse en caso de impacto directo en el interior de sus vehículos. La tecnología usada para estos fines es el moldeo por compresión con fibras como lino, sisal, algodón y una mezcla de lino / algodón; utilizando diversas matrices como polipropileno (termoplástico), epoxy, poliuretano y resinas fenólicas (termofijas), dependiendo de la aplicación.

1.5.1 Los Biocompuestos

Los primeros Compuestos que fueron diseñados a partir de fibras naturales se denominaron “bioCompuestos”, por estar formados parcial o totalmente por materiales de origen natural [49]. Sin embargo, las matrices poliméricas de estos primeros biocompuestos eran polímeros convencionales (polipropileno, polietileno, epoxis, etc.) [50,98]. Estos biocompuestos se usan fundamentalmente en el campo de la construcción y del automóvil y son ya una alternativa real frente al uso de los compuestos procedentes de fuentes no renovables [51,52].

El diseño de biocompuestos formados a partir de una matriz biodegradable y fibras naturales (compuestos verdes) es una alternativa más ambiciosa y respetuosa con el medio ambiente que la anterior. El diseño de este tipo de biocomposite permite: la conservación de los recursos petrolíferos, la completa degradación biológica de sus componentes, la reducción del volumen de los residuos poliméricos, la reducción de la cantidad de dióxido de carbono emitido a la atmósfera y nuevas aplicaciones para los recursos agrícolas. Por tanto, el diseño de "compuestos verdes" presenta grandes ventajas, no sólo por cubrir una serie de aplicaciones específicas, sino por ser una alternativa de gestión de residuos [53, 54].

1.5.2 Clasificación de los biocompuestos

Como ya se ha dicho, los biocompuestos son materiales compuestos de fibras naturales y polímeros no biodegradables sintéticos (PP, PE y epoxis) o polímeros biodegradables (PLA, PHA). También se usa este término para definir a los compuestos conformados a partir de fibras sintéticas y polímeros biodegradables. De entre todos los tipos de biocompuestos, los más respetuosos con el medio ambiente, se conocen como "compuestos verdes" y están formados por fibras naturales y polímeros biodegradables [**49**,55]. En la Figura 1.16 se esquematiza de forma sencilla la clasificación de los biocompuestos de fibras naturales.

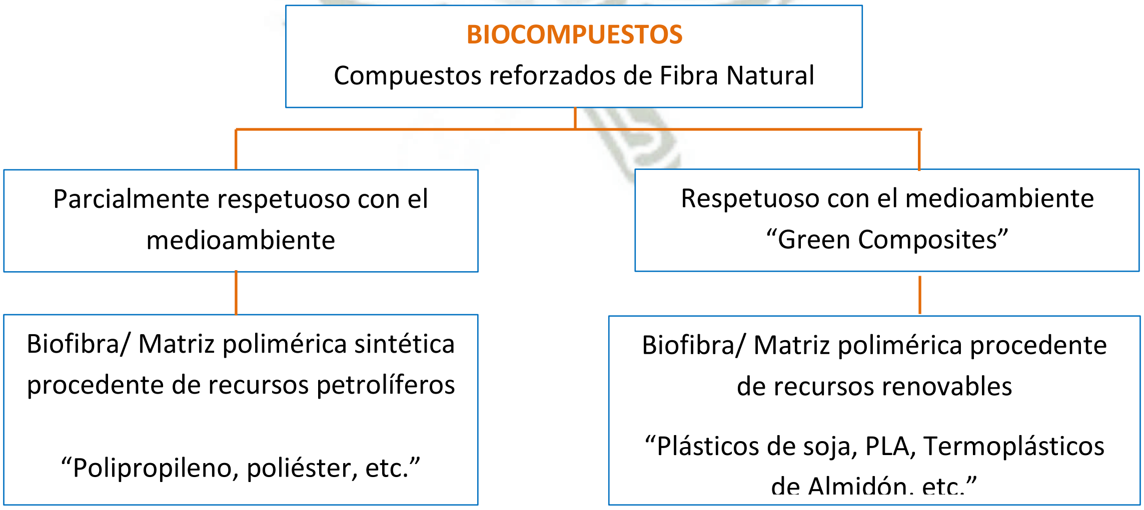


Figura 1.17. *Clasificación de los biocompuestos de fibras naturales [49]*

a) Compuestos verdes

Actualmente un gran número de investigadores centran sus esfuerzos en el desarrollo de los Compuestos verdes [56, 57]. Las características más atractivas de estos bioCompuestos es que tanto la matriz polimérica como las fibras naturales son materiales biodegradables, que pueden ser depositados en la naturaleza sin que su eliminación suponga ningún tipo de riesgo medioambiental.

Dependiendo de los materiales poliméricos escogidos como matriz polimérica, se puede realizar una subclasificación [58].

Compuestos basados en termoplásticos biodegradables

Dentro de esta categoría se encuentran aquellos Compuestos cuyas matrices poliméricas están conformadas por:

- Polímeros biodegradables sintéticos tales como el ácido poli (glicol) (PGA), el poli (vinil alcohol) (PVA), etc.
- Polímeros biodegradables naturales tales como el ácido poli(láctico) (PLA), el almidón termoplastificado (TPS) y sus mezclas, la celulosa regenerada, etc.

Imam y col. [59] evaluaron la estabilidad térmica de los Compuestos diseñados a partir de la mezcla de poli(vinil alcohol), almidón y fibras lignocelulósicas. En este estudio, el análisis termogravimétrico se utilizó para confirmar la formulación escogida, asegurándose la estabilidad del composite durante su procesado y vida útil. Además se estudió la degradación de los materiales base y del biocomposite. Se demostró que la celulosa y el almidón se degradaban más rápidamente que el PVA. Pero que al adicionar la fibra natural la velocidad de degradación del PVA aumentaba.

b) Compuestos híbridos

El término híbrido se usa para describir el fenómeno de un aumento sinergético en las propiedades de las Compuestos al mezclar dos o más tipos de fibras en su formulación. El comportamiento de los Compuestos híbridos ideales es la suma del comportamiento de los componentes individuales, las propiedades de un tipo de fibra pueden complementar a las propiedades de las otras. El diseño de Compuestos híbridos implica un balance entre el coste y el uso [60]. Las propiedades de un composite híbrido dependen mayoritariamente del contenido de fibra, de la longitud individual de la fibra, de la orientación, del tipo de enlaces entre la fibra y la matriz y el empaquetamiento de ambas fibras. Para conseguir diseñar un composite híbrido competitivo, cabe tener en cuenta tanto a la matriz polimérica como a la fibra natural y al sistema fibras/matriz polimérica que deben ser química, mecánica, física y térmicamente estables.

1.5.3 Comportamiento de los Materiales Compuestos

1.5.3.1 Materiales compuestos reforzados con fibras continua

Las propiedades mecánicas de los materiales compuestos de fibra larga (LFRTP’s) dependen del contenido y de la orientación de las fibras.

Un factor importante es la relación de forma, ésta se define como el cociente entre la longitud de la fibra y su diámetro; por tanto las fibras con un parámetro relación de forma alta, son largas y delgadas, mientras que las fibras con un parámetro bajo son más cortas y más anchas. La relación de forma de una fibra vegetal individual procedente del tallo o de la corteza de las plantas es más alta que las fibras madereras. Algunos autores, consideran ventajoso conseguir la máxima longitud posible de fibra, ya que a más alta relación de forma se puede alcanzar una mayor eficacia de refuerzo [54].

Se determinará la relación entre las propiedades elásticas de una lámina unidireccional y las propiedades de los constituyentes y se comparan con los resultados experimentales. Esto viene seguido por una descripción de las relaciones correspondientes para láminas de fibras largas en disposición aleatoria. En ambas láminas, la unidireccional y la de fibras largas en disposición aleatoria, pueden ignorarse los efectos asociados a las puntas de las fibras, excepto cuando se consideren los procesos de rotura. Esto no es verdad para materiales compuestos de fibra corta.

1.5.3.2 Materiales compuestos reforzados con fibras corta

Desde el punto de vista tecnológico, los materiales compuestos con fases dispersas en forma de fibras son los más importantes. A menudo se diseñan materiales compuestos reforzados con fibras con la finalidad de conseguir elevada resistencia y rigidez con una baja densidad. Estas características se expresan mediante los parámetros resistencia específica y módulo específico, que corresponden, respectivamente, a las relaciones entre la resistencia a la tracción y el peso específico y entre el módulo de elasticidad y el peso específico. Utilizando materiales de baja densidad, tanto para la matriz como para las fibras, se fabrican compuestos reforzados con fibras que tienen resistencias y módulos específicos excepcionalmente elevados.

Los materiales compuestos reforzados con fibras se pueden clasificar utilizando como criterio la longitud de la fibra (Figura 1.17). En el caso de fibras cortas, éstas suelen ser demasiado cortas como para conseguir un apreciable aumento de la resistencia.

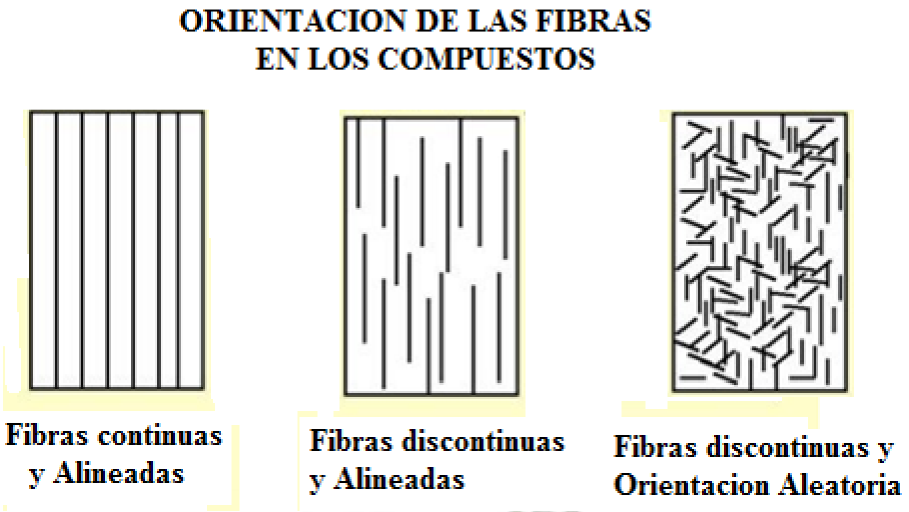


***Figura 1.18** Clasificación de los materiales compuestos reforzados con fibras.*

El reforzamiento con fibras continuas, como regla general, es más caro que otras formas de reforzar el material. Además los procesos de fabricación de materiales compuestos reforzados con fibra continua suelen ser más lentos e inflexibles.

El precio a pagar por el uso de tales técnicas de producción en masa es un acortamiento de la longitud de la fibra. Esta reducción en la longitud de la fibra se debe en parte a las exigencias de la técnica de procesamiento, pero algunos procesos que involucran acciones mecánicas de corte y mezcla también pueden promover una rotura de fibras considerable. El daño de las fibras es especialmente notable en el moldeo por inyección, extrusión y mezcla de materiales compuestos moldeados a base de poliéster.

Se pueden considerar tres grupos de materiales de fibra corta.

- En el primer grupo de materiales las fibras cortas están orientadas paralelamente unas con otras. Se requieren métodos especiales para alinear las fibras cortadas, con el fin de conseguir un fieltro, que se impregna luego con resina. Se puede obtener también un alto grado de orientación usando procesos de extrusión y de moldeo por inyección con un control cuidadoso de la forma del flujo. Las variables principales, además de las propiedades de la matriz, son la longitud de la fibra, la distribución de longitudes de fibra y la resistencia de la intercara.
- El segundo grupo de materiales contiene fibras largas cortadas orientadas aleatoriamente en un plano (fieltro de mechas cortadas, CSM)

Figura 1.19 *Fieltro de mechas cortadas, CSM*

En la práctica normal, las fibras reciben un tratamiento de preparación para mantenerlas unidas en la mecha de forma que los paquetes o haces de fibras mantengan su integridad. De esta manera, la "unidad" de refuerzo es el paquete de fibras. Puede obtenerse un efecto parecido con los compuestos para hojas continuas (SMC, sheet moulding compound) y los "premix" (compuestos de moldeo en pasta, DMC) que están basados también en resinas termoestables. La preparación de las fibras puede seleccionarse para obtener diferentes grados de dispersión de la fibra durante el moldeo, que conduzcan a diferentes propiedades, particularmente con respecto a la tenacidad de la rotura.

- El tercer grupo de materiales está basado en productos de moldeo por inyección que tienen fibras cortas como elemento reforzador. La matriz es normalmente un polímero termoplástico, aunque se usan algunas matrices termoestables. Las fibras pueden tener una disposición completamente aleatoria, pero normalmente muestran un grado de preferencia en la orientación a causa del campo de flujo. Pueden producirse grandes diferencias de orientación de una parte del moldeado a otra.

CAPÍTULO II

METODOLOGÍA EXPERIMENTAL

2.1. Materiales y equipos.

2.1.1. Materiales

- Ácido Poliláctico (PLA).

Figura 2.1 Ácido poliláctico en Pellets

- Cascara de Arroz.

Figura 2.2 Cáscara de Arroz molido.

- Alcohol poli vinílico.

***Figura* 2.3** *Alcohol Poli vinílico*

- Tinner

***Figura* 2.4** *Tinner*

- Cera Desmoldante

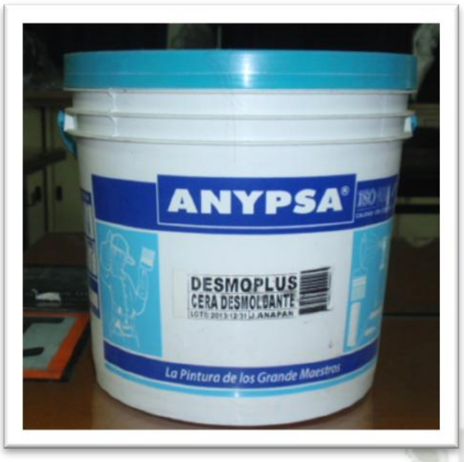


Figura 2.5 *Cera desmoldante*

- Waype industrial.

Figura 2.6 *Waype industrial*

- Espátula

Figura 2.7 *Espátula*

- Escobilla de bronce.

Figura 2.8 *Escobilla de Bronce*

- Brochas

Figura 2.9 *Brochas*

- Guantes

Figura 2.10 *Guantes protectores*

- Llave mixta ¾"'

Figura 2.11 Imagen de la llave mixta

- Extensión ½"

Figura 2.12 Imagen de la extensión ½''

- Dado ½"

Figura 2.13 Imagen del dado ½''

- Material de vidrio: Placas Petri, vasos de precipitado, baguetas.

2.1.2. Equipos

- Brabender.

Figura 2.14 *Equipo Brabender*

- Prensa para fabricar probetas.

Figura 2.15 *Prensa para fabricar probetas*

- Ro-tap

Figura 2.16 *Ro-tap*

- Tamices

Figura 2.17 *Tamices Nº 8, 10, 25 y 50*

- Paletas para fabricación de probetas

Figura 2.18 *Paletas de acero Inoxidable*

- Balanza

Figura 2.19 *Balanza*

- **Equipo de Ensayo de tracción**

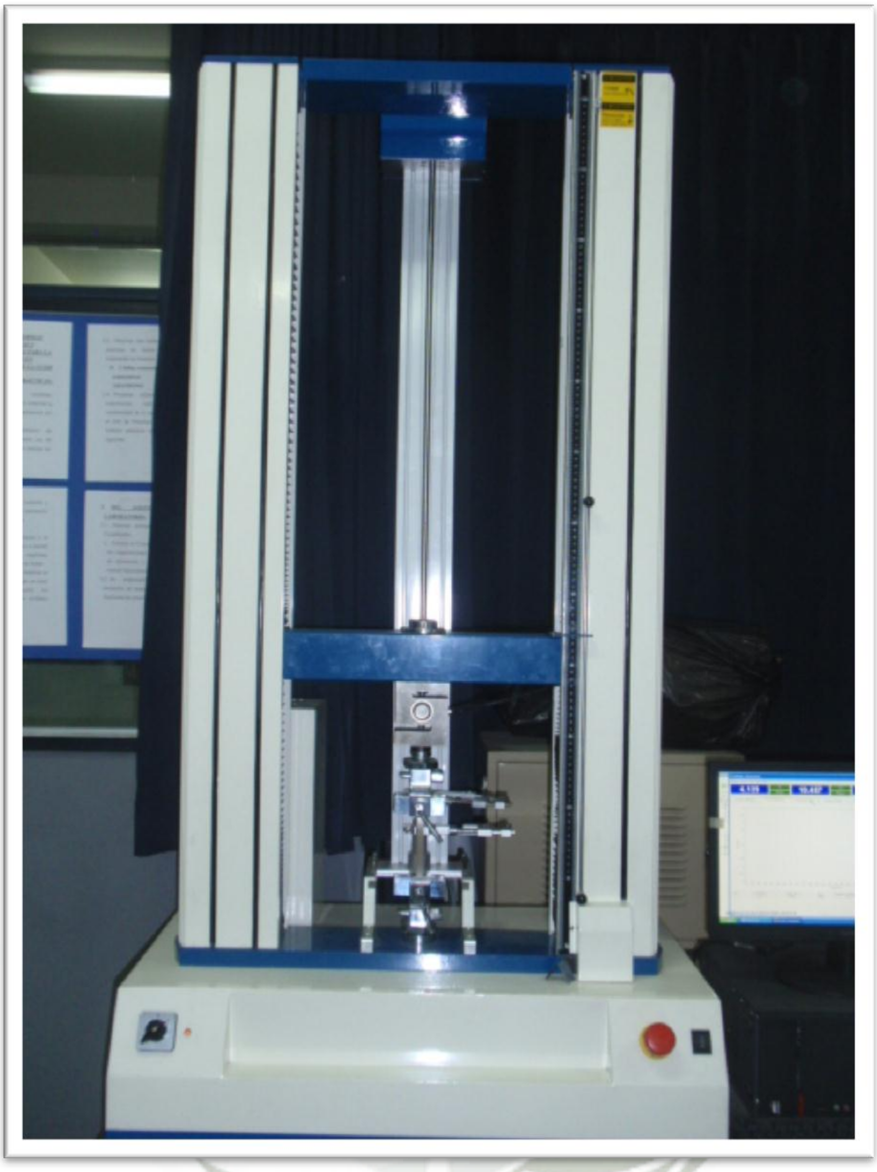


Figura 2.20 *Equipo de Ensayo de tracción*

- Maquina Universal de Ensayo

Figura 2.21 *Maquina Universal de Ensayo*

- Sierra Caladora

Figura 2.22 *Sierra Caladora*

- Durómetro Shore D

***Figura* 2.23** *Durómetro Shore D*

- Equipo para ensayo de Charpy

***Figura* 2.24** *Equipo para ensayo de Charpy*

- Plastómetro

***Figura* 2.25** *Plastómetro*

- Microscopio Óptico simple Hund Wetzlar

***Figura* 2.26** *Microscopio*

2.2. Metodológica

2.2.1. Elaboración del material compuesto

Antes de la elaboración del material compuesto debemos acondicionar la cascara de arroz:

- Primero sacudimos la cascara de arroz en el Ro-tap durante 15 minutos en la malla Nº 8 para eliminar la tierra e impurezas que pueda contener.
- Luego trituramos la cáscara de arroz en una licuadora.
- Después procedemos a tamizar la cascara de arroz molida, usamos las mallas Nº 8, 10, 25 y 50 para hacer un análisis granulométrico.
- Para la preparación de los compuestos usamos el pasante de la malla 50.

Tabla Nº 2.1. Mallas Usadas para el Tamizado

Numero	Serie de Tyler	Abertura (mm)
1	8	2.38
2	10	2.00
3	25	0.707
4	50	0.297

Para realizar análisis granulométrico se siguió el siguiente procedimiento:

- Limpiar los tamices que serán usados.
- Ordenar y colocar los tamices de acuerdo a la tabla Nº 2.1 (contando desde arriba) sobre el Ro–tap, en la parte inferior colocar el fondo contenedor del pasante del último tamiz y en la parte superior colocar la tapa.
- Pesar la muestra a ensayar y verter en el tamiz superior.
- Prender el Equipo Ro-tap, programar el tiempo por un periodo de 15 – 20 minutos e iniciar el ensayo.
- Pasado el tiempo de ensayo pesar el retenido de cada malla y también lo contenido en el fondo de la serie de tamices.

2.2.1.1. Parámetros para la elaboración del PLA

La preparación de las mezclas se efectúa haciendo uso del brabender del Laboratorio de Ensayo de Materiales de la Universidad Católica de Santa María (figura 2.14). Antes de procesar limpiamos el brabender con solventes orgánicos (Acetona o tinner) en nuestro caso usamos tinner.

Se prepararon compuestos con tres porcentajes de cáscara de arroz diferentes (ver tabla 2.2).

Tabla Nº 2.2. Relación de porcentajes y pesos de los materiales en cada mezcla

Código	**Compuesto de Cascara de Arroz con PLA**	**Matriz PLA**		**Reforzante YUTE**	
		(g)	**%**	**(g)**	**%**
CAM-0.50	Cascara de Arroz Molido	20.00	97.50	0.50	2.50
CAM-0.75	Cascara de Arroz Molido	20.00	96.25	0.75	3.75
CAM-1.00	Cascara de Arroz Molido	20.00	95.00	1.00	5.00

Fuente: Elaboración propia

Se prepararon las tres mezclas con las mismas condiciones:

- ✓ La temperatura de trabajo es de 145 ºC.
- ✓ El tiempo de ablandamiento del PLA 3 minutos.
- ✓ El tiempo de mezclado con la cascara de arroz 2 minutos.

El procedimiento fue el siguiente:

- La cascara de arroz usada para la preparación de los compuestos es la pasante de la malla Nº 50.
- De acuerdo a la tabla Nº 2.2 pesar por separado el PLA y la cascara de arroz.
- Cuando el equipo de brabender este a la temperatura de 145 ºC verter el PLA en la cámara de mezcla encendida.
- Esperar 3 minutos hasta que el PLA se ablande.
- Luego verter la cascara de arroz y mezclar por dos minutos.
- Pasado el tiempo de mezcla apagar el brabender y abrir la cámara de mezcla usando la llave mixta, la extensión y el dado (Ver figura 2.27).

- Extraer toda la mezcla haciendo que todo el material forme una sola masa redonda (Ver figura 2.29)

Figura 2.27 *Abertura de la cámara de mezcla del Brabender*

Figura 2.28 *Extracción del compuesto de la Cámara de mezcla abierta del Brabender*

Figura 2.29 *Bola formada a la salida del brabender.*

2.2.1.2. Elaboración de láminas de material compuesto por compresión

Se prepara dos placas de cada mezcla por moldeo por compresión, usando una prensa de Laboratorio de Ensayo de Materiales de la Universidad Católica de Santa María (LEM-UCSM) con las condiciones de procesamiento siguiente:

- 3 minutos a 180°C sin presión para calentar las placas de acero inoxidable
- 1 minutos a 180°C sin presión para ablandar el material,
- 8 minutos a 180°C con presión constante.
- Se deja enfriar a temperatura de ambiente.

El procedimiento fue el siguiente:

a. Preparación de las placas de acero Inoxidable:
    - ✓ Primero limpiamos las placas con la escobilla de bronce, espátula y guaipe.
    - ✓ Con guaipe y tiñer volvemos a limpiar.
    - ✓ Añadir una capa cera desmoldante y pulir las placas.
    - ✓ Usando guaipe y alcohol polivilico mojar una cara de las dos placas, esperar que seque y volver a echar una capa de alcohol polivinílico.
    - ✓ Esperar que el alcohol seque por completo.

Figura 2.30 *Preparación de las placas de Acero inoxidable.*

b. Elaboración de láminas

- ✓ Cuando la prensa de termoformado este a la temperatura de 180 °C calentar las placas de acero inoxidable por tres minutos.
- ✓ Una vez obtenido la bola a la salida del brabender esta se coloca en los platos ya preparados (ver figura 2.31).
- ✓ Ablandar el material sin presión durante 1 minuto a 180°C (ver figura 2.32).
- ✓ Luego ejercer presión constante a una temperatura de 180°C durante 8 minutos.
- ✓ Extraer los platos, enfriar a temperatura ambiente y extraer la lámina formada.

Figura 2.31 *Material extraído del Brabender colocado en las paletas de acero inoxidable.*

Figura 2.32*Ablandamiento del compuesto.*

2.2.2. Obtención de probetas normalizadas

Según la norma ASTM D 882-02 las probetas consistirán en tiras de uniforme anchura y espesor. La anchura nominal de las muestras no deberá ser menor de 5,0 mm (0,20 pulg.) o mayor que 25,4 mm (1 pulg.).

Para la fabricación de las probetas según norma ASTM D 882-02 se siguió el siguiente procedimiento:

- Marcar la lámina delgada cada 10 mm de ancho y después trazar líneas de corte sobre las marcas antes hechas.
- Colocar la lámina marcada entre dos planchas metálicas para que la sujeten, ajustando el borde de las planchas cerca de la línea de corte.
- Colocar dos sujetadores para mantener inmovilizada la lámina a la hora del corte.
- Con la sierra caladora hacer un corte encima de la línea dibujada.
- Se quitan los bordes excedentes de las probetas con lijar Nº 220 y 400 para evitar fisuras o concentradores de esfuerzos.

Figura 2.33 *Corte para la fabricación de probetas*

Siempre que sea posible, se deben seleccionar los especímenes de ensayo de manera que el espesor sea uniforme.

Se midió el espesor de cada probeta en la zona central antes del ensayo con un micrómetro DIGITAL MITUTOYO con una resolución de 1µm.

También se midió la longitud y ancho de las probetas con un vernier digital MITUTOYO con una resolución de 0.01mm.

2.3. Técnicas de caracterización mecánica de las probetas fabricadas

2.3.1. Ensayos de tracción de acuerdo con ASTM D 3039-08

Al final del proceso de moldeo por compresión se obtuvieron láminas de material compuesto de tres configuraciones de cascara de arroz. Como se muestra en la Tabla 2.2. Con estas láminas se fabricaron probetas de tracción, flexión e impacto para caracterizar a cada uno de los materiales, de acuerdo al procedimiento que se detalla a continuación:

- ☞ Se eliminaron residuos del procesamiento y se desecharon los extremos de la lámina que normalmente no presentaban un buen acabado.
- ☞ Se cortaron las probetas de acuerdo a la norma establecida mediante una sierra manual.
- ☞ La probetas fueron lijadas en sus bordes con una lija número 400 para evitar fisuras o concentradores de esfuerzos.

Las pruebas de tracción fueron realizadas de acuerdo a la norma ASTMD- 3039-08 en una máquina universal LIYI TECH, como se muestra en la Figura 2.20, a una temperatura de aproximadamente 22 °C, y la velocidad de la mordaza superior fue de 10 mm/min. Con esta prueba se busca conocer cómo influye en el comportamiento mecánico las cargas de cascara de arroz de las diferentes mezclas de los compuestos obtenidos, teniendo como referencia el PLA puro.

El procedimiento seguido para el uso del equipo fue el siguiente:

- Seleccione un rango de carga de tal manera que se produzca el fallo dentro de sus dos tercios superiores. Unas ejecuciones de prueba pueden ser necesarias para seleccionar una combinación adecuada de rango de carga y el ancho de muestra.

- Medir el área de la sección transversal de la muestra en varios puntos a lo largo de su longitud. Mida el ancho con un vernier a una exactitud de 0,25 mm (0,010 in.). Mida el espesor con una precisión de 0,0025 mm (0,0001 in.)
- Ajuste la velocidad de separación de las mordazas, basado en la distancia inicial entre las mordazas.
- Colocar la muestra de ensayo en las mordazas de la máquina de prueba, teniendo cuidado de alinear el eje largo de la muestra con una línea imaginaria que une los puntos de unión de las mordazas a la máquina. Apretar las mordazas de manera uniforme y con firmeza al grado necesario para reducir al mínimo el deslizamiento de la muestra durante la prueba.
- Ingresar los datos de la probeta, como longitud de ensayo, ancho y espesor de la probeta.
- Inicie la carga de la máquina.
- Al observar el rompimiento de la probeta detener el ensayo.
- Retirar la probeta.
- Medir la longitud final de la probeta.

Cálculos

A) Resistencia a la tracción (nominal) se calculará dividiendo la carga máxima entre el área de la sección transversal original mínimo de la muestra. El resultado se expresará en fuerza por unidad de área, usualmente Mega pascales (o libras-fuerza por pulgada cuadrada).

$$\sigma = F_{max}/A \qquad \text{Ecuación 2.1}$$

Donde F es la fuerza máxima medida en Newton, A es el área de la sección trasversal inicial de la probeta en mm²,

B) Tensión de rotura (nominal) de la misma manera como la resistencia a la tracción, excepto que la carga en ruptura se utiliza en lugar de la carga máxima.

$$\sigma = F_{rup}/A \qquad \text{Ecuación 2.2}$$

C) Porcentaje de alargamiento a la rotura se calculará dividiendo la extensión en el momento de la ruptura del espécimen por la longitud de referencia inicial de la muestra y multiplicando por 100.

$$Ɛ = (\Delta L_0/L_0)x100 \qquad \text{Ecuación 2.3}$$

Donde L_0 la longitud de referencia de la probeta en mm y ΔL_0 es el incremento de la longitud entre las marcas de referencia en mm.

D) Módulo Elástico se calculará mediante la elaboración de una recta tangente a la porción lineal inicial de la curva de carga-extensión, seleccionando cualquier punto de esta tangente, y dividiendo la resistencia a la tracción por el área correspondiente.

2.3.2. Ensayo de flexión de acuerdo con ASTM D 7264-07

El ensayo de flexión se hará siguiendo el procedimiento de la norma ASTM 7264-07, se hará uso de la maquina Universal de ensayo (Figura 2.21). Este método de ensayo determina la rigidez a la flexión y propiedades de resistencia de los materiales compuestos de matriz polimérica, se cuentan con dos procedimientos.

Procedimiento A: De tres puntos del sistema de carga que utiliza carga central en una viga simplemente apoyada.

Procedimiento B: De cuatro puntos el sistema de carga utiliza dos puntos de carga igualmente separados de su soporte, con una distancia entre los puntos de carga de la mitad del tramo de apoyo.

De la norma se escogió trabajar con el procedimiento A, el esquema se muestra en la Figura Nº 2.32.

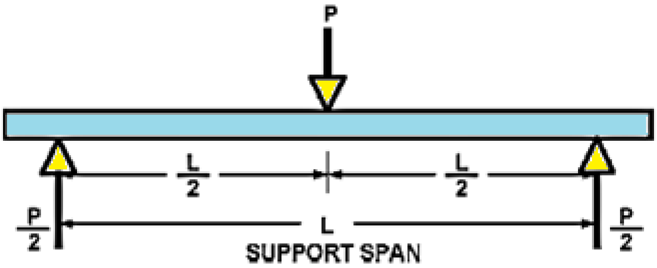


Figura 2.34. Procedimiento A

- En este trabajo se usó el procedimiento A.
- Antes de la prueba, medir y registrar la anchura y el espesor en la sección media de la muestra, así como también la longitud de las muestras.
- Definir la Velocidad de ensayo.
- Alinear el punzón de la carga (P) y soportar de manera que los ejes de las superficies cilíndricas sean paralelas. Para el procedimiento A, la carga estará a medio camino entre los soportes.
- Centrar la muestra en los soportes, con el eje largo de la muestra perpendicular al punzón de la carga y apoyos.
- Aplicar la fuerza a la muestra a la velocidad de ensayo antes definida.
- Mida y registre los datos de esfuerzo-deformación.

Cálculos

Resistencia a la flexión, la tensión máxima en el exterior de la superficie de una muestra de ensayo de flexión correspondiente al pico más alto de fuerza aplicada antes del fallo a la flexión.

Módulo de flexión, la relación de rango de esfuerzos para rango de tensión correspondiente a una muestra de ensayo cargados en flexión

Modos de Falla; para obtener resistencia a la flexión válida, es necesario que la rotura de la probeta se produzca en cualquiera de sus superficies exteriores, sin un fallo de cizalla interlaminar anterior o un fracaso aplastante bajo un soporte de carga. El fallo en la superficie puede ser una grieta, mientras que en la superficie de compresión puede ser de pandeo local. El pandeo puede ser que se manifiesta como la fibra de micro-pandeo o deformación a nivel de capa. Pandeo Ply-nivel puede resultar en, o ser precedido por delaminación de la hoja externa.

La flexión máxima, Procedimiento A - Cuando un haz de material homogéneo, elástico se prueba en flexión viga simplemente apoyada en dos puntos y se carga en el punto medio, la tensión máxima en la superficie exterior se produce a la mitad del tramo. El estrés puede calcularse para cualquier punto de la curva de carga-deflexión por la siguiente ecuación:

$$\sigma = \frac{3PL}{2bh^2}$$

Ecuación 2.4

Dónde:

s = tensión en la superficie exterior en la mitad del tramo, MPa [psi],

P = fuerza aplicada, N [lbf],

L = longitud de apoyo, mm [pulg.],

b = ancho del haz, mm [pulg.], y

h = espesor del haz, mm [pulg.].

2.3.3. Ensayo de resistencia al impacto Charpy de acuerdo con ASTM D6110

Estos métodos de pruebas cubren la determinación de la resistencia de los plásticos al impacto de tipo de péndulos-martillos estandarizados. La tenacidad es una medida de la cantidad de energía que un material puede absorber antes de fracturar. Evalúa la habilidad de un material de soportar un impacto sin fracturarse. Esta propiedad se valora mediante una prueba sencilla en una máquina de ensayos de impacto.

Los resultados son reportados en términos de energía absorbida por unidad de ancho del espécimen o por unidad de área transversal bajo la hendidura.

El ensayo de impacto genera datos útiles cuantitativos en cuanto a la resistencia del material al impacto.

Existen dos maneras distintas de realizar el ensayo el método Izod y el método Charpy; la diferencia radica en la posición de la probeta entallada.

En este trabajo de investigación se usaran como probetas de ensayo, aquellas probetas obtenidas después del uso del plastometro.

Para el ensayo de charpy se siguió el siguiente procedimiento:

Al menos deben de ser ensayadas 5 probetas de cada muestra a ser probada.

Si la maquina está equipada con un sistema indicador digital, seguir las indicaciones del fabricante para corregir la fricción y demás perdidas de energía.

a) Conectar y encender el equipo, seleccionar en el modo las condiciones requeridas; masa del martillo, unidades de la energía (joule).
b) Subir el martillo a la posición más elevada
c) Medir la longitud (mm) y el área transversal (mm^2) en la zona de entalladura.
d) Colocar la probeta de ensayo en el equipo.
e) Ingresar los datos de la probeta (ítems d)
f) Realizar el ensayo liberando el martillo para que impacte la probeta
g) Es necesario que utilice varias probetas para este ensayo de manera que al final pueda determinar el promedio del material que está ensayando.
h) Registre los datos.

2.3.4. Ensayo de Índice de Fluidez ASTM D 1238

Las propiedades reológicas de los materiales son caracterizadas por su índice de fluidez. Se determinó el índice de Fluidez (MFI) empleando un equipo KE JIAN Instrument CO. , LTD KJ – 3092 Metl Flow Indexer. El ensayo se realizó a una temperatura de 158°C y un peso de 7.160 Kg aplicando un precalentamiento de 3 minutos. Se tomaron un mínimo de 7 medidas en el intervalo de 90 segundos que fueron promediados para el cálculo del índice de fluidez.

Un esquema de la composición de un MFI se representa en la figura 2.35.

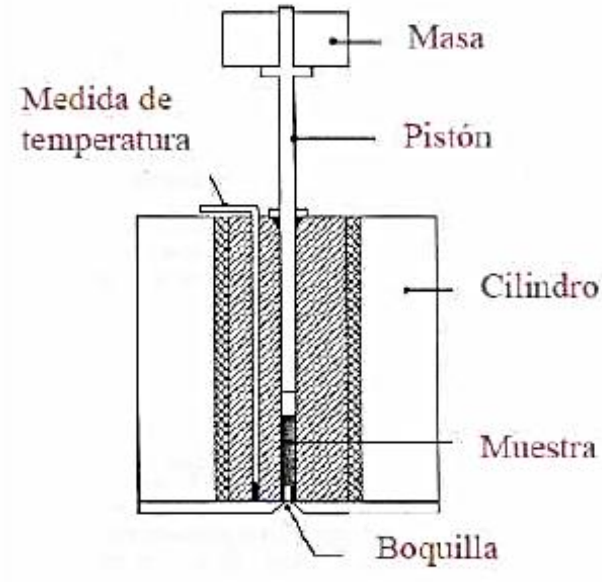


***Figura* 2.35.** *Esquema de la composición de un MFI.*

El espécimen de prueba puede estar en cualquier forma por ejemplo: polvo, pellets, films, placas, etc.

Se sigue el siguiente procedimiento**:**

- Revisar el dado, el cilindro y las dimensiones de posición.
- Limpiar el aparato para cada prueba.
- Fijar la temperatura del plastometro de acuerdo al material.
- Asegurarse que el agujero del plastometro de extrusión este alineado en la dirección vertical.
- Colocar el pistón cargado en su lugar e inmediatamente programe el corte en un tiempo de acuerdo al material
- Registrar la masa extraída de cada probeta de modo que se obtendrá la tasa de flujo en gramos/10min.

$$\text{IF (Temperatura, Carga)} = \frac{m \times T_{ref}}{t} \qquad \text{Ecuación 2.5}$$

Dónde:

m: masa (g) en un tiempo de corte

T_{ref}: tiempo de referencia (10 min) (600s)

t: Tiempo de intervalo para el corte.

2.3.5. Ensayo de dureza ASTM D 2240

Este método permite la medición de la dureza mediante indentación de la muestra utilizando un penetrador estándar. La medición de la dureza se llevó acabo siguiendo la norma ASTM D2240. El método consiste en dentar el espécimen usando un indentador de acero endurecido con la geometría y fuerza específica, basado en la escala escogida de las mediciones. El desplazamiento de la punta del indentador se mide para el cálculo de la dureza del material.

2.3.5.1 Obtención de probetas para el ensayo

Las muestras deben cumplir con lo siguiente:

- ❖ El espesor de la muestra debe ser al menos 6,0 mm.
- ❖ Las caras superficiales deben de ser paralelas
- ❖ No deben de presentar rugosidad
- ❖ El área de ensayo mínimo de 2.5 cm^2

El procedimiento seguido es el siguiente:

- ✓ Montar la muestra en la base inferior. Del durómetro
- ✓ Colocar la aguja roja en cero.
- ✓ Aplicar la carga.
- ✓ Observar la magnitud en el reloj marcador de la aguja roja.

