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Influencia de los tratamientos: alcalino, hornificación y látex con humo de sílice en la conductividad térmica de los morteros reforzados con fibras de abacá

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George Becerra

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Este trabajo se lo dedico a mis padres, a mis abuelos, a mis hermanos, por todo el apoyo que me han dado, por el amor y los ánimos que siempre me dieron para seguir adelante, por todos los consejos que me dieron, y finalmente por cuidarme siempre.

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RESUMEN

En este trabajo se tiene el propósito de analizar el comportamiento térmico del mortero reforzado con fibra de abacá con los diferentes tratamientos planteados que son: fibra tratada con hidróxido de sodio (NaOH 3%), tratada aplicando goma con humo de sílice, y pasando un proceso denominado de hornificación. Se realizará la comparativa de los tres tipos de mortero y adicionalmente con un mortero convencional para determinar el mejor comportamiento entre los tres tipos de morteros mencionados; para lo cual se realizó una muestra de cada tipo de mortero donde se aplicó a los veintiocho días el ensayo de conductividad térmica y adicionalmente se realizaron ensayos de densidad y flujo respectivamente de cada tipo de mortero. Se presentará paso a paso cada tratamiento que se realiza a la fibra de abacá y el proceso de elaboración de las muestras. El análisis de la conductividad térmica se centrará en el mortero simple versus el mortero reforzado con fibra de abacá previamente tratada con hidróxido de sodio debido que este tratamiento presenta un mejor comportamiento en el análisis termogravimétrico.

Palabras Claves: (abacá, mortero, tratamiento, hidróxido de sodio, humo de sílice, hornificación y conductividad térmica)

ABSTRACT

In this work it is intended to analyze the thermal behavior of mortar reinforced with abaca fiber with the different treatments proposed that are: fiber treated with sodium hydroxide (NaOH 3%), treated by applying rubber with silica smoke, and passing a process called baking. The three types of mortar will be benchmarked and additionally with a conventional mortar to determine the best behavior among the three types of mortars mentioned; for which a sample of each type of mortar was made where the thermal conductivity test was applied at twenty-eight days and additionally density and flow tests were carried out respectively of each type of mortar. Each treatment carried out to the abaca fiber and the sample-making process will be presented step by step. Thermal conductivity analysis will focus on simple mortar versus abaca fiber-reinforced mortar previously treated with sodium hydroxide because this treatment has better behavior in thermogravimetric analysis.

Key words: (abaca, mortar, treatment, sodium hydroxide, silica smoke, baking and thermal conductivi

INTRODUCCIÓN

Antecedentes

El ser humano en el paso del tiempo siempre ha buscado alternativas de construcción para mejorar su calidad de vida, durante siglos las fibras fueron utilizadas como un material más de construcción donde en estas últimas décadas creció su interés debido que se ha demostrado gracias a la investigación de (Freire, 2019) que las fibras vegetales mejoran las propiedades mecánicas del mortero en cantidades adecuadas, mostrando eficiencia en el control de la fisuración por tracción; sin embargo el tratamiento previo de la fibra es fundamental y varía dependiendo del tipo de tratamiento que se le dé, es necesario que la fibra pase por este proceso debido a la degradación que podría sufrir al entrar en contacto con los componentes del mortero. Partiendo de la premisa anterior se analizará el comportamiento térmico del mortero convencional y del mortero reforzado aplicando tres tratamientos, para determinar cuál presenta una resistencia térmica o tiene un mejor comportamiento al ser sometido al calor. En el trabajo de investigación de (Gómez, 2017) se realizó un análisis similar, pero con fibra de coco no tratada, los resultados presentados en este trabajo llaman la atención debido a que el mortero con fibra aporta una resistencia térmica convirtiéndolo en un material más aislante.

Objetivo general

Analizar y comparar el comportamiento térmico del mortero reforzado con fibra de abacá previamente sometida a los diferentes tratamientos (hidróxido de sodio, humo de sílice y hornificación) versus un mortero tradicional.