2.3.6. Determinación de la densidad del compuesto

a. Este ensayo se hace por triplicado por cada tipo de mezcla.

b. Se corta una porción de material en forma de cuadrado o rectángulo

c. Se toman las medidas de largo, alto y espesor de las muestras con el vernier y micrómetro.

d. A continuación se pesan las muestras en una balanza con 0.01 g de precisión.

e. Realizar los cálculos

$$\text{Densidad } (g/cm^3) = \frac{\text{masa } (g)}{\text{Volumen } (cm^3)} \qquad \text{Ecuación 2.6}$$

$$\text{Volumen } (cm^3) = \text{Largo } (cm) \times \text{Ancho } (cm) \times \text{Espesor } (cm) \qquad \text{Ecuación 2.7}$$

CAPÍTULO III

ANÁLISIS Y DISCUSIÓN DE RESULTADOS

3.1 Resultados del análisis granulométrico

Tabla 3.1: Análisis granulométrico de la cascara de arroz molida

MALLA	ABERTURA (µm)	PESO RETENIDO (g)	% Peso Retenido	% retenido acumulado	% pasante
8	2360	14.27	10.48	10.48	89.52
10	2000	24.55	18.02	28.50	71.50
25	710	88.14	64.71	93.22	6.78
50	300	8.82	6.48	99.69	0.31
-50	….	0.42	0.31	100.00	0.00
	Total	**136.2**	100.00		

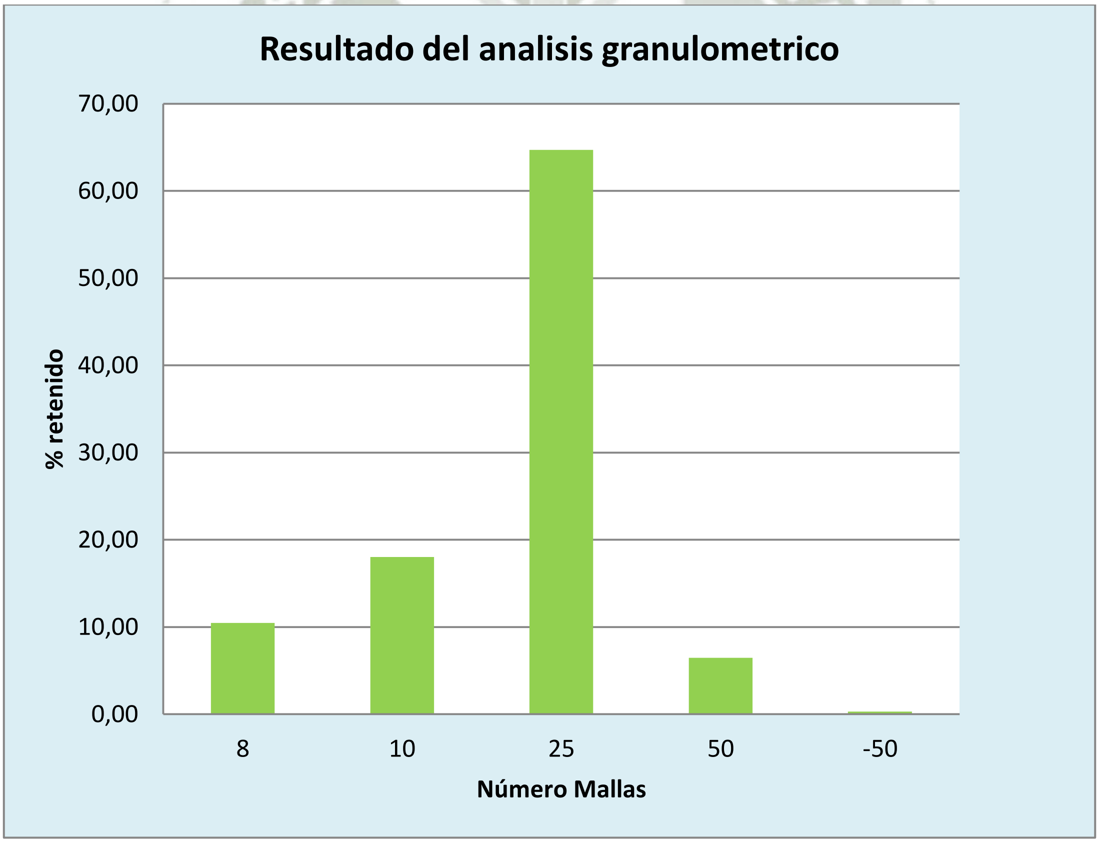


Figura 3.1: Análisis granulométrico de la cascara de arroz.

Descripción: Al observar la gráfica 3.1 que relaciona el número de malla con el % retenido se aprecia que el 64.71% del material tamizado es retenido en la malla Nº 25 con una abertura de 710 um, sin embargo en el presente trabajo se utiliza el pasante de la malla 50 para que la mezcla del biocompuesto sea homogénea y se evite la formación de poros o vacíos por el tamaño de la cascara de arroz.

3.2 Resultados Propiedades Mecánicas

3.2.1 Análisis Mecánico por Resistencia a la Tensión

a) Carga de 0.5 gramos de cascara de Arroz

Tabla 3.2: Datos de las dimensiones de las probetas: Largo, ancho y espesor. De la mezcla de Cascara de Arroz de 0.5g con PLA.

No.	Espesor (mm)	Ancho (mm)	Área (mm^2)
1	1.213	10.22	12.4
2	1.156	9.92	11.465
3	1.306	9.48	12.378

Tabla 3.3: Resultados de los ensayos de tracción de la mezcla de Cascara de Arroz de 0.5g con 20 PLA (2.5%).

No.	resistencia a la tracción (MPa)	Carga Máxima (N)
1	44.797	352.55
2	35.627	220.356
3	36.562	367.554
Valor Máximo	44.797	367.554
Valor Mínimo	35.627	220.356
Valor Medio	38.995	313.487

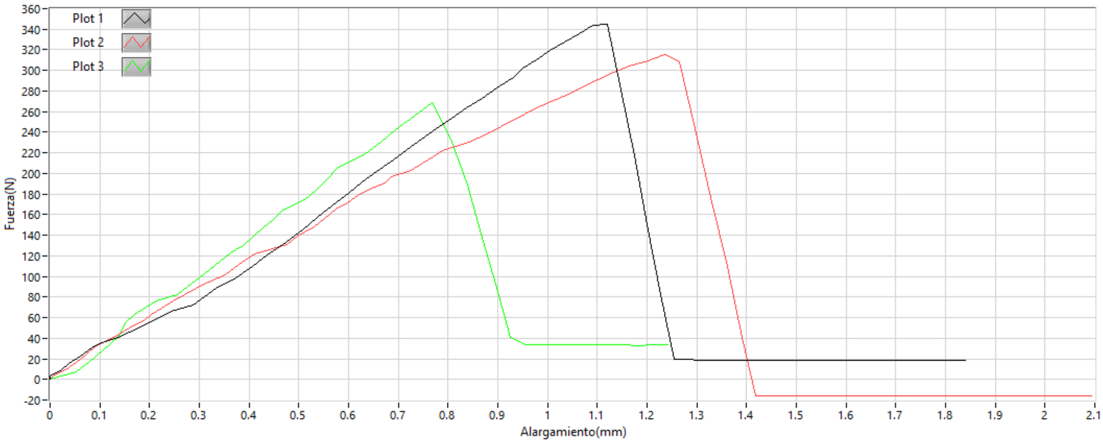


***Figura 3.2**: Gráficas de carga (KN) desplazamiento (mm) para una carga de 0.5 g en 20 g de PLA*

b) Carga de 0.75 gramos de cascara de Arroz

Tabla 3.4: Datos de las dimensiones de las probetas: Largo, ancho y espesor. PLA con Cascara de Arroz de 0.75g

No.	Espesor (mm)	Ancho (mm)	Área (mm^2)
1	1.213	10.22	12.4
2	1.156	9.92	11.465
3	1.306	9.48	12.378

Tabla 3.5: Resultados de los ensayos de tracción

No.	resistencia a la tracción (MPa)	Carga Máxima (N)
1	27.787	344.558
2	27.5	315.285
3	21.7	268.604
Valor Máximo	27.787	344.558
Valor Mínimo	21.7	268.604
Valor Medio	25.662	309.482

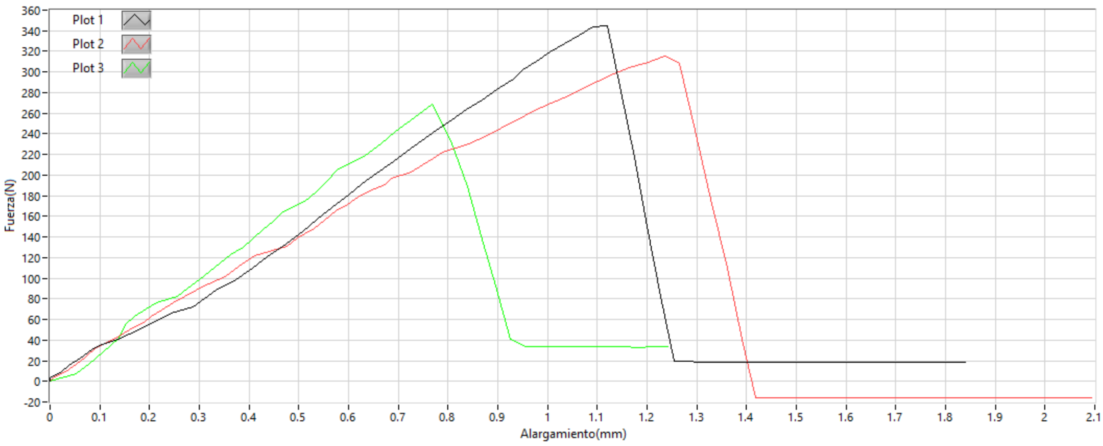


***Figura* 3.3**: *Gráficas de carga (KN) desplazamiento (mm) para una carga de 0.75 g en 20 g de PLA*

c) Carga de 1.00 gramos de cascara de Arroz

Tabla 3.6: Datos de las dimensiones de las probetas: Largo, ancho y espesor. De PLA con Cascara de Arroz de 1.00g

No.	Espesor (mm)	Ancho (mm)	Área (mm^2)
1	1.278	9.17	11.722
2	1.101	10.15	11.178
3	1.101	10.15	11.178

Tabla 3.7: Resultados de los ensayos de tracción

No.	resistencia a la tracción (MPa)	Carga Máxima (N)
1	23.283	272.919
2	21.358	238.744
3	24.512	273.998
Valor Máximo	24.512	273.998
Valor Mínimo	21.358	238.744
Valor Medio	23.051	261.887

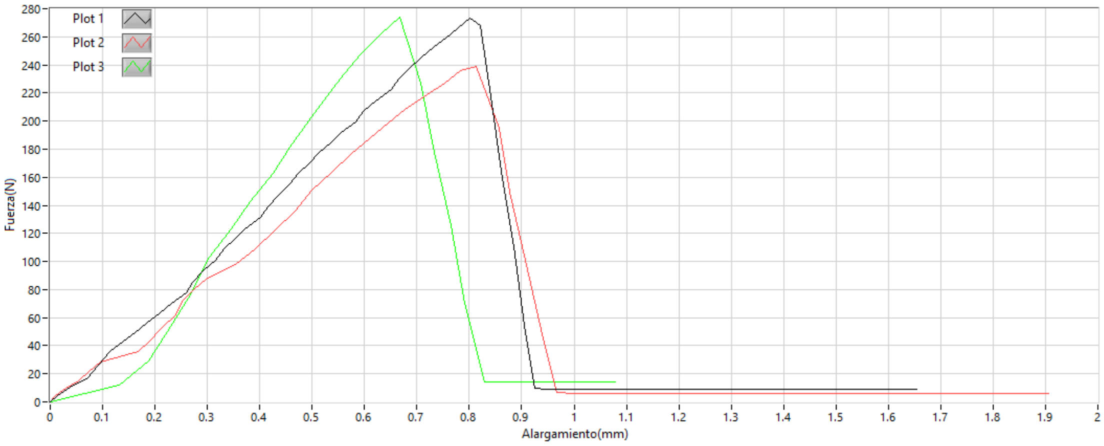


***Figura 3.4**: Gráficas de carga (KN) desplazamiento (mm) para el compuesto de 1.00 g con 20 g de PLA*

Tabla 3.8 Comparación de las Resistencias a la tracción respecto a la carga.

% de carga	Carga (g)	Resistencia a la tracción en (Mpa)
0.00	0.0 *	68.763
2.50	0.5	38.995
3.75	0.75	25.662
5.00	1	23.051
*Fuente: Obtención y caracterización mecánica de compósitos con matriz de biopolímero termoplástico de PLA. Espezúa E., Ponce E. 2014		

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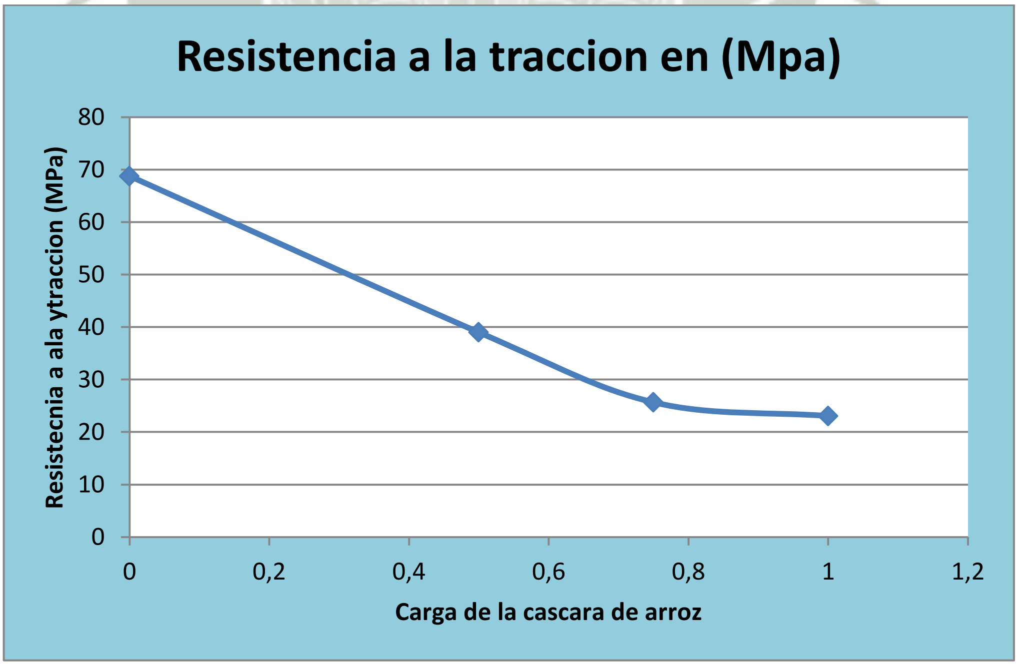

***Figura 3.5**: Comparación de las Resistencias a la tracción respecto a la carga*

Comentario: En la figura 3.5 se muestra la resistencia a la tracción promedio de los compuestos en relación a la carga de cascara de arroz (g). Se puede observar que existe una disminución de la resistencia a la tracción desde un 43.29% hasta un 66.48% al aumentar la carga (g) de cascara de arroz en 2.5 % y 5 % respectivamente. A pesar de esta disminución los valores obtenidos son mayores a los del Polietileno (PE) que es uno de los polímeros más usados actualmente (ver tabla 3.9).

Tabla 3.9 Resistencia a la tracción polímeros

Material	Resistencia a la tracción (Mpa)
PE-UHMW	17
PE-HD	22
PS-HI	26
PP	33
ABS	41
PA 6	45
PVDF	50
PVC-U	55
PPE	55

Material	Resistencia a la tracción (Mpa)
PC	65
PSU	70
PMMA	70
PA 6.6	80
PET	81
PEI	105
PP-30 GF	57
POM	63
PVC-C	57

3.2.2 Análisis Mecánico por Flexión

Tabla 3.10: Medidas de las probetas

PROBETA	CODIGO	Espesor (mm)					Ancho (mm)				
		1	2	3	4	Prom.	1	2	3	4	Prom.
1	CAM-0.50	3.23	3.50	3.51	3.33	3.39	18.08	17.69	17.50	17.09	17.59
2	CAM-0.75	3.18	3.28	3.51	3.51	3.37	17.86	18.01	18.09	17.81	17.94
3	CAM-1.00	4.50	4.76	4.75	4.29	4.58	16.44	16.38	16.87	16.99	16.67

Tabla 3.11 Resultados del ensayo de Flexión

PROBETA	Código	Espesor Promedio (mm)	Ancho	Carga Máxima (N)	Tensión en la superficie exterior en la mitad del tramo (MPa)
1	PLA Puro *	5.00	13.00	54.00	12.49
2	CAM-0.50	3.39	15.00	42.00	15.56
3	CAM-0.75	3.37	15.00	39.60	14.58
4	CAM-1.00	4.58	15.00	75.60	16.25
*** Dato obtenido de la tesis de Erick Ponce y Edson Espezúa**					

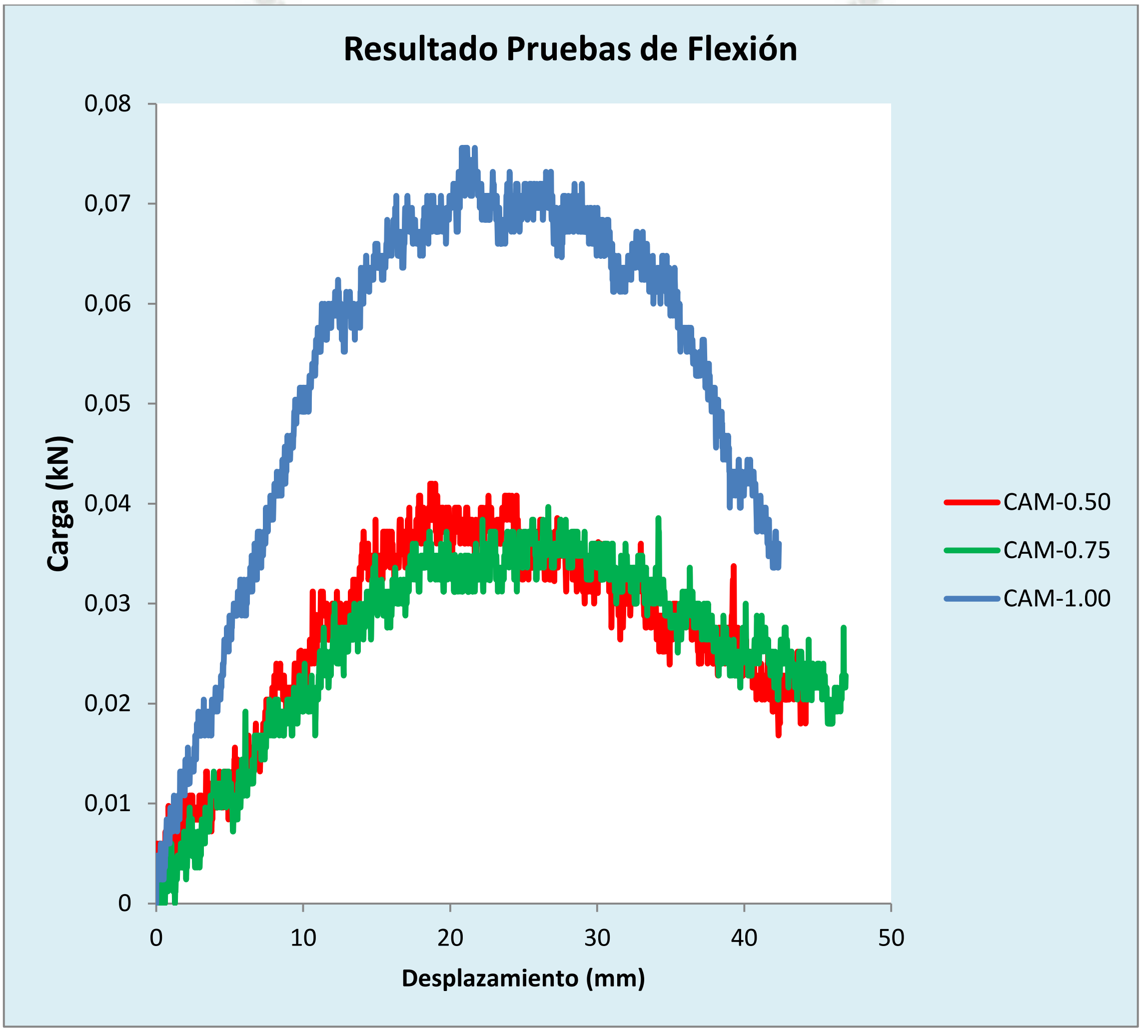

Figura 3.6 *Relación carga (KN) versus Desplazamiento (mm)*

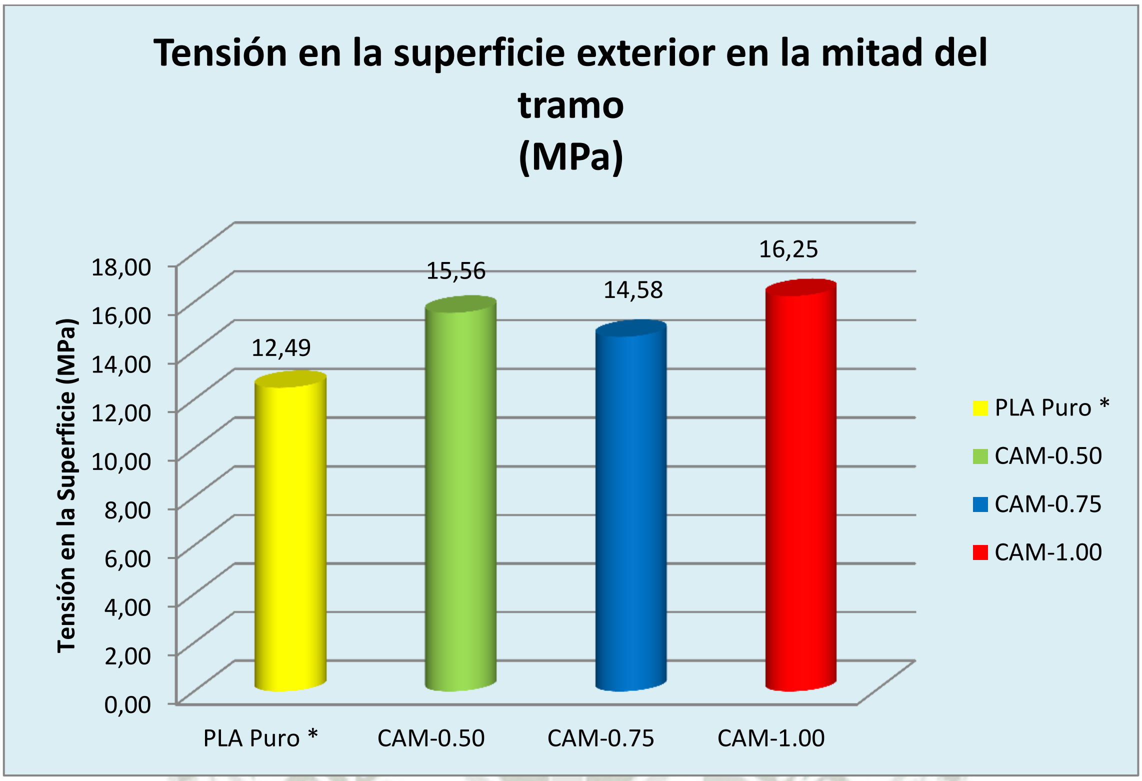

Figura 3.7 *Tensión en la superficie exterior en la mitad del tramo*

Comentario: En la figura 3.7 se muestra la tensión en la Superficie (MPa) en relación a las cargas de cascara de arroz. Se observa que los compuestos de PLA con cascara de arroz poseen mayor valor de la tensión en la superficie que el PLA puro, es así que a 1 g presentó una tensión superficial de 16.25 MPa, esta resistencia fue superior en comparación a las otras mezclas y al mismo PLA puro.

3.2.3 Análisis Mecánico por Dureza

Tabla 3.12 Resultados pruebas dureza

PROBETA	Código	D-1	D-2	D-3	D-4	D-5	Dureza SHORE D
1	PLA Puro *	81	79	80	81	81	80.4
2	CAM-0.50	79	80	80.5	80	80.5	80.0
3	CAM-0.75	84	86	84	85	85	84.8
4	CAM-1.00	77	79	78	77	78	77.8

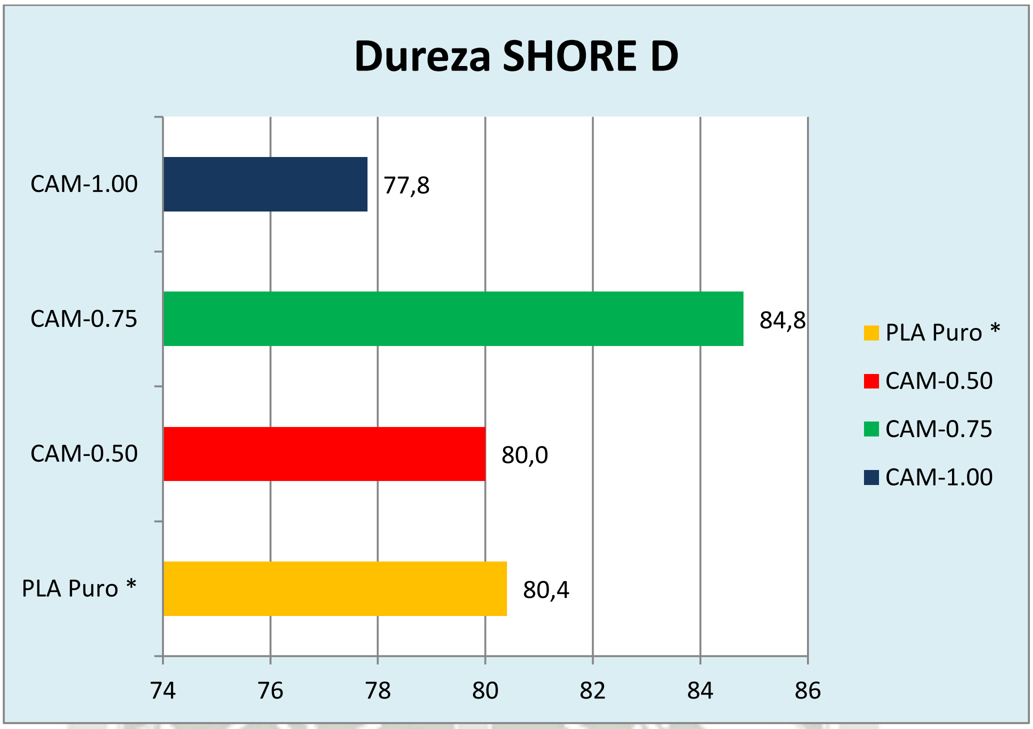

Figura 3.8: *Comparación de las Durezas de las diferentes mezclas*

Comentario: En la gráfica 3.8 se puede observar que la cascara de arroz modifica la dureza del compuesto final obtenido, es así que a 0.75 gramos de cascara de arroz en 20 g de PLA se da el mayor aumento de dureza shore A (84.8) en comparación con el PLA puro (80.4), sin embargo a 1.00 gramo de cascara de arroz en 20 g de PLA se produce una disminución a 77.8.

3.2.4 Análisis del Ensayo de Charpy

Tabla 3.13 Resultados del ensayo de Charpy para el compuesto de PLA y cascara de arroz.

	Energía Absorbida (J/m)			
Nº	PLA puro	CAM-0.50	CAM-0.75	CAM-1.00
1	41.9687	45.4635	38.7358	54.1507
2	52.5708	40.8623	46.9806	44.1303
3	47.3578	46.6192	54.5567	46.9806
4	50.71.31	43.7129	51.2378	47.7009
5	54.7534	44.1645	49.4662	49.0634
PROMEDIO	49.1627	44.1645	48.1954	48.4052

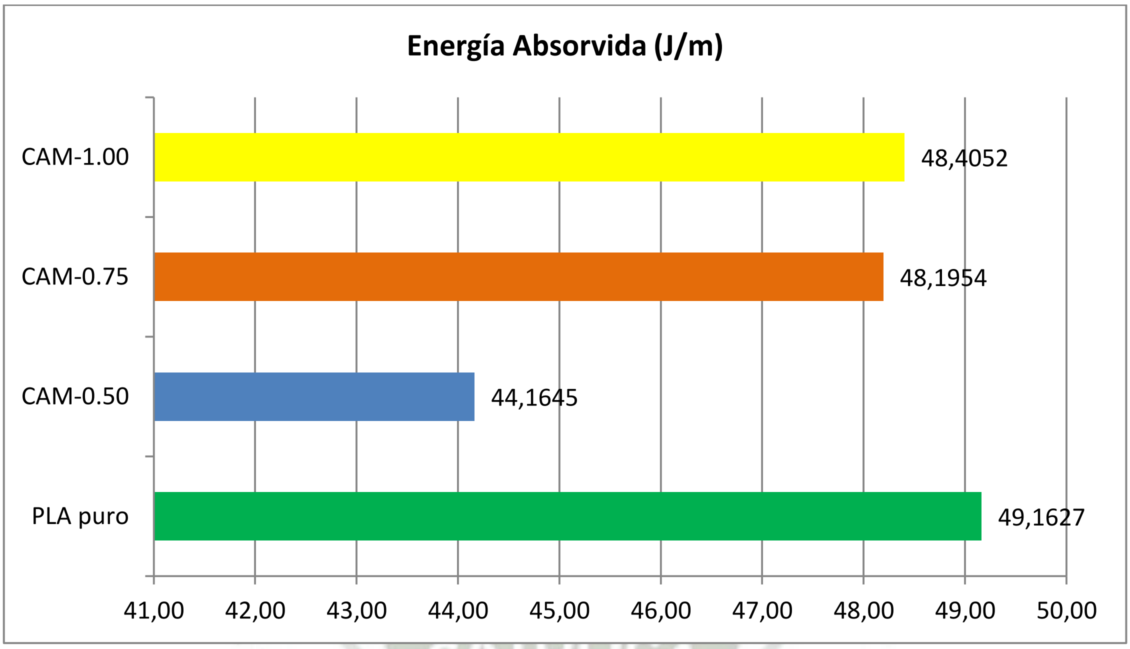

Figura 3.9: *Comparación de los resultados del ensayo de Charpy de todas las mezclas y PLA puro*

Comentario: En la figura 3.9 se aprecia que la energía absorbida (J/m) disminuye en los compuestos de PLA con cascara de arroz, es así que para el PLA puro el valor promedio que le corresponde es 49.1627 J/m y los otros valores son 44.1645, 48.1954 y 48.4052 para las cargas de 0.5g, 0.75g y 1.0g respectivamente que están contenidos en 20g de PLA.

3.3 Análisis del Índice de Fluidez

Tabla Nº 3.14: Valores en gramos del Plastometro de cada mezcla, en 90 segundos de tiempo de corte.

Código	PLA puro (g)	CAM-0.50 (g)	CAM-0.75 (g)	CAM-1.00 (g)
1	0.40	0.37	0.55	0.47
2	0.43	0.40	0.43	0.56
3	0.43	0.42	0.40	0.38
4	0.40	0.49	0.51	0.39
5	0.46	0.44	0.51	0.44
6	0.46		0.60	0.36
7				0.50
8				0.62
9				0.65
PROMEDIO	0.43	0.42	0.50	0.49

Figura 3.10: ***a)*** *Pesada de las muestras,* ***b)*** *colocado de la muestra en el equipo* ***c)*** *Colocado de las pesas y* ***d)*** *Muestras Obtenidas*

Tabla 3.15: Valores del Índices de fluidez obtenidos para las diferentes mezclas

Código	Resultado Plastometro (g)	IF (158ºC/7160 g) g/10min
PLA puro	0.43	2.87
CAM-0.50	0.42	2.83
CAM-0.75	0.50	3.33
CAM-1.00	0.49	3.24

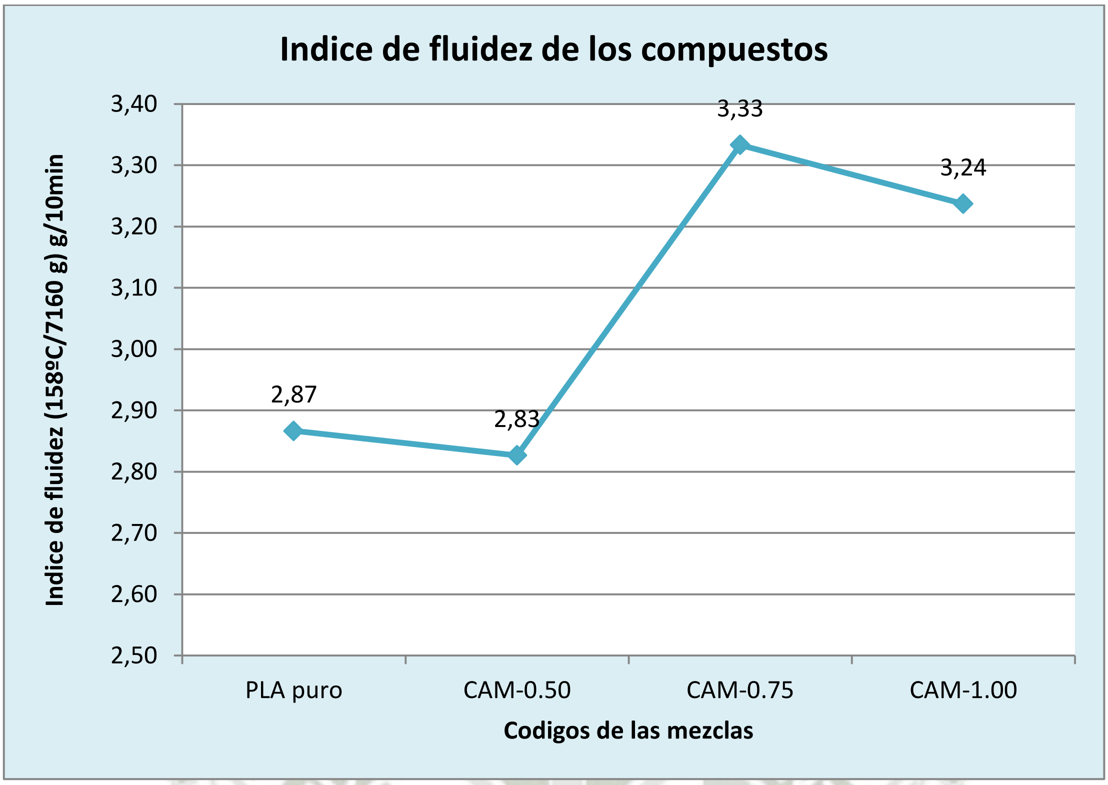

Figura 3.11: *Comparación Índice de fluidez de todas las mezclas del compuesto de PLA con cascara de arroz y PLA puro*

Comentario: De acuerdo a la figura 3.11 se puede observar que el índice de fluidez se mantiene constante para la mezcla de PLA con 0.5 g de cascara de arroz respecto a los resultados del PLA puro. Por otro lado se observa un incremento de este en las mezclas de 0.75 g y 1.00 g

3.4 Análisis de la Densidad

Tabla 3.16: Valores de las medidas de las probetas en el ensayo de densidad

Probeta	Código	Peso (g)	Largo (mm)				Ancho (mm)				Espesor (mm)			
			1	2	3	Prom	1	2	3	Prom	1	2	3	Prom
1	PLA Puro *	1.55				45.81				42.92				0.65
2	CAM-0.50	1.13	30.85	31.11	31.14	31.03	31.23	31.08	30.56	30.96	1.03	1.04	1.00	1.02
3	CAM-0.75	0.82	25.78	25.92	25.83	25.84	26.62	26.61	26.07	26.43	1.00	1.03	0.98	1.00
4	CAM-1.00	1.88	30.36	30.19	30.51	30.35	30.66	30.61	30.41	30.56	1.71	1.70	1.73	1.71

* Dato obtenido de la tesis de Erick Ponce y Edson Espezúa

Tabla 3.17: Valores de la densidad de las probetas de cascara de arroz con PLA.

PROBETA	Código	Peso (g)	Espesor (mm)	Largo (mm)	Ancho (mm)	DENSIDAD (g/cm^3)
1	PLA Puro *	1.55	0.65	45.81	42.92	1.22
2	CAM-0.50	1.13	1.02	31.03	30.96	1.15
3	CAM-0.75	0.82	1.00	25.84	26.43	1.20
4	CAM-1.00	1.88	1.71	30.35	30.56	1.18

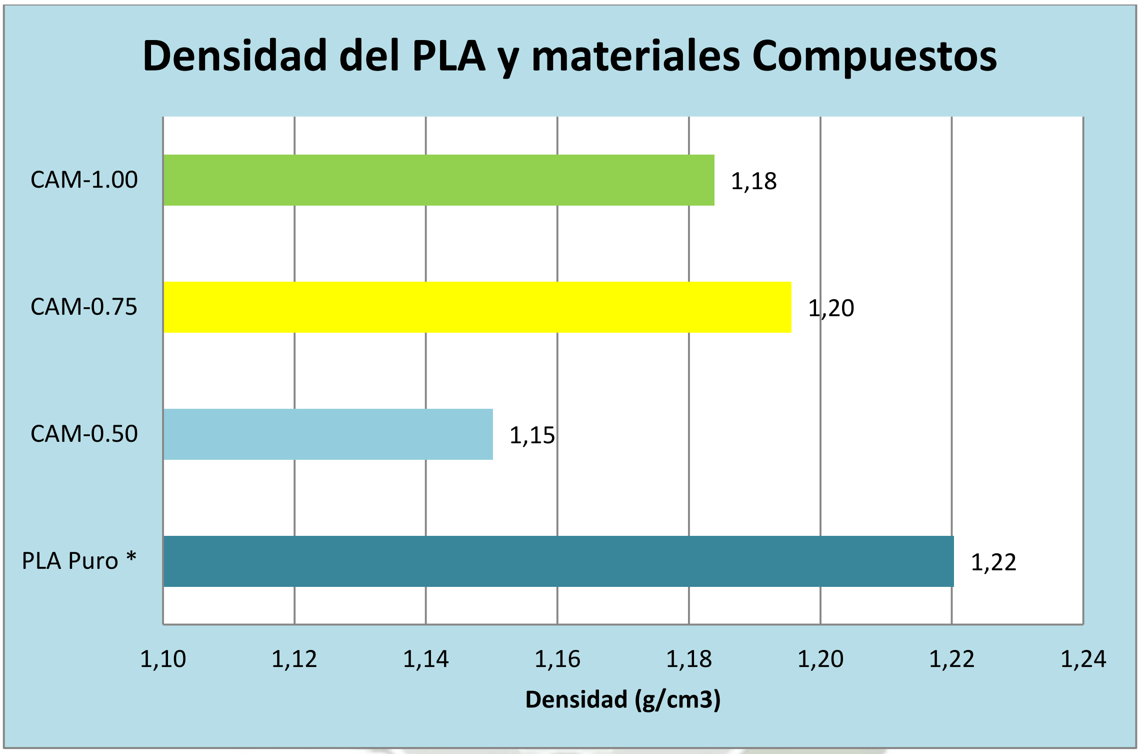

***Figura 3.12:** Comparación de los valores de densidad obtenidos para los compuestos de PLA con cascara de arroz*

Comentario: En la gráfica 3.12 se puede apreciar que los valores de densidad disminuyen respecto a valor obtenido para el PLA puro, es decir existe menor masa por unidad de volumen.

3.5 Análisis Termogravimétrico (ATG)

La Termogravimetría está basada en la medida de la variación de la masa de una muestra cuando dicha muestra se somete a un programa de temperatura en una atmósfera controlada. Esta variación de masa puede ser una pérdida de masa o una ganancia de masa.

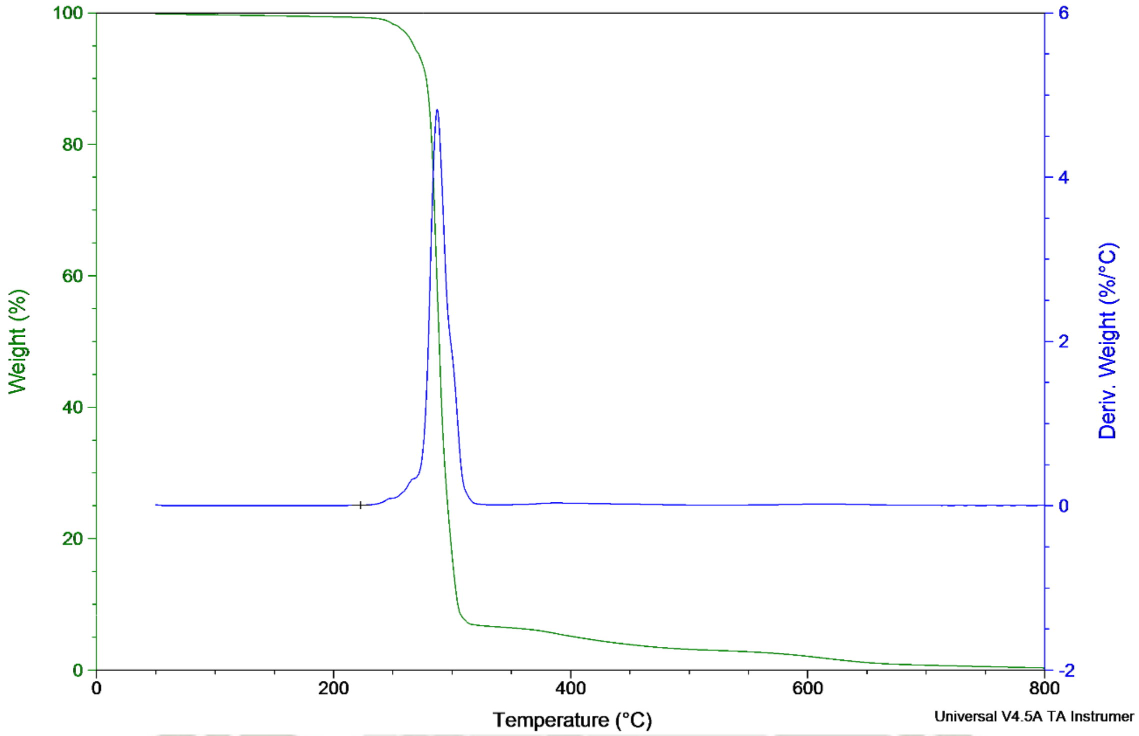

Figura 3.13: *Curva ATG del PLA puro*

Fuente: *Laboratorio Universidad de Lille 1*

Comentario: La caracterización mediante ATG nos indica la temperatura máxima de degradación que puede soportar el polímero, el PLA comienza su degradación a los 223.4 °C, por lo cual solo hasta esta temperatura el polímero puede ser sometido en su proceso de transformación.

3.6 Análisis por Calorimetría diferencial de barrido

Es una técnica termoanalítica en la que la diferencia de calor entre una muestra y una referencia es medida como una función de la temperatura. La muestra y la referencia son mantenidas aproximadamente a la misma temperatura a través de un experimento.

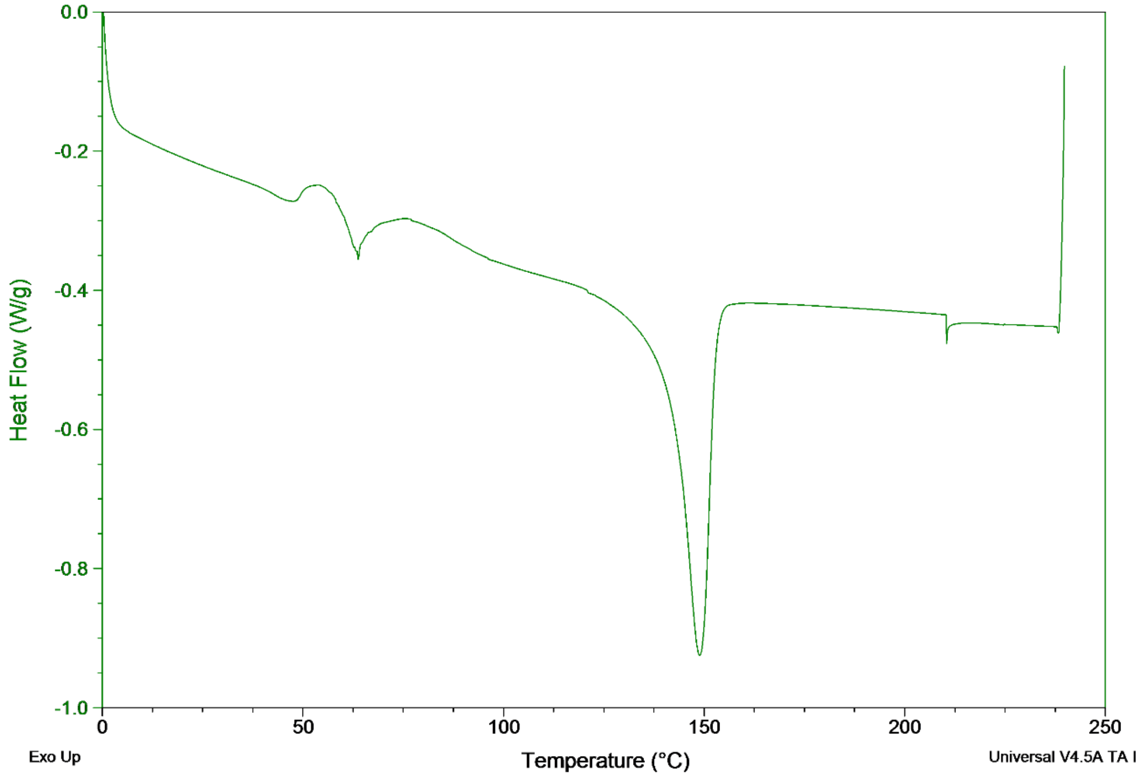

Figura 3.14: *Curva CDB del PLA puro*

Fuente: *Laboratorio Universidad de Lille 1*

Comentario: Mediante esta caracterización podemos observar que el PLA presenta dos picos de fusión a 64.03 °C y 149.06 °C, con lo cual tendremos los parámetros máximos de trabajo, para evitar la degradación del polímero.

3.7 Espectroscopia de Infrarrojo por Transformada de Fourier FTIR

Se basa en la absorción de radiación infrarroja por el material que se analiza. Los análisis fueron realizados en un FTIR Nicolet modelo de Impacto 400D, el método ATR (análisis del sólido).

Figura 3.15: *Espectro FTIR PLA puro*

Fuente: *Laboratorio Universidad de Lille 1*

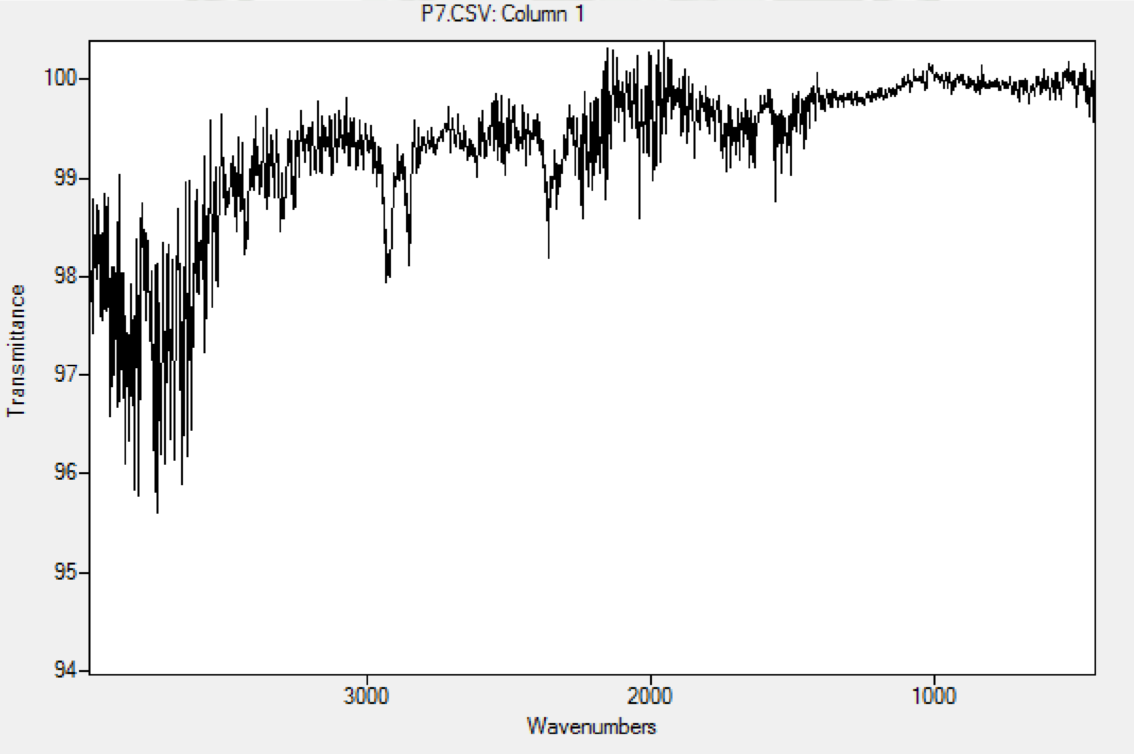

Figura 3.16: *Espectro FTIR CAM-0.50*

Fuente: *Laboratorio Universidad de Lille 1*

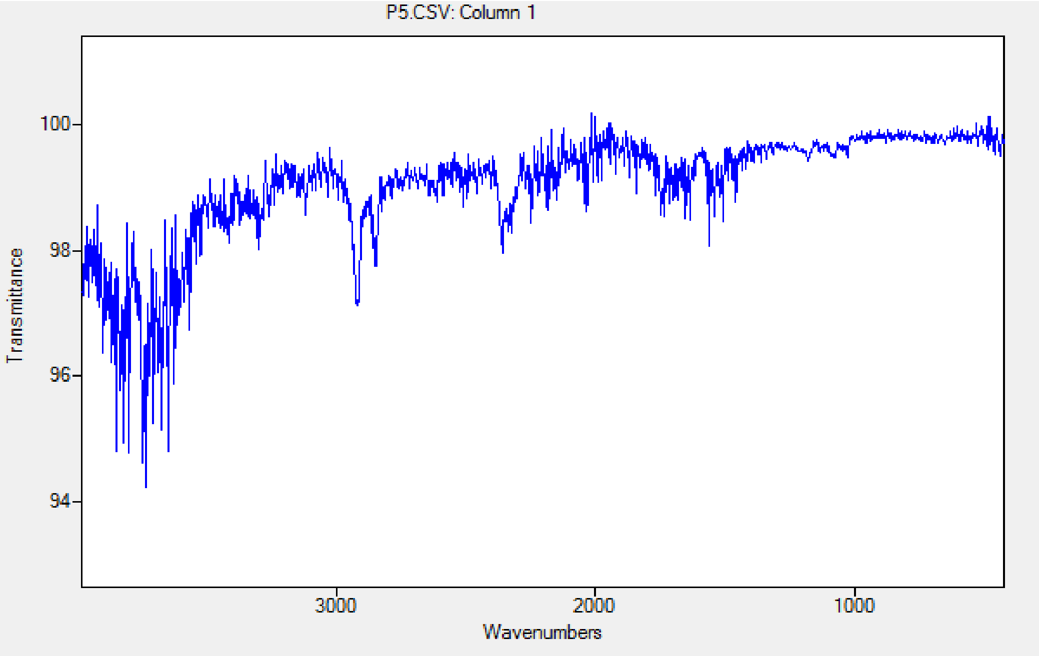

Figura 3.17: *Espectro FTIR CAM-0.75*

Fuente: *Laboratorio Universidad de Lille 1*

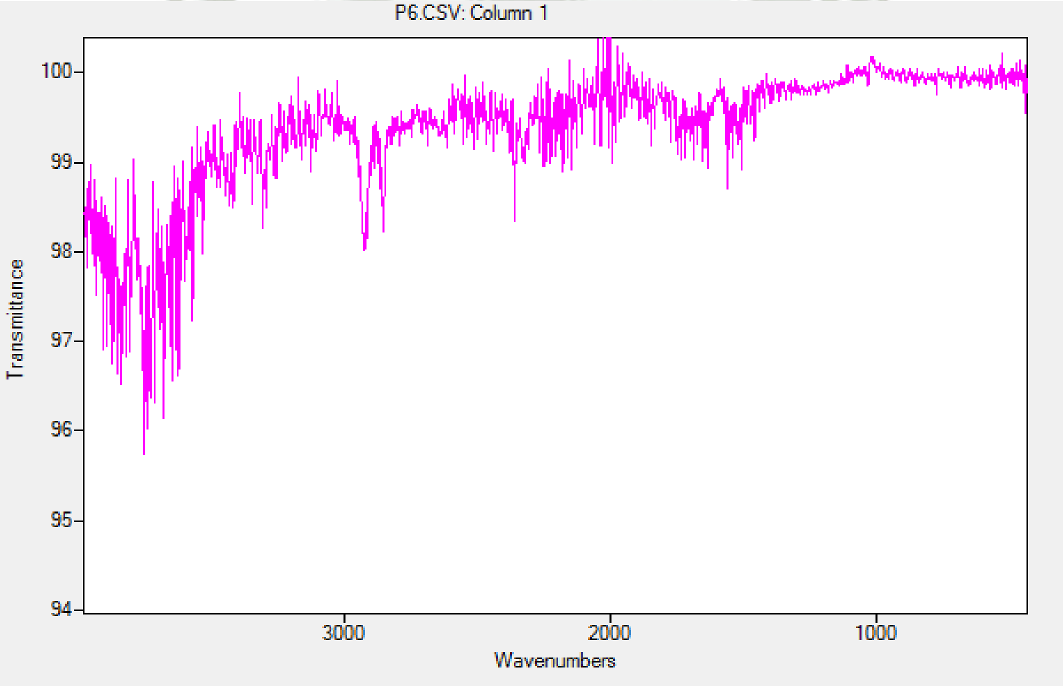

Figura 3.18: *Espectro FTIR CAM-1.00*

Fuente: *Laboratorio Universidad de Lille 1*

Comentario: Podemos indicar que las mezclas de PLA con la cascara de arroz presentan una variación con respecto al PLA puro por lo tanto esto nos indica que se dio una mezcla de PLA y cascara de arroz.

3.8 Microscopia de probetas del compuesto PLA y Cascara de arroz

Consiste en hacer pasar luz visible de una fuente (difractada, reflejada o refractada en el sujeto de estudio) a través de lentes ópticos simples, para lograr una vista ampliada de la muestra. La imagen resultante puede ser detectada directamente por el ojo humano.

Figura 3.19: *Probeta de PLA con 1 gr de Cascara de arroz vista al microscopio*

Fuente: *Laboratorio del Instituto Honorio Delgado Espinoza*

Figura 3.20: *Probeta de PLA con 0.75 gr de Cascara de arroz vista al microscopio*

Fuente: *Laboratorio del Instituto Honorio Delgado Espinoza*

Figura 3.21: *Probeta de PLA con 0.5 gr de Cascara de arroz vista al microscopio*

Fuente: *Laboratorio del Instituto Honorio Delgado Espinoza*

Comentario: Podemos observar que las tres mezclas de PLA con cascara de arroz presentan lo siguiente:

- La mezcla que contiene 1gr de cascara de arroz presenta una dispersión poco homogénea de la carga.
- La mezcla que contiene 0.75gr de cascara de arroz presenta una dispersión más homogénea de la carga.
- La mezcla que contiene 0.5gr de cascara de arroz presenta burbujas y vacíos.
- Se puede observar que la cascara de arroz está presente en forma de microfibrillas en las tres probetas obtenidas.

CONCLUSIONES

- Los compuestos producidos en el laboratorio han presentado un incremento en la Energía Absorbida con respecto al PLA puro, el incremento de esta energía se da en forma proporcional al incremento de las cargas.
- Se logró incrementar la Tensión en la superficie exterior en la mitad del tramo (MPa) en un 30.11 % usando un 5 % de cascara de arroz en el compuesto con PLA
- Existe una disminución de la resistencia a la tracción desde un 43.29% hasta un 66.48% al aumentar la carga (g) de cascara de arroz en 2.5 % y 5 % respectivamente.
- La dureza del PLA se incrementó en un 5.47% con 3.75 % de cascara de arroz, pero con 5 % de cascara de arroz disminuye el valor en 2.74%.
- Las mezclas muestran un aumento de la fluidez con la adición de la cascara de arroz, es así que el índice de fluidez para el compuesto de 2.5 % de cascara de arroz con PLA es muy similar al valor obtenido para el PLA puro, sin embargo con las cargas de 3.75 % y 5 % aumenta el valor del índice de fluidez en 28.79 % y 12.79 % respectivamente.

RECOMENDACIONES

1. Realizar un estudio micro estructural para determinar el tipo de unión producido entre el PLA y la cascara de arroz, sabiendo que los porcentajes de sílice que contiene la cascara de arroz son elevados, y su influencia en las propiedades del polímero puede ser determinante.
2. Generar un nuevo estudio mediante la incorporación de agentes compatibilizantes que puedan ayudar a mejorar la interfaz matriz-fibra.
3. La disminución de la elasticidad del PLA se genera al incorporar la cascara de arroz, el vuelve más rígido al PLA, es posible realizar estudios incorporando plastificantes para mejorar esta propiedades del polímero.

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ANEXOS

Designation: D 882 – 02

Standard Test Method for Tensile Properties of Thin Plastic Sheeting[1]

This standard is issued under the fixed designation D 882; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

These test methods have been approved for use by agencies of the Department of Defense to replace Method 1013 of Federal Test Method Standard 406.

1. Scope *

1.1 This test method covers the determination of tensile properties of plastics in the form of thin sheeting, including film (less than 1.0 mm (0.04 in.) in thickness).

NOTE 1—Film has been arbitrarily defined as sheeting having nominal thickness not greater than 0.25 mm (0.010 in.).

NOTE 2—Tensile properties of plastics 1.0 mm (0.04 in.) or greater in thickness shall be determined according to Test Method D 638.

1.2 This test method may be used to test all plastics within the thickness range described and the capacity of the machine employed.

1.2.1 *Static Weighing, Constant-Rate-of-Grip Separation Test*—This test method employs a constant rate of separation of the grips holding the ends of the test specimen.

1.3 Specimen extension may be measured in these test methods by grip separation, extension indicators, or displacement of gage marks.

1.4 A procedure for determining the tensile modulus of elasticity is included at one strain rate.

NOTE 3—The modulus determination is generally based on the use of grip separation as a measure of extension; however, the desirability of using extensometers, as described in 5.2, is recognized and provision for the use of such instrumentation is incorporated in the procedure.

1.5 Test data obtained by this test method is relevant and appropriate for use in engineering design.

1.6 The values stated in SI units are to be regarded as the standard. The values in parentheses are provided for information only.

1.7 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

NOTE 4—This test method is similar to ISO 527-3, but is not considered technically equivalent. ISO 527-3 allows for additional specimen configurations, specifies different test speeds, and requires an extensometer or gage marks on the specimen.

2. Referenced Documents

2.1 *ASTM Standards:*
D 618 Practice for Conditioning Plastics for Testing[2]
D 638 Test Method for Tensile Properties of Plastics[2]
D 4000 Classification System for Specifying Plastic Materials[3]
D 5947 Test Methods for Physical Dimensions of Solid Plastic Specimens[4]
D 6287 Practice for Cutting Film and Sheeting Test Specimens[4]
E 4 Practices for Force Verification of Testing Machines[5]
E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[6]

2.2 *ISO Standard:*
ISO 527-3 Plastics—Determination of Tensile Properties—Part 3: Test Conditions for Films and Sheets[7]

3. Terminology

3.1 *Definitions*—Definitions of terms and symbols relating to tension testing of plastics appear in the Annex to Test Method D 638.

3.1.1 *line grips*—grips having faces designed to concentrate the entire gripping force along a single line perpendicular to the direction of testing stress. This is usually done by combining one standard flat face and an opposing face from which protrudes a half-round.

3.1.2 *tear failure*—a tensile failure characterized by fracture initiating at one edge of the specimen and progressing across the specimen at a rate slow enough to produce an anomalous load-deformation curve.

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[1] These test methods are under the jurisdiction of ASTM Committee D20 on Plastics and are the direct responsibility of Subcommittee D20.10 on Mechanical Properties.

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[2] *Annual Book of ASTM Standards*, Vol 08.01.
[3] *Annual Book of ASTM Standards*, Vol 08.02.
[4] *Annual Book of ASTM Standards*, Vol 08.03.
[5] *Annual Book of ASTM Standards*, Vol 03.01.
[6] *Annual Book of ASTM Standards*, Vol 14.02.
[7] Available from American National Standards Institute, 25 W. 43rd St., 4th Floor, New York, NY 10036.

***A Summary of Changes section appears at the end of this standard.**

4. Significance and Use

4.1 Tensile properties determined by this test method are of value for the identification and characterization of materials for control and specification purposes. Tensile properties may vary with specimen thickness, method of preparation, speed of testing, type of grips used, and manner of measuring extension. Consequently, where precise comparative results are desired, these factors must be carefully controlled. This test method shall be used for referee purposes, unless otherwise indicated in particular material specifications. For many materials, there may be a specification that requires the use of this test method, but with some procedural modifications that take precedence when adhering to the specification. Therefore, it is advisable to refer to that material specification before using this test method. Table 1 in Classification D 4000 lists the ASTM materials standards that currently exist.

4.2 Tensile properties may be utilized to provide data for research and development and engineering design as well as quality control and specification. However, data from such tests cannot be considered significant for applications differing widely from the load-time scale of the test employed.

4.3 The tensile modulus of elasticity is an index of the stiffness of thin plastic sheeting. The reproducibility of test results is good when precise control is maintained over all test conditions. When different materials are being compared for stiffness, specimens of identical dimensions must be employed.

4.4 The tensile energy to break (TEB) is the total energy absorbed per unit volume of the specimen up to the point of rupture. In some texts this property has been referred to as *toughness*. It is used to evaluate materials that may be subjected to heavy abuse or that might stall web transport equipment in the event of a machine malfunction in end-use applications. However, the rate of strain, specimen parameters, and especially flaws may cause large variations in the results. In that sense, caution is advised in utilizing TEB test results for end-use design applications.

4.5 Materials that fail by tearing give anomalous data which cannot be compared with those from normal failure.

5. Apparatus

5.1 *Testing Machine*—A testing machine of the constant rate-of-crosshead-movement type and comprising essentially the following:

5.1.1 *Fixed Member*—A fixed or essentially stationary member carrying one grip.

5.1.2 *Movable Member*—A movable member carrying a second grip.

5.1.3 *Grips*—A set of grips for holding the test specimen between the fixed member and the movable member of the testing machine; grips can be either the fixed or self-aligning type. In either case, the gripping system must minimize both slippage and uneven stress distribution.

5.1.3.1 Fixed grips are rigidly attached to the fixed and movable members of the testing machine. When this type of grip is used, care must be taken to ensure that the test specimen is inserted and clamped so that the long axis of the test specimen coincides with the direction of pull through the center line of the grip assembly.

5.1.3.2 Self-aligning grips are attached to the fixed and movable members of the testing machine in such a manner that they will move freely into alignment as soon as a load is applied so that the long axis of the test specimen will coincide with the direction of the applied pull through the center line of the grip assembly. The specimens should be aligned as perfectly as possible with the direction of pull so that no rotary motion that may induce slippage will occur in the grips; there is a limit to the amount of misalignment self-aligning grips will accommodate.

5.1.3.3 The test specimen shall be held in such a way that slippage relative to the grips is prevented insofar as possible. Grips lined with thin rubber, crocus-cloth, or pressure-sensitive tape as well as file-faced or serrated grips have been successfully used for many materials. The choice of grip surface will depend on the material tested, thickness, etc. Line grips padded on the round face with 1.0 mm (40 mil) blotting paper or filter paper have been found superior. Air-actuated grips have been found advantageous, particularly in the case of materials that tend to "neck" into the grips, since pressure is maintained at all times. In cases where samples frequently fail at the edge of the grips, it may be advantageous to increase slightly the radius of curvature of the edges where the grips come in contact with the test area of the specimen.

5.1.4 *Drive Mechanism*—A drive mechanism for imparting to the movable member a uniform, controlled velocity with respect to the stationary member. The velocity shall be regulated as specified in Section 9.

5.1.5 *Load Indicator*—A suitable load-indicating mechanism capable of showing the total tensile load carried by the test specimen held by the grips. This mechanism shall be essentially free of inertial lag at the specified rate of testing (see Note 5). Unless a suitable extensometer is used (see 5.2), the motion of the weighing system shall not exceed 2 % of the specimen extension within the range being measured. The load indicator shall determine the tensile load applied to the specimen with an accuracy of ±1 % of the indicated value, or better. The accuracy of the testing machine shall be verified in accordance with Practices E 4.

5.1.6 *Crosshead Extension Indicator*— A suitable extension-indicating mechanism capable of showing the amount of change in the separation of the grips, that is, crosshead movement. This mechanism shall be essentially free of inertial lag at the specified rate of testing (see Note 5) and shall indicate the crosshead movement with an accuracy of ±1 % of the indicated value, or better.

5.2 *Extensometer (Optional)*—A suitable instrument may, if desired, be used for determining the distance between two designated points on the test specimen as the specimen is stretched. This apparatus, if employed, shall be so designed as to minimize stress on the specimen at the contact points of the specimen and the instrument (see 8.3). It is desirable that this instrument automatically record the distance, or any change in it, as a function of the load on the test specimen or of the elapsed time from the start of the test, or both. If only the latter is obtained, load-time data must also be taken. This instrument must be essentially free of inertial lag at the specified speed of testing (see Note 5).

5.2.1 *Modulus of Elasticity and Low-Extension Measurements*—Extensometers used for modulus of elasticity and low-extension (less than 20 % elongation) measurements shall, at a minimum, be accurate to ±1 % and comply with the requirements set forth in Practice E 83 for a Class C instrument.

5.2.2 *High-Extension Measurements*—Instrumentation and measuring techniques used for high-extension (20 % elongation or greater) measurements shall be accurate to ±10 % of the indicated value, or better.

NOTE 5—A sufficiently high response speed in the indicating and recording system for the load and extension data is essential. The response speed required of the system will depend in part on the material tested (high or low elongation) and the rate of straining.

5.3 *Thickness Gage*—A dead-weight dial micrometer as prescribed in Method C of Test Methods D 5947, or an equivalent measuring device, reading to 0.0025 mm (0.0001 in.) or less.

5.4 *Width-Measuring Devices*—Suitable test scales or other width measuring devices capable of measuring 0.25 mm (0.010 in.) or less.

5.5 *Specimen Cutter*—For the apparatus and techniques for cutting film and sheeting used in this test method, refer to Practice D 6287.

5.5.1 Devices that use razor blades have proven especially suitable for materials having an elongation-at-fracture above 10 to 20 %.

5.5.2 The use of punch press or striking dies are not recommended because poor and inconsistent specimen edges may be produced.

6. Test Specimens

6.1 The test specimens shall consist of strips of uniform width and thickness at least 50 mm (2 in.) longer than the grip separation used.

6.2 The nominal width of the specimens shall be not less than 5.0 mm (0.20 in.) or greater than 25.4 mm (1.0 in.).

6.3 A width-thickness ratio of at least eight shall be used. Narrow specimens magnify effects of edge strains or flaws, or both.

6.4 The utmost care shall be exercised in cutting specimens to prevent nicks and tears which are likely to cause premature failures (Note 6). The edges shall be parallel to within 5 % of the width over the length of the specimen between the grips.

NOTE 6—Microscopical examination of specimens may be used to detect flaws due to sample or specimen preparation.

6.5 Wherever possible, the test specimens shall be selected so that thickness is uniform to within 10 % of the thickness over the length of the specimen between the grips in the case of materials 0.25 mm (0.010 in.) or less in thickness and to within 5 % in the case of materials greater than 0.25 mm (0.010 in.) in thickness but less than 1.00 mm (0.040 in.) in thickness.

NOTE 7—In cases where thickness variations are in excess of those recommended in 6.5, results may not be characteristic of the material under test.

6.6 If the material is suspected of being anisotropic, two sets of test specimens shall be prepared having their long axes respectively parallel with and normal to the suspected direction of anisotropy.

6.7 For tensile modulus of elasticity determinations, a specimen gage length of 250 mm (10 in.) shall be considered as standard. This length is used in order to minimize the effects of grip slippage on test results. When this length is not feasible, test sections as short as 100 mm (4 in.) may be used if it has been shown that results are not appreciably affected. However, the 250-mm gage length shall be used for referee purposes. The speed of testing of shorter specimens must be adjusted in order for the strain rate to be equivalent to that of the standard specimen.

NOTE 8—Two round robin tests[8] have shown that, for materials of less than 0.25-mm (10-mil) thickness, line grips padded on the round side with 1.0-mm (40-mil) blotting paper give the same results with a 100-mm test section as a 250-mm test section produces with flat-face grips.

NOTE 9—Excessive jaw slippage becomes increasingly difficult to overcome in cases where high modulus materials are tested in thicknesses greater than 0.25 mm (0.010 in.).

7. Conditioning

7.1 *Conditioning*—Condition the test specimens at 23 ± 2°C (73.4 ± 3.6°F) and 50 ± 5 % relative humidity for not less than 40 h prior to test in accordance with Procedure A of Practice D 618 unless otherwise specified by contract or the relevant ASTM material specification. Reference pre-test conditioning, to settle disagreements, shall apply tolerances of ±1°C (1.8°F) and ±2 % relative humidity.

7.2 *Test Conditions*—Conduct the tests at 23 ± 2°C (73.4 ± 3.6°F) and 50 ± 5 % relative humidity unless otherwise specified by contract or the relevant ASTM material specification. Reference testing conditions, to settle disagreements, shall apply tolerances of ±1°C (1.8°F) and ±2 % relative humidity.

8. Number of Test Specimens

8.1 In the case of isotropic materials, at least five specimens shall be tested from each sample.

8.2 In the case of anisotropic materials, at least ten specimens, five normal and five parallel with the principal axis of anisotropy, shall be tested from each sample.

8.3 Specimens that fail at some obvious flaw or that fail outside the gage length shall be discarded and retests made, unless such flaws or conditions constitute a variable whose effect is being studied. However, jaw breaks (failures at the grip contact point) are acceptable if it has been shown that results from such tests are in essential agreement with values obtained from breaks occurring within the gage length.

NOTE 10—In the case of some materials, examination of specimens, prior to and following testing, under crossed optical polarizers (polarizing films) provides a useful means of detecting flaws which may be, or are, responsible for premature failure.

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[8] Supporting data are available from ASTM Headquarters. Request RR: D20-1058.

9. Speed of Testing

9.1 The speed of testing is the rate of separation of the two members (or grips) of the testing machine when running idle (under no load). This rate of separation shall be maintained within 5 % of the no-load value when running under full-capacity load.

9.2 The speed of testing shall be calculated from the required initial strain rate as specified in Table 1. The rate of grip separation may be determined for the purpose of these test methods from the initial strain rate as follows:

$$A = BC \tag{1}$$

where:

A = rate of grip separation, mm (or in.)/min,
B = initial distance between grips, mm (or in.), and
C = initial strain rate, mm/mm·min (or in./in.·min).

9.3 The initial strain rate shall be as in Table 1 unless otherwise indicated by the specification for the material being tested.

NOTE 11—Results obtained at different initial strain rates are not comparable; consequently, where direct comparisons between materials in various elongation classes are required, a single initial strain rate should be used. For some materials it may be advisable to select the strain rates on the basis of percent elongation at yield.

9.4 In cases where conflicting material classification, as determined by percent elongation at break values, results in a choice of strain rates, the lower rate shall be used.

9.5 If modulus values are being determined, separate specimens shall be used whenever strain rates and specimen dimensions are not the same as those employed in the test for other tensile properties.

10. Procedure

10.1 Select a load range such that specimen failure occurs within its upper two thirds. A few trial runs may be necessary to select a proper combination of load range and specimen width.

10.2 Measure the cross-sectional area of the specimen at several points along its length. Measure the width to an accuracy of 0.25 mm (0.010 in.) or better. Measure the thickness to an accuracy of 0.0025 mm (0.0001 in.) or better for films less than 0.25 mm (0.010 in.) in thickness and to an accuracy of 1 % or better for films greater than 0.25 mm (0.010 in.) but less than 1.0 mm (0.040 in.) in thickness.

10.3 Set the initial grip separation in accordance with Table 1.

10.4 Set the rate of grip separation to give the desired strain rate, based on the initial distance between the grips, in accordance with Table 1. Zero the calibrated load weighing system, extension indicator(s) and recording system.

NOTE 12—Extensometers may be used for modulus of elasticity determinations with the expectation of obtaining more accurate values than may be obtained using grip separation as the effective gage length. Precautions should be taken to ensure that extensometer slippage and undue stressing of the specimen do not occur. Refer also to 6.7.

10.5 In cases where it is desired to measure a test section other than the total length between the grips, mark the ends of the desired test section with a soft, fine wax crayon or with ink. Do not scratch these marks onto the surface since such scratches may act as stress raisers and cause premature specimen failure. Extensometers may be used if available; in this case, the test section will be defined by the contact points of the extensometer.

NOTE 13—Measurement of a specific test section is necessary with some materials having high elongation. As the specimen elongates, the accompanying reduction in area results in a loosening of material at the inside edge of the grips. This reduction and loosening moves back into the grips as further elongation and reduction in area takes place. In effect, this causes problems similar to grip slippage, that is, exaggerates measured extension.

10.6 Place the test specimen in the grips of the testing machine, taking care to align the long axis of the specimen with an imaginary line joining the points of attachment of the grips to the machine. Tighten the grips evenly and firmly to the degree necessary to minimize slipping of the specimen during test.

10.7 Start the machine and record load versus extension.

10.7.1 When the total length between the grips is used as the test area, record load versus grip separation.

10.7.2 When a specific test area has been marked on the specimen, follow the displacement of the edge boundary lines with respect to each other with dividers or some other suitable device. If a load-extension curve is desired, plot various extensions versus corresponding loads sustained, as measured by the load indicator.

10.7.3 When an extensometer is used, record load versus extension of the test area measured by the extensometer.

10.8 If modulus values are being determined, select a load range and chart rate to produce a load-extension curve of between 30 and 60° to the X axis. For maximum accuracy, use the most sensitive load scale for which this condition can be met. The test may be discontinued when the load-extension curve deviates from linearity.

10.9 In the case of materials being evaluated for secant modulus, the test may be discontinued when the specified extension has been reached.

TABLE 1 Crosshead Speeds and Initial Grip Separation

Percent Elongation at Break	Initial Strain Rate, mm/mm·min (in./in.·min)	Initial Grip Separation		Rate of Grip Separation	
		mm	in.	mm/min	in./min
Modulus of Elasticity Determination					
	0.1	250	10	25	1.0
Determinations other than Elastic Modulus					
Less than 20	0.1	125	5	12.5	0.5
20 to 100	0.5	100	4	50	2.0
Greater than 100	10.0	50	2	500	20.0

10.10 If tensile energy to break is being determined, some provision must be made for integration of the stress-strain curve. This may be either an electronic integration during the test or a subsequent determination from the area of the finished stress-strain curve (see Annex A2).

11. Calculation

11.1 Toe compensation shall be made in accordance with Annex A1 unless it can be shown that the toe region of the curve is not due to the takeup of slack, seating of the specimen, or other artifact, but rather is an authentic material response.

11.2 *Breaking Factor* (nominal) shall be calculated by dividing the maximum load by the original minimum width of the specimen. The result shall be expressed in force per unit of width, usually newtons per metre (or pounds per inch) of width, and reported to three significant figures. The thickness of the film shall always be stated to the nearest 0.0025 mm (0.0001 in.).

Example—Breaking Factor = 1.75 kN/m (10.0 lbf/in.) of width for 0.1300-mm (0.0051-in.) thickness.

NOTE 14—This method of reporting is useful for very thin films (0.13 mm (0.005 in.) and less) for which breaking load may not be proportional to cross-sectional area and whose thickness may be difficult to determine with precision. Furthermore, films which are in effect laminar due to orientation, skin effects, nonuniform crystallinity, etc., have tensile properties disproportionate to cross-sectional area.

11.3 *Tensile Strength* (nominal) shall be calculated by dividing the maximum load by the original minimum cross-sectional area of the specimen. The result shall be expressed in force per unit area, usually megapascals (or pounds-force per square inch). This value shall be reported to three significant figures.

NOTE 15—When tear failure occurs, so indicate and calculate results based on load and elongation at which tear initiates, as reflected in the load-deformation curve.

11.4 *Tensile Strength at Break* (nominal) shall be calculated in the same way as the tensile strength except that the load at break shall be used in place of the maximum load (Note 15 and Note 16).

NOTE 16—In many cases tensile strength and tensile strength at break are identical.

11.5 *Percent Elongation at Break* shall be calculated by dividing the extension at the moment of rupture of the specimen by the initial gage length of the specimen and multiplying by 100. When gage marks or extensometers are used to define a specific test section, only this length shall be used in the calculation; otherwise the distance between the grips shall be used. The result shall be expressed in percent and reported to two significant figures (Note 15).

11.6 *Yield Strength*, where applicable, shall be calculated by dividing the load at the yield point by the original minimum cross-sectional area of the specimen. The result shall be expressed in force per unit area, usually megapascals (or pounds-force per square inch). This value shall be reported to three significant figures. Alternatively, for materials that exhibit Hookean behavior in the initial part of the curve, an offset yield strength may be obtained as described in the Appendix of Test Method D 638. In this case the value should be given as "yield strength at —% offset."

11.7 *Percent Elongation at Yield*, where applicable, shall be calculated by dividing the extension at the yield point by the initial gage length of specimen and multiplying by 100. When gage marks or extensometers are used to define a specific test section, only this length shall be used in the calculation. Before calculating, correct the extension for "toe compensation" as described in Annex A1. The results shall be expressed in percent and reported to two significant figures. When offset yield strength is used, the elongation at the offset yield strength may be calculated.

11.8 *Elastic Modulus* shall be calculated by drawing a tangent to the initial linear portion of the load-extension curve, selecting any point on this tangent, and dividing the tensile stress by the corresponding strain. Before calculating, correct the extension for "toe compensation" as described in Annex A1. For purposes of this determination, the tensile stress shall be calculated by dividing the load by the average original cross section of the test section. The result shall be expressed in force per unit area, usually megapascals (or pounds-force per square inch), and reported to three significant figures.

11.9 *Secant Modulus*, at a designated strain, shall be calculated by dividing the corresponding stress (nominal) by the designated strain. Elastic modulus values are preferable and shall be calculated whenever possible. However, for materials where no proportionality is evident, the secant value shall be calculated. Draw the tangent as directed in A1.3 and Fig. A1.2 of Annex A1, and mark off the designated strain from the yield point where the tangent line goes through zero stress. The stress to be used in the calculation is then determined by dividing the load at the designated strain on the load-extension curve by the original average cross-sectional area of the specimen.

11.10 *Tensile Energy to Break*, where applicable, shall be calculated by integrating the energy per unit volume under the stress-strain curve or by integrating the total energy absorbed and dividing it by the volume of the original gage region of the specimen. As indicated in Annex A2, this may be done directly during the test by an electronic integrator, or subsequently by computation from the area of the plotted curve. The result shall be expressed in energy per unit volume, usually in megajoules per cubic metre (or inch-pounds-force per cubic inch). This value shall be reported to two significant figures.

11.11 For each series of tests, the arithmetic mean of all values obtained shall be calculated to the proper number of significant figures.

11.12 The standard deviation (estimated) shall be calculated as follows and reported to two significant figures:

$$s = \sqrt{(\Sigma X^2 - n\bar{X}^2)/(n-1)} \qquad (2)$$

where:
s = estimated standard deviation,
X = value of a single observation,
n = number of observations, and
$\bar{X}$ = arithmetic mean of the set of observations.

12. Report

12.1 Report the following information:

12.1.1 Complete identification of the material tested, including type, source, manufacturer's code number, form, principal dimensions, previous history, and orientation of samples with respect to anisotropy (if any),

12.1.2 Method of preparing test specimens,

12.1.3 Thickness, width, and length of test specimens,

12.1.4 Number of specimens tested,

12.1.5 Strain rate employed,

12.1.6 Grip separation (initial),

12.1.7 Crosshead speed (rate of grip separation),

12.1.8 Gage length (if different from grip separation),

12.1.9 Type of grips used, including facing (if any),

12.1.10 Conditioning procedure (test conditions, temperature, and relative humidity if nonstandard),

12.1.11 Anomalous behavior such as tear failure and failure at a grip,

12.1.12 Average breaking factor and standard deviation,

12.1.13 Average tensile strength (nominal) and standard deviation,

12.1.14 Average tensile strength at break (nominal) and standard deviation,

12.1.15 Average percent elongation at break and standard deviation,

12.1.16 Where applicable, average tensile energy to break and standard deviation,

12.1.17 In the case of materials exhibiting "yield" phenomenon: average yield strength and standard deviation; and average percent elongation at yield and standard deviation,

12.1.18 For materials which do not exhibit a yield point: average —% offset yield strength and standard deviation; and average percent elongation at —% offset yield strength and standard deviation,

12.1.19 Average modulus of elasticity and standard deviation (if secant modulus is used, so indicate and report strain at which calculated), and

12.1.20 When an extensometer is employed, so indicate.

13. Precision and Bias

13.1 Two interlaboratory tests have been run for these tensile properties. The first was run for modulus only, in 1977, in which randomly drawn samples of four thin (~ 0.025 mm (0.001-in.)) materials were tested with five specimens in each laboratory. Elastic (tangent) modulus measurements were made by six laboratories, and secant (1 %) modulus measurements were taken by five laboratories. The relative precision obtained in this interlaboratory study is in Table 2.

13.1.1 In deriving the estimates in Table 2, statistical outliers were not removed, in keeping with Practice E 691.[9]

13.1.2 The within-lab standard deviation of a mean value, $S_{\bar{x}}$, in each case was determined from the standard deviation, $S_{\bar{x}}$, of the five individual specimens as follows: $S_{\bar{x}} = S_x/(5)^{1/2}$. The $S_{\bar{x}}$ values were pooled among laboratories for a given material to obtain the within-lab standard deviation, S_r, of a test result (mean of five specimens). See 13.3-13.3.2 for definitions of terms in the tables.