Objetivos específicos

- Realizar y analizar para los tipos de fibra tratada el ensayo de Análisis Termogravimétrico (TGA).
- Llevar un control del comportamiento del mortero aplicando los ensayos de flujo y densidad de cada fundición.
- Comparar resultados del mortero reforzado con fibra de abacá y sin fibra aplicando el ensayo de conductividad térmica.

Hipótesis

El mortero reforzado con fibra de abacá tratada con hidróxido de sodio tiene un comportamiento térmico diferente que el mortero simple.

Justificación

En la investigación de (Freire, 2019), basada en el comportamiento de morteros reforzados con fibra de abacá para mampostería elaborado bajo la norma INEN (Instituto Ecuatoriano de Normalización, 2015), se determinó que la fibra aporta una mejora en sus propiedades mecánicas y se determinó que el tratamiento de hidróxido de sodio es el más efectivo, que evita la degradación de la fibra. Posteriormente se reflejó en los resultados del trabajo de investigación de (Cubillo & Chasiguasín, 2020) luego que se aplicará los ciclos de envejecimiento. Adicionalmente se obtuvo de la investigación de (Calle, 2019); el tamaño y la dosificación óptima de la fibra que como base de esta nueva investigación para determinar la resistencia térmica del mortero reforzado con fibra de abacá debido que es un parámetro interesante para tomar en cuenta según (Cotrina, 2019).

En la actualidad la preocupación por el estado del planeta ha crecido y se buscan maneras de producir dinero sin dañar el medio ambiente, esta investigación será un aporte positivo debido que la fibra de abacá puede ser aprovechada en el sector de la construcción sin crear un daño ambiental y mejorar las propiedades mecánicas del mortero demostrado en la investigación de (Freire, 2019).

CAPÍTULO I

1. MARCO TEÓRICO

Este apartado, se centrará en la presentación teórica del proyecto, los temas se dividen en bases del mortero y aplicación de la fibra de abacá previamente tratada por hidróxido de sodio, humo de sílice y hornificación. Adicionalmente se dará una revisión bibliográfica del método de placa caliente para el análisis térmico del mortero.

1.1.Mortero

El mortero es un elemento utilizado en el área de la construcción, principalmente en la mampostería para unir los bloques de las paredes y como revestimiento de estas que ayudan a protegerlas del agente climático. Este material está compuesto fundamentalmente de agregado fino(arena), agua y cemento, adicionalmente según (Cubillo & Chasiguasín, 2020) se puede agregar aditivos que ayudan a su maleabilidad, y como un plus se puede reforzar con fibras sintéticas o naturales, que mejoran sus propiedades mecánicas.

1.2.Propiedades del mortero

Según el (INSTITUTO ECUATORIANO DE NORMALIZACIÓN, 2010),el mortero se presenta en dos estados importantes que son: plástico y endurecido, cada estado debe cumplir con propiedades específicas para que se considere un mortero óptimo.

1.2.1. Mortero en estado plástico

En este estado las propiedades del mortero determinan su trabajabilidad en la construcción que facilita su aplicación en la mampostería, adicionalmente mide la retención de agua, el flujo y el tiempo de fraguado según (INSTITUTO ECUATORIANO DE NORMALIZACIÓN, 2010).

1.2.2. Mortero en estado endurecido

En estado endurecido se determina el comportamiento posterior de la mampostería donde se puede analizar la adherencia, la durabilidad, la resistencia a compresión y la elasticidad (INSTITUTO ECUATORIANO DE NORMALIZACIÓN, 2010).

1.3. Agua

El agua es de los componentes principales del mortero donde su relación con respecto al cemento es fundamental para obtener las características óptimas de nuestra mezcla dado que puede afectar directamente a sus propiedades como su resistencia y trabajabilidad, este componente debe cumplir lo que dice la norma NTE INEN 2518 (INSTITUTO ECUATORIANO DE NORMALIZACIÓN, 2010), nos dice que ¨El agua debe ser limpia y estar libre de aceites, ácidos, álcalis, sales, materiales orgánicos, u otras substancias que sean perjudicial es para los morteros o para cualquier metal en la pared¨(p.3).