TABLE 2 Precision Data for Modulus

Material	Thickness, mils	Average, 10^3 psi	S_r, 10^3 psi	S_R, 10^3 psi	I_r, 10^3 psi	I_R, 10^3 psi
Tangent Modulus						
LDPE	1.4	53.9	1.81	8.81	5.12	24.9
HDPE	1.6	191	5.47	16.2	15.5	45.9
PP	1.1	425	10.3	31.5	29.0	89.1
PET	0.9	672	13.8	55.5	39.1	157.1
Secant Modulus						
LDPE	1.4	45.0	2.11	3.43	5.98	9.70
HDPE	1.6	150	3.29	9.58	9.30	27.1
PP	1.1	372	4.66	26.5	13.2	74.9
PET	0.9	640	10.0	27.5	28.4	77.8

13.2 An interlaboratory test was run for all the other tensile properties except modulus in 1981, in which randomly drawn samples of six materials (one of these in three thicknesses) ranging in thickness from 0.019 to 0.178 mm (0.00075 to 0.007 in.) were tested in seven laboratories. A test result was defined as the mean of five specimen determinations. However, each laboratory tested eight specimens, and the $S_{\bar{x}}$ was determined from $S_{\bar{x}} = S_x/(5)^{1/2}$ as above. This was done to improve the quality of the statistics while maintaining their applicability to a five-specimen test result. The materials and their thicknesses are identified in Tables 3-7, each of which contain data for one of the following properties: tensile yield stress, yield elongation, tensile strength, tensile elongation at break, and tensile energy at break (see Note 17).[10]

NOTE 17—Subsequent to filing the research report, examination of the LDPE used in this study between crossed polarizers revealed lengthwise lines representing substantial widthwise variation in molecular orientation that probably was not successfully randomized out of the between-labs component of variance.

NOTE 18—**Caution:** The following explanations of I_r and I_R (13.3-13.3.3) are only intended to present a meaningful way of considering the *Approximate* precision of this test method. The data in Table 2 should not be rigorously applied to the acceptance or rejection of material, as those data are specific to the round robin and may not be representative of other lots, conditions, materials, or laboratories. Users of this test method should apply the principles outlined in Practice E 691 to generate data specific to their laboratory and materials, or between specific laboratories. The principles of 13.3-13.3.3 would then be valid for such data.

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[9] Supporting data are available from ASTM Headquarters. Request RR: D20-1084.

[10] Supporting data are available from ASTM Headquarters. Request RR: D20-1101.

TABLE 3 Precision Data for Yield Stress

Material	Thickness, mils	Average, 10^3 psi	(S_r)[A] 10^3 psi	(S_R)[B] 10^3 psi	$I(r)$[C] 10^3 psi	$I(R)$[D] 10^3 psi
LDPE	1.0	1.49	0.051	0.13	0.14	0.37
HDPE	1.0	4.33	0.084	0.16	0.24	0.44
PP	0.75	6.40	0.13	0.52	0.37	1.46
PC	4.0	8.59	0.072	0.29	0.20	0.82
CTA	5.3	11.4	0.12	0.50	0.34	1.43
PET	4.0	14.3	0.12	0.23	0.34	0.66
PET	2.5	14.4	0.14	0.54	0.40	1.52
PET	7.0	14.4	0.13	0.36	0.37	1.03

[A] S_r is the within-laboratory standard deviation of the average.

[B] S_R is the between-laboratories standard deviation of the average.

[C] $I_r = 2.83\ S_r$.

[D] $I_R = 2.83\ S_R$.

TABLE 4 Precision Data for Yield Elongation

Material	Thickness, mils	Average, %	(S_r)[A], %	(S_R)[B], %	$I(r)$[C], %	$I(R)$[D], %
PP	0.75	3.5	0.15	0.41	0.42	1.2
PET	2.5	5.2	0.26	0.92	0.74	2.6
PET	4.0	5.3	0.25	0.60	0.71	1.7
PET	7.0	5.4	0.14	1.05	0.40	3.0
CTA	5.3	5.4	0.19	0.99	0.54	2.8
PC	4.0	6.9	0.24	0.98	0.68	2.8
HDPE	1.0	8.8	0.32	1.82	0.91	5.2
LDPE	1.0	10.0	0.55	3.41	1.56	9.6

NOTE 1—See Table 3 for footnote explanation.

TABLE 5 Precision Data for Tensile Strength

Material	Thickness, mils	Average, 10^3 psi	(S_r)[A] 10^3 psi	(S_R)[B] 10^3 psi	$I(r)$[C] 10^3 psi	$I(R)$[D] 10^3 psi
LDPE	1.0	3.42	0.14	0.53	0.40	1.5
HDPE	1.0	6.87	0.27	0.81	0.76	2.3
PC	4.0	12.0	0.34	0.93	0.96	2.6
CTA	5.3	14.6	0.20	1.37	0.57	3.9
PP	0.75	28.4	1.57	4.56	4.4	12.9
PET	4.0	28.9	0.65	1.27	1.8	3.6
PET	7.0	30.3	0.83	1.32	2.3	3.7
PET	2.5	30.6	1.22	2.64	3.4	7.5

NOTE 1—See Table 3 for footnote explanation.

TABLE 6 Precision Data for Elongation at Break

Material	Thickness, mils	Average, %	(S_r)[A], %	(S_R)[B], %	$I(r)$[C], %	$I(R)$[D], %
CTA	5.3	26.4	1.0	4.3	3	12
PP	0.75	57.8	4.4	12.7	12	36
PET	2.5	120	8.0	14.6	23	41
PET	7.0	132	5.8	10.6	16	30
PET	4.0	134	4.4	12.2	12	35
PC	4.0	155	5.4	17.1	15	48
LDPE	1.0	205	24.4	73.3	69	210
HDPE	1.0	570	26.0	91.7	74	260

NOTE 1—See Table 3 for footnote explanation.

13.3 For the purpose of compiling summary statistics, a test result has been defined to be the average of five replicate measurements of a property for a material in a laboratory, as specified in this test method. Summary statistics are given in Table 3. In each table, for the material indicated, $S(r)$ is the pooled within-laboratory standard deviation of a test result, $S(R)$ is the between-laboratory standard deviation of a test result, where r equals $2.83 \times S(r)$ (see 13.3.1) and R equals $2.83 \times S(R)$ (see 13.3.2).

13.3.1 *Repeatability*, I_r (Comparing two test results for the same material, obtained by the same operator using the same equipment on the same day)—The two test results should be judged not equivalent if they differ by more than the I_r value for that material.

13.3.2 *Reproducibility*—In comparing two mean values for the same material obtained by different operators using different equipment on different days, either in the same

TABLE 7 Precision Data for Tensile Energy to Break

Material	Thickness, mils	Average, 10^3 in./lb/in.3	$(S_r)^A 10^3$ in./lb/in.3	$(S_R)^B 10^3$ in./lb/in.3	$I(r)^C 10^3$ in./lb/in.3	$I(R)^D 10^3$ in./lb/in.3
CTA	5.0	3.14	0.14	0.70	0.4	2.0
LDPE	1.0	5.55	0.84	2.47	2.4	7.0
PP	0.75	11.3	1.19	3.11	3.4	8.8
PC	4.0	12.9	0.59	1.55	1.7	4.4
HDPE	1.0	26.0	1.87	5.02	5.3	14.2
PET	2.5	26.1	2.13	4.20	6.0	11.9
PET	4.0	27.1	1.42	2.75	4.0	7.8
PET	7.0	28.4	1.71	2.72	4.8	7.7

NOTE 1—See Table 3 for footnote explanation.

laboratory or in different laboratories, the means should be judged not equivalent if they differ by more than the *R* value for that material.

13.3.3 Any judgment made in accordance with 13.3.1 and 13.3.2 would have an approximate 95 % (0.95) probability of being correct.

13.3.4 For further information, see Practice E 691.

13.4 *Bias*—The systematic error which contributes to the difference between a test result and a true (or reference) value. There are no recognized standards on which to base an estimate of bias for these test methods.

14. Keywords

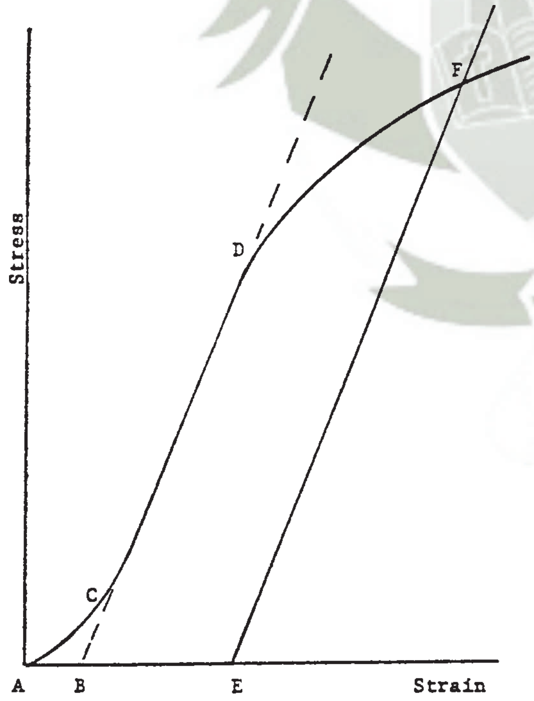
14.1 modulus of elasticity; plastic film; plastic sheeting; tensile properties; tensile strength; toughness; yield stress

ANNEXES

(Mandatory Information)

A1. TOE COMPENSATION

A1.1 In a typical stress-strain curve (Fig. A1.1) there is a toe region, *AC*, which does not represent a property of the material. It is an artifact caused by a takeup of slack, and alignment or seating of the specimen. In order to obtain correct values of such parameters as modulus, strain, and offset yield point, this artifact must be compensated for to give the corrected zero point on the strain or extension axis.

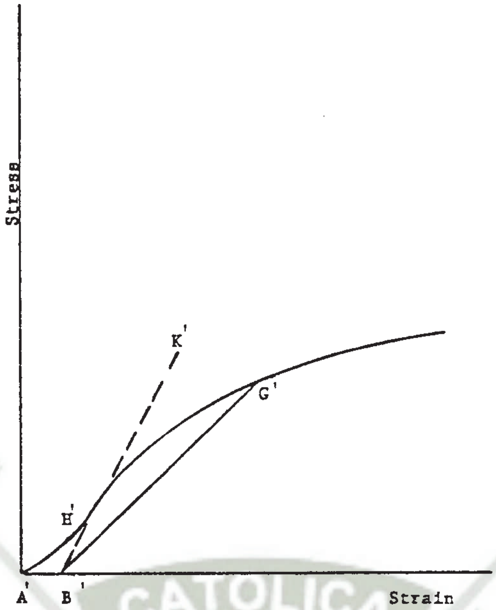


NOTE 1—Some chart recorders plot the mirror image of this graph.

FIG. A1.1 Material with Hookean Region

A1.2 In the case of a material exhibiting a region of Hookean (linear) behavior (Fig. A1.1), a continuation of the linear (*CD*) region of the curve is constructed through the zero-stress axis. This intersection (*B*) is the corrected zero-strain point from which all extensions or strains must be measured, including the yield offset (*BE*), if applicable. The elastic modulus can be determined by dividing the stress at any point along the line *CD* (or its extension) by the strain at the same point (measured from point *B*, defined as zero-strain).

A1.3 In the case of a material that does not exhibit any linear region (Fig. A1.2), the same kind of toe correction of the zero-strain point can be made by constructing a tangent to the maximum slope at the inflection point (H'). This is extended to intersect the strain axis at point B', the corrected zero-strain point. Using point B' as zero strain, the stress at any point (G') on the curve can be divided by the strain at that point to obtain a secant modulus (slope of line $B' G'$). For those materials with no linear region, any attempt to use the tangent through the inflection point as a basis for determination of an offset yield point may result in unacceptable error.



NOTE 1—Some chart recorders plot the mirror image of this graph.

FIG. A1.2 Material with No Hookean Region

A2. DETERMINATION OF TENSILE ENERGY TO BREAK

A2.1 Tensile energy to break (TEB) is defined by the area under the stress-strain curve, or

$$\text{TEB} = \int_0^{\epsilon_T} S \, d\epsilon \qquad \text{(A2.1)}$$

where S is the stress at any strain, ϵ, and ϵ_T is the strain at rupture. The value is in units of energy per unit volume of the specimen's initial gage region. TEB is most conveniently and accurately measured with a tension tester equipped with an integrator. The calculation is then:

$$TEB = (I/K)\,\frac{\text{(full scale load) (chart speed) (crosshead speed/chart speed)}}{\text{(mean caliper) (specimen width) (gage length)}} \qquad \text{(A2.2)}$$

where I is the integrator count reading and K is the maximum possible count per unit time for a constant full scale load. This whole calculation is typically done electronically. The results are best expressed in megajoules per cubic metre (or inch-pounds-force per cubic inch).

A2.2 Without an integrator, the area under the recorded stress-strain curve can be measured by planimeter, counting squares, or weighing the cut-out curve. These techniques are time-consuming and likely to be less accurate, since the load scale on some chart paper is not in round-number dimensions. Moreover, if the curve coordinates are in terms of force and extension instead of stress and strain, the calculated energy, corresponding to the measured area, must be divided by the product of gage length, specimen width, and mean caliper:

$$\text{TEB} = \frac{\text{(curve area) (force per unit chart scale) (extension per unit chart travel)}}{\text{(mean caliper) (specimen width) (gage length)}} \qquad \text{(A2.3)}$$

A2.3 For example, if the area under a force-extension curve is 60 000 mm^2, the load coordinate is 2.0 N/mm of chart scale, the extension coordinate is 0.25 mm of extension per mm of chart travel, and the specimen dimensions are 0.1 mm caliper, 15 mm width and 100 mm gage length, then the calculation for tensile energy to break is:

$$\text{TEB} = \frac{(60\ 000\ \text{mm}^2)\ (2.0\ \text{N/mm})\ (0.25 \times 10^{-3}\ \text{m/mm})}{(0.1 \times 10^{-3}\,\text{m})\ (15 \times 10^{-3}\,\text{m})\ (100 \times 10^{-3}\,\text{m})} \qquad \text{(A2.4)}$$

$$\text{TEB} = 200\ \text{MJ/m}^3$$

SUMMARY OF CHANGES

This section identifies the location of selected changes to this test method. For the convenience of the user, Committee D20 has highlighted those changes that may impact the use of this test method. This section may also include descriptions of the changes or reasons for the changes, or both.

D 882 – 02:
(*1*) Revised 7.1 and 7.2.
D 882 – 01:
(*1*) Section 5.5 rewritten.
(*2*) Note 6 deleted.
(*3*) Added Practice D 6287 to Referenced Documents section.
D 882 – 00:
(*1*) Added 11.1.
D 882 – 97:
(*1*) Note 3 rewritten and moved.
(*2*) ISO equivalency statement changed in Note 4.
(*3*) ISO reference changed in 2.2.
(*4*) Apparatus section (Section 5) rewritten.
(*5*) Table 1 deleted. Table 2 renumbered as Table 1.
(*6*) Sections 10.3 and 10.4 rewritten.
(*7*) Note 13 deleted. New Note 12 added.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

Designation: D 1238 – 04

Standard Test Method for Melt Flow Rates of Thermoplastics by Extrusion Plastometer[1]

This standard is issued under the fixed designation D 1238; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This test method covers measurement of the rate of extrusion of molten resins through a die of a specified length and diameter under prescribed conditions of temperature, load, and piston position in the barrel as the timed measurement is being made.

1.2 Procedure A is a manual cutoff operation based on time used for materials having flow rates that fall generally between 0.15 and 50 g/10 min. Procedure B is an automatically timed flow rate measurement used for materials having flows from 0.50 to 900 g/10 min. By both procedures, the piston travel is generally the same during the timed measurement; the piston foot is about 46 and 20.6 mm above the die. Comparable flow rates have been obtained by these procedures in interlaboratory round-robin measurements of several materials described in 13.1. Provision is made for calculation of melt volume-flow rate as well as melt mass-flow rate.

NOTE 1—Round-robin testing indicates this test method may be suitable at flow rates up to 1500 g/10 min if the timing clock resolves the elapsed time to the nearest 0.01 s.

NOTE 2—This test method and ISO 1133-1991 are technically equivalent.

1.3 *This standard does not purport to address the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific precautionary statements are given in 5.7, 10.2.12, and 14.1.2.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 618 Practice for Conditioning Plastics for Testing
D 883 Terminology Relating to Plastics
E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 *ANSI Standard:*

B46.1 on Surface Texture[3]

2.3 *ISO Standard:*

ISO 1133-1991 Determination of the Melt-Mass Flow Rate (MFR) and the Melt Volume-Flow Rate (MVR) of Thermoplastics[3]

3. Terminology

3.1 *General:*

3.1.1 For definition of some of the technical terms used in this test method refer to Terminology D 883.

4. Significance and Use

4.1 This test method is particularly useful for quality control tests on thermoplastics.

NOTE 3—Polymers having flow rates less than 0.15 or greater than 900 g/10 min may be tested by the procedures in this test method; however, precision data have not been developed.

4.2 This test method serves to indicate the uniformity of the flow rate of the polymer as made by an individual process and, in this case, may be indicative of uniformity of other properties. However, uniformity of flow rate among various polymers as made by various processes does not, in the absence of other tests, indicate uniformity of other properties.

4.3 The flow rate obtained with the extrusion plastometer is not a fundamental polymer property. It is an empirically defined parameter critically influenced by the physical properties and molecular structure of the polymer and the conditions of measurement. The rheological characteristics of polymer melts depend on a number of variables. Since the values of these variables occurring in this test may differ substantially from those in large-scale processes, test results may not correlate directly with processing behavior.

4.4 The flow rate of a material may be measured under any of the conditions listed for it in 8.2. Additional characterization of a material can be obtained if more than one condition is used. In case two conditions are employed, a Flow Rate Ratio (FRR) may be obtained by dividing the flow rate at one condition by the flow rate at the other condition.

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[1] This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.30 on Thermal Properties (Section D20.30.08).

Current edition approved March 1, 2004. Published April 2004. Originally approved in 1965. Last previous edition approved in 2001 as D 1238 - 01$^{\epsilon 1}$.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

***A Summary of Changes section appears at the end of this standard.**

5. Apparatus

5.1 *Plastometer*:

5.1.1 The apparatus shall be a dead-weight piston plastometer consisting of a thermostatically controlled heated steel cylinder with a die at the lower end and a weighted piston operating within the cylinder. The essential features of the plastometer, illustrated in Figs. 1 and 2, are described in 5.2-5.8. All dimensional measurements shall be made when the article being measured is at 23 ± 5°C.

5.1.2 Relatively minor changes in the design and arrangement of the component parts have been shown to cause differences in results among laboratories. It is important, therefore, for the best interlaboratory agreement that the design adhere closely to the description herein; otherwise, it should be determined that modifications do not influence the results.

5.2 *Cylinder*—The steel cylinder shall be 50.8 mm in diameter, 162 mm in length with a smooth, straight hole 9.5504 ± 0.0076 mm in diameter, displaced 4.8 mm from the cylinder axis. Wells for a thermal sensor (thermoregulator, thermistor, etc.) and thermometer shall be provided as shown in Fig. 1. A 3.2-mm plate shall be attached to the bottom of the cylinder to retain the die. A hole in this plate, centered under the die and countersunk from below, allows free passage of the extrudate. The cylinder may be supported by at least two 6.4-mm high-strength screws at the top (radially positioned at right angles to the applied load) or by at least two 10-mm diameter rods screwed into the side of the cylinder for attaching to a vertical support. The essential dimensions of a satisfactory cylinder of this type are shown in Fig. 1 (Note 4). The cylinder bore should be finished by techniques known to produce approximately 12 rms or better in accordance with ANSI B46.1.

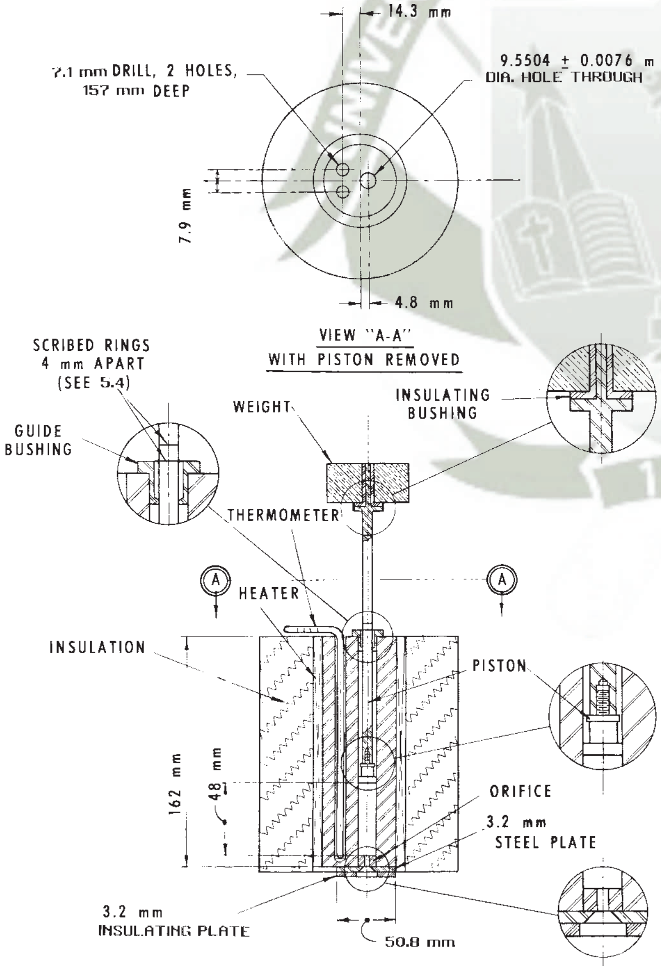

FIG. 1 General Arrangement of Extrusion Plastometer

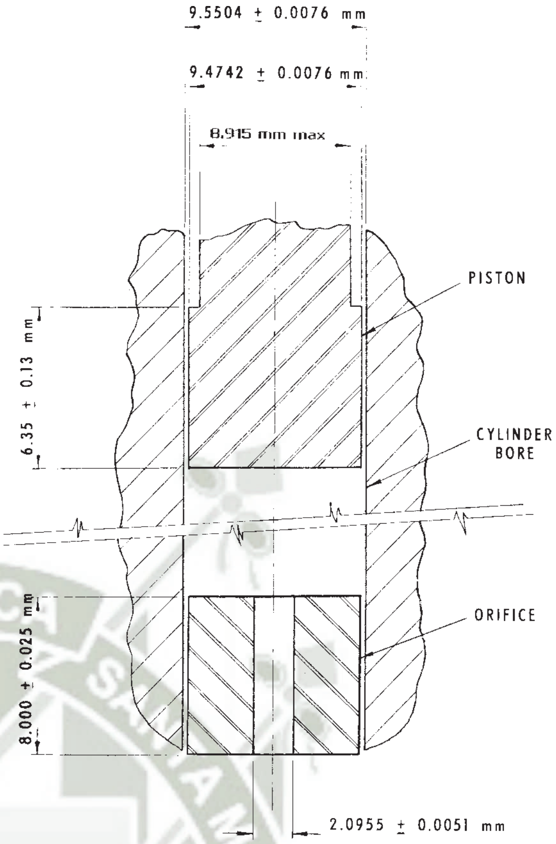

FIG. 2 Details of Extrusion Plastometer

NOTE 4—Cylinders made of SAE 52100 or other equivalent steel heat-hardened to 60–65 Rockwell Hardness Scale C give good service when used at temperatures below 200°C. Cylinder liners of cobalt-chromium-tungsten alloy are also satisfactory to 300°C.

5.3 *Die*—The outside of the steel die shall be such diameter that it will fall freely to the bottom of the 9.5504 ± 0.0076 mm diameter hole in the cylinder (Note 5). The die shall have a smooth straight bore 2.0955 ± 0.0051 mm in diameter and shall be 8.000 ± 0.025 mm in length. The bore and its finish are critical. It shall have no visible drill or other tool marks and no detectable eccentricity. The die bore shall be finished by techniques known to produce approximately 12 rms or better in accordance with ANSI B46.1.

NOTE 5—Recommended die material is tungsten carbide. Also satisfactory are steel, synthetic sapphire, and cobalt-chromium-tungsten alloy.

5.4 *Piston*:

5.4.1 The piston shall be made of steel with an insulating bushing at the top as a barrier to heat transfer from the piston to the weight. The land of the piston shall be 9.4742 ± 0.0076 mm in diameter and 6.35 ± 0.13 mm in length. The piston

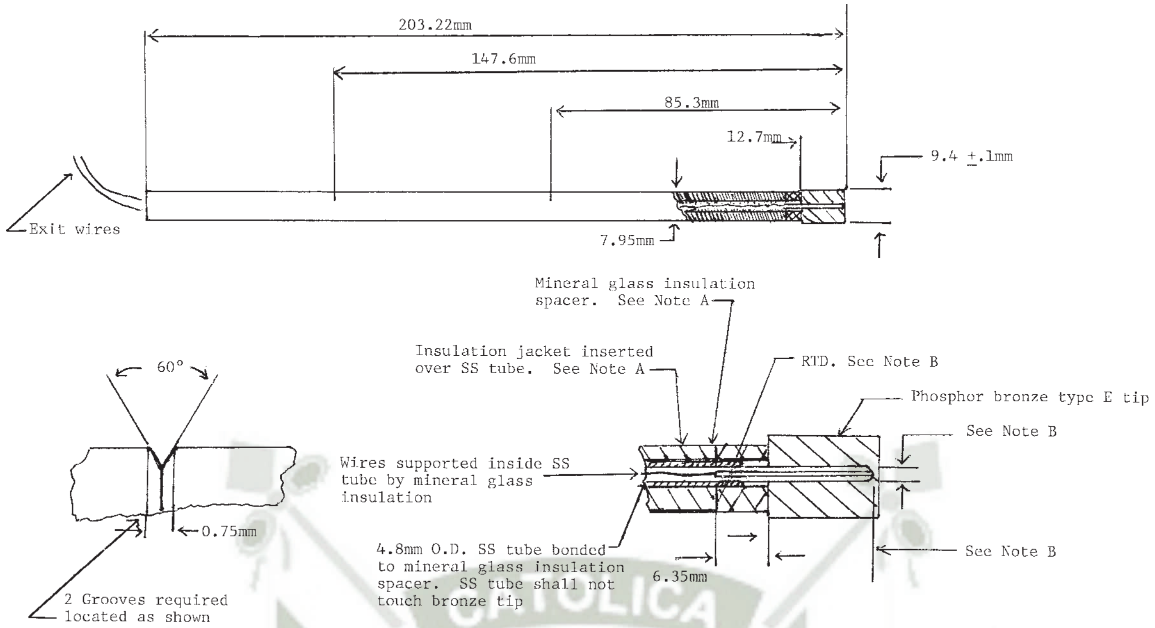

FIG. 3 Example of a Temperature Calibration Device

Note A—Mineral glass insulation or equivalent spacer shall be bonded to tip and SS tube. Bond material shall be low conductivity type, 400°C minimum rating. Insulation jacket material shall be low conductivity type (400°C minimum rating preferred, see Note 5).

Note B—The RTD shall be inserted into bronze tip and bonded using high conductivity, 400°C rated material. Tip of RTD element shall touch the bronze tip. Minimum insertion depth of 11.2 mm clearance between RTD and tip wall shall be minimized.

design may incorporate means for land replacement, for example, having threads and flats immediately above the land. Above the land, the piston shall be no larger than 8.915 mm in diameter (Note 6). The finish of the piston foot shall be 12 rms in accordance with ANSI B46.1. If wear or corrosion is a problem, the piston should be of stainless steel and equipped with a detachable foot for ease of replacement.

NOTE 6—To improve standardization it is preferable that the piston be guided with a loose-fitting metal sleeve at the top of the cylinder.

NOTE 7—Pistons of SAE 52100 steel with the bottom 25 mm, including the foot, hardened to a Rockwell hardness, C scale, of 55 to 59 have been found to give good service when used at temperatures below 200°C.

5.4.2 The piston shall be scribed with two reference marks 4 mm apart in such fashion that when the lower mark coincides with the top of the cylinder or other suitable reference point, the bottom of the piston is 48 mm above the top of the die (see Fig. 1).

5.4.3 The combined weight of piston and load shall be within a tolerance of ±0.5 % of the selected load.

5.5 *Heater*:

5.5.1 The equipment must have a heater capable of heating the apparatus so that the temperature at 10 mm above the die can be maintained within ±0.2°C of the desired temperature during the test. The temperature of the barrel, from 10 mm to 75 mm above the top of the die, must be maintained within ±1 % of the set temperature (°C).

NOTE 8—At temperatures higher than 200°C this degree of temperature control may be more difficult to obtain.

5.5.2 Calibrate the temperature-indicating device by means of a light-gage probe-type thermocouple or a platinum-resistance temperature sensor having a short sensing length.[4] The thermocouple should be encased in a metallic sheath having a diameter of approximately 1.6 mm with its hot junction grounded to the end of the sheath. Insert the temperature sensor into the melt from the top of the cylinder so that it is 10 ± 1 mm above the upper face of the die. The temperature sensors shall be used with a potentiometer having a sensitivity of at least 0.005 mV, or a temperature readout having a sensitivity of at least 0.1°C. Calibration should also be verified at 75 mm above the upper face of the die. An alternate technique for calibration is to use a sheathed thermocouple or platinum-resistance temperature sensor with tip diameter of 9.4 ± 0.1 mm for insertion in the bore without material present. An example of this is shown in Fig. 3. Calibration of the temperature-indicating device shall be verified at each run temperature.

NOTE 9—The response of the temperature sensing device may be affected by immersion level. Take care to ensure adequate insulation of the device sensor and stabilization of the barrel temperature.

5.5.3 Heat shall be supplied by electric band heater(s) covering the entire length of the cylinder. The heater(s) may be single- or multi-element, depending upon the manufacturer's control means. The heater(s) plus control system must be capable of maintaining the temperature within the required ±0.2°C of the set point. The temperature sensor and readout equipment must be calibrated to a traceable national standard

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[4] Round-robin data showing flow rate and corresponding temperature profile of the melt obtained using probe-type thermocouples and platinum resistance temperature sensors can be obtained from ASTM Headquarters. Request RR:D20-1094.

(that is, NIST) at least once per year. The cylinder with the heater(s) shall be lagged with 38 mm of foamed-glass insulation. An insulating plate 3.2 mm in thickness shall be attached to the bottom of the cylinder to minimize heat loss at this point.

5.6 *Temperature Controller*—The type of controller and sensor must be capable of meeting the required control tolerance specified in 5.5.1.

5.7 *Thermometer*— Thermometers having a range of 4°C graduated in 0.2°C divisions may be used to indicate temperature. The temperature at this point may not necessarily be the temperature of the material 10 mm above the die. The thermometer may be used to monitor indirectly the temperature of the material 10 mm above the die and may be calibrated by reference to a thermocouple or platinum resistance temperature sensor inserted in the material 10 mm above the die. See 5.5.2 for a description of a method for measuring temperature. **Warning—**Caution should be observed with the use of a mercury-filled thermometer. Mercury vaporization occurs if the thermometer is broken. Mercury thermometers are not to be used at or above the boiling point of mercury, which is 357°C.

NOTE 10—**Warning:**

5.8 *Level*—Provision shall be made for vertical alignment of the bore of the extrusion plastometer. This is necessary to minimize subtractive loads resulting from rubbing or friction between the piston tip and sidewall. Means of alignment are discussed in Appendix X1.

5.9 *Accessory Equipment*—Necessary accessories include equipment for charging samples to the cylinder, a funnel, a die plug, a tool for cutting off the extruded sample, a timer or stop watch, cleaning equipment, go/no-go gages, a balance accurate to ±0.001 g, and, when required, a weight or weight-piston support.

NOTE 11—Satisfactory operation of the apparatus for polyethylenes can be ascertained by making measurements on NIST Standard Reference Materials (SRMs) certified for melt flow rate. The four SRMs certified under condition 190/2.16 are SRM 1473 with a flow rate of 1.29 g/min, SRM 1474 with a flow rate of 5.03 g/10 min, SRM 1496 with a flow rate of 0.26 g/10 min, and SRM 1497 with a flow rate of 0.19 g/10 min. SRM 1475a is certified under condition 190/3.25 with a flow rate of 2.20 g/10 min.[5]

6. Test Specimen

6.1 The test specimen may be in any form that can be introduced into the bore of the cylinder, for example, powder, granules, strips of film, or molded slugs. It may be desirable to preform or pelletize a powder.

7. Conditioning

7.1 Many thermoplastic materials do not require conditioning prior to testing. Materials which contain volatile components, are chemically reactive, or have other special characteristics most probably require appropriate conditioning procedures. Moisture not only affects reproducibility of flow rate measurement but, in some types of materials, degradation is accelerated by moisture at the high temperatures used in testing. Check the applicable material specification for any conditioning requirements before using this test. See Practice D 618 for appropriate conditioning practices.

8. Procedural Conditions

8.1 Standard conditions of test are given in Table 1. Test conditions shall be shown as: Condition _ _ _ / _ _ _, where the temperature in degrees Celsius is shown first, followed by the weight in kilograms. For example: Condition 190/2.16.

8.2 The following conditions have been found satisfactory for the material listed:

Material	Condition	
Acetals (copolymer and homopolymer)	190/2.16	190/1.05
Acrylics	230/1.2	230/3.8
Acrylonitrile-butadiene-styrene	200/5.0	230/3.8
	220/10	
Acrylonitrile/butadiene/styrene/polycarbonate blends	230/3.8	250/1.2
	265/3.8	265/5.0
Cellulose esters	190/0.325	190/2.16
	190/21.60	210/2.16
Ethylene-chlorotrifluoroethylene copolymer	271.5/2.16	
Ethylene-tetrafluoroethylene copolymer	297/5.0	
Nylon	275/0.325	235/1.0
	235/2.16	235/5.0
	275/5.0	
Perfluoro(ethylene-propylene) copolymer	372/2.16	
Perfluoroalkoxyalkane	372/5.0	
Polycaprolactone	125/2.16	80/2.16
Polychlorotrifluorethylene	265/12.5	
Polyether sulfone (PES)	380/2.16	360/10
	343/2.16	
Polyethylene	125/0.325	125/2.16
	250/1.2	
	190/0.325	190/2.16
	190/21.60	190/10
	310/12.5	
Polycarbonate	300/1.2	
Polymonochlorotrifluoroethylene	265/21.6	
	265/31.6	
Polypropylene	230/2.16	
Polyphenyl sulfone (PPSU)	365/5.0	380/2.16
Polystyrene	200/5.0	230/1.2
	230/3.8	190/5.0
Polysulfone (PSU)	343/2.16	360/10
Polyterephthalate	250/2.16	210/2.16
	285/2.16	
Poly(vinyl acetal)	150/21.6	
Poly(vinylidene fluoride)	230/21.6	
	230/5.0	
Poly(phenylene sulfide)	315/5.0	
Styrene acrylonitrile	220/10	230/10
	230/3.8	
Styrenic Thermoplastic Elastomer	190/2.16	200/5.0
Thermoplastic Elastomer-Ether-Ester	190/2.16	220/2.16
	230/2.16	240/2.16
		250/2.16
Thermoplastic elastomers (TEO)	230/2.16	
Vinylidene fluoride copolymers	230/21.6	
	230/5.0	
	for T_m= 100° use 120/5.0 or 21.6	

NOTE 12—Some materials may require special materials of construction or handling for performing this test. Please refer to the material specification for appropriate recommendations.

8.3 If more than one condition is used and the material is polyethylene, the determination of Flow Rate Ratio (FRR) has been found to be useful. The FRR is a dimensionless number derived by dividing the flow rate at Condition 190/10 by the flow rate at Condition 190/2.16.

NOTE 13—When determining such a ratio of flow rates for a material at

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[5] These standard polyethylenes are available from the National Institute of Standards and Technology, Office of Standard Reference Materials, Washington, DC 20234.

TABLE 1 Standard Test Conditions, Temperature, and Load

Condition	Temperature, °C	Total Load Including Piston, kg	Approximate Pressure	
Standard Designation			kPa	psi
80/2.16	80	2.16		
125/0.325	125	0.325	44.8	6.5
125/2.16	125	2.16	298.2	43.25
150/2.16	150	2.16	298.2	43.25
190/0.325	190	0.325	44.8	6.5
190/2.16	190	2.16	298.2	43.25
190/21.60	190	21.60	2982.2	432.5
200/5.0	200	5.0	689.5	100.0
230/1.2	230	1.2	165.4	24.0
230/3.8	230	3.8	524.0	76.0
265/12.5	265	12.5	1723.7	250.0
275/0.325	275	0.325	44.8	6.5
230/2.16	230	2.16	298.2	43.25
190/1.05	190	1.05	144.7	21.0
190/10.0	190	10.0	1379.0	200.0
300/1.2	300	1.2	165.4	24.0
190/5.0	190	5.0	689.5	100.0
235/1.0	235	1.0	138.2	20.05
235/2.16	235	2.16	298.2	43.25
235/5.0	235	5.0	689.5	100.0
250/2.16	250	2.16	298.2	43.25
310/12.5	310	12.5	1723.7	250.0
210/2.16	210	2.16	298.2	43.25
285/2.16	285	2.16	298.2	43.25
315/5.0	315	5.0	689.5	100.0
372/2.16	372	2.16	298.2	43.25
372/5.0	372	5.0	689.5	100
297/5.0	297	5.0	689.5	100
230/21.6	230	21.6	2982.2	432.5
230/5.0	230	5.0	689.5	100
265/21.6	265	21.6	2982.2	432.5
265/31.6	265	31.6	4361.2	632.5
271.5/2.16	271.5	2.16	298.2	43.25
220/10	220	10.0	1379.0	200.0
250/1.2	250	1.2	165.4	24.0
265/3.8	265	3.8	524.0	76.0
265/5	265	5.0	689.5	100.0

the same temperature under different loads, it has been found that precision is maximized when one operator uses one Procedure (A or B), the same plastometer, and the same die for both measurements (the die need not be removed from the plastometer between the two determinations).

9. Procedure A—Manual Operation

9.1 Select conditions of temperature and load from Table 1 in accordance with material specifications such that flow rates will fall between 0.15 to 50 g/10 min.

9.2 Ensure that the bore of the extrusion plastometer is properly aligned in the vertical direction. (See Appendix X1.)

9.3 Inspect the apparatus and die for cleanliness. If it is not clean, see 9.11.

NOTE 14—The degree of cleanliness can significantly influence the flow rate results, therefore a thorough method of cleaning must be established. It has been found that three swabs of the barrel is satisfactory for most materials and that the die, barrel, and piston are more easily cleaned while hot.

9.4 Check the die bore diameter with appropriately sized no-go/go gages prior to testing. Make frequent checks to determine whether the die diameter (tested with die at 23 ± 5°C) is within the tolerances given in 5.3.

NOTE 15—Cleaning and usage can result in a die diameter that is out of specifications. Data has shown that erroneous results will be obtained if the die diameter is not within the appropriate tolerances.

9.5 Verify that the temperature is stable and within ±0.2°C of the appropriate test temperature as specified in 5.5.1.

9.6 Insert the die and the piston. The temperature of the cylinder with the piston and die in place must be stable at the appropriate test temperature 15 min before testing is begun. When equipment is used repetitiously, it should not be necessary to heat the piston and die for 15 min.

NOTE 16—The reduction in heating time when the unit is being used repetitiously is only allowed when runs of the same or similar material are being measured over a continuous time frame. If the piston and die are removed and cleaned, they should be considered "cold" and the full 15 minutes heating stabilization time required.

9.7 Remove the piston and place it on an insulated surface. Charge the cylinder within 1 min with a weighed portion of the sample in accordance with the expected flow rate, as given in Table 2. Reinsert the piston and add the appropriate weight.

NOTE 17—Experience has shown that for the best reproducibility the piston should operate within the same part of the cylinder for each measurement. The piston is scribed so the starting point for each extrusion is roughly the same. Some excess of material over the minimum required for the actual flow measurement portion of the test is provided by the charging weights shown in Table 2. This is necessary to achieve a void-free extrudate and flow equilibrium before start of rate measurements.

NOTE 18—It is frequently helpful to take interim cuts of the extrudate at uniform time intervals during the specified extrusion time. Weights of

TABLE 2 Standard Test Conditions, Sample Mass,[A] and Testing Time[B]

Flow Range, g/10 min	Suggested Mass of Sample in Cylinder, g	Time Interval, min	Factor for Obtaining Flow Rate in g/10 min
0.15 to 1.0	2.5 to 3.0	6.00	1.67
>1.0 to 3.5	3.0 to 5.0	3.00	3.33
>3.5 to 10	4.0 to 8.0	1.00	10.00
>10 to 25	4.0 to 8.0	0.50	20.00
>25	4.0 to 8.0	0.25	40.00

[A] This is a suggested mass for materials with melt densities of about 0.7 g/cm^3. Correspondingly, greater quantities are suggested for materials of greater melt densities. Density of the molten resin (without filler) may be obtained using the procedure described by Terry, B. W., and Yang, K., "A New Method for Determining Melt Density as a Function of Pressure and Temperature," *SPE Journal*, SPEJA, Vol. 20, No. 6, June 1964, p. 540 or the procedure described by Zoller, Paul, "The Pressure-Volume-Temperature Properties of Polyolefins," *Journal of Applied Polymer Science*, Vol 23, 1979, p. 1051. It may also be obtained from the weight of an extruded known volume of resin at the desired temperature. For example, 25.4 mm (1 in.) of piston movement extrudes 1.804 cm^3 of resin. An estimate of the density of the material can be calculated from the following equation:

$$\text{resin density at test temperature} = M/1.804$$

where:
M = mass of extruded resin.

[B] See 9.14.

these individual cuts give an indication of the presence of bubbles which may be masked due to their size or to opacity of the sample. This technique is particularly helpful in the case of highly pigmented materials. Forcing out some of the resin manually during the preheat period often eliminates bubbles in the test extrudate.

9.8 Allow time for the material to soften and begin to melt, and then purge some material to a position such that subsequent travel of the piston will position the lower scribe mark at the reference start position 7.0 ± 0.5 min from the completion of the charge. Purge must be completed at least 2 min prior to start of the test for materials having melt flow rates less than 10 g/10 min.

NOTE 19—It has been found that purging within 60 s of the start time will result in higher variability in the data.

NOTE 20—There may be cases where the 7.0 ± 0.5 min is too much or not enough preheat time. For those materials, provisions must be in the material documents. It is necessary to refer to the appropriate material document before beginning any test.

NOTE 21—Additional care may be necessary to prevent thermal degradation in the extrusion plastometer. This is sometimes done by the addition of an appropriate antioxidant. For highly unstable materials, it may be necessary to use alternative techniques as an indication of flow characteristics.

9.9 For materials with flow rates greater than 10 g/10 min, a weight (and if needed, a piston) support must be used after the initial purge. The support shall be removed at such a time as to allow the test to begin within 7 ± 0.5 min of the completion of the charge. The piston/weight support should be of such a length that the lower scribe mark of the supported piston/weight will be 25 mm above the top of the guide bushing or other suitable reference mark.

NOTE 22—It has been found that the effect of choosing plugging, weight support, or both, is significant to the flow rate results. The choice of piston support was made to cover all conditions and flow rates 10 to 50 g/10 min.

9.10 For all tests, start collecting a timed extrudate when requirements for the piston position are met, provided this is within 7.0 ± 0.5 min from the end of charging; otherwise, discard the charge and repeat the test with readjusted piston position after the initial purge, or change weights. Requirements are that the top scribed mark on the piston be visible above the cylinder or index and that the lower scribe mark be in the cylinder or below the index. As the lower scribed mark approaches the top of the cylinder or index, reset the timer to zero, then simultaneously start the timer and make the initial cut-off when the position requirements are met. Make the final cut-off exactly when the time interval given in Table 2 is reached. Collect the timed extrudate. If the extrudate contains visible bubbles, discard the complete charge and begin the test again.

NOTE 23—The charge weight should only be increased if no material is being purged and there is still not enough material to complete the test.

9.11 Discharge the remainder of the specimen and push the die out through the top of the cylinder. Swab out the cylinder with cloth patches after the manner of cleaning a pistol barrel. The die may be cleaned by dissolving the residue in a solvent. A better method is pyrolytic decomposition of the residue in a nitrogen atmosphere. Place the die in a tubular combustion furnace or other device for heating to 550 ± 10°C and clean with a small nitrogen purge through the die. This method is preferable to flame or solvent cleaning, being faster than solvent cleaning and less detrimental to the die than an open flame. In certain cases where materials of a given class having similar flow characteristics are being tested consecutively, interim die cleaning may be unnecessary. In such cases, however, the effect of cleaning upon flow rate determination must be shown to be negligible if this step is avoided.

9.12 Once the extrudate is cool, weigh to the nearest 1 mg.

9.13 Multiply the weight of the extrudate by the appropriate factor shown in Table 2 to obtain the flow rate in grams per 10 min.

NOTE 24—Frequently, errors in test technique, apparatus geometry, or test conditions, which defy all but the most careful scrutiny exist, causing discrepancy in flow rate determinations. The existence of such errors is readily determined by periodically measuring a reference sample of known flow rate. The flow rate value and range to be tolerated can be determined using a statistically correct test program composed of multiple determinations with various instruments. Standard samples of polyethylene, linear or branched, are available from the National Institute of Standards and Technology.

9.14 In case a specimen has a flow rate at the borderline of the ranges in Table 2 and slightly different values are obtained at different time intervals, the referee value shall be obtained at the longer time interval.

10. Procedure B—Automatically Timed Flow Rate Measurement

10.1 *Apparatus*:

10.1.1 Extrusion plastometer and auxiliary equipment are detailed in Section 4 and below.

10.1.2 A timing device shall electrically, optically, or mechanically time piston movement within the specified travel range. The requirements of the system are as follows:

10.1.2.1 Sense and indicate the piston travel time within ±0.01 s (see Note 1).

10.1.2.2 Measure piston travel within ±0.4 % of the nominal preset value (see 10.1.2.4 and 10.1.2.5) for use in the flow rate calculations.

10.1.2.3 Any effects on the applied load must be included in the allowable tolerance given in 5.4.3.

10.1.2.4 It should be preset or be settable for measuring piston travel of 6.35 ± 0.25 mm for flow rates up to 10 g/10 min.

10.1.2.5 It should be preset or be adjustable for measuring piston travel of 25.4 ± 0.25 mm for flow rates greater than 10 g/10 min.

10.1.2.6 To ensure high interlaboratory reproducibility, it is important that the timing device operates within a fixed portion of the cylinder. This is defined as the portion of the cylinder between 46 ± 2 mm and 20.6 ± 2 mm above the top of the die.

10.1.2.7 Check die, cylinder, and position dimensions for conformance to 5.2-5.4 and Figs. 1 and 2.

10.2 *Procedure*:

10.2.1 Refer to Table 1 for selection of conditions of temperature and load in accordance with the material specification.

10.2.2 Check the die bore diameter with appropriately sized no-go/go gages prior to testing. Make frequent checks to determine whether the die diameter (tested with die at 23 ± 5°C) is within the tolerances given in 5.3 (see Note 15).

10.2.3 Ensure that the bore of the extrusion plastometer is properly aligned in the vertical direction (see Appendix X1).

10.2.4 Inspect the apparatus and die for cleanliness. If it is not clean, see 9.11 and Note 14.

10.2.5 Check the die bore diameter with appropriately sized no-go/go gages before beginning the test. Make frequent checks to determine whether the die diameter is within the tolerances given in 5.3 (see Note 15).

10.2.6 Verify that the temperature is stable and within ±0.2°C of the appropriate test temperature as specified in 5.5.1.

10.2.7 Insert the die and the piston. The temperature of the cylinder with the piston and die in place must be stable at the appropriate test temperature 15 min before testing is begun. When equipment is used repetitiously, it should not be necessary to heat the piston and die for 15 min.

10.2.8 Adjust the travel arm to 6.35 ± 0.25 mm for measuring materials with expected flow rates of up to 10 g/10 min or 25.40 ± 0.25 mm for measuring materials with expected flow rates of 10 g/10 min or higher.

NOTE 25—It has been found that for some materials the melt flow rates obtained on a material will be different depending on which timer length is chosen; therefore, it is important to adhere to the protocol in 10.2.8 to compare interlaboratory results.

10.2.9 Remove the piston and place it on an insulated surface. Charge the cylinder within 60 s with a weighted portion of the sample in accordance with the expected flow rate, as given in Table 2. Reinsert the piston and add weight.

10.2.10 Allow time for the material to soften and begin to melt, and then purge some material to a position such that subsequent travel of the piston will position the lower scribe mark at the reference start position 7.0 ± 0.5 min from the completion of the charge. Purge must be completed at least 2 min prior to start of the test for materials having melt flow rates less than 10 g/10 min (see Note 19).

10.2.11 Weight and piston support, if needed, must be used after the initial purge. The support will be removed at such a time as to allow the timer to activate within 7.0 ± 0.5 min after completion of the charge. If the timer is not activated within 7 ± 0.5 min after the completion of the charge, the test must be repeated with readjusted piston position after the initial purge, or change weights. The piston/weight support should be of such a length that the lower scribe mark of the supported piston/weight will be at least 25 mm above the top of the cylinder. Only use piston support if there is excessive material flow (see Notes 22 and 23).

10.2.12 For materials greater than 50 g/10 min a die plug must be used in addition to the piston/weight support. The die plug is inserted before charge and is removed prior to removing the piston/weight support. The initial charge should be adjusted to reduce excess flow. If the timer arm is not activated within 7 ± 0.5 min after the completion of the charge the test must be repeated with readjusted piston position, or change weights (see Notes 22 and 23). **Warning—**Rapid expulsion of material when die plug is removed may be hazardous.

NOTE 26—**Warning:**

10.2.13 If the timed extrudate contains visible bubbles, repeat the test (see Note 24).

10.2.14 Record the time to the nearest 0.01 s for the piston to complete the calibrated distance of travel.

10.2.15 Discharge any remaining resin and clean the die, piston, and cylinder as detailed in 9.11.

11. Procedure C— Automatically Timed Flow Rate Measurement for High Flow Rate Polyolefins Using Half-Height, Half Diameter Die

11.1 *Apparatus*:

11.1.1 Extrusion plastometer and auxiliary equipment are detailed in Sections 5 and 10.

11.1.2 For polyolefins with a MFR of 75 or greater using the standard die (See 5.3), an alternate die can be used to reduce the flow rate of these materials and improve the reproducibility of results. The alternate die dimensions shall be: Height 3.985 ± 0.025 mm; bore diameter 1.048 ± 0.005 mm. No spacer shall be used with this die. Bore and finish requirements are the same as 5.3.

11.1.3 For calibration of the temperature indicating device, 5.5.2 shall be used with the variation that temperatures are

TABLE 3 Factors for Calculation of Flow Rate

Material (Unpigmented)	Temperature, °C	Piston Travel, *L*, cm [in.]	Factor for Calculation of Flow Rate, *F*[A]
Polyethylene	190	2.54 [1]	826
Polyethylene	190	0.635 [0.25]	207
Polypropylene	230	2.54 [1]	799
Polypropylene	230	0.635 [0.25]	200

[A] Factors calculated using melt-density values of 0.7636 g/cm^3 for polyethylene and 0.7386 g/cm^3 for polypropylene, as expressed in article by Zoller, Paul, "The Pressure-Volume-Temperature Properties of Polyolefins," *Journal of Applied Polymer Science*, Vol 23, 1979, P. 1051. The base densities at 23°C for which the melt densities are reported were 0.917 g/dm^3 for annealed low-density polyethylene and polypropylene homopolymer.

measured at 14 ± 1 mm and at a nominal 79 mm above the upper surface of the die.

11.1.4 If a thermometer as described in 5.7 is used to indicate temperature, it can be used to monitor indirectly the temperature of the material 14 mm above the upper surface of the die and may be calibrated via 11.1.3.

11.2 *Procedure*:

11.2.1 Use procedure described in 10.2 with the exception that the die diameter and tolerances are given in 11.1.2.

12. Calculation (Procedures B and C)

12.1 Calculate the flow rate in grams per 10 min or volume rate in cm^3 per 10 min as follows (see Note 24):

$$\text{Flow rate} = (426 \times L \times d)/t$$

or

$$\text{Volume rate} = 426 \times L/t$$

where:

L = length of calibrated piston travel, cm,
d = density of resin at test temperature, g/cm^3(see reference under Table 2),
t = time of piston travel for length L, s, and
426 = mean of areas of piston and cylinder × 600.

NOTE 27—Factors that may be substituted in the following equation are given for some materials in Table 3.

$$\text{Flow rate, g/10 min} = F/t$$

where:

F = factor from Table 3, and
t = time of piston travel for length L, s.

12.2 Agreement between Procedures A and B may be optimized if an average melt density for a particular type of material is determined with the actual equipment used and that value is substituted into the equation given in 12.1.

13. Report

13.1 Report the following information:

13.1.1 Statement indicating the nature and physical form of the material charged to the cylinder.

13.1.2 Temperature and load at which the test is run shall be reported. The results and test conditions can be referred to as FR-condition, where the standard designation for the condition from Table 1 is shown (for example: FR-190/2.16).

NOTE 28—It has become customary to refer to the flow rate of polyethylene as "melt index" when obtained under Condition 190/2.16. However, for all other materials the use of melt index or any term other than "flow rate" is discouraged, regardless of the condition used.

13.1.3 Flow rate reported as the rate of extrusion in grams per 10 min or volume rate in cm^3 per 10 min.

13.1.4 Procedure used (A, B, or C).

13.1.5 Any unusual behavior of the test specimen such as discoloration, sticking, extrudate surface irregularity or roughness, etc.

13.1.6 Details of conditioning, if any.

14. Precision and Bias (Procedures A, B, and C)

14.1 *Precision*:

14.1.1 Tables 4 and 5 are based on a round robin[6] conducted in 1986 and 1987, involving polypropylene, polyethylene, polystyrene, polycarbonate and acrylic materials. Tables 6 and 7 are based on a round robin[7] completed in 1997 involving low and high density polyethylene, polypropylene, polystyrene, polycarbonate, PMMA, and acetal. The number of participating laboratories is shown for each material. Data for Tables 4 and 5 were generated through each lab testing two specimens for each material on three different days, while data for Tables 6 and 7 were generated through each lab testing two specimens for each material on two different days. The analysis in Practice E 691 is based on a test result being the average of two specimens.

14.1.2 Table 8 is based on a round robin[8] conducted in 1980 using Procedure B. Four polypropylene samples having flow

TABLE 4 Precision, Procedure A (Values in g/10 min)

Material	Condition	Average	S_r[A]	S_R[B]	I_r[C]	I_R[D]	Number of Laboratories
Polyethylene	190/2.16	0.27	0.008	0.022	0.023	0.063	9
Polyethylene	190/2.16	0.40	0.012	0.038	0.035	0.108	9
Polyethylene	190/2.16	2.04	0.026	0.079	0.073	0.224	9
Polyethylene	190/2.16	44.1	0.919	1.232	2.560	3.486	7
Polypropylene	230/2.16	2.23	0.106	0.226	0.299	0.639	9
Polypropylene	230/2.16	7.09	0.222	0.471	0.627	1.331	9
Polypropylene	230/2.16	32.8	0.581	1.051	1.644	2.974	9
Polystyrene	200/5	1.67	0.024	0.122	0.068	0.344	6
Polystyrene	200/5	8.82	0.190	0.667	0.538	1.886	6
Polystyrene	200/5	13.3	0.305	0.925	0.864	2.617	6
Polycarbonate	300/1.2	2.41	0.076	0.115	0.215	0.326	4
Polycarbonate	300/1.2	10.5	0.429	0.647	1.215	1.830	4
Polycarbonate	300/1.2	16.2	0.155	1.109	0.438	3.140	4
Acrylic	230/3.8	2.59	0.051	0.051	0.145	0.145	3

[A] S_r = within-laboratory standard deviation of the average.
[B] S_R = between-laboratories standard deviation of the average.
[C] $I_r = 2.83\ S_r$, and
[D] $I_R = 2.83\ S_R$.

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[6] Supporting data are available from ASTM Headquarters. Request RR: D20-1164.

[7] Supporting data are available from ASTM Headquarters.

[8] Supporting data are available from ASTM Headquarters. Request RR: D20-1124.

TABLE 5 Precision, Procedure B (Values in g/10 min)

Material	Condition	Average	S_r[A]	S_R[B]	I_r[C]	I_R[D]	Number of Laboratories
Polyethylene	190/2.16	0.27	0.009	0.014	0.026	0.039	8
Polyethylene	190/2.16	0.40	0.016	0.027	0.045	0.076	8
Polyethylene	190/2.16	2.04	0.040	0.094	0.112	0.266	9
Polyethylene	190/2.16	43.7	0.997	1.924	2.819	5.443	8
Polypropylene	230/2.16	2.25	0.052	0.214	0.1466	0.604	8
Polypropylene	230/2.16	7.16	0.143	0.589	0.4051	1.666	8
Polypropylene	230/2.16	32.6	0.693	0.945	1.959	2.672	8
Polystyrene	200/5	1.65	0.037	0.166	0.106	0.470	4
Polystyrene	200/5	8.39	0.144	0.423	0.406	1.197	4
Polystyrene	200/5	13.0	0.108	0.387	0.306	1.097	4

[A] S_r = within-laboratory standard deviation of the average.
[B] S_R = between-laboratories standard deviation of the average.
[C] $I_r = 2.83\ S_r$, and
[D] $I_R = 2.83\ S_R$.

TABLE 6 Precision, Procedure A (Values in g/10 min)

Material	Condition	Average	S_r[A]	S_R[B]	r[C]	R[D]	Number of Labs
PMMA	230/3.8	1.51	0.013	0.086	0.037	0.242	7
LDPE	190/2.16	1.74	0.027	0.052	0.075	0.144	11
Polystyrene	200/5.0	1.86	0.040	0.085	0.112	0.238	9
HDPE	190/2.16	5.35	0.049	0.103	0.137	0.288	11
Polypropylene	230/2.16	10.94	0.088	0.473	0.247	1.324	10
Polycarbonate	300/1.2	13.59	0.109	0.233	0.305	0.653	4[E]
Acetal	190/2.16	25.30	0.235	0.571	0.658	1.599	7

[A] S_r = within-laboratory standard deviation of the average.
[B] S_R = between-laboratories standard deviation of the average.
[C] $r = 2.83\ S_r$.
[D] $R = 2.83\ S_R$.
[E] Insufficient laboratories to meet Practice E 691.

TABLE 7 Precision, Procedure B (Values in g/10 min)

Material	Condition	Average	S_r[A]	S_R[B]	r[C]	R[D]	Number of Labs
PMMA	230/3.8	1.54	0.026	0.102	0.072	0.286	6
LDPE	190/2.16	1.76	0.015	0.053	0.042	0.149	11
Polystyrene	200/5.0	1.89	0.042	0.102	0.117	0.285	7
HDPE	190/2.16	5.41	0.041	0.113	0.114	0.316	10
Polypropylene	230/2.16	10.96	0.357	0.491	0.999	1.376	10
Polycarbonate	300/1.2	13.79	0.104	0.477	0.292	1.335	3[E]
Acetal	190/2.16	25.64	0.182	0.822	0.508	2.302	6

[A] S_r = within-laboratory standard deviation of the average.
[B] S_R = between-laboratories standard deviation of the average.
[C] $r = 2.83\ S_r$.
[D] $R = 2.83\ S_R$.
[E] Insufficient laboratories to meet Practice E 691.

TABLE 8 Precision, Procedure B (Values in g/10 min)

Material	Condition	Average	S_r[A]	S_R[B]	I_r[C]	I_R[D]
Polypropylene	230/2.16	245	13.2	16.6	37.4	46.9
Polypropylene	230/2.16	482	31.8	40.0	89.9	113
Polypropylene	230/2.16	837	20.9	58.6	59.1	166
Polypropylene	230/2.16	1603	129	243	365	688

[A] S_r = within-laboratory standard deviation of the average.
[B] S_R = between-laboratories standard deviation of the average.
[C] $I_r = 2.83\ S_r$, and
[D] $I_R = 2.83\ S_R$.

rates from 250 to 1500 were tested in nine laboratories. **Warning—**The following explanations of I_r and I_R (14.1.4-14.1.6) are only intended to present a meaningful way of considering the approximate precision of this test method. The data in Tables 4-8 should not be vigorously applied to acceptance or rejection of material since those data are specific to the round robin and may not be representative of other lots, conditions, materials or laboratories. Users of this test method should apply the principles outlined in Practice E 691 to generate data specific to their laboratory and materials. The principles of 14.1.4-14.1.7 would then be valid for such data.

14.1.3 Table 9 is based on a round robin conducted in 1999 on Procedure C. Data for seven of the eight participating

TABLE 9 Precision Data for High Melt Flow Polyolefins Procedure C (Values in g/10 min)

Materials[A]	Average	S_r	S_R	r	R
PE-A (35)	4.67	0.068	0.119	0.191	0.334
PE-B (185)	24.30	0.688	1.168	1.928	3.270
PE-C (2350)	315.7	10.81	19.89	30.27	55.69
PE-D (122)	16.20	0.188	0.348	0.526	0.975

[A] Numbers in parentheses are approximate melt flow rate values of materials using standard die (5.3).

laboratories were included in the statistics for this table. Four polyethylene materials were tested with melt flow rates using the standard die ranging from approximately 35 to 2350 g/10 min using the half-height, half-diameter die.

14.1.4 *Concept of* I_r *and* I_R—Relevant if S_r and S_R have been calculated from a large enough body of data, and if test results are averages obtained from testing two specimens.

14.1.5 *Repeatability,* I_r—In comparing two test results for the same material, obtained by the same operator using the same equipment on the same day, the two test results should be judged not equivalent if they differ by more than the I_r value for that material.

14.1.6 *Reproducibility,* I_R— In comparing two test results for the same material, obtained by different operators using different equipment on different days, the two test results should be judged not equivalent if they differ by more than the I_R value for that material.

14.1.7 Any judgment in accordance with 14.1.4 and 14.1.6 would have an approximate 95 % (0.95) probability of being correct.

14.2 *Bias*—There are no recognized standards by which to estimate bias of this test method.

15. Keywords

15.1 melt flow rate; melt index; volume flow rate

APPENDIXES

(Nonmandatory Information)

X1. EXTRUSION PLASTOMETER BORE ALIGNMENT

X1.1 A fixture consisting of a circular level mounted on a shaft having two bearing points 9.47 + 0.00 − 0.0076 mm in diameter that can be inserted into the bore has been found suitable. A circular level that can be rigidly mounted on the piston rod for insertion into the bore may also be satisfactory. A circular level having a sensitivity of 20 min/2.5 mm has been found satisfactory. Other alignment techniques that give comparable alignment sensitivity would be considered satisfactory.

X2. TROUBLESHOOTING GUIDE

INTRODUCTION

Appendix X2 is offered in an effort to help a laboratory improve melt flow rate testing and to get to the root cause of problems which may be caused by equipment, environment, or testing technique. This guide is not meant to be an all-inclusive trouble-shooting check list, but merely tries to help users to evaluate testing to some degree.

X2.1 Basic Programs

X2.1.1 The following are basic programs in which all laboratories should participate:

X2.1.1.1 *Standard Reference Materials*—If available, SRMs can usually be obtained from NIST. These SRMs provide accurate information on the melt flow rate of these materials. However, these SRMs are expensive and are available for a very limited number of materials.

X2.1.1.2 *Internal Controls*—An internal control for each type of material should be set up. This involves setting aside enough material to last a long time (at least one year). These materials will have to be tested many times to establish statistical parameters. Each time an internal standard is run, the results should be plotted on a SQC chart so that any problems or trends can be detected quickly. A SQC chart should be set up for each extrusion plastometer in the laboratory. A replacement standard should be introduced before the old standard runs out and should be compared to the old standard to ensure that any shifts seen in the SQC chart are due to the material and not to the equipment.

X2.1.1.3 *Proficiency Tests*—Participation in proficiency test programs is important for demonstrating how the laboratory's results compare with other laboratories. Over a period of time, laboratory bias can be demonstrated, if bias exists.

X2.1.1.4 *ASTM Round Robins (Interlaboratory Tests)*—These programs are similar to proficiency test programs but are limited to only a few laboratories and a few materials. However, these programs do provide information on how well the laboratory performs the test.

X2.1.1.5 *Calibration, Verification, and Maintenance*—The extrusion plastometer should be calibrated. Proper maintenance of the instrument will help to ensure proper calibration.

(*1*) As a first step to obtaining reproducible results, operators should be well trained. Using internal standards can demonstrate that repeatable results can be provided. The operators should also understand the test and know what can affect the results.

(*2*) Before starting the test, the following should be verified:

(*a*) The protocol is understood by the operator.

(*b*) The barrel, piston, and die orifice have been properly cleaned.

(*c*) The extrusion plastometer, including the piston and die, are at equilibrium at the proper temperature.

(*d*) The extrusion plastometer is level.

(*e*) A standard has been run and the results fall within established parameters.

(*3*) When a problem arises, the following questions should be asked:

(*a*) Did anything unusual happen?

(*b*) Does the extrudate contain air bubbles?

(*c*) Were the proper weights applied?

(*d*) Is the unit at the proper temperature?

(*e*) Was the piston stored in the barrel?

(*f*) Is the die damaged, that is, chipped?

(*g*) Is the die bore worn, that is, the diameter is larger than the maximum specified?

(*h*) Was the proper amount of material used?

(*i*) Has the balance been properly calibrated?

(*j*) Was the proper purge time used?

(*k*) Was the plug pulled at the proper time?

(*l*) Was the correct warm-up time used?

(*m*) Was the barrel cleaned properly?

(*n*) Is the piston rod straight?

(*o*) Is the piston tip diameter OK?

X2.2 Understanding How Melt Flow Rate is Affected

X2.2.1 *Levelness of the Instrument*—The piston must be free to move in a vertical position. If the instrument is not level, the piston can be slowed by friction as it touches the side of the barrel. This will not only introduce an error into the results but may also scratch the barrel. The piston must move in an exact vertical plane, indicated by a small bubble level that should be placed on the top of the barrel or on the top of the piston when placed in the barrel. The level should be checked on a regular schedule.

X2.2.2 *Die Orifice Diameter*—An undersized die orifice (which can result from a buildup of residue) will cause low results. Conversely, an oversized orifice (which can result from wear) will cause high results. It is important that the die orifice be cleaned after each test and that the die orifice diameter be verified frequently using a calibrated go/no-go gage. Remember, the calibrated pin gage can also wear and the diameter should be verified regularly.

X2.2.3 *Die Cleanliness*—The die should be completely cleaned after each test. Any residue left in the orifice will eventually char and be very difficult, if not impossible, to clean. Buildup of material will reduce the diameter of the die and change the surface smoothness, resulting in erroneous results.

X2.2.4 *Temperature in the Barrel*—Melt flow rates are very dependent on temperature. The temperature within the barrel is the only important temperature. Temperature indicators must be calibrated to the temperature within the barrel. High flow rates will result from high temperatures and low flow rates from low temperatures. Defective heaters may be difficult to detect and can cause variable results.

X2.2.5 *Preheat Time*—Proper preheat time is required to allow the material in the barrel to fully melt and to come to temperature equilibrium throughout the barrel. If material is not fully melted, die plugging and low melt flow rates can result. If not at temperature equilibrium, the melt flow rate will change as the test is conducted.

X2.2.5.1 It is important that the die and piston be at temperature equilibrium with the rest of the system before any sample is introduced into the barrel. If the piston and die are not at temperature, the large mass of metal involved must be heated to temperature during testing and the energy required may not allow the polymer to reach thermal equilibrium. This can result in erroneous data.

X2.2.6 *Barrel Condition*—The barrel should be properly cleaned after each test. Failure to do so can result in contamination of the next sample, and buildup and degradation of material in the barrel, resulting in a decrease in the diameter of the barrel. This can cause friction with the piston tip resulting in low melt flow rates.

X2.2.6.1 Frequently overlooked is the condition of the barrel itself. In addition to being clean, the barrel wall must be smooth and of the proper diameter. The inside diameter of the barrel is as important as the diameter of the die orifice. The barrel diameter should be measured regularly, and changed if so indicated. The melt flow rate is a function of the fourth power of the barrel diameter.

X2.2.7 *Piston Parameters*—The diameter of the piston foot should be checked. If it is worn, material can flow back past the tip. This would result in erroneous data.

X2.2.7.1 The piston tip, or foot, is sometimes screwed into the end of the piston and can be changed easily. However, because it is easily unscrewed, it can work itself loose. The piston tip must be checked frequently and kept tightly screwed into the piston.

X2.2.7.2 Care must be taken not to bend a piston. Even a slight, almost non-detectable curvature in the piston can result in the force not being applied directly to the vertical position, resulting in excessive pressure on the wall of the barrel. This will cause low results and can also scratch the barrel wall.

X2.2.7.3 The piston has two reference marks. The lower scribe mark on the piston must be at the reference start position (top of the guide ring or barrel) at 7.0 ± 0.5 min, as stated in 9.8 and 10.2.10. Starting each test at a different position can give variable results.

X2.2.8 *Sample Mass*—Small variations in sample mass can cause significant variability in melt flow rates. Any balance used to weigh the sample should be calibrated and verified on a regular schedule.

X2.2.9 *Moisture in Sample*—Samples should be dried before testing. Some materials may be affected more than others by moisture. However, the presence of moisture during the test, in general, will affect the melt flow rates of most materials.

X2.2.10 *Sample Purge Time*—Purging material from the barrel before the actual test starts serves two primary functions: (*1*) to expel entrapped air or volatiles before applying the full test load, and (*2*) to move the lower scribe mark on the piston to the reference start position. For flow rate consistency, it is important that the extrudate be free of voids and that the test always starts with the piston in the same position in the barrel. Whether in the extrudate for Procedure A or in the barrel for Procedure B, voids will affect test results.

X2.2.11 *Load Weight*—The actual weight (load) applied to the material during the test will affect the test results. Higher weights will produce higher results. The load weight, which includes the piston weight, should be verified at regular intervals.

X2.2.12 *Extrudate Cut Technique*—It is important that good extrudate cutting technique be developed. A sharp, clean tool should always be used for this operation. The timing of the cut is critical since a shorter than target cut time will produce low results and a longer than target cut time will give high results. The timing intervals should be verified with certified timers. When cut, the extrudate end should not be ragged or stringy. These variables, if not controlled, can cause poor reproducibility of test results.

X2.2.13 *Purging*—Purging the barrel between runs or when changing materials is sometimes necessary. Purging with the same material which will be tested after the purge is best. However, if for some reason another purge material is used, run the test material through the barrel before the actual test run in order to ensure that the purge material is no longer in the barrel.

X2.2.13.1 Purging does not replace cleaning. After purging, the equipment must be cleaned properly to avoid the effects of contamination, resin buildup, and so forth, as discussed previously.

X2.2.14 *Melt Density*—It is important that the correct melt density be used, that is, 0.7636 g/cm^3 for PE at 190°C and 0.7386 g/cm^3 for PP at 230°C. These values may be different for copolymers or when additives are incorporated into the resin. Small errors in these values can affect the end results. If not known, the melt density can be determined as described at the end of Footnote A of Table 2.

X2.2.15 *Piston (Flag) Travel Distance*—The setting of the proper piston travel distance (6.35 ± 0.25 mm for MFRs up to 10 g/10 min and 25.40 ± 0.25 mm for MFRs greater than 10 g/10 min) is important in Procedure B. The reproducibility of Procedure B is better if these parameters are strictly adhered to. A calibrated distance verification device will be required to maintain the proper piston travel.

X2.2.16 *Calculation Factors*—When trouble shooting, always check the calculations and the factors used.

X2.2.17 *Power (Electrical) Fluctuation*—Constant power (voltage) is important to maintain the temperature desired. Periodic changes in voltage will cause changes in the temperature of the unit, creating test values that fluctuate because of inconsistent sample temperatures. Though this is a rare situation, this fluctuation has been found to cause erratic test results.

REFERENCES

(**1**) "Polyethylene Insulation and Sheathing for Electrical Cables," *Government Department Electrical Specification No. 27*, Great Britain, 1950.

(**2**) Tordella, J. P., and Jolly, R. E., "Melt Flow of Polyethylene," *Modern Plastics*, Vol 31, No. 2, 1953, p. 146.

(**3**) Dexter, F. D., "Plasticity Grading of Fluorothenes," *Modern Plastics*, Vol 30, No. 8, 1953, p. 125.

(**4**) Harban, A. A., and McClamery, R. M., "Limitations on Measuring Melt Flow Rates of Polyethylene and Ethylene Copolymers by Extrusion Plastometer," *Materials Research and Standards*, Vol 3, No. 11, 1963, p. 906.

(**5**) Rudin, A., and Schreiber, H. P., "Factors in Melt Indexing of Polyolefins," *SPE Journal*, Vol 20, No. 6, 1964, p. 533.

SUMMARY OF CHANGES

This section identifies the location of selected changes to this test method. For the convenience of the user, Committee D20 has highlighted those changes that may impact the use of this test method. This section may also include descriptions of the changes or reasons for the changes, or both.

D 1238 – 04:

(*1*) Editorially added sulfone plastic melt flow rate conditions as found in Specification D 6394 for Sulfone Plastics (SP).

(*2*) Additional melt flow rate conditions for polyether sulfone and polyphenylsulfone placed in 8.2.

D 1238 – 01:

(*1*) Procedure C for high flow polyolefins including precision data for the procedure, as well as Table 9 in Section 14 was added.

D 1238 – 00:

(*1*) Added Appendix X2, Troubleshooting Guide.

D 1238-99:

(*1*) Revised title to include the word "melt."

(*2*) Added conditions for polycaprolactone to 8.2 and Table 1.

(*3*) In Note 21, replaced "melt indexer" with "extrusion plastometer."

(*4*) In Note 25, replaced incorrect reference to 10.2.9 with 10.2.8.

(*5*) Editorially corrected equation in Footnote A of Table 2.

D 1238-98:

(*1*) Revision to Section 14.

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Designation: D 2240 – 97$^{\epsilon 1}$

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Standard Test Method for
Rubber Property—Durometer Hardness[1]

This standard is issued under the fixed designation D 2240; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

ϵ^1 Note—A footnote was removed from Note 5 in February 1999.

1. Scope

1.1 This test method covers seven types of durometers A, B, C, D, DO, O and OO, and the procedure for determining indentation hardness of substances classified as rubber, cellular materials, elastomeric materials, thermoplastic elastomers and some hard plastics.

1.2 This test method is not applicable to the testing of fabrics.

1.3 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 618 Practice for Conditioning Plastics for Testing[2]

D 785 Test Method for Rockwell Hardness of Plastics and Electrical Insulating Materials[2]

D 1349 Practice for Rubber—Standard Temperatures For Testing[3]

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries[3]

3. Summary of Test Method

3.1 This test method permits hardness measurements based on either initial indentation or indentation after a specified period of time, or both.

Note 1—Durometers with maximum reading pointers used to determine initial hardness values may yield lower hardness when the maximum pointer is used.

4. Significance and Use

4.1 This test method is based on the penetration of a specific type of indentor when forced into the material under specified conditions. The indentation hardness is inversely related to the penetration and is dependent on the elastic modulus and viscoelastic behavior of the material. The shape of the indentor and the applied force influence the results obtained so there may be no simple relationship between the results obtained with one type of durometer and those obtained with another type of durometer or other instruments for measuring hardness. This test method is an empirical test intended primarily for control purposes. No simple relationship is known to exist between indentation hardness determined by this test method and any fundamental property of the material tested. For specification purposes it is recommended that Test Method D 785 be used for hard material.

Note 2—Durometer scale comparison chart only. This is not and cannot be used as a conversion reference.

Type A	10 20 30 40 50 60 70 80 90 100
Type B	10 20 30 40 50 60 70 80 90 100
Type C	10 20 30 40 50 60 70 80 90 100
Type D	10 20 30 40 50 60 60 70 80 90 100
Type DO	10 20 30 40 50 60 70 80 90 100
Type O	10 20 30 40 50 60 70 80 90 100
Type OO	10 20 30 40 50 60 70 80 90 100

5. Apparatus

5.1 Hardness measuring apparatus or durometer consisting of the following components:

5.1.1 *Presser Foot*, with a hole having a diameter as specified in Fig. 1(*a*), (*b*), or (*c*) with its center at least 6 mm (0.25 in.) from any edge of the foot.

5.1.2 *Indentor*, formed from hardened steel rod and shaped in accordance with Fig. 1(*a*), (*b*), or (*c*) with full extension adjustable between 2.46 to 2.54 mm (0.97 to 0.100 in.).

5.1.3 *Indentor Extension Indicating Device* (analog or electronic), having a scale reading from 0 to 100 with equal divisions throughout the range. The scale reading is an inverse function of the indentor extension. The device shall have a pointer that moves on the scale at a rate of one hardness point for each 0.025 mm (0.001 in.) of indentor movement.

Note 3—Type A Shore Durometers serial numbers 1 through 16 300 and 16 351 through 16 900 and Type A-2 Shore Durometers numbers 1 through 8077 do not meet the requirement of 2.46 to 2.54 mm (0.097 to 0.100 in.) extension of the indentor at zero reading. These durometers will give readings which are low by amounts ranging from 3 units at 30

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[1] This test method is under the jurisdiction of ASTM Committee D-11 on Rubber and is the direct responsibility of Subcommittee D11.10 on Physical Testing.

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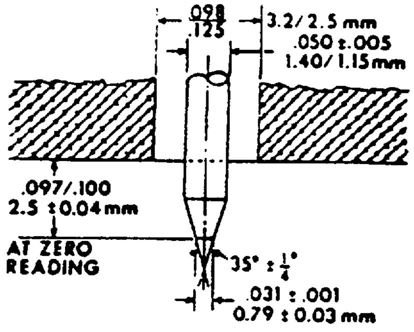
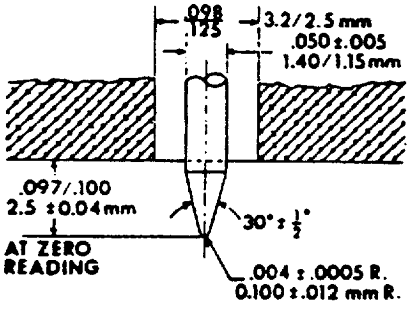
[2] *Annual Book of ASTM Standards*, Vol 08.01.

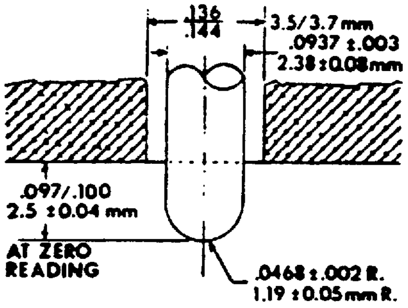
[3] *Annual Book of ASTM Standards*, Vol 09.01.



(*a*) Indentor for Type A and C Durometers



(*b*) Indentor for Type B and D Durometers



(*c*) Indentor for Type DO, O, and OO Durometers

NOTE 1—Spring Force Combinations:

$$\text{Force, N} = 0.550 + 0.075\ H_x$$

where H_x = hardness reading on Type A, B and O durometers.

$$\text{Force, N} = 0.4445\ H_y$$

where H_y = hardness reading on Type C, D and DO durometers.

$$\text{Force, N} = 0.203 + 0.00908\ H_{oo}$$

where H_{oo} = hardness reading on Type OO durometers.

FIG. 1 Durometer, Indentor and Spring Force Combinations

hardness to 1 unit at 90 hardness.

5.1.4 *Timing Device* (optional), capable of being set to a desired elapsed time, signalling the operator or holding the hardness reading when the desired elapsed time has been reached. The timer should be automatically activated when the presser foot is in firm contact with the specimen being tested.

5.1.5 *Calibrated Spring*, for applying force to the indentor in accordance with Fig. 1.

6. Test Specimen

6.1 The test specimen shall be at least 6 mm (0.25 in.) in thickness unless it is known that results equivalent to the 6 mm values are obtained with a thinner specimen (see Note 4). A specimen may be composed of plied pieces to obtain the necessary thickness, but determinations made on such specimens may not agree with those made on solid specimens because the surface faces between plies may not be in complete contact. The lateral dimensions of the specimen shall be sufficient to permit measurements at least 12 mm (0.5 in.) from any edge unless it is known that identical results are obtained when measurements are made at lesser distance from an edge. The surfaces of the specimen shall be flat and parallel over a sufficient area to permit the presser foot to contact the specimen over an area having a radius of at least 6 mm (0.25 in.) from the indentor point. A suitable hardness determination cannot be made on a rounded, uneven, or rough surface.