1.4. Arena

Este componente del mortero es un material granular fino y el único material granular de la mezcla, es el responsable que le de ese color gris al mortero. (Cubillo & Chasiguasín, 2020) nos dice que agregado fino se puede obtener principalmente de canteras o ríos donde tienen sus diferencias debido a que la arena de río es más redondeada que la de cantera y se deposita al final de este. La arena utilizada en esta investigación es recolectada del Río Boliche ubicado en la provincia del Guayas, respetando la norma ASTM C 144 – 04 se presenta la siguiente caracterización.

Tabla 1 Granulometría de la arena utilizada en la investigación.

TAMIZ	PESO RETENIDO	% RETENIDO	%RETENIDO ACUMULADO	% PASANTE
4 (4,75 mm)	0	0%	0%	100%
8 (2,36 mm)	40,5	3%	3%	98%
16 (1,18 mm)	218,7	14%	16%	84%
30 (600 μm)	461,7	29%	45%	56%
50 (300 μm)	534,6	33%	77%	23%
100 (150 μm)	226,8	14%	91%	9%
200 (75 μm)	97,2	6%	97%	3%
FONDO	40,5	3%	100%	0%
TOTAL	1620			

Nota: Fuente: (Cubillo & Chasiguasín, 2020)

1.5. Cemento

El cemento es un material que al estar en contacto con el agua o la humedad del ambiente tiene una reacción química que genera calor, se endurece como un pegamento y tiene una buena resistencia después de 28 días, a esto se le conoce como pasta de cemento y al agregar la arena se denomina mortero. El cemento que se utilizara en la investigación es el cemento hidráulico tipo GU de (Holcim Ecuador S.A., 2018), este cemento está diseñado para todo tipo de construcción y cumple sobresalientemente con todos los estándares de la norma NTE INEN 2380 (INSTITUTO ECUATORIANO DE NORMALIZACIÓN, 2012).

En la siguiente ilustración se muestran los estándares de la norma NTE INEN 2380, donde Holcim Fuerte tipo GU corresponde a la categoría M.

REQUISITO	TIPOS DE CEMENTO PARA MORTERO		
	N	S	M
Finura, retenido sobre el tamiz de 45 µm (No. 325), % máximo.	24	24	24
Expansión en autoclave, % máximo	1,0	1,0	1,0
Tiempo de fraguado por el método de Gillmore: Fraguado inicial, minutos, no menor a Fraguado inicial, minutos, no mayor a	 120 1 000	 90 1 000	 90 1 000
Resistencia a la compresión, (promedio de tres cubos): Resistencia a la compresión de cubos de mortero, elaborado en volumen, de 1 parte de cemento y 3 partes de mezcla de arenas (50% de arena graduada normalizada y 50% de arena normalizada 20 – 30) preparados y ensayados de acuerdo con esta norma, debe ser igual o mayor que los valores especificados para las edades indicadas a continuación: 7 días, MPa. 28 días, MPa.	 3,5 6,2	 9,0 14,5	 12,4 20,0
Resistencia de adherencia en flexión. 28 días, mínimo, MPa	 0,5	 0,7	 0,8
Contenido de aire del mortero: % Mínimo, en volumen % Máximo, en volumen	 8 17	 8 15	 8 15
Retención de agua, % mínimo respecto al flujo original	70	70	70

Ilustración 1 Requisitos físicos del cemento para mampostería

Fuente: (INSTITUTO ECUATORIANO DE NORMALIZACIÓN, 2012)

1.6.Fibras vegetales

Las fibras vegetales se utilizaban mucho antes del descubrimiento de muchos materiales de construcción que persisten en la actualidad. Debido a la preocupación por el medio ambiente que se da en esta época, por ello se han buscado maneras de mejorar la eficiencia y las propiedades de los materiales de construcción, las fibras vegetales han llamado la atención en estos últimos años según (Kosmatka et al., 2004) por su abundancia en el país y por su bajo costo se ha realizado investigaciones para ser utilizada como un refuerzo en la construcción.