NOTE 4—The minimum requirement for the thickness of the specimen is dependent on the extent of penetration of the indentor into the specimen; that is, thinner specimens may be used for materials having hardness values at the upper end of the scale. The minimum distance from the edge at which measurements may be made likewise decreases as the hardness increases. For materials having hardness values above 50 Type D durometer, the thickness of the specimen should be at least 3 mm (0.12 in.) and measurements should not be made closer than 6 mm (0.25 in.) to any edge.

7. Calibration

7.1 The durometer spring shall be calibrated by supporting the durometer in a vertical position and applying a measurable force to the indentor tip (see Fig. 2). The device used to apply the force may be a dead weight or electronic load cell device capable of measuring applied force at 50 % of the calibration tolerance. Care should be taken to ensure that the force is applied vertically to the indentor tip, as side loads will cause errors in calibration. Spring calibration shall be verified on all durometer at scale readings of 20, 30, 40, 50, 60, 70, 80 and 90. The measured force (9.8 × mass in kilograms) shall be equivalent to the force calculated by the equation in Fig. 1. The measured force for Type A, B and O durometers shall be within ±0.08 N. For Type C, D and DO durometers it shall be within ±0.44 N, and for Type OO durometers it shall be within ±0.025 N.

NOTE 5—Instruments specifically designed for the calibration of durometers may be used.

7.2 Indentor extension and shape must be in accordance with 5.1.2. With the durometer placed firmly on a flat surface the indicator should read a number equal to the indentor extension measured in inches × 1000, within ±0.5 durometer points.

NOTE 6—When performing the procedure in 7.2 on Type B and D durometers care should be used not to damage the indentor tip.

7.3 Test blocks (rubber or spring type) provided for checking durometer operation are not to be relied upon as calibration standards. The calibration procedures outlined in 7.1 and 7.2 are the only valid calibration methods.

8. Conditioning

8.1 Tests shall be made at 23 ± 2°C (73.4± 3.6°F). For materials whose hardness depends on relative humidity, the specimen shall be conditioned in accordance with Procedure A of Practice D 618 and tested under the same conditions.

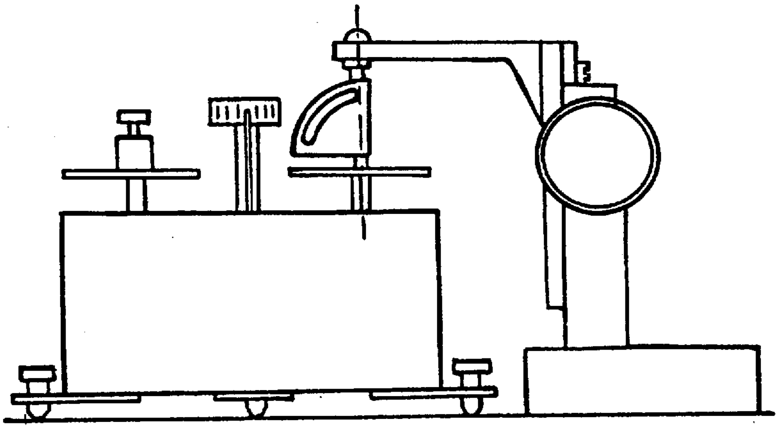


FIG. 2 Apparatus for Calibration of Durometer Spring

NOTE 7—No conclusive evaluation has been made on durometers at temperatures other than 23 ± 2°C (73.4 ± 3.6°F). Conditioning at temperatures other than the above may show changes in calibration. Durometer use at temperatures other than the above should be decided locally (see Practice D 1349).

9. Procedure

9.1 Place the specimen on a hard, horizontal surface. Hold the durometer in a vertical position with the point of the indentor at least 12 mm (0.5 in.) from any edge of the specimen, unless it is known that identical results are obtained when measurements are made with the indentor at a lesser distance. Apply the presser foot to the specimen as rapidly as possible, without shock, keeping the foot parallel to the surface of the specimen. Apply just sufficient pressure to obtain firm contact between presser foot and specimen.

NOTE 8—Better repeatability may be obtained by using a mass centered on the axis of the indentor. Recommended masses are 1 kg for Type A, B and O durometers, 5 kg for Type C, D and DO durometers, and 400 g for Type OO durometers. Durometer stands using the masses above as a constant load and a controlled descent speed, without shock, produce maximum repeatability.

9.2 For any material covered in 1.1, after the presser foot is in firm contact with the specimen, the scale reading is to be taken within 1 s or after any period of time agreed upon between supplier and user unless the durometer has a maximum indicator, in which case the maximum reading is taken. The hardness reading may progressively decrease with time delay.

9.3 Make one measurement at each of three or five different points distributed over the specimen at least 6 mm (0.25 in.) apart using the median of these measurements for the hardness value.

NOTE 9—The type of durometer should be selected with the knowledge that readings below 10 or above 90 are not considered reliable by the manufacturer. It is suggested that readings in these ranges not be recorded.

10. Report

10.1 Report the following information:

10.1.1 Hardness value obtained,

10.1.2 Complete identification of the material tested,

10.1.3 Vulcanization date,

10.1.4 Description of specimen, including thickness and number of pieces plied, if less than 6 mm (0.25 in.),

10.1.5 Temperature of test if other than 23°C,

10.1.6 Relative humidity when hardness of material is dependent on humidity,

10.1.7 Type and serial number of durometer,

10.1.8 Indentation hardness time interval at which reading was taken, and

10.1.9 Date of test.

NOTE 10—Readings may be reported in the form: A/45/15 where A is the type of durometer, 45 the reading, and 15 the time in seconds that the pressure foot is in firm contact with the specimen. Similarly, D/60/1 indicates a reading of 60 on the Type D durometer obtained either within 1 s or from a maximum indicator.

11. Precision and Bias [4]

11.1 These precision and bias statements have been prepared in accordance with Practice D 4483. Refer to this Practice for terminology and other testing and statistical concepts.

11.2 The Type 1 precision for both Type A and D methods was determined from an interlaboratory program with three materials of varying hardness, with six participating laboratories. Tests were conducted on two separate days in each laboratory for both A and D testing programs. All materials were supplied from a single source.

11.3 A test result for hardness (both A and D) was the median of five individual hardness readings on each day in each laboratory.

11.4 Table 1 shows the precision results for Type A method. Table 2 gives the precision results for Type D method.

11.5 The precision results in this precision and bias section give an estimate of the precision of this test method with the materials (rubbers) used in the particular interlaboratory program as described above. The precision parameters should not be used for acceptance or rejection testing, or both, of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols that include this test method.

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[4] Supporting data are available from ASTM Headquarters. Request RR:D11-1029.

TABLE 1 Type 1 Precision—Type A Durometer Method

Material	Average Level	Within Laboratories			Between Laboratories		
		Sr[A]	*r*[B]	(*r*)[C]	*SR*[D]	*R*[E]	(*R*)[F]
1	51.4	0.646	1.83	3.56	1.56	4.41	8.59
2	65.3	0.878	2.48	3.81	2.14	6.06	9.27
3	68.0	0.433	1.23	1.80	2.28	6.45	9.49
Pooled	61.6	0.677	1.92	3.11	2.018	5.72	9.28

[A] *Sr* = repeatability standard deviation, measurement units.
[B] *r* = repeatability = 2.83 × *Sr*, measurement units.
[C] (*r*) = repeatability, relative, (that is, in percent).
[D] *SR* = reproducibility standard deviation, measurement units.
[E] *R* = reproducibility = 2.83 × *SR*, measurement units.
[F] (*R*) = reproducibility, relative, (that is, in percent).

TABLE 2 Type 1 Precision—Type D Durometer Method

Material	Average Level	Within Laboratories			Between Laboratories		
		Sr[A]	*r*[B]	(*r*)[C]	*SR*[D]	*R*[E]	(*R*)[F]
1	42.6	0.316	0.894	2.10	2.82	7.98	18.7
2	54.5	0.791	2.24	4.11	3.54	10.0	18.4
3	82.3	1.01	2.86	3.47	3.54	10.0	12.2
Pooled	59.8	0.762	2.16	3.61	3.32	9.40	15.7

[A] *Sr* = repeatability standard deviation, measurement units.
[B] *r* = repeatability = 2.83 × *Sr*, measurement units.
[C] (*r*) = repeatability, relative, (that is, in percent).
[D] *SR* = reproducibility standard deviation, measurement units.
[E] *R* = reproducibility = 2.83 × *SR*, measurement units.
[F] (*R*) = reproducibility, relative, (that is, in percent).

11.6 *Precision*—The precision of this test method may be expressed in the format of the following statements which use as appropriate value *r*, *R*, (*r*) or (*R*), that is, that value to be used in decisions about test results (obtained with the test method). The appropriate value is that value of *r* or *R* associated with a mean level in Table 1 and Table 2 closest to the mean level under consideration (at any given time, for any given material) in routine testing operations.

11.6.1 *Repeatability*—The repeatability, *r*, of this test method has been established as the appropriate value tabulated in Table 1 and Table 2. Two single test results, obtained under normal test method procedures, that differ by more than this tabulated *r* (for any given level) must be considered as derived from different or nonidentical sample populations.

11.6.2 *Reproducibility*—The reproducibility, *R*, of this test method has been established as the appropriate value tabulated in Table 1 and Table 2. Two single test results obtained in two different laboratories, under normal test method procedures, that differ by more than the tabulated *R* (for any given level) must be considered to have come from different or nonidentical sample populations.

11.6.3 Repeatability and reproducibility expressed as a percentage of the mean level, (*r*) and (*R*), have equivalent application statements as above for *r* and *R*. For the (*r*) and (*R*) statements, the difference in the two single test results is expressed as a percentage of the arithmetic mean of the two test results.

11.7 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (or true) test property value. Reference values do not exist for this test method since the value (of the test property) is exclusively defined by this test method. Bias, therefore, cannot be determined.

12. Keywords

12.1 durometer hardness

Designation: D 6110 – 04

Standard Test Method for Determining the Charpy Impact Resistance of Notched Specimens of Plastics[1]

This standard is issued under the fixed designation D 6110; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This test method is used to determine the resistance of plastics to breakage by flexural shock as indicated by the energy extracted from standardized (see Note 1) pendulum-type hammers, mounted in standardized machines, in breaking standard specimens with one pendulum swing. This test method requires specimens to be made with a milled notch (see Note 2). The notch produces a stress concentration which promotes a brittle, rather than a ductile, fracture. The results of this test method are reported in terms of energy absorbed per unit of specimen width (see Note 3).

NOTE 1—The machines with pendulum-type hammers have been standardized in that they must comply with certain requirements including a fixed height of hammer fall, which results in a substantially fixed velocity of the hammer at the moment of impact. Hammers of different initial energies (produced by varying their effective weights), however, are recommended for use with specimens of different impact resistance. Moreover, manufacturers of the equipment are permitted to use different lengths and constructions of pendulums with possible differences in pendulum rigidities resulting (see Section 5). Be aware that other differences in machine design do exist.

NOTE 2—The specimens are standardized in that they have a fixed length and fixed depth, however, the width of the specimens is permitted to vary between limits. One design of milled notch is allowed. The notch in the specimen serves to concentrate the stress, minimize plastic deformation, and direct the fracture to the part of the specimen behind the notch. Scatter in energy-to-break is thus reduced. Because of differences in the elastic and viscoelastic properties of plastics, however, response to a given notch varies among materials.

NOTE 3—Caution must be exercised in interpreting the results of this test method. The following testing parameters have been shown to affect test results significantly: method of specimen fabrication, including but not limited to processing technology, molding conditions, mold design, and thermal treatment; method of notching; speed of notching tool; design of notching apparatus; quality of the notch; time between notching and test; test specimen thickness; test specimen width under notch; and environmental conditioning.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

NOTE 4—This standard resembles ISO 179 in title only. The content is significantly different.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 618 Practice for Conditioning Plastics for Testing
D 647 Practice for Design of Molds for Test Specimens of Plastic Molding Materials[3]
D 883 Terminology Relating to Plastics
D 4000 Classification System for Specifying Plastic Materials
D 4066 Classification System for Nylon Injection and Extrusion Materials
D 5942 Test Method for Determination of Charpy Impact Strength
D 5947 Test Methods for Physical Dimensions of Solid Plastics Specimens
E 691 Practice for Conducting an Interlaboratory Test Program to Determine the Precision of Test Methods

3. Terminology

3.1 *Definitions*—For definitions related to plastics, see Terminology D 883.

4. Summary of Test Method

4.1 A notched specimen is supported as a horizontal simple beam and is broken by a single swing of the pendulum with the impact line midway between the supports and directly opposite the notch.

5. Significance and Use

5.1 Before proceeding with this test method, refer to the material specification for the material being tested. Any test specimen preparation, conditioning, dimensions and testing parameters required by the materials specification shall take

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[1] This test method are under the jurisdiction of ASTM Committee D20 on Plastics and are the direct responsibility of Subcommittee D20.10 on Mechanical Properties.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] *Discontinued 1994; Replaced by D 1896, D 3419, D 3641, D 4703, D 5227.*

***A Summary of Changes section appears at the end of this standard.**

precedence over those required by this test method. Table 1 of Classification D 4000 lists the ASTM materials standards that currently exist. If there is no material specification, then the requirements of this test method apply.

5.2 The excess energy pendulum impact test indicates the energy to break standard test specimens of specified size under stipulated conditions of specimen mounting, notching (stress concentration), and pendulum velocity at impact.

5.3 For this test method, the energy lost by the pendulum during the breakage of the specimen is the sum of the energies required to initiate fracture of the specimen; to propagate the fracture across the specimen; to throw the free ends of the broken specimen (toss energy); to bend the specimen; to produce vibration in the pendulum arm; to produce vibration or horizontal movement of the machine frame or base; to overcome friction in the pendulum bearing and in the excess energy indicating mechanism, and to overcome windage (pendulum air drag); to indent or deform, plastically, the specimen at the line of impact; and to overcome the friction caused by the rubbing of the striking nose over the face of the bent specimen.

NOTE 5—The toss energy, or the energy used to throw the free ends of the broken specimen, is suspected to represent a very large fraction of the total energy absorbed when testing relatively dense and brittle materials. No procedure has been established for estimating the toss energy for the Charpy method.

5.4 For tough, ductile, fiber-filled, or cloth-laminated materials, the fracture propagation energy is usually large compared to the fracture initiation energy. When testing these materials, energy losses due to fracture propagation, vibration, friction between the striking nose and the specimen has the potential to become quite significant, even when the specimen is accurately machined and positioned, and the machine is in good condition with adequate capacity (see Note 6). Significant energy losses due to bending and indentation when testing soft materials have also been observed.

NOTE 6—Although the frame and the base of the machine should be sufficiently rigid and massive to handle the energies of tough specimens without motion or excessive vibration, the pendulum arm cannot be made very massive because the greater part of its mass must be concentrated near its center of percussion at its striking nose. Locating the striking nose precisely at the center of percussion reduces the vibration of the pendulum arm when used with brittle specimens. Some losses due to pendulum arm vibration (the amount varying with the design of the pendulum) will occur with tough specimens even when the striking nose is properly positioned.

5.5 In a well-designed machine of sufficient rigidity and mass the losses due to vibration and friction in the pendulum bearing and in the excess energy indicating mechanism should be very small. Vibrational losses are observed when wide specimens of tough materials are tested in machines of insufficient mass, or in machines that are not securely fastened to a heavy base.

5.6 Since this test method permits a variation in the width of the specimens and since the width dictates, for many materials, whether a brittle, low-energy break (as evidenced by little or no drawing down or necking and by a relatively low energy absorption) or a ductile, high-energy break (as evidenced by considerable drawing or necking down in the region behind the notch and by a relatively high energy absorption) will occur, it is necessary that the width be stated in the specification covering that material and that the width be stated along with the impact value.

5.7 This test method requires that the specimen break completely. Results obtained when testing materials with a pendulum that does not have sufficient energy to complete the breaking of the extreme fibers and toss the broken pieces shall be considered a departure from standard and should not be reported as a standard result. Impact values cannot be directly compared for any two materials that experience different types of failure.

5.8 The value of this impact test method lies mainly in the areas of quality control and materials specification. If two groups of specimens of supposedly the same material show significantly different energy absorptions, critical widths, or critical temperatures, it is permitted to assume that they were made of different materials or were exposed to different processing or conditioning environments. The fact that a material shows twice the energy absorption of another under these conditions of test does not indicate that this same relationship will exist under another set of test conditions.

6. Apparatus

6.1 *Pendulum Impact Machine*—The machine shall consist of a massive base on which are mounted a pair of supports for holding the specimen and to which is connected, through a rigid frame and bearings, one of a number of pendulum-type hammers having an initial energy suitable for use with the particular specimen to be tested (or one basic pendulum designed to accept add-on weights), plus a pendulum holding and releasing mechanism and a mechanism for indicating the excess energy remaining in the pendulum after breaking specimen. The specimen anvil, pendulum, and frame shall be sufficiently rigid to maintain correct alignment of the striking edge and specimen, both at the moment of impact and during the propagation of the fracture, and to minimize energy losses due to vibration. The base shall be sufficiently massive so that the impact will not cause it to move. The machine shall be designed, constructed, and maintained so that energy losses due to pendulum air drag (windage), friction in the pendulum bearings, and friction and inertia in the excess energy indicating mechanism are held to a minimum.

6.1.1 *Pendulum*—The simple pendulum shall consist of a single or multi-membered arm with a bearing on one end and a head, containing the striking nose, on the other. Although a large proportion of the mass of the pendulum should be concentrated in the head, the arm must be sufficiently rigid to maintain the proper clearances and geometric relationships between the machine parts and the specimen and to minimize vibrational energy losses, which are always included in the measured impact value. A machine with a simple pendulum design is illustrated in Fig. 1. Instruments with a compound-pendulum design also have been found to be acceptable for use. A compound-pendulum design is illustrated in Fig. 2.

6.1.1.1 The machine shall be provided with a basic pendulum capable of delivering an energy of 2.7 ± 0.14 J [2.0 ± 0.10 ft-lbf]. This pendulum shall be used for specimens that extract less than 85 % of this energy when breaking a specimen. Heavier pendulums or additional weights designed to

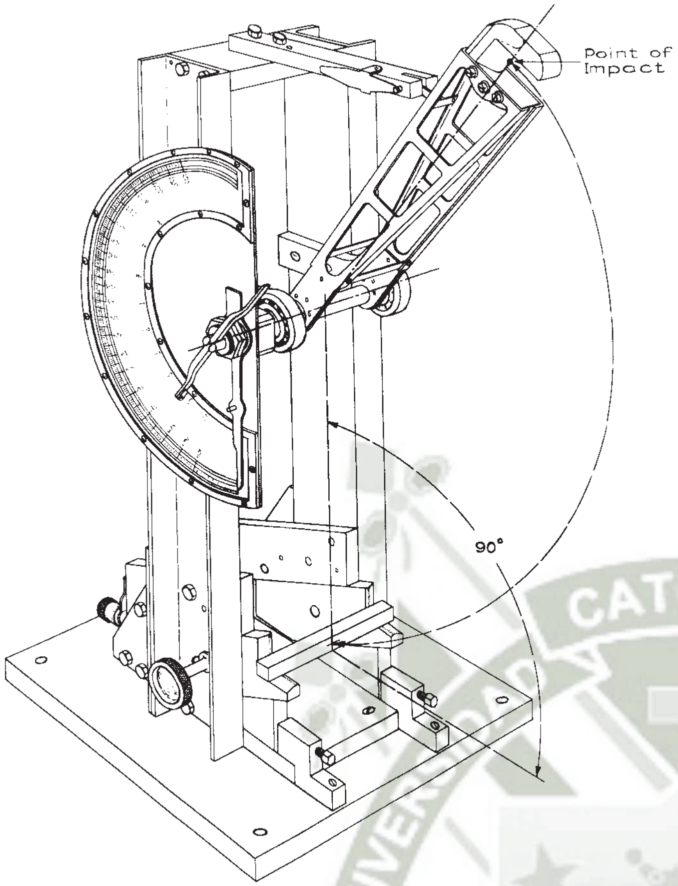

FIG. 1 Simple Beam (Charpy-Type) Impact Machine

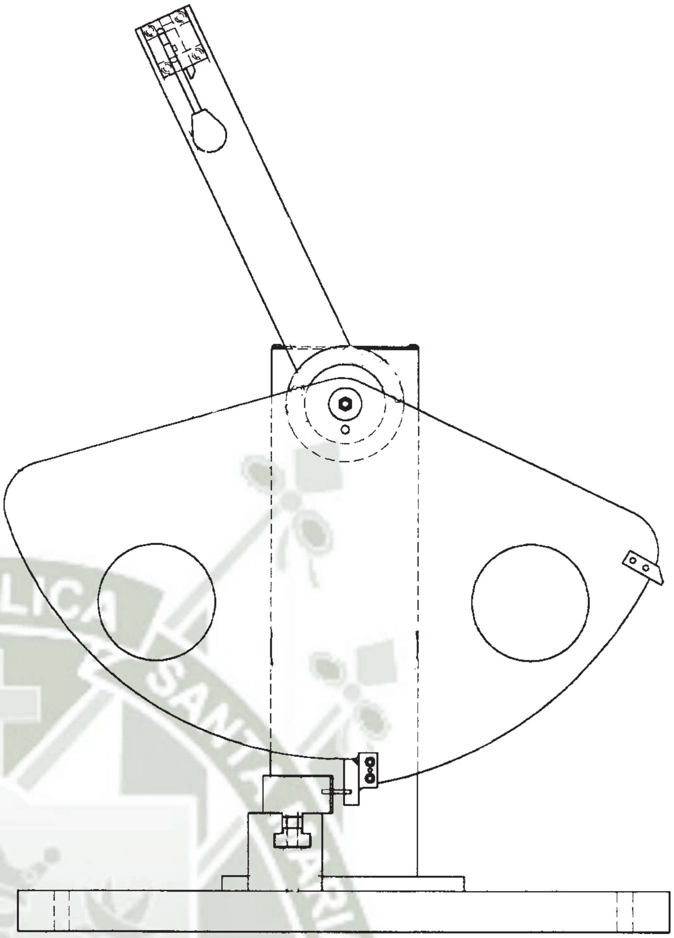

FIG. 2 Example of Compound–Pendulum–Type Machine

attach to the basic pendulum shall be provided for specimens that require more energy to break. A series of pendulums such that each has twice the energy of the next lighter one has been found convenient.

6.1.1.2 The effective length of the pendulum shall be between 0.325 and 0.406 m [12.8 an 16.0 in.] so that the required elevation of the striking nose is obtained by raising the pendulum to an angle between 60 and 30° above the horizontal.

6.1.2 *Striking Edge*—The striking edge (nose) of the pendulum shall be made of hardened steel, tapered to have an included angle of 45 ± 2° and shall be rounded to a radius of 3.17 ± 0.12 mm [0.125 ± 0.005 in.]. The pendulum shall be aligned in such a way that when it is in its free hanging position, the center of percussion of the pendulum shall lie within ±2.54 mm [0.10 in.] of the middle of the line of contact made by the striking nose upon the face of a standard specimen of square cross section. The distance from the axis of support to the center of percussion is determined experimentally from the period of motion of small amplitude oscillations of the pendulum by means of the following equation:

$$L = (g/4\pi^2)\, p^2 \qquad (1)$$

where:

L = distance from the axis of support to the center of percussion, m,

g = local gravitational acceleration (known to an accuracy of one part in one thousand), m/s^2

π = 3.1416 ($4\pi^2$= 39.48), and

p = period, in s, of a single complete swing (to and fro) determined from at least 20 consecutive and uninterrupted swings. The angle of swing shall be less than 5° each side of center.

6.1.3 *Pendulum Holding and Releasing Mechanism*—The mechanism shall be designed, constructed, and operated so that it will release the pendulum without imparting acceleration or vibration to the pendulum. The position of the pendulum holding and releasing mechanism shall be such that the vertical height of fall of the striking nose shall be 610 ± 2 mm [24.0 ± 0.005 in.]. This will produce a velocity of the striking nose at the moment of impact of approximately 3.46 m [11.4 ft]/s as determined by the following equation:

$$v = \sqrt{2gh} \qquad (2)$$

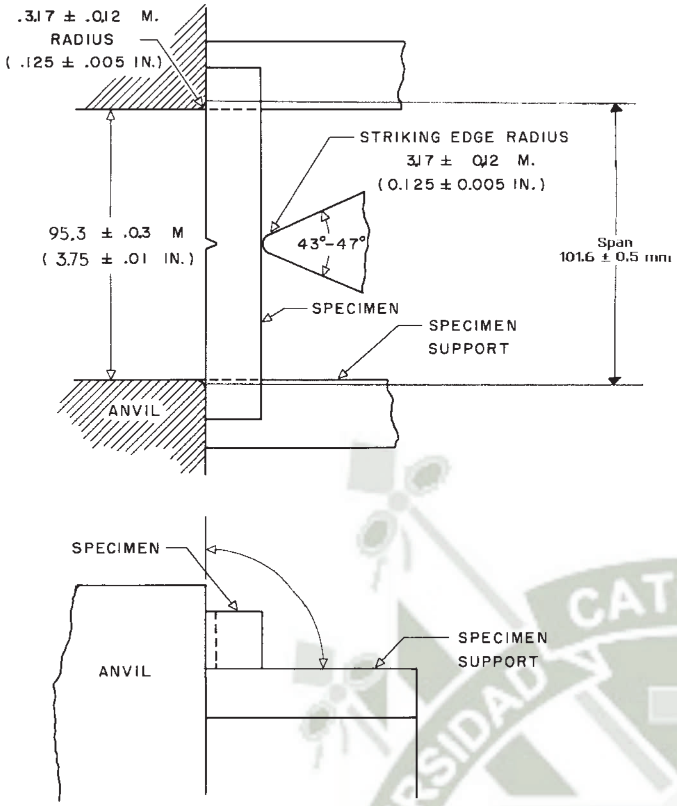

FIG. 3 Relationship of Anvil, Specimen, and Striking Edge to Each Other for Charpy Test Method

where:

v = velocity of the striking nose at the moment of impact,

g = local gravitational acceleration, and

h = vertical height of fall of the striking nose.

This assumes no windage or friction.

6.1.4 *Specimen Supports*—The test specimen shall be supported against two rigid anvils in such a position that its center of gravity and the center of the notch shall lie on tangent to the arc of travel of the center of percussion of the pendulum drawn at the position of impact. The edges of the anvils shall be rounded to a radius of 3.17 ± 0.12 mm [0.125 ± 0.005 in.] and the anvils' lines of contact (span) with the specimen shall be 101.6 ± 0.5 mm [4.0 ± 0.02 in.] apart (see Fig. 3). A jig for positioning the specimen on the supports should be supplied by the machine manufacturer.

NOTE 7—Some machines currently in use employ a 108.0–mm span. Data obtained under these conditions are valid.[4]

6.1.5 *Excess Energy Indicator*—Means shall be provided for determining energy remaining in the pendulum after breaking a specimen. One acceptable method is the use of a pointer and dial mechanism which indicate the height of rise of the pendulum beyond the point of impact in terms of energy removed from that specific pendulum. More modern instruments use an electronic digital display or computer to measure the energy loss and indicate the breaking energy of the specimen. The indicated remaining energy must be corrected for pendulum bearing friction, pointer friction, pointer inertia, and pendulum windage. The equipment manufacturer should provide graphs or tables to aid in the calculation of the correction friction and windage. Instructions for making these corrections are found in Annex A1 and Annex A2. Many digital indicating systems automatically correct for windage and friction. The equipment manufacturer should be consulted for information on how this is performed.

6.1.6 The calibration procedure in Appendix X2 should be used to establish the accuracy of the equipment. A check of the calibration of an impact machine is difficult to make under dynamic conditions. The basic parameters normally are checked under static conditions. If the machine passes the static tests, then it is assumed to be accurate. Appendix X2, however, also describes a dynamic test for checking certain features of the machine and specimen. For some machine designs, it might be necessary to change the recommended method of obtaining the required calibration measurements. Additional instructions for adjusting a particular machine should be supplied by the manufacturer. Other methods of performing the required checks are acceptable provided that they are proven to result in an equivalent accuracy.

6.2 *Specimen Notching Machine*—Notching shall be done on a milling machine, engine lathe, or other suitable machine tool. A carbide-tipped or industrial diamond-tipped notching cutter is recommended. Both cutter speed and feed rate shall be controllable. Provision for cooling the specimen is recommended. Water and compressed air are suitable coolants for many plastics.

6.2.1 The profile of the cutting tooth or teeth shall be such as to produce a notch in the test specimen of the contour and depth specified in Fig. 4 and in the manner specified in Section 8.

6.2.2 A single-tooth cutter shall be used for notching the specimen, unless it is demonstrated that notches of an equivalent quality are produced with a multi-tooth cutter. Single-tooth cutters are preferred because of the ease of grinding the cutter to the specimen contour and because of the smoother cut on the specimen. The cutting edge shall be ground and honed carefully to ensure sharpness and freedom from nicks and burrs. Tools with no rake and a work relief angle of 15 to 20° have been found satisfactory.

6.3 *Micrometers*—Apparatus for measurement of the width of the specimen shall comply with the requirements of Test Methods D 5947. Apparatus for the measurement of the depth of plastic material remaining in the specimen under the notch shall comply with requirements of Test Methods D 5947, provided however that the one anvil or presser foot shall be a tapered blade conforming to the dimensions given in Fig. 5. The opposing anvil or presser foot shall be flat and conforming to Test Methods D 5947.

7. Test Specimen

7.1 The test specimen shall conform to the dimensions and geometry of Fig. 4, except as modified in accordance with 7.2-7.5. To ensure the correct contour and conditions of the specified notch, all specimens shall be notched in accordance with Section 8.

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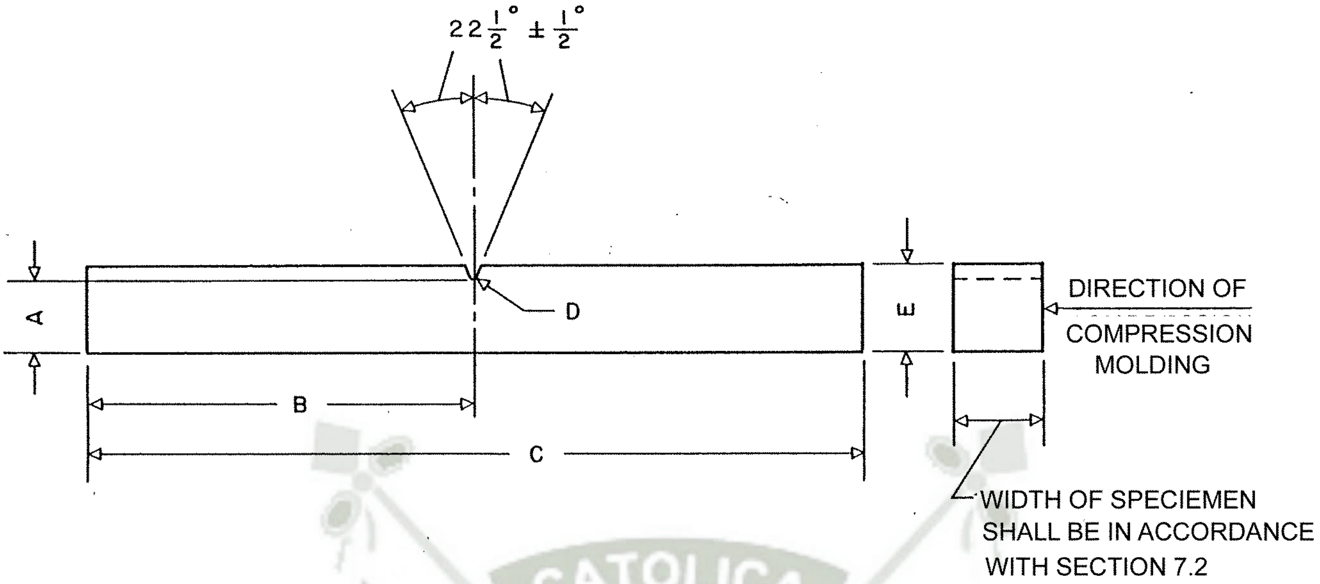
[4] Supporting data is available from ASTM Headquarters. Request Research Report RR: D20–1033.



A	10.16 ± 0.05	0.400 ± 0.002
B	63.5 max	2.50 max
	61.0 min	2.40 min
C	127.0 max	5.00 min
	124.5 min	4.90 min
D	0.25R ± 0.05	0.010R ± 0.002
E	12.70 ± 0.15	0.500 ± 0.006

FIG. 4 Dimensions of Simple Beam, Charpy Type, Impact Test Specimen

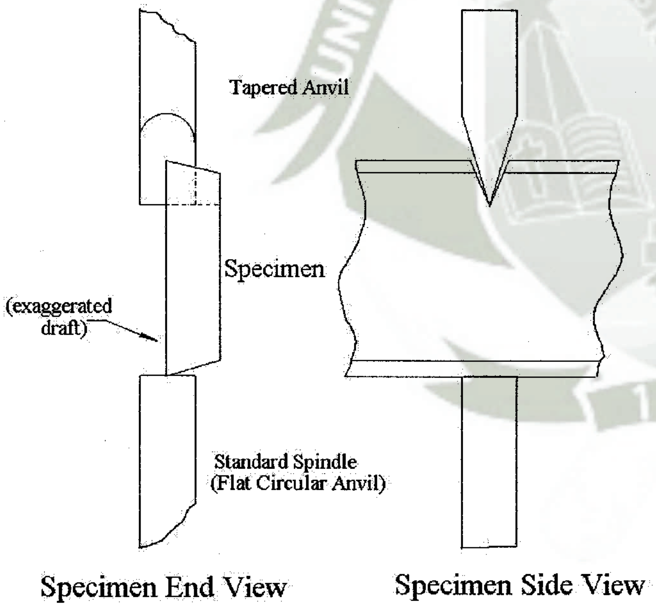


FIG. 5 Notch Depth Measurement on Test Specimens

7.2 Molded specimens shall have a width between 3.00 and 12.7 mm [0.118 and 0.500 in.]. Use the specimen width as specified in the material specification or as agreed upon between the supplier and the customer.

7.2.1 The type of mold and molding machine used and the flow behavior in the mold cavity will influence the strength obtained. It is possible that results from a specimen taken from one end of a molded bar will give different results than a specimen taken from the other end. Cooperating laboratories should therefore agree on standard molds conforming to Practice D 647, and upon a standard molding procedure for the material under investigation.

7.2.2 A critical investigation of the mechanics of impact testing has shown that tests made upon specimens under 6.35 mm [0.250 in.] in width absorb more energy due to crushing, bending, and twisting than do wider specimens. Specimens 6.35 mm [0.250 in.] or over in width are therefore recommended. The responsibility for determining the minimum specimen width shall be the investigator's, with due reference to the specification for that material.

7.2.3 The impact resistance of a plastic material will be different if the notch is perpendicular to, rather than parallel to, the direction of molding.

7.3 For sheet materials, the specimens shall be cut from the sheet in both the lengthwise and crosswise directions unless otherwise specified. The width of the specimen shall be the thickness of the sheet if the sheet thickness is between 3.00 and 12.7 mm [0.118 and 0.500 in.]. Sheet material thicker than 12.7 mm [0.500 in.] shall be machined down to 12.7 mm [0.500 in.]. It is acceptable to test specimens with a 12.7–mm [0.500–in.] square cross section either edgewise or flatwise as cut from the sheet. When specimens are tested flatwise, the notch shall be made on the machined surface if the specimen is machined on one face only. When the specimen is cut from a thick sheet, notation shall be made of the portion of the thickness of the sheet from which the specimen was cut, for example, center, top, or bottom surface.

7.3.1 The impact resistance of a plastic material will be different if the notch is perpendicular to, rather than parallel to, the grain of an anisotropic bar cut from a sheet. Specimens cut from sheets that are suspected of being anisotropic shall be prepared and tested both lengthwise and crosswise to the direction of the anisotropy.

7.4 The practice of cementing, bolting, clamping, or otherwise combining specimens of substandard width to form a composite test specimen is not recommended and should be avoided since test results will be seriously affected by interface effects or effects of solvents and cements on energy absorption of composite test specimens, or both. If Charpy test data on such thin materials are required, however, and if possible sources of error are recognized and acceptable, the following technique of preparing composites ought to be utilized. The test specimens shall be a composite of individual thin specimens totaling 6.35 to 12.7 mm [0.125 to 0.500 in.] in width. Individual members of the composite shall be aligned accurately with each other and clamped, bolted, or cemented together. Care must be taken to select a solvent or adhesive that will not affect the impact resistance of the material under test. If solvents or solvent–containing adhesives are employed, a conditioning procedure shall be established to ensure complete removal of the solvent prior to test. The composite specimens shall be machined to proper dimensions and then notched. In all such cases, the use of composite specimens shall be noted in the report of test results.

7.5 Each specimen shall be free of twist and shall be bounded by mutually perpendicular pairs of plane, paralleled surfaces and free from scratches, pits, and sink marks. The specimens shall be checked for conformity with these requirements by visual observation against straight edges, squares or flat plates, and by measuring with micrometer calipers. Any specimen showing observable or measurable departure from one or more of these requirements shall be rejected or machined to the proper size and shape before testing. A specimen that has a slight twist to its notched face of 0.05 mm [0.002 in.] at the point of contact with the pendulum striking edge will be likely to have a characteristic fracture surface with considerable greater fracture area than for a normal break. In this case, the energy to break and toss the broken section will be considerably larger (20 to 30 %) than for a normal break.

8. Notching Test Specimens

NOTE 8—When testing a material for the first time, it is necessary to study the effect of all variations in the notching conditions, including cutter dimensions, notch depth, cutter speed, and feed rate. To establish that the notching parameters are suitable, it is advisable to notch several specimens of the material and inspect both the tool entrance and tool exit side of each notched specimen, in accordance with Appendix X1. Adjust the notching machine as required. The specimens used to determine notching conditions shall not be used to make determinations of impact resistance.

8.1 *Notch Dimensions*—The included angle of the notch shall be 45 ± 1° with a radius of curvature at the apex of 0.25 ± 0.05 mm [0.010 ± 0.002 in.]. The plane bisecting the notch angle shall be perpendicular to the face of the test specimen within 2°.

8.1.1 The notch is a critical factor of this test. It is extremely important, therefore, that dimensions of the notch in the specimen are verified. There is evidence that the contour of notches cut in materials of widely differing physical properties by the same cutter will differ. It is sometimes necessary to alter the cutter dimensions in order to produce the required notch contour for certain materials.

8.1.2 Both the notch and the cutter used to make the notch shall be inspected, at a minimum, after every 500 notches. The specimen used to verify the notch shall be the same material that is being prepared for testing. Follow the procedure in Appendix X1 when inspecting and verifying the notch in the specimen. If the angle or radius of the notch does not meet the requirements of 8.1, the cutter should be replaced.

NOTE 9—The contour of the notch made using multi-tooth cutters is checked by measuring the contour of the notch on a strip of soft metal that is inserted between two specimens during the notching process.

NOTE 10—When the same material is being tested on a repetitive basis, and it is demonstrated that the notch in the specimen takes the contour of the tip of the cutter and that the notch meets the contour requirements when checked in accordance with Appendix X1, then it is acceptable to check the contour of the tip of the cutter instead of the notch in the specimen.

8.2 *Notch Depth*—The depth of the plastic material remaining in the specimen under the notch shall be 10.16 ± 0.05 mm [0.400 ± 0.002 in.]. This dimension shall be measured with apparatus in accordance with 6.3. The tapered blade will be fitted to the notch. The specimen will be approximately vertical between the anvils. Position the edge of the non-cavity (wider edge) surface centered on the micrometer's flat circular anvil.

8.3 *Cutter Speed and Feed Rate*—The cutter speed and feed speed should be selected based on the material being tested. The quality of the notch will be adversely affected by thermal deformations and stresses induced during the cutting operation if proper conditions are not selected.[5] The notching parameters used shall not alter the physical state of the material, such as by raising the temperature of a thermoplastic above its glass transition temperature.

8.3.1 In general, high cutter speeds, slow feed rates, and lack of coolant induce more thermal damage than a slow cutter speed, fast feed speed, and the use of a coolant. Too high a feed speed/cutter speed ratio, however, has been shown to cause impacting and cracking of the specimen. The range of cutter speed/feed ratios possible to produce acceptable notches has been shown to be extended by the use of a suitable coolant.

8.3.1.1 For some thermoplastics, suitable notches have been produced using cutter speeds from 54 to 150 m/min and a feed rate of 89 to 160 mm/min without a water coolant. Satisfactory notches also have been produced using the same cutter speeds at feed speeds of from 36 to 160 mm/min with water coolant.

8.3.1.2 Embedded thermocouples have been used to determine the temperature rise in the material near the apex of the notch during machining. Thermal stresses induced during the notching operation have been observed in transparent materials by viewing the specimen at low magnification between crossed

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[5] Supporting data is available from ASTM Headquarters. Request Research Report RR: D20–1066.

polars in monochromatic light. The specimens used to determine temperature rise shall not be used to make determinations of impact resistance.

8.3.2 The feed rate and the cutter speed shall remain constant throughout the notching operation.

8.4 It is acceptable to notch specimens individually or in a group. In either case, however, an unnotched backup or dummy bar shall be placed behind the last specimen in the sample holder to prevent distortion and chipping by the cutter as it exits from the last test specimen.

8.5 All specimens having one dimension less than 12.7 mm [0.500 in.] shall have the notch cut on the shorter side. Compression molded specimens shall be notched on the side parallel to the direction of application of molding pressure. The impact resistance of a plastic material will be different if the notch is perpendicular to rather than parallel to the direction of molding, as with or across the grain of an anisotropic bar cut from a plate.

9. Conditioning

9.1 Check the materials specification for the material that is being tested. If there are no conditioning requirements stated by the materials specification, the test specimens shall be conditioned at 23 ± 2°C [73 ± 3.6°F] and 50 ± 5 % relative humidity for not less than 40 h after notching and prior to testing in accordance with Procedure A of Practice D 618. unless documented (between supplier and customer) that shorter conditioning time is sufficient for a given material to reach equilibrium of impact resistance.

9.2 For hygroscopic materials, such as nylons, the material specifications (for example, Classification System D 4066) call for testing dry–as–molded specimens. Such requirements take precedence over the above routine preconditioning to 50 % relative humidity. These specimens shall be sealed in water vapor-impermeable containers as soon as molded. When notching these specimens, minimize the exposure time during notching and return the specimens to a dry container after notching to allow for full cooling of the specimens prior to testing.

9.3 *Test Conditions*—Conduct tests in the standard laboratory atmosphere of 23 ± 2°C [73 ± 3.6°F] and 50 ± 5 % relative humidity, unless otherwise specified. In cases of disagreement, the tolerances shall be ±1°C and ±2 % relative humidity.

10. Procedure

10.1 *Specimen Preparation*:

10.1.1 Prepare the test specimens in accordance with the procedures in Section 7. At least five and preferably ten or more individual determinations of impact resistance shall be made to determine the average impact resistance for a particular sample. The specimens shall be of nominal width only.

10.1.2 Notch the specimens in accordance with the procedure in Section 8.

10.1.3 Condition the specimens in accordance with the materials specification for the material that is being tested. If there are no conditioning requirements detailed in the materials specification, follow the conditioning requirements in Section 9.

10.2 *Machine Preparation*:

10.2.1 Estimate the breaking energy for the sample and select a pendulum of suitable energy. Select the lightest standard pendulum that is expected to break all specimens in the group with an energy loss of not more than 85 % of its capacity (see 6.1). If the breaking energy cannot be estimated, the correct pendulum can be determined by performing trial runs. Caution should be used to avoid damaging the pendulum by selecting a pendulum that is too light for a particular sample.

NOTE 11—Ideally, an impact test would be conducted at a constant test velocity. In a pendulum-type test, however, the velocity decreases as the fracture progresses. For specimens that have an impact energy approaching the capacity of the pendulum, there is insufficient energy to complete the break and toss. By avoiding the higher 15 % scale energy readings, the velocity of the pendulum will not be reduced below 1.33 m/s. On the other hand, the use of a pendulum that is too heavy would reduce the sensitivity of the reading.

10.2.2 After installing the selected pendulum on the machine, check the machine for conformity with the requirements of Section 6 before starting the tests.

10.2.3 When using a machine equipped with a pointer and dial mechanism or an electronic indicator that does not automatically correct for windage and friction, determine the windage and friction correction factors for the machine before testing specimens. Windage and friction correction factors shall be determined on a daily basis and shall be calculated each time weights are added to the pendulum or the pendulum is changed. Refer to Annex A1 for information on constructing windage and friction correction charts or refer to Annex A2 for a procedure to calculate the windage and friction correction. If excessive friction is indicated (see X2.12 and X2.13) the machine shall be adjusted before testing specimens. Follow the machine manufacturer's instructions to correct for excessive windage and friction.

NOTE 12—The actual correction factors for windage and friction will be smaller than these factors in an actual test because the energy absorbed by the specimen prevents the pendulum from making a full swing. The indicated breaking energy of the specimen, therefore, must be included in the calculation of the machine correction.

10.2.4 Some machines equipped with an electronic digital display or computer automatically compensate for windage and friction.

10.3 *Specimen Testing*:

10.3.1 Check all of the specimens in the sample group for conformity with the requirements of Sections 7 and 8 and 10.1.

10.3.2 Measure and record the width of each specimen after notching to the nearest 0.025 mm [0.001 in]. Measure the width in one location adjacent to the notch centered about the anticipated fracture plane.

10.3.3 Measure and record the depth of material remaining in the specimen under the notch of each specimen to the nearest 0.025 mm [0.001 in]. The tapered blade will be fitted to the notch. The specimen will be approximately vertical between the anvils. Position the edge of the non-cavity (wider edge) surface so that it is centered on the micrometer's flat circular anvil. See Fig. 5.

10.3.4 Position a test specimen horizontally on the supports and against the anvils so that it will be impacted on the face opposite the notch (see Fig. 3). The notch should be centered between the anvils. A centering jig is useful for this purpose.

10.3.5 Raise and secure the pendulum in the release mechanism. Zero the excess energy indicating mechanism.

10.3.6 Release the pendulum, allowing the striking edge of the pendulum to impact the specimen. Note the indicated breaking energy.

10.3.7 Calculate the net breaking energy (see 11.1). If the net breaking energy is greater than 85 % of the pendulum's nominal energy, the wrong pendulum was used. Discard the result. Select and install a pendulum with a greater available energy or add additional weight to the pendulum, determine the windage and friction correction factor, and repeat the test on a new specimen.

10.3.8 If the proper pendulum was used, test the remaining specimens as described in 10.3.1-10.3.6. Results from specimens that do not break should be discarded. A specimen that does not break completely into two or more pieces is not considered to be broken.

10.3.9 After all of the specimens for the sample have been tested, calculate the impact resistance, in joules per metre, for each individual specimen (see 11.2).

10.3.10 Calculate the average impact resistance for the group of specimens (see 11.3). Values obtained from specimens that did not break completely shall not be included in the average.

10.3.11 Calculate the standard deviation for the group of specimens (see 11.4).

11. Calculation

11.1 *Net Breaking Energy*—Subtract the windage and friction loss energy from the indicated breaking energy.

11.2 *Impact Resistance*—Divide the net breaking energy by the measured width of each individual specimen.

11.3 Calculate the average impact resistance for a group of specimens by adding the individual impact resistance values for the group and dividing the sum by the total number of specimens in the group.

11.4 Calculate the standard deviation as follows and report it to two significant figures:

$$s = \sqrt{(\Sigma X^2 - n\,\bar{X}^2/(n-1)} \qquad (3)$$

where:

s = estimated standard deviation,
X = value of single observation,
n = number of observations, and
$\bar{X}$ = arithmetic mean of the set of observations.

12. Report

12.1 Report the following information:

12.1.1 Complete identification of the material tested, including type source, manufacturer's code number, and previous history.

12.1.2 A statement of how the specimens were prepared, the testing conditions used, the number of hours the specimens were conditioned after notching, and for sheet materials, the direction of testing with respect to anisotropy, if any.

12.1.3 The capacity of the pendulum, J.

12.1.4 The span.

12.1.5 The width and depth under the notch of each specimen tested.

12.1.6 The total number of specimens tested per sample of material (that is five, ten, or more).

12.1.7 The average impact resistance, J/m. Impact resistance is not to be reported for other than complete breaks. Reporting results in kJ/m^2 is optional (see Appendix X4).

12.1.8 The standard deviation of the values of the impact resistance of the specimens in 10.3.11.

13. Precision and Bias

13.1 Table 1 is based on a round robin[6] conducted in 1987 in accordance with Practice E 691, involving five materials tested by nine laboratories. For each material, all samples were prepared at one source, but the individual specimens were notched and conditioned at the laboratories which tested them. Each laboratory tested an average of nine specimens for each material. **Warning—**The explanations of *r* and *R* (13.2-13.2.3) are intended only to present a meaningful way of considering the approximate precision of this test method. The data presented in Table 1 should not be applied to acceptance or rejection of materials, as these data apply only to the materials tested in the round robin and are unlikely to be rigorously representative of other lots, formulations, conditions, materials, or laboratories. Users of this test method should apply the principles outlined in Practice E 691 to generate data specific to their materials and laboratory, or between specific laboratories. The principles of 13.2-13.2.3 would then be valid for such data.

13.2 *Concept of r and R in Table 1* —If S_r and S_R have been calculated from a large enough body of data, and for test results that were averages from testing nine specimens for each test result, then:

13.2.1 *Repeatability*—*r* is the interval representing the critical difference between two test results for the same material, obtained by the same operator using the same equipment on the

TABLE 1 Precision for Charpy Test

Values in ft·lbf/in. of Width

Material	Average	S_r[A]	S_R[B]	r[C]	R[D]	Number of Laboratories
Phenolic	0.55	0.029	0.050	0.08	0.14	7
Reinforced nylon	1.98	0.065	0.143	0.18	0.40	7
Polycarbonate	2.85	0.083	0.422	0.23	1.19	8
Polypropylene	4.06	0.151	0.422	0.42	1.19	9
ABS	10.3	0.115	0.629	0.32	1.78	9

[A] S_r = within-laboratory standard deviation for the indicated material. It is obtained by pooling the within-laboratory standard deviations of the test result from all of the participating laboratories:

$$S_r = [\,[(S_1)^2 + (S_2)^2 \ldots + (Sn)^2]\,/n]^{1/2}$$

[B] S_R = between-laboratories reproducibility, expressed as standard deviation:

$$S_R = [\,S_r^2 + S_L^2\,]^{1/2}$$

where S_L = standard deviation of laboratory means.
[C] r = within-laboratory critical interval between two test results = 2.8 × S_r.
[D] R = between laboratories critical interval between two test results = 2.8 × S_R.

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[6] Supporting data is available from ASTM Headquarters. Request Research Report RR: D20–1041 and 1134.

same day in the same laboratory. Two tests results shall be judged not equivalent if they differ by more than the *r* value for that material.

13.2.2 *Reproducibility—R* is the interval representing the critical difference between two test results for the same material, obtained by different operators using different equipment in different laboratories, not necessarily on the same day. Two test results shall be judged not equivalent if they differ by more than the *R* value for that material.

13.2.3 Any judgement in accordance with 13.2.1 or 13.2.2 would have an approximate 95 % (0.95) probability of being correct.

13.3 There are no recognized standards by which to estimate bias of this test method.

14. Keywords

14.1 Charpy impact; impact resistance; notch sensitivity; notched specimen

ANNEXES

(Mandatory Information)

A1. INSTRUCTIONS FOR THE CONSTRUCTION OF A WINDAGE AND FRICTION CORRECTION CHART

A1.1 The construction and use of the chart herein described is based upon the assumption that the friction and windage losses are proportional to the angle through which these loss torques are applied to the pendulum. Fig. A1.1 shows the assumed energy loss versus the angle of the pendulum position during the pendulum swing. The correction chart to be described is principally the left half of Fig. A1.1. The windage and friction correction charts should be available from commercial testing machine manufacturers. The energy losses designated as A or B are described in 10.3.

A1.2 Start the construction of the correction chart (Fig. A1.2) by laying off to some convenient linear scale on the abscissa of a graph the angle of pendulum position for the portion of the swing beyond the free hanging position. For convenience, place the free hanging reference point on the right end of the abscissa with the angular displacement increasing linearly to the left. The abscissa is referred to as Scale C. Although angular displacement is the quantity to be represented linearly on the abscissa, this displacement is more conveniently expressed in terms of indicated energy read from the machine dial. This yields a nonlinear Scale C with indicated pendulum energy increasing to the right.

A1.3 On the right hand ordinate lay off a linear Scale B starting with zero at the bottom and stopping at the maximum expected pendulum friction and windage value at the top.

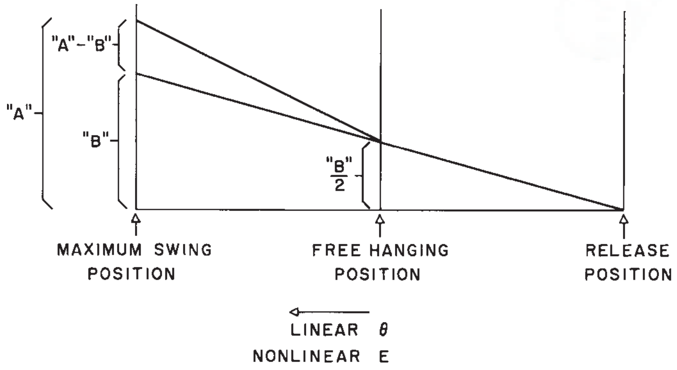


FIG. A1.1 Method of Construction of a Windage and Friction Correction Chart

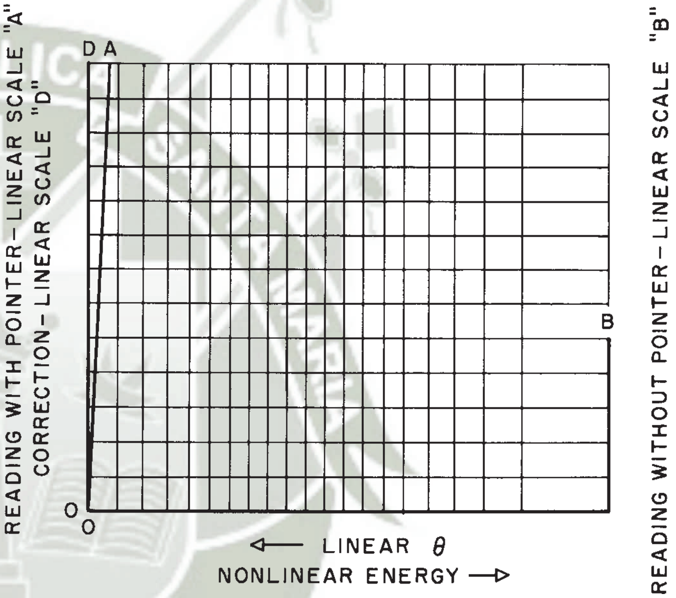


FIG. A1.2 Sample Windage and Friction Correction Chart

A1.4 On the left ordinate construct a linear Scale D ranging from zero at the bottom to 1.2 times the maximum ordinate value appearing on Scale B, but make the scale twice the scale used in the construction of Scale B.

A1.5 Adjoining Scale D draw a curve OA which is the focus of points whose coordinates have equal values of energy correction on Scale D and indicated energy on Scale C. This curve is referred to as Scale A and utilizes the same divisions and numbering system as the adjoining Scale D.

A1.6 *Instructions for Using Chart*:

A1.6.1 Locate and mark on Scale A the reading A obtained from the free swing of the pendulum with the pointer prepositioned in the free hanging or maximum indicated energy position on the dial.

A1.6.2 Locate and mark on Scale B the reading B obtained after several free swings with the pointer pushed up close to zero indicated energy position of the dial by the pendulum in accordance with instructions in 10.3.

A1.6.3 Connect the two points thus obtained by a straight line.

A1.6.4 From the indicated impact energy on Scale C project up to the constructed line and across to the left to obtain the correction for windage and friction from Scale D.

A1.6.5 Subtract this correction from the indicated impact reading to obtain the energy delivered to the specimen.

A2. PROCEDURE FOR THE CALCULATION OF WINDAGE AND FRICTION CORRECTION

A2.1 The procedure for the calculation of the windage and friction correction in this annex is based on the equations developed by derivation in Appendix X3. This procedure is acceptable as a substitute for the graphical procedure described in Annex A1 and is applicable to small electronic calculator and computer analysis.

A2.2 Calculate *L*, the distance from the axis of support to the center of percussion as indicated in 6.3. It is assumed here that the center of percussion is approximately the same as the center of strike.

A2.3 Measure the maximum height, h_M, of the center of percussion (center of strike) of the pendulum at the start of the test as indicated in X2.16.

A2.4 Measure and record the energy correction, E_A, for windage of the pendulum plus friction in the dial, as determined with the first swing of the pendulum with no specimen in the testing device. This correction must be read on the energy scale, E_M, appropriate for the pendulum used.

A2.5 Without resetting the position of the indicator obtained in A2.4, measure the energy correction, E_B, for pendulum windage after two additional releases of the pendulum with no specimen in the testing device.

A2.6 Calculate β_{max} as follows:

$$\beta_{max} = \cos^{-1}\{1 - [(h_M/L)(1 - E_A/E_M)]\} \quad (A2.1)$$

where:
E_A = energy correction for windage of pendulum plus friction in dial, J [ft·lbf],
E_M = full-scale reading for pendulum used, J [ft·lbf],
L = distance from fulcrum to center of strike of pendulum, m [ft],
h_M = maximum height of center of strike of pendulum at start of test, m [ft], and
β_{max} = maximum angle pendulum will travel with one swing of the pendulum.

A2.7 Measure specimen breaking energy, E_S, J [ft·lbf].

A2.8 Calculate β for specimen measurement E_s as:

$$\beta = \cos^{-1}\{1 - [(h_M/L)(1 - E_S/E_M)]\} \quad (A2.2)$$

where:
β = angle pendulum travels for a given specimen, and
E_S = dial reading breaking energy for a specimen, J [ft·lbf].

A2.9 Calculate total correction energy, E_{TC} as:

$$E_{TC} = (E_A - (E_B/2))(\beta/\beta_{max}) + (E_B/2) \quad (A2.3)$$

where:
E_{TC} = total correction energy for the breaking energy, E_s, of a specimen, J [ft·lbf], and
E_B = energy correction for windage of the pendulum, J [ft·lbf].

A2.10 Calculate the impact resistance using the following formula:

$$I_s = (E_s - E_{TC})/t \quad (A2.4)$$

where:
I_s = impact resistance of specimen, J/m [ft·lbf/in.] of width, and
t = width of specimen or width of notch, m [in.]

APPENDIXES

(Nonmandatory Information)

X1. PROCEDURE FOR THE INSPECTION AND VERIFICATION OF NOTCH

X1.1 The purpose of this procedure is to describe the microscopic method to be used for determining the radius and angle of the notch. These measurements could also be made using a comparator if available.

NOTE X1.1—The notch shall have a radius of 0.25 ± 0.05 mm [0.010 ± 0.002 in.] and an angle of 45 ± 1°.

X1.2 *Apparatus*:

X1.2.1 *Optical Device*, with minimum magnification of 60×, Filar glass scale and camera attachment.

X1.2.2 *Transparent Template*, that will be developed in this procedure.

X1.2.3 *Ruler*.

X1.2.4 *Compass*.

X1.2.5 *Plastic Drafting Set Squares (Triangles),* 45–45–90°.

X1.3 A transparent template must be developed for each magnification and for each microscope used. It is preferable that each laboratory standardize on one microscope and one magnification. It is not necessary for each laboratory to use the same magnification because each microscope and camera combination have somewhat different blowup ratios.

X1.3.1 Set the magnification of the optical device at a suitable magnification with a minimum magnification of 60×.

X1.3.2 Place the Filar glass slide on the microscope platform. Focus the microscope so the most distinct of the Filar scale is visible.

X1.3.3 Take a photograph of the Filar scale (see Fig. X1.1).

X1.3.4 Create a template similar to that shown in Fig. X1.2.

X1.3.4.1 Find the approximate center of the piece of paper.

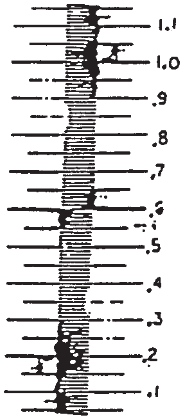
X1.3.4.2 Draw a set of perpendicular coordinates through the center point.

X1.3.4.3 Draw a family of concentric circles that are spaced in accordance with the dimensions of the Filar scale. This task is accomplished by first setting a mechanical compass at a distance of 0.1 mm [0.004 in.] as referenced by the magnified photograph of the Filar eyepiece. Subsequent circles shall be spaced 0.02 mm apart [0.001 in.], as rings, with the outer ring being 0.4 mm [0.016 in.] from the center.

X1.3.5 Photocopy the paper with the concentric circles to make a transparent template of the concentric circles.

X1.3.6 Construct Fig. X1.3 by taking a second piece of paper, finding its approximate center, and marking this point. Draw one line through this center point. Label this line zero degree (0°). Draw a second line perpendicular to the first line through this center point. Label this line 90°. From the center draw a line that is 44° relative to the 0°. Label the line 44°. Draw another line at 46°. Label the line 46°.

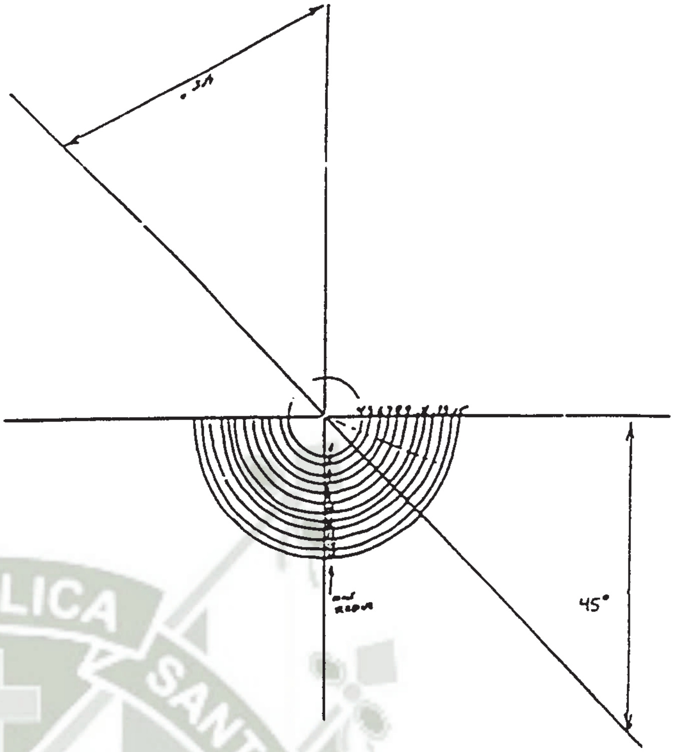
X1.4 Place a microscope glass slide on the microscope platform. Place the notched specimen on top of the slide. Focus the microscope. Move the specimen around using the platform adjusting knobs until the specimen's notch is centered and near the bottom of the viewing area. Take a picture of the notch.



NOTE 1—100× reference.

NOTE 2—0.1 mm major scale; 0.01 mm minor scale.

FIG. X1.1 Filar Scale



NOTE 1—Magnification = 100×.

FIG. X1.2 Example of Transparent Template for Determining Radius of Notch

X1.4.1 *Determination of Notching Radius (*Fig. X1.4*)*:

X1.4.1.1 Place the picture on a sheet of paper. Position the picture so that bottom of the notch in the picture faces downwards and is about 64 mm [2.5 in.] from the bottom of the paper. Tape the picture down to the paper.

X1.4.1.2 Draw two lines along the sides of the notch projecting down to a point where they intersect below the notch Point I (see Fig. X1.4B).

X1.4.1.3 Open the compass to about 51 mm [2 in.]. Using Point I as a reference, draw two arcs intersecting both sides of the notch (see Fig. X1.4C). These intersections are called 1a and 1b.

X1.4.1.4 Close the compass to about 38 mm [1.5 in.]. Using Point 1a as the reference point, draw an arc (2a) above the notch, draw a second arc (2b) that intersects with arc 2a at Point J. Draw a line between I and J. This establishes the centerline of the notch (see Fig. X1.4D)

X1.4.1.5 Place the transparent template on top of the picture and align the center of the concentric circles with the drawn centerline of the notch (see Fig. X1.4E).

X1.4.1.6 Slide the template down the centerline of the notch until one concentric circle touches both sides of the notch. Record the radius of the notch and compare it against the limits of 0.2 to 0.3 mm [0.008 to 0.012 in.].

X1.4.1.7 Examine the notch to ensure that there are no flat spots along the measured radius.

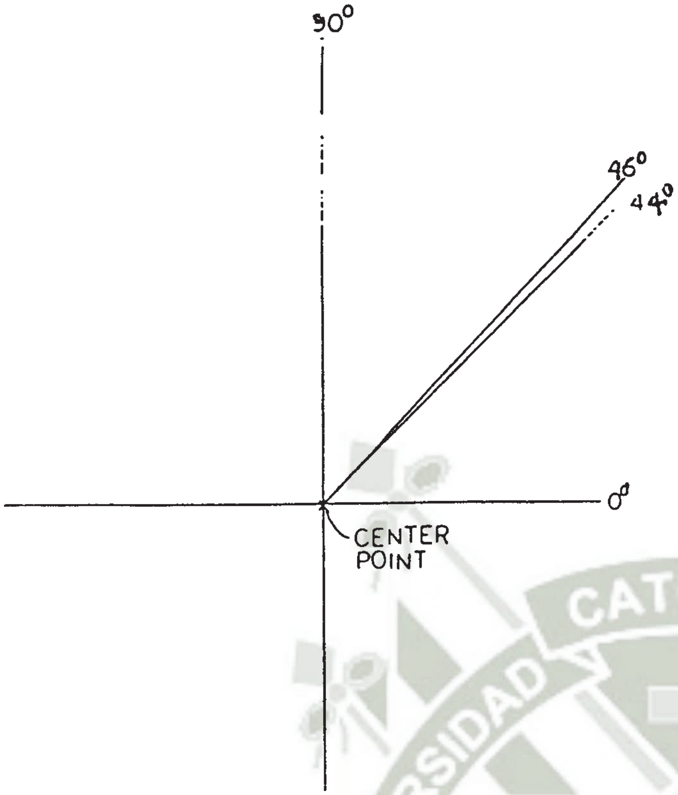


FIG. X1.3 Example of Transparent Template for Determining Angle of Notch

X1.4.2 *Determination of Notch Angle*—Place transparent template for determining notch angle (Fig. X1.3) on top of the photograph attached to the sheet of paper. Rotate the picture so that the notch tip is pointed towards you. Position the center point of the template on top of the Point I established in 0° axis of the template with the right side straight portion of the notch. Check the left side straight portion of the notch to ensure that this portion falls between the 44° and 46° lines. If not, replace the blade.

X1.5 A picture of a notch shall be taken at least every 500 notches or if a control sample gives a value outside its 3-sigma limits for that test.

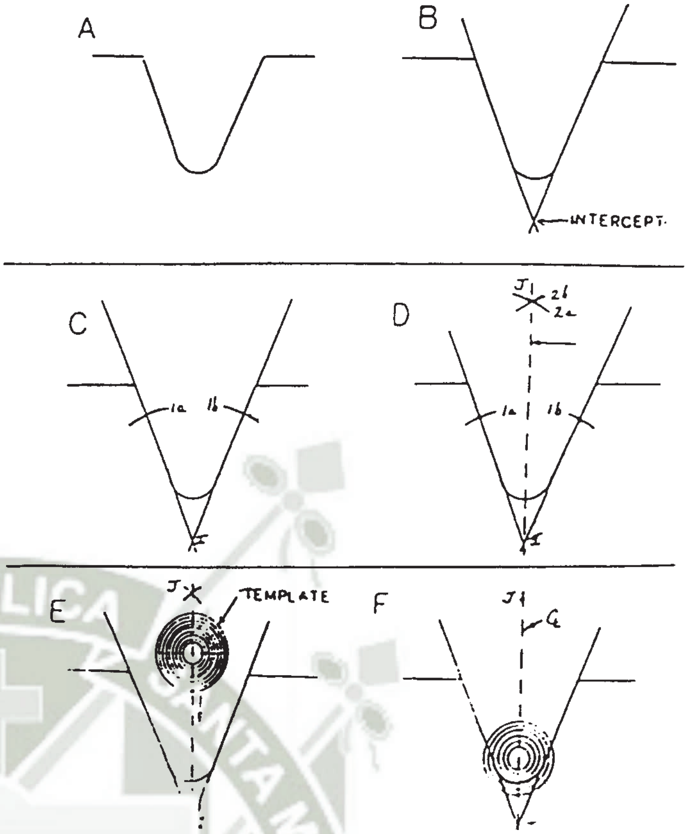


FIG. X1.4 Determining Radius

X1.6 If the notch in the control specimen is not within the requirements, a picture of the notching blade should be taken and analyzed by the same procedure used for the specimen notch. If the notching blade does not meet ASTM requirements or shows damage, it should be replaced with a new blade which has been checked for proper dimensions.

X1.7 If a cutter has the correct dimensions, but does not cut the correct notch in the specimen, it will be necessary to evaluate other conditions (cutter and feed speeds) to obtain the correct notch dimension for that material.

X2. CALIBRATION OF PENDULUM-TYPE HAMMER IMPACT MACHINES FOR USE WITH PLASTIC SPECIMENS

X2.1 This calibration procedure applies specifically to the Charpy impact machine.

X2.2 Locate the impact machine on a sturdy base. It shall not walk on the base and the base shall not vibrate appreciably. Loss of energy from vibrations will give high readings. It is recommended that the impact tester be bolted to a base having a mass of at least 23 kg if it is used at capacities higher than 2.7 J [2 ft·lbf].

X2.3 Check the level of the machine in both directions on the plane of the base with spirit levels mounted in the base, by a machinist's level if a satisfactory reference surface is available, or with a plumb bob. The machine should be made level to within $\tan^{-1}$ 0.001 in the plane of swing and to within $\tan^{-1}$ 0.002 in the place perpendicular to the swing.

X2.4 Contact the machine manufacturer for a procedure to ensure the striker radius is in tolerance (3.17 ± 0.12 mm) (see 6.3).

X2.5 Check the transverse location of the center of the pendulum striking edge that shall be within 0.40 mm [0.016 in.] of the center of the anvil. Readjust the shaft bearings or relocate the anvil or straighten the pendulum shaft as necessary to attain the proper relationship between the two centers.

X2.6 Check the pendulum arm for straightness within 1.2

mm [0.05 in.] with a straightedge or by sighting down the shaft. This arm is sometimes bent by allowing the pendulum to slam against the catch when high–capacity weights are on the pendulum.

X2.7 Center a notched 12.7–mm square metal bar having opposite sides parallel within 0.025 mm and 125 mm long on the Charpy anvils. Place a thin oil film on the striking edge of the pendulum with an oiled cloth and let the striking edge rest gently against the bar. A thin line of oil should be transferred across the entire width of the bar, thereby verifying that the striking edge is in contact across the entire specimen width.

X2.8 When the pendulum is hanging free in its lowest position, the energy reading must be within 0.2 % of full scale.

X2.9 Swing the pendulum to a horizontal position, and support it by the striking edge in this position with a vertical bar. Allow the other end of this bar to rest at the center of a load pan on a balanced scale. Subtract the weight of the bar from the total weight to find the effective weight of the pendulum. The effective pendulum weight should be within 0.4 % of the required weight for that pendulum capacity. If weight must be added or removed, take care to balance the added or removed weight without affecting the center of percussion relative to the striking edge. It is not advisable to add weight to the opposite side of the bearing axis from the striking edge to decrease the effective weight of the pendulum since the distributed mass has the potential to result in large energy losses from vibration of the pendulum.

X2.10 Calculate the effective length of the pendulum arm or the distance to the center of percussion from the axis of rotation by the procedure in Note 5. The effective length must be within the tolerance stated in 6.3.

X2.11 Determine the vertical distance of fall of the pendulum striking edge from its latched height to its lowest point. This distance should be 610 ± 2 mm. This measurement is made with a half-width specimen positioned on the anvils. Place a thin oil film on the specimen and bring the striking edge against it. The upper end of the oil line on the striking edge is the center of strike. Measure the change in vertical height of the center of strike from the latched to the free hang position (the lowest point). This vertical fall distance is adjusted by varying the position of the pendulum latch.

X2.12 If a pointer and dial mechanism is used to indicate the energy, the pointer friction should be adjusted so that the pointer will just maintain its position anywhere on the scale. The striking pin of the pointer should be securely fastened to the pointer. Friction washers with glazed surfaces should be replaced with new washers. Friction washers should be on either side of the pointer collar. The last friction washer installed should be backed by a heavy metal washer. Pressure on this metal washer is produced by a thin bent spring washer and locknuts. If the spring washer is placed next to the fiber friction washer, the pointer will tend to vibrate during impact.

X2.13 The free-swing reading of the pendulum (without specimen) from the latched height should be less than 2.5 % of pendulum capacity on the first swing. If the reading is higher than this, the friction in the indicating mechanism is excessive or the bearings are dirty. To clean the bearings, dip them in grease solvent and spin dry in an air jet. Clean the bearings until they spin freely or replace them. Oil very lightly with instrument oil before replacing. A reproducible method of starting the pendulum from the proper height must be devised.

X2.14 The shaft about which the pendulum rotates shall have no detectable radial play, less than 0.05 mm [0.002 in.]. An end play of 0.25 mm [0.010 in.] is permissible when a 9.8–N [2.2–lbf] axial force is applied in alternate directions.

X2.15 The machine should not be used to indicate more than 85 % of the energy capacity of the pendulum. Extra weight added to the pendulum will increase available energy of the machine. This weight must be added so as to maintain the center of percussion within the tolerance stated in 6.3. Correct effective weight for any range is calculated as follows:

$$W = E_p / h \tag{X2.1}$$

where:

W = the effective pendulum weight, N [lbf] (see X2.13),
E_p = potential or available energy of the machine, J [ft × lbf], and
h = the vertical distance of fall of the pendulum striking edge, m [ft] (see X2.11).

Each 4.5 N [1 lbf] of added effective weight increases the capacity of the machine by 2.7 J [2 ft × lbf].

NOTE X2.1—If the pendulum is designed for use with add-on weight, it is recommended that they be obtained through the equipment manufacturer.

X3. DERIVATION OF PENDULUM IMPACT CORRECTION EQUATIONS

X3.1 From right triangle distances in Fig. X3.1:

$$L - h = L \cos \beta \tag{X3.1}$$

X3.2 The potential energy gain of pendulum, E_p, is:

$$E_p = hW_pg \tag{X3.2}$$

X3.3 Combining Eq X3.1 and Eq X3.2 gives the following:

$$L - E_p/W_pg = L \cos \beta \tag{X3.3}$$

X3.4 The maximum energy of the pendulum is the potential energy at the start of the test, E_M, or

$$E_M = h_MW_pg \tag{X3.4}$$

X3.5 The potential energy gained by the pendulum, E_p, is related to the absorption of energy of a specimen, E_s, by the following equation:

$$E_M - E_s = E_p \tag{X3.5}$$

X3.6 Combining Eq X3.3-X3.5 gives the following:

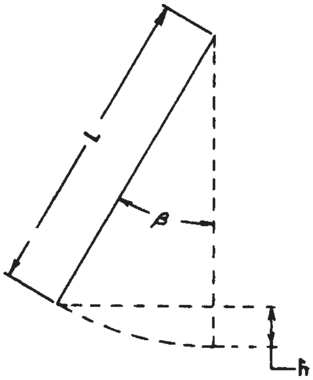


FIG. X3.1 Swing of Pendulum from Its Rest Position

$$(E_M - E_S)/E_M = L/h_M\,(1 - \cos\beta) \tag{X3.6}$$

X3.7 Solving Eq X3.6 for β gives the following:

$$\beta = \cos^{-1}\{1 - [(h_M/L)(1 - E_S/E_M)]\} \tag{X3.7}$$

X3.8 From Fig. X3.2, the total energy correction, E_{TC}, is given as:

$$E_{TC} = m\beta + b \tag{X3.8}$$

X3.9 At the zero point of the pendulum the potential energy is:

$$E_B/2 = m(0) + b$$

or (X3.9)

$$b = E_B/2 \tag{X3.9}$$

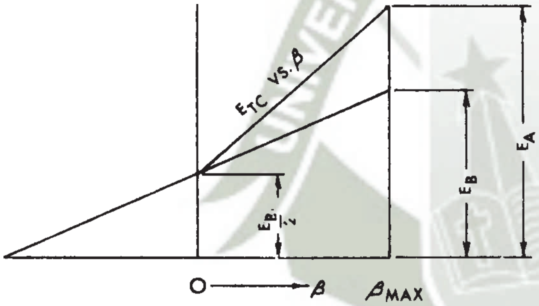


FIG. X3.2 Total Energy Correction for Pendulum Windage and Dial Friction as a Function of Pendulum Position

X3.10 The energy correction, E_A, on the first swing of the pendulum occurs at the maximum pendulum angle, β_{max}. Substituting in Eq X3.8 gives the following:

$$E_A = m\beta_{max} + (E_B/2) \tag{X3.10}$$

X3.11 Combining Eq X3.8 and Eq X3.11 gives the following:

$$E_{TC} = (E_A - (E_B/2))(\beta/\beta_{max}) + (E_B/2) \tag{X3.11}$$

X3.12 *Nomenclature*:

b = intercept of total correction energy straight line,
E_A = energy correction, including both pendulum windage plus dial friction, J,
E_B = energy correction for pendulum windage only, J,
E_M = maximum energy of the pendulum (at the start of test), J,
E_p = potential energy gain of pendulum from the pendulum rest position, J,
E_S = uncorrected breaking energy of specimen, J,
E_{TC} = total energy correction for a given breaking energy, E_S, J,
g = acceleration of gravity, m/s^2,
h = distance center of gravity of pendulum rises vertically from the rest position of the pendulum, m,
h_m = maximum height of the center of gravity of the pendulum, m,
m = slope of total correction energy straight line,
L = distance from fulcrum to center of gravity of pendulum, m,
W_p = weight of pendulum, as determined in X2.13, kg, and
β = angle of pendulum position from the pendulum rest position.

X4. UNIT CONVERSIONS

X4.1 Joules per metre cannot be converted directly into kilojoules per square metre.

NOTE X4.1—The optional units of kJ/m^2 [ft·lbf/in.^{2}] also may be required; therefore, the cross-sectional area under the notch must be reported.

X4.2 The following examples are approximations:

1ft·lbf/39.37 in. = 1.356 J/m
1ft·lbf/in. = (39.37)(1.356) J/m
1ft·lbf/in. = 53.4 J/m
1ft·lbf/in. = 0.0534 kJ/m

1ft·lbf/1550 in.^{2} = 1.356 J/m^2
1ft·lbf/in.^{2} = (1550)(1.356) J/m^2
1ft·lbf/in.^{2} = 2101 J/m^2
1ft·lbf/in.^{2} = 2.1 kJ/m^2

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SUMMARY OF CHANGES

This section identifies the location of selected changes to this test method. For the convenience of the user, Committee D20 has highlighted those changes that impact the use of this test method. This section also includes descriptions of the changes or reasons for the changes, or both.

D6110-04:
(*1*) Add reference to D5947 in paragraph 2.1.
(*2*) Changed requirements for micrometer in paragraph 6.3.
(*3*) Removed requirement to mill draft angle from specimens.
(*4*) Changed notch depth dimensions to 10.16 ± 0.05 mm in order to agree with Figure 4; and clarified procedure to measure notch depth in paragraph 8.2.
(*5*) Added paragraphs 10.3.2 and 10.3.3 on clarifying notch depth measuring technique. Renumbered subsequent sections.
(*6*) Added Figure 6.
(*7*) Permissive language removed.
(*8*) Imperial units added for reference.

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Designation: D7264/D7264M – 07

Standard Test Method for Flexural Properties of Polymer Matrix Composite Materials[1]

This standard is issued under the fixed designation D7264/D7264M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method determines the flexural stiffness and strength properties of polymer matrix composites.

1.1.1 *Procedure A*—A three-point loading system utilizing center loading on a simply supported beam.