1.7.Abacá

El cáñamo de manila (musa textiles) o mayormente conocido como abacá es una planta herbácea, (Freire, 2019) nos dice que se confunde con el banano debido a que es similar en su apariencia, pero sus propiedades y usos son diferentes. Esta planta mayormente se produce en la costa ecuatoriana debido a las características agro- ecológicas especiales que esta requiere, en el mercado se puede obtener con diferentes niveles de calidad y resistencias. Según (Páez, 2007) el abacá se puede utilizar para fabricar redes de pesca por su gran resistencia al agua salada, adicionalmente sirve para la elaboración de filtros de maquinaria, textiles para hospitales, papel de seguridad, cables de conducción eléctrica y muchos productos más.

1.8.Fibras de abacá

Las fibras de abacá tienen unas propiedades muy interesantes, que han llamado la atención en los investigadores del país debido a su dureza, flexibilidad y bajo costo. La fibra de abacá en el Ecuador se produce mayormente en usos artesanales, pero actualmente gracias a la investigación de (Freire, 2019) para darle un uso en la construcción aprovechando al máximo sus propiedades mecánicas, por ello este trabajo la utilizara como un refuerzo al mortero de mampostería, las fibras que se utilizara tienen 3 cm de longitud con un porcentaje en la mezcla del 0.2 con respeto a la arena y el cemento basándose en el trabajo de investigación de (Calle, 2019).

1.9.Tratamientos

La fibra de abacá tiene buenas propiedades mecánicas, pero sufre con el tiempo una degradación al estar en contacto con el cemento demostrado en la investigación de (Alcívar, 2010), afectando a los beneficios mecánicos que aportaba al mortero inicialmente, por ello es necesario aplicar un tratamiento previo y para este estudio se han aplicado tres tipos de tratamientos cada uno único que aporta una mejora mecánica a la fibra, pero en el trabajo de (Calle, 2019); se determinó que el tratamiento de hidróxido de sodio es el que arroja mejores resultados en los ensayos planteados en esta investigación. En la obtenidos 2 se muestra los beneficios de los tres tratamientos utilizados en la investigación, obtenidos en investigaciones previas en diferentes tipos de fibra.

Tabla 2 Tratamientos utilizados en la investigación.

Fibra	Tipo de Tratamiento	Beneficios
Coco	Solución adhesiva: látex natural, revestimiento: humo de sílice	Presento un esfuerzo a flexión 42,2% más alto que la fibra sin tratar y 73,4% más alto que sin fibra. También demostró el mejor módulo elástico, con un aumento de 80,5% a los 28 días (Silva, y otros, 2017).
Sisal, Yute, Curaua	Hornificación	La resistencia a la tracción de las fibras naturales aumentó. Con el tratamiento de hornificación fue posible alcanzar un mayor enlace matriz-fibra. (Ferreira, Silva, Lima, & Toledo Filho, 2017)
Hoja	Hidróxido de sodio (NaOH)	Mejoran las propiedades mecánicas. Ofrece un óptimo aislamiento térmico (Jiang et al.,2018).

Nota: Fuente: (Freire, 2019)

1.10. Hidróxido de sodio

Este tratamiento alcalino usando hidróxido de sodio (NaOH) es el mejor procedimiento para mejorar las propiedades mecánicas de la fibra. El hidróxido de sodio se presenta en su estado sólido es altamente peligroso según el (Consejo Colombiano de Seguridad, 2005) al estar en contacto con la humedad o peor aún ser sumergido en el agua debido a que genera calor y genera un gas que tóxico que puede dañar los pulmones al ser inhalado, y si se expone a la piel puede provocar quemaduras, por ello su venta es restringida y solo se puede manipular con todos los equipos de protección requeridos.

Tabla 3 Tratamiento de hidróxido de sodio aplicado a diferentes fibras naturales.