1.1.2 *Procedure B*—A four-point loading system utilizing two load points equally spaced from their adjacent support points, with a distance between load points of one-half of the support span.

NOTE 1—Unlike Test Method D6272, which allows loading at both one-third and one-half of the support span, in order to standardize geometry and simplify calculations this standard permits loading at only one-half the support span.

1.2 For comparison purposes, tests may be conducted according to either test procedure, provided that the same procedure is used for all tests, since the two procedures generally give slightly different property values.

1.3 The values stated in either SI units or inch-pound units are to be regarded separately as standard. Within the text, the inch-pound units are shown in brackets. The values stated in each system are not exact equivalents; therefore, each system must be used independently of the other. Combining values from the two systems may result in nonconformance with the standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D790 Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials

D2344/D2344M Test Method for Short-Beam Strength of Polymer Matrix Composite Materials and Their Laminates

D3878 Terminology for Composite Materials

D5229/D5229M Test Method for Moisture Absorption Properties and Equilibrium Conditioning of Polymer Matrix Composite Materials

D5687/D5687M Guide for Preparation of Flat Composite Panels with Processing Guidelines for Specimen Preparation

D6272 Test Method for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials by Four-Point Bending

D6856 Guide for Testing Fabric-Reinforced "Textile" Composite Materials

E4 Practices for Force Verification of Testing Machines

E6 Terminology Relating to Methods of Mechanical Testing

E18 Test Methods for Rockwell Hardness of Metallic Materials

E122 Practice for Calculating Sample Size to Estimate, With Specified Precision, the Average for a Characteristic of a Lot or Process

E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E456 Terminology Relating to Quality and Statistics

E1309 Guide for Identification of Fiber-Reinforced Polymer-Matrix Composite Materials in Databases

E1434 Guide for Recording Mechanical Test Data of Fiber-Reinforced Composite Materials in Databases

2.2 *Other Documents:*

ANSI Y14.5-1999 Dimensioning and Tolerancing—Includes Inch and Metric[3]

ANSI B46.1-1995 Surface Texture (Surface Roughness, Waviness and Lay)[3]

3. Terminology

3.1 *Definitions*—Terminology D3878 defines the terms relating to high-modulus fibers and their composites. Terminology E6 defines terms relating to mechanical testing. Terminology E456 and Practice E177 define terms relating to statistics. In the event of a conflict between terms, Terminology D3878 shall have precedence over the other documents.

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[1] This test method is under the jurisdiction of ASTM Committee D30 on Composite Materials and is the direct responsibility of Subcommittee D30.04 on Lamina and Laminate Test Methods.

Current edition approved April 1, 2007. Published April 2007. Originally approved in 2006. Last previous edition approved in 2006 as D7264/D7264M – 06. DOI: 10.1520/D7264_D7264M-07.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *flexural strength*, *n*—the maximum stress at the outer surface of a flexure test specimen corresponding to the peak applied force prior to flexural failure.

3.2.2 *flexural modulus*, *n*—the ratio of stress range to corresponding strain range for a test specimen loaded in flexure.

3.3 *Symbols*:

b = specimen width
CV = sample coefficient of variation, in percent
E_f^{chord} = flexural chord modulus of elasticity
E_f^{secant} = flexural secant modulus of elasticity
h = specimen thickness
L = support span
m = slope of the secant of the load-deflection curve
n = number of specimens
P = applied force
s_{n-1} = sample standard deviation
x_i = measured or derived property
$\bar{x}$ = sample mean
δ = mid-span deflection of the specimen
ε = strain at the outer surface at mid-span of the specimen
σ = stress at the outer surface at mid-span of the specimen

4. Summary of Test Method

4.1 A bar of rectangular cross section, supported as a beam, is deflected at a constant rate as follows:

4.1.1 *Procedure A*—The bar rests on two supports and is loaded by means of a loading nose midway between the supports (see Fig. 1).

4.1.2 *Procedure B*—The bar rests on two supports and is loaded at two points (by means of two loading noses), each an equal distance from the adjacent support point. The distance between the loading noses (that is, the load span) is one-half of the support span (see Fig. 2).

4.2 Force applied to the specimen and resulting specimen deflection at the center of span are measured and recorded until the failure occurs on either one of the outer surfaces, or the deformation reaches some pre-determined value.

4.3 The major difference between four-point and three-point loading configurations is the location of maximum bending moment and maximum flexural stress. With the four-point configuration the bending moment is constant between the central force application members. Consequently, the maximum flexural stress is uniform between the central force application members. In the three-point configuration, the maximum flexural stress is located directly under the center force application member. Another difference between the three-point and four-point configurations is the presence of resultant vertical shear force in the three-point configuration everywhere in the beam except right under the mid-point force application member whereas in the four-point configuration, the area between the central force application members has no resultant vertical shear force. The distance between the outer support members is the same as in the equivalent three-point configuration.

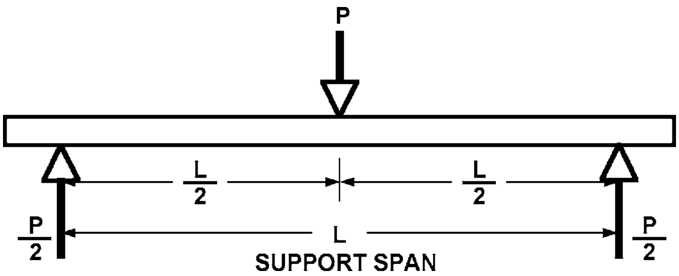


FIG. 1 Procedure A—Loading Diagram

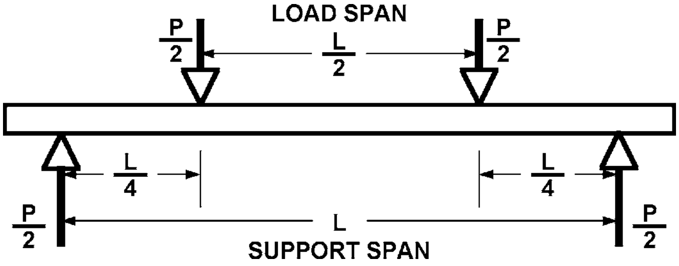


FIG. 2 Procedure B—Loading Diagram

4.4 The test geometry is chosen to limit out-of-plane shear deformations and avoid the type of short beam failure modes that are interrogated in Test Method D2344/D2344M.

5. Significance and Use

5.1 This test method determines the flexural properties (including strength, stiffness, and load/deflection behavior) of polymer matrix composite materials under the conditions defined. Procedure A is used for three-point loading and Procedure B is used for four-point loading. This test method was developed for optimum use with continuous-fiber-reinforced polymer matrix composites and differs in several respects from other flexure methods, including the use of a standard span-to-thickness ratio of 32:1 versus the 16:1 ratio used by Test Methods D790 (a plastics-focused method covering three-point flexure) and D6272 (a plastics-focused method covering four-point flexure).

5.2 This test method is intended to interrogate long-beam strength in contrast to the short-beam strength evaluated by Test Method D2344/D2344M.

5.3 Flexural properties determined by these procedures can be used for quality control and specification purposes, and may find design applications.

5.4 These procedures can be useful in the evaluation of multiple environmental conditions to determine which are design drivers and may require further testing.

5.5 These procedures may also be used to determine flexural properties of structures.

6. Interferences

6.1 Flexural properties may vary depending on which surface of the specimen is in compression, as no laminate is perfectly symmetric (even when full symmetry is intended); such differences will shift the neutral axis and will be further affected by even modest asymmetry in the laminate. Flexural properties may also vary with specimen thickness, conditioning and/or testing environments, and rate of straining. When evaluating several datasets these parameters should be equivalent for all data in the comparison.

6.2 For multidirectional laminates with a small or moderate number of laminae, flexural modulus and flexural strength may be affected by the ply-stacking sequence and will not necessarily correlate with extensional modulus, which is not stacking-sequence dependent.

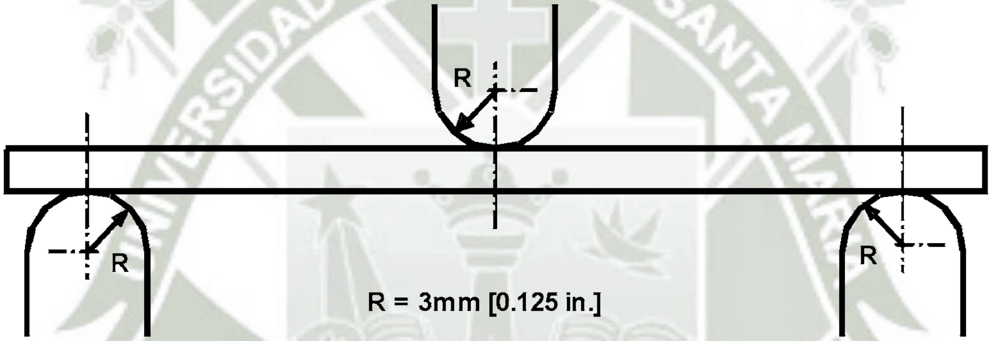
6.3 The calculation of the flexural properties in Section 13 of this standard is based on beam theory, while the specimens in general may be described as plates. The differences may in some cases be significant, particularly for laminates containing a large number of plies in the ±45° direction. The deviations from beam theory decrease with decreasing width.

6.4 Loading noses may be fixed, rotatable or rolling. Typically, for testing composites, fixed or rotatable loading noses are used. The type of loading nose can affect results, since non-rolling paired supports on either the tension or compression side of the specimen introduce slight longitudinal forces and resisting moments on the beam, which superpose with the intended loading. The type of supports used is to be reported as described in Section 14. The loading noses should also uniformly contact the specimen across its width. Lack of uniform contact can affect flexural properties by initiating damage by crushing and by non-uniformly loading the beam. Formulas used in this standard assume a uniform line loading at the specimen supports across the entire specimen width; deviations from this type of loading is beyond the scope of this standard.

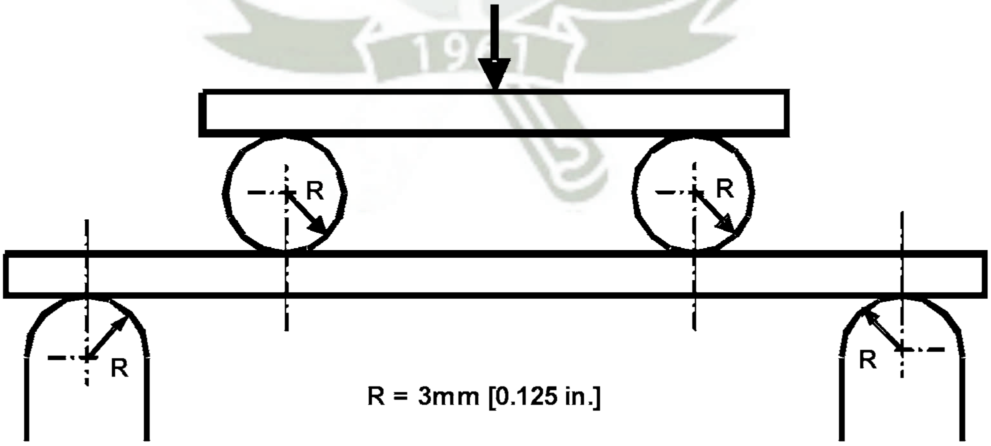
7. Apparatus

7.1 *Testing Machine*—Properly calibrated, which can be operated at a constant rate of crosshead motion, and in which the error in the force application system shall not exceed ±1 % of the full scale. The force indicating mechanism shall be essentially free of inertia lag at the crosshead rate used. Inertia lag shall not exceed 1 % of the measured force. The accuracy of the testing machine shall be verified in accordance with Practices E4.

7.2 *Loading Noses and Supports*—The loading noses and supports shall have cylindrical contact surfaces of radius 3.00 mm [0.125 in.] as shown in Fig. 3, with a hardness of 60 to 62 HRC, as specified in Test Methods E18, and shall have finely



Three-Point Loading Configuration with Fixed Supports and Loading Nose



Four-Point Loading Configuration with Fixed Supports and Rolling Loading Noses

FIG. 3 Example Loading Nose and Supports for Procedures A (top) and B (bottom) ground surfaces free of indentation and burrs with all sharp edges relieved. Loading noses and supports may be arranged in a fixed, rotatable or rolling arrangement. Typically, with composites, rotatable or fixed arrangements are used.

7.3 *Micrometers*—For width and thickness measurements the micrometers shall use a 4 to 7 mm [0.16 to 0.28 in.] nominal diameter ball-interface on an irregular surface such as the bag side of a laminate, and a flat anvil interface on machined edges or very smooth tooled surfaces. A micrometer or caliper with flat anvil faces shall be used to measure the length of the specimen. The accuracy of the instrument(s) shall be suitable for reading to within 1 % or better of the specimen dimensions. For typical section geometries, an instrument with an accuracy of ±0.02 mm [±0.001 in.] is desirable for thickness and width measurement, while an instrument with an accuracy of ±0.1 mm [±0.004 in.] is adequate for length measurement.

7.4 *Deflection Measurement*—Specimen deflection at the common center of the loading span shall be measured by a properly calibrated device having an accuracy of ±1 % or better of the expected maximum displacement. The device shall automatically and continuously record the deflection during the test.

7.5 *Conditioning Chamber*—When conditioning materials at non-laboratory environments, a temperature/vapor-level controlled environmental conditioning chamber is required that shall be capable of maintaining the required temperature to within ±1°C [±2°F] and the required vapor level to within ±3 % relative humidity, as outlined in Test Method D5229/D5229M. Chamber conditions shall be monitored either on an automated continuous basis or on a manual basis at regular intervals.

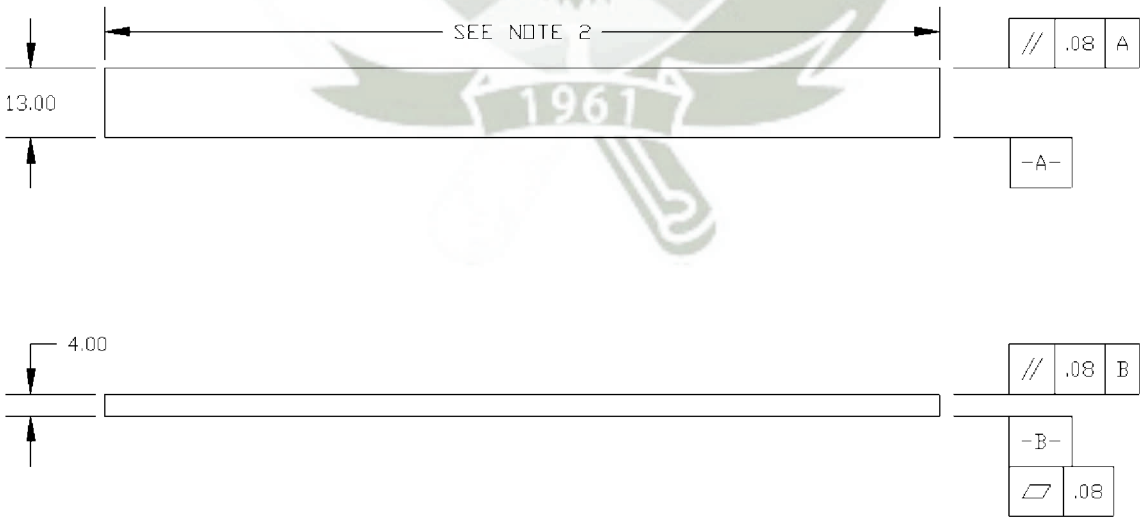
7.6 *Environmental Test Chamber*—An environmental test chamber is required for test environments other than ambient testing laboratory conditions. This chamber shall be capable of maintaining the test specimen at the required temperature within ±3°C [±5°F] and the required vapor level to within ±5 % relative humidity.

8. Test Specimens

8.1 *Specimen Preparation*—Guide D5687/D5687M provides recommended specimen preparation practices and should be followed when practical.

8.2 *Specimen Size* is chosen such that the flexural properties are determined accurately from the tests. For flexural strength, the standard support span-to-thickness ratio is chosen such that failure occurs at the outer surface of the specimens, due only to the bending moment (see Notes 2 and 3). The standard span-to-thickness ratio is 32:1, the standard specimen thickness is 4 mm [0.16 in.], and the standard specimen width is 13 mm [0.5 in.] with the specimen length being about 20 % longer than the support span. See Figs. 4 and 5 for a drawing of the standard test specimen in SI and inch-pound units, respectively. For fabric-reinforced textile composite materials, the width of the specimen shall be at least two unit cells, as defined in Guide D6856. If the standard specimen thickness cannot be obtained in a given material system, an alternate specimen thickness shall be used while maintaining the support span-to-thickness ratio [32:1] and specimen width. Optional support span-to-thickness ratios of 16:1, 20:1, 40:1, and 60:1 may also be used provided it is so noted in the report. Also, the data obtained from a test using one support span-to-thickness ratio may not be compared with the data from another test using a different support span-to-thickness ratio.

8.2.1 Shear deformations can significantly reduce the apparent modulus of highly orthotropic laminates when they are tested at low support span-to-thickness ratios. For this reason, a high support span-to-thickness ratio is recommended for flexural modulus determinations. In some cases, separate sets of specimens may have to be used for modulus and strength determination.



NOTE 1—Drawing interpretation per ANSI Y14.5-1999 and ANSI B46.1-1995.
NOTE 2—See 8.2 and 11.3 of this test standard for the required values of span and overall length.

FIG. 4 Standard Flexural Test Specimen Drawing (SI)

NOTE 1—Drawing interpretation per ANSI Y14.5-1999 and ANSI B46.1-1995.

NOTE 2—See 8.2 and 11.3 of this test standard for the required values of span and overall length.

FIG. 5 Standard Flexural Test Specimen Drawing (Inch-Pound)

NOTE 2—A support span-to-thickness ratio of less than 32:1 may be acceptable for obtaining the desired flexural failure mode when the ratio of the lower of the compressive and tensile strength to out-of-plane shear strength is less than 8, but the support span-to-thickness ratio must be increased for composite laminates having relatively low out-of-plane shear strength and relatively high in-plane tensile or compressive strength parallel to the support span.

NOTE 3—While laminate stacking sequence is not limited by this test method, significant deviations from a lay-up of nominal balance and symmetry may induce unusual test behaviors and a shift in the neutral axis.

9. Number of Test Specimens

9.1 Test at least five specimens per test condition unless valid results can be gained through the use of fewer specimens, such as in the case of a designed experiment. For statistically significant data the procedures outlined in Practice E122 should be consulted. Report the method of sampling.

10. Conditioning

10.1 The recommended pre-test specimen condition is effective moisture equilibrium at a specific relative humidity as established by Test Method D5229/D5229M; however, if the test requester does not explicitly specify a pre-test conditioning environment, conditioning is not required and the test specimens may be tested as prepared.

NOTE 4—The term *moisture*, as used in Test Method D5229/D5229M, includes not only the vapor of a liquid and its condensate, but the liquid itself in large quantities, as for immersion.

10.2 The pre-test specimen conditioning process, to include specified environmental exposure levels and resulting moisture content, shall be reported with the data.

10.3 If there is no explicit conditioning process, the conditioning process shall be reported as “unconditioned” and the moisture content as “unknown.”

11. Procedure

11.1 Condition the specimens as required. Store the specimens in the conditioned environment until test time.

11.2 Following final specimen machining and any conditioning but before testing, measure and record the specimen width and thickness at the specimen mid–section, and the specimen length, to the specified accuracy.

11.3 Measure the span accurately to the nearest 0.1 mm [0.004 in.] for spans less than 63 mm [2.5 in.] and the nearest 0.3 mm [0.012 in.] for spans greater than or equal to 63 mm [2.5 in.]. Use the measured span for all calculations. See Annex A1 for information on the determination of and setting of the span.

11.4 *Speed of Testing*—Set the speed of testing at a rate of crosshead movement of 1.0 mm/min [0.05 in./min] for a specimen with standard dimensions. For specimens with dimensions that vary greatly from the standard dimensions, a crosshead rate that will give a similar rate of straining at the outer surface can be obtained via the method outlined in Test Methods D790 for Procedure A and Test Method D6272 for Procedure B.

11.5 Align the loading nose(s) and supports so that the axes of the cylindrical surfaces are parallel. For Procedure A, the loading nose shall be midway between the supports. For Procedure B, the load span shall be one-half of the support span and symmetrically placed between the supports. The parallelism may be checked by means of plates with parallel grooves into which the loading nose(s) and supports will fit when properly aligned. Center the specimen on the supports, with the long axis of the specimen perpendicular to the loading noses and supports. See Annex A1 for setting and measuring span.

11.6 Apply the force to the specimen at the specified crosshead rate. Measure and record force-deflection data at a rate such that a minimum of 50 data points comprise the force deflection curve. (A higher sampling rate may be required to properly capture any nonlinearities or progressive failure of the specimen.) Measure deflection by a transducer under the specimen in contact with it at the center of the support span, the transducer being mounted stationary relative to the specimen supports. Do not use the measurement of the motion of the loading nose relative to the supports as this will not take into account the rotation of the specimen about the load and support noses, nor account for the compliance in the loading nose or crosshead.

11.7 *Failure Modes*—To obtain valid flexural strength, it is necessary that the specimen failure occurs on either one of its outer surfaces, without a preceding interlaminar shear failure or a crushing failure under a support or loading nose. Failure on the tension surface may be a crack while that on the compression surface may be local buckling. Buckling may be manifested as fiber micro-buckling or ply-level buckling. Ply-level buckling may result in, or be preceded by delamination of the outer ply.

11.7.1 *Failure Identification Codes*—Record the mode, area, and location of failure for each specimen. Choose a standard failure identification code based on the three-part code shown in Fig. 6. A multimode failure can be described by including each of the appropriate failure-mode codes between the parentheses of the M failure mode.

12. Validation

12.1 Values for properties at failure shall not be calculated for any specimen that breaks at some obvious, fortuitous flaw, unless such flaws constitute a variable being studied. Specimens that fail in an unacceptable failure mode shall not be included in the flexural property calculations. Retests shall be made for any specimen for which values are not calculated. If a significant fraction (>50 %) of the specimens fail in an unacceptable failure mode then the span-to-thickness ratio (for excessive shear failures) or the loading nose diameter (crushing under the loading nose) should be reexamined.

13. Calculation

NOTE 5—In determination of the calculated value of some of the properties listed in this section it is necessary to determine if the toe compensation (see Annex A2) adjustment must be made. This toe compensation correction shall be made only when it has been shown that the toe region of the curve is due to take up of the slack, alignment, or seating of the specimen and is not an authentic material response.

13.1 *Maximum Flexural Stress, Procedure A*—When a beam of homogenous, elastic material is tested in flexure as a beam simply supported at two points and loaded at the midpoint, the maximum stress at the outer surface occurs at mid-span. The stress may be calculated for any point on the load-deflection curve by the following equation (Note 6):

$$\sigma = \frac{3PL}{2bh^2} \quad (1)$$

where:

σ = stress at the outer surface at mid-span, MPa [psi],
P = applied force, N [lbf],
L = support span, mm [in.],
b = width of beam, mm [in.], and
h = thickness of beam, mm [in.].

NOTE 6—Eq 1 applies strictly to materials for which the stress is linearly proportional to strain up to the point of rupture and for which the strains are small. Since this is not always the case, a slight error will be introduced in the use of this equation. The equation will however, be valid for comparison data and specification values up to the maximum fiber strain of 2 % for specimens tested by the procedure herein described. It should be noted that the maximum ply stress may not occur at the outer surface of a multidirectional laminate.[4] Laminated beam theory must be applied to determine the maximum tensile stress at failure. Thus, Eq 1 yields an apparent strength based on homogeneous beam theory. This apparent strength is highly dependent on the ply-stacking sequence for multidirectional laminates.

13.2 *Maximum Flexural Stress, Procedure B*—When a beam of homogeneous, elastic material is tested in flexure as a beam simply supported at two outer points and loaded at two central points separated by a distance equal to ½ the support span and at equal distance from the adjacent support point, the maximum stress at the outer surface occurs between the two central loading points that define the load span (Fig. 2). The stress may be calculated for any point on the load-deflection curve by the following equation (Note 7):

$$\sigma = \frac{3PL}{4bh^2} \quad (2)$$

where:

σ = stress at the outer surface in the load span region, MPa [psi],
P = applied force, N [lbf],
L = support span, mm [in.],
b = width of beam, mm [in.], and

First Character

Failure Mode	Code
Tension	T
Compression	C
Buckling	B
interlaminar Shear	S
Multi-mode	M(xyz)
Other	O

Second Character

Failure Area	Code
At loading nose	A
Between loading noses	B
at Support nose	S
between Load and support nose	L
Unknown	U

Third Character

Failure Location	Code
Top	T
Bottom	B
Left	L
Right	R
Middle	M
Various	V
Unknown	U

FIG. 6 Flexure Test Specimen Three-Part Failure Identification Code

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[4] For the theoretical details, see Whitney, J. M., Browning, C. E., and Mair, A., "Analysis of the Flexure Test for Laminated Composite Materials," *Composite Materials: Testing and Design (Third Conference), ASTM STP 546* , 1974, pp. 30-45.

h = thickness of beam, mm [in.].

NOTE 7—The limitations defined for Eq 1 in Note 6 apply also to Eq 2.

13.3 *Flexural Strength*—The flexural strength is equal to the maximum stress at the outer surface corresponding to the peak applied force prior to failure. (for multidirectional laminates, see Note 6). It is calculated in accordance with Eq 1 and 2 by letting P equal the peak applied force.

13.4 *Flexural Stress at a Given Strain*—The maximum flexural stress at any given strain may be calculated in accordance with Eq 1 and 2 by letting P equal the applied force read from the force-deflection curve at the deflection corresponding to the desired strain (for multidirectional laminates, see Note 6). Equations for calculating strains from the measured deflection are given in 13.5 and 13.6.

13.5 *Maximum Strain, Procedure A*—The maximum strain at the outer surface also occurs at mid-span, and it may be calculated as follows:

$$\varepsilon = \frac{6\delta h}{L^2} \tag{3}$$

where:

ε = maximum strain at the outer surface, mm/mm [in./in.],
δ = mid-span deflection, mm [in.],
L = support span, mm [in.], and
h = thickness of beam, mm [in.].

13.6 *Maximum Strain, Procedure B*—The maximum strain at the outer surface also occurs at mid-span, and it may be calculated as follows:

$$\varepsilon = \frac{4.36\delta h}{L^2} \tag{4}$$

where:

δ = mid-span deflection, mm [in.],
ε = maximum strain at the outer surface, mm/mm [in./in.],
L = support span, mm [in.], and
h = thickness of beam, mm [in.].

13.7 *Flexural Modulus of Elasticity*:

13.7.1 *Flexural Chord Modulus of Elasticity*—The flexural chord modulus of elasticity is the ratio of stress range and corresponding strain range. For calculation of flexural chord modulus, the recommended strain range is 0.002 with a start point of 0.001 and an end point 0.003. If the data is not available at the exact strain range end points (as often occurs with digital data), use the closest available data point. Calculate the flexural chord modulus of elasticity from the stress-strain data using Eq 5 (for multidirectional or highly orthotropic composites, see Note 8).

$$E_f^{chord} = \frac{\Delta\sigma}{\Delta\varepsilon} \tag{5}$$

where:

E_f^{chord} = flexural chord modulus of elasticity, MPa [psi],
$\Delta\sigma$ = difference in flexural stress between the two selected strain points, MPa [psi], and
$\Delta\varepsilon$ = difference between the two selected strain points (nominally 0.002).

13.7.1.1 Report the chord modulus of elasticity in MPa [psi] for the strain range 0.001 to 0.003. If a different strain range is used in the calculations, also report the strain range used.

NOTE 8—Shear deformation can seriously reduce the apparent flexural modulus of highly orthotropic laminates when they are tested at low span-to-thickness ratios.[5] For this reason, a high span-to-thickness ratio is recommended for flexural modulus determinations. In some cases, separate sets of specimens may have to be used for modulus and strength determination.

13.7.2 *Flexural Secant Modulus of Elasticity*—The flexural secant modulus of elasticity is the ratio of stress to corresponding strain at any given point on the stress-strain curve. The flexural secant modulus is same as the flexural chord modulus in which the initial strain point is zero. It shall be expressed in MPa [psi]. It is calculated as follows (for multidirectional or highly orthotropic composites, see Note 8):

13.7.2.1 *For Procedure A*:

$$E_f^{secant} = \frac{L^3 m}{4bh^3} \tag{6}$$

where:

E_f^{secant} = flexural secant modulus of elasticity, MPa [psi],
L = support span, mm [in.],
b = width of beam, mm [in.],
h = thickness of beam, mm [in.] and
m = slope of the secant of the force-deflection curve.

13.7.2.2 *For Procedure B*:

$$E_f^{secant} = \frac{0.17L^3 m}{bh^3} \tag{7}$$

where E_f^{secant}, m, L, b, and h are the same as for Eq 6.

13.7.3 Chord modulus of elasticity shall be reported although other definitions of moduli may also be used. However, when other definitions of moduli are used, it should be clearly indicated in the report.

13.8 *Statistics*—For each series of tests calculate the average value, standard deviation, and coefficient of variation for each property determined:

$$\bar{x} = \frac{1}{n}\left(\sum_{i=1}^{n} x_i\right) \tag{8}$$

$$s_{n-1} = \sqrt{\frac{\left(\sum_{i=1}^{n} x_i^2 - n\bar{x}^2\right)}{n-1}}$$

$$CV = 100 \cdot \frac{s_{n-1}}{\bar{x}}$$

where:

$\bar{x}$ = average value or sample mean,
x_i = value of single measured or derived property,
n = number of specimens,
s_{n-1} = estimated standard deviation,
CV = coefficient of variation in percentage.

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[5] For discussion of these effects, see Zweben C., Smith, W. S., and Wardle, M. W., "Test Methods for Fiber Tensile Strength, Composite Flexural Modulus, and Properties of Fabric-Reinforced Laminates," *Composite Materials: Testing and Design (Fifth Conference), ASTM STP 674*, 1979, pp. 228-262.

14. Report

14.1 The information reported for this test method includes material identification and mechanical testing data. These data shall be reported in accordance with Guides E1309 and E1434. At a minimum, the following should be reported:

14.1.1 The revision level or date of issue of the test method used.

14.1.2 The date(s) and location(s) of the testing.

14.1.3 The name(s) of the test operator(s).

14.1.4 The test Procedure used (A or B).

14.1.5 Any variations to this test method, anomalies noticed during testing, or equipment problems occurring during testing.

14.1.6 Identification of the material tested including: material specification, material type, material designation, manufacturer, manufacturer's lot or batch number, source (if not from the manufacturer), date of certification, expiration of certification, filament diameter, tow or yarn filament count and twist, sizing, form or weave, fiber areal weight, matrix type, prepreg matrix content, and prepreg volatiles content.

14.1.7 Description of the fabrication steps used to prepare the laminate including: fabrication start date, fabrication end date, process specification, cure cycle, consolidation method, and a description of the equipment used.

14.1.8 Ply orientation stacking sequence of the laminate.

14.1.9 If requested, report density, reinforcement volume fraction, and void content test methods, specimen sampling method and geometries, test parameters, and test data.

14.1.10 Average ply thickness of the material.

14.1.11 Results of any nondestructive evaluation tests.

14.1.12 Method of preparing the test specimens, including specimen labeling scheme and method, specimen geometry, sampling method, and specimen cutting method.

14.1.13 Calibration dates and methods for all measurement and test equipment.

14.1.14 Type of test machine, grips, jaws, alignment data, and data acquisition sampling rate and equipment type.

14.1.15 Dimensions of each specimen to at least three significant figures, including specimen width, thickness, and overall length.

14.1.16 Conditioning parameters and results, and the procedure used if other than that specified in this test method.

14.1.17 Relative humidity and temperature of the testing laboratory.

14.1.18 Environment of the test machine environmental chamber (if used) and soak time at environment.

14.1.19 Number of specimens tested.

14.1.20 Load-span length, support-span length, and support span-to-thickness ratio.

14.1.21 Loading and support nose type and dimensions.

14.1.22 Speed of testing.

14.1.23 Transducer placement on the specimen, transducer type, and calibration data for each transducer used.

14.1.24 Force-deflection curves for each specimen. Note method and offset value if toe compensation was applied to force-deflection curve.

14.1.25 Tabulated data of flexural stress versus strain for each specimen.

14.1.26 Individual flexural strengths and average value, standard deviation, and coefficient of variation (in percent) for the population. Note if the failure load was less than the maximum load prior to failure.

14.1.27 Individual strains at failure and the average value, standard deviation, and coefficient of variation (in percent) for the population.

14.1.28 Strain range used for the flexural chord modulus of elasticity determination.

14.1.29 Individual values of flexural chord modulus of elasticity, and the average value, standard deviation, and coefficient of variation (in percent) for the population.

14.1.30 If an alternate definition of flexural modulus of elasticity is used in addition to chord modulus, describe the method used, the resulting correlation coefficient (if applicable), and the strain range used for the evaluation.

14.1.31 Individual values of the alternate (see above) flexural modulus of elasticity, and the average value, standard deviation, and coefficient of variation (in percent) for the population.

14.1.32 Individual maximum flexural stresses, and the average, standard deviation, and coefficient of variation (in percent) values for the population. Note any test in which the failure load was less than the maximum load before failure.

14.1.33 For flexural modulus only tests: maximum load applied, strain at maximum applied load, and calculated flexural modulus of elasticity (E_f).

14.1.34 Individual maximum flexural strains and the average, standard deviation, and coefficient of variation (in percent) values for the population. Note any test that was truncated to 2 % strain.

14.1.35 Failure mode and location of failure for each specimen.

15. Precision and Bias

15.1 *Precision*—The data required for the development of precision is not currently available for this test method.

15.2 *Bias*—Bias cannot be determined for this test method as no acceptable reference standard exists.

16. Keywords

16.1 fiber-reinforced composites; flexural properties; stiffness; strength

ANNEXES

(Mandatory Information)

A1. MEASURING AND SETTING SPAN

A1.1 For flexural fixtures that have adjustable spans, it is important that the span between the supports is maintained constant or the actual measured span is used in the calculation of flexural stress, flexural modulus and strain, and the loading noses are positioned and aligned properly with respect to the supports. Some simple steps as follows can improve the repeatability of results when using adjustable span fixtures.

A1.2 *Measurement of Span*:

A1.2.1 This technique is needed to ensure that the correct span, not an estimated span, is used in calculation of results.

A1.2.2 Scribe a permanent line or mark at the exact center of the support where the specimen makes complete contact. The type of mark depends on whether the supports are fixed or rotatable (see Figs. A1.1 and A1.2).

A1.2.3 Using a vernier caliper with pointed tips that is readable to at least 0.1 mm [0.004 in.], measure the distance between the supports, and use this measurement of span in the calculations.

A1.3 *Setting the Span and Alignment of Loading Nose(s)*—To ensure a constant day-to-day setup of the span and ensure the alignment and proper positioning of the loading nose(s), simple jigs should be manufactured for each of the standard setups used. An example of a jig found to be useful is shown in Fig. A1.3.

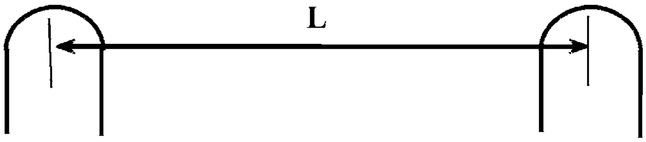

FIG. A1.1 Markings on Fixed Specimen Supports

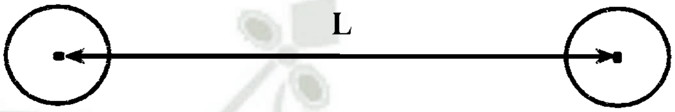

FIG. A1.2 Markings on Rotatable Specimen Supports

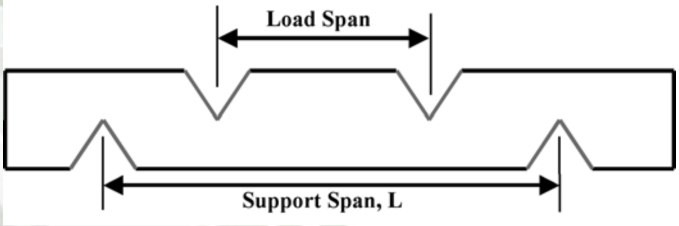

FIG. A1.3 Fixture Used to Align Loading Noses and Supports

A2. TOE COMPENSATION

A2.1 In a typical force-deflection curve (see Fig. A2.1) there is a toe region, AC, which does not represent a property of the material. It is an artifact caused by a take-up of slack and alignment, or seating of the specimen. In order to obtain correct values of such parameters as flexural modulus, and deflection at failure, this artifact must be compensated for to give the corrected zero point on the deflection, or extension axis.

A2.2 In the case of a material exhibiting a region of Hookean (linear) behavior (see Fig. A2.1), a continuation of the linear (CD) region is constructed through the zero axis. This intersection (B) is the corrected zero deflection point from which all deflections must be measured. The slope can be determined by dividing the change in force between any two points along the line CD (or its extension) by the change in deflection at the same two points (measured from Point B, defined as zero-deflection).

A2.3 In the case of a material that does not exhibit any linear region (see Fig. A2.2), the same kind of toe correction of zero-deflection point can be made by constructing a tangent to the maximum slope at the inflection Point H'. This is extended to intersect the deflection axis at Point B', the corrected zero-deflection point. Using Point B' as zero deflection, the force at any point (G') on the curve can be divided by the deflection at that point to obtain a flexural chord modulus (slope of Line B'G').

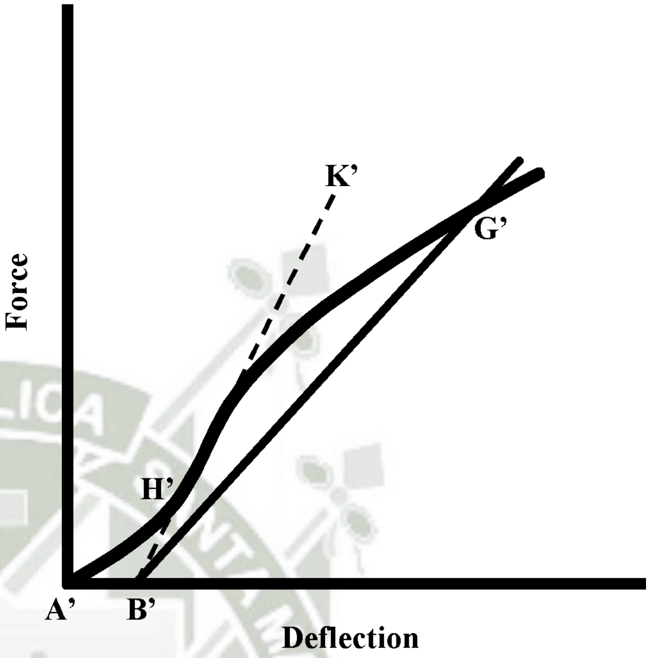

FIG. A2.2 Material without a Hookean Region

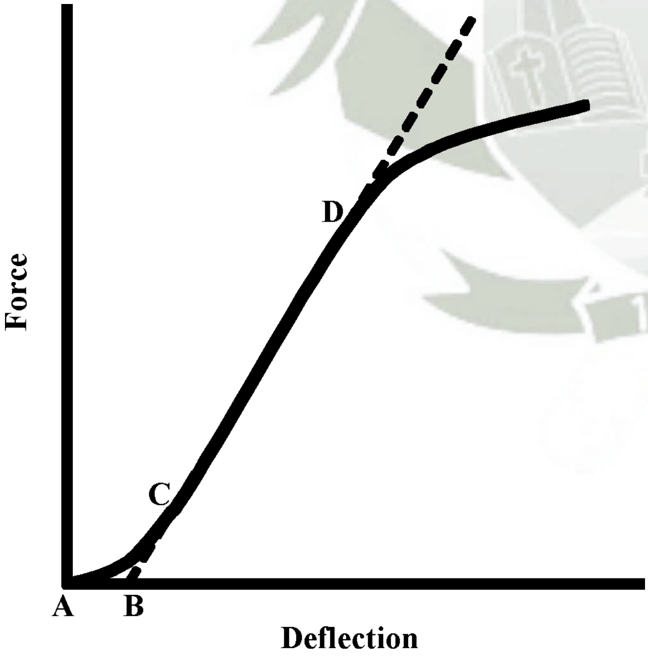

FIG. A2.1 Material with a Hookean Region

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