FIBRA	TIPO DE TRATAMIENTO	BENEFICIOS
Hoja	Hidróxido de sodio (NaOH)	Mejoran las propiedades mecánicas. Ofrece un óptimo aislamiento térmico (Jiang et al., 2018).
Abacá		Con un 5% de solución se ven mejoras de un 8% en el esfuerzo a tracción y un 38% en el módulo de Young. (Cai et al., 2016)
Lino		Ayuda a controlar el tamaño y el número de grietas. Demuestra mejora en la resistencia a tracción y en el módulo de Young (Snoeck et al., 2015)

Nota: Fuente: (Calle, 2019)

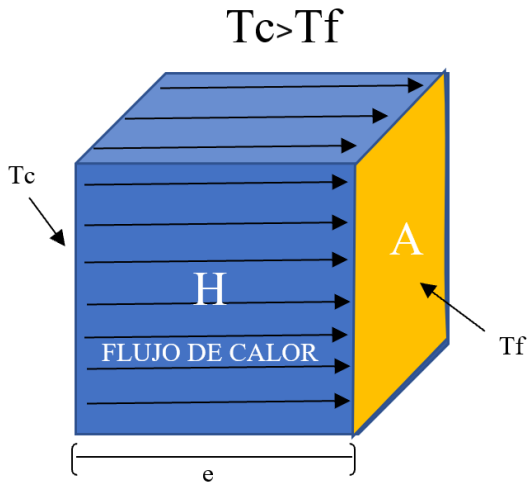
1.11. Método analítico para análisis térmico

El método que se utilizará en esta investigación para la determinación de la conductividad térmica de las muestras es mediante la aplicación del método de placa caliente de la norma ASTM-C-177 o ISO 8302, que emplea la ley de calentamiento de Newton.

1.12. Determinación de la conductividad térmica

La conductividad térmica es una propiedad física que determina la capacidad de los materiales en conducir calor y entre menor sea esa capacidad el material será más aislante al calor. Según (Gómez, 2017) la conductividad térmica en otras palabras es la resistencia de las moléculas de los materiales al calor y depende de la composición del material, por ejemplo, si el material es afectado por la humedad y otros factores puede variar su resistencia inicial.

$$\frac{\Delta Q}{\Delta T} = KA\frac{Tc - Tf}{e}$$



Donde

$\Delta Q = calor\ tranferido\ en\ el\ intervalo\ de\ tiempo\ \Delta t$

$Tc = temperatura\ del\ foco\ caliente$

$Tf = temperatura\ del\ foco\ frio$

$A = Area\ tranversal$

$e = espesor\ de\ la\ lámina$

$k = constante\ de$ conductividad térmica

Ilustración 2 Esquema representativo del flujo de calor a través de una superficie.

Nota: Fuente: Autor

En la ilustración 2 se puede ver un cubo compuesto de un material cualquiera donde se quiere determinar su resistencia térmica, para ello el foco de temperatura elevada se transmite en una cara de este por una cantidad de calor y tiempo específicos, el flujo de calor (H) atravesará el espesor(e) del bloque hacia la otra cara donde se encuentra el foco de menor temperatura. Este proceso se basa en la ecuación de la Ley de Fourier donde determinando ¨k¨ obtendremos la constante de conductividad térmica del material analizado.

1.13. Método de Placa Caliente (ASTM-C-177)

Este método mide principalmente materiales aislantes y de alta resistencia térmica, se recomienda para materiales no homogéneos; aislantes de baja densidad como fibras también para materiales con baja o intermedia conductividad térmica como; minerales, vidrios, cerámicas, fibras minerales, adicionalmente para morteros, hormigones y materiales completos que consisten en diferentes componentes. La norma (ASTM C177-13, 2013) nos dice que el método consiste en aplicar calor a la muestra mediante una placa que se encontrara en una cara de la muestra donde se genera un flujo de calor unidimensional en el espesor y otra placa fría en el lado opuesto que toma la temperatura receptada que determina su resistencia térmica tomando en cuenta el tiempo, área de la cara y su espesor. El (INSTITUTO DE INVESTIGACIÓN GEOLÓGICO Y ENERGÉTICO, 2020) nos dice que el rango medible de conductividad térmica es de 0,002 W/m-K hasta 3 W/m-K y las muestran varían de tamaño dependiendo el tipo de material, para el tipo aislante el ensayo requiere muestras de gran tamaño, donde las medidas son 500 mm de ancho, 500 mm de longitud y con un espesor mínimo de 12 mm hasta 100 mm, para no aislantes sus medidas óptimas son de un cubo de 150 mm en sus 3 dimensiones. La temperatura media de este ensayo es de 10°C a 40°C.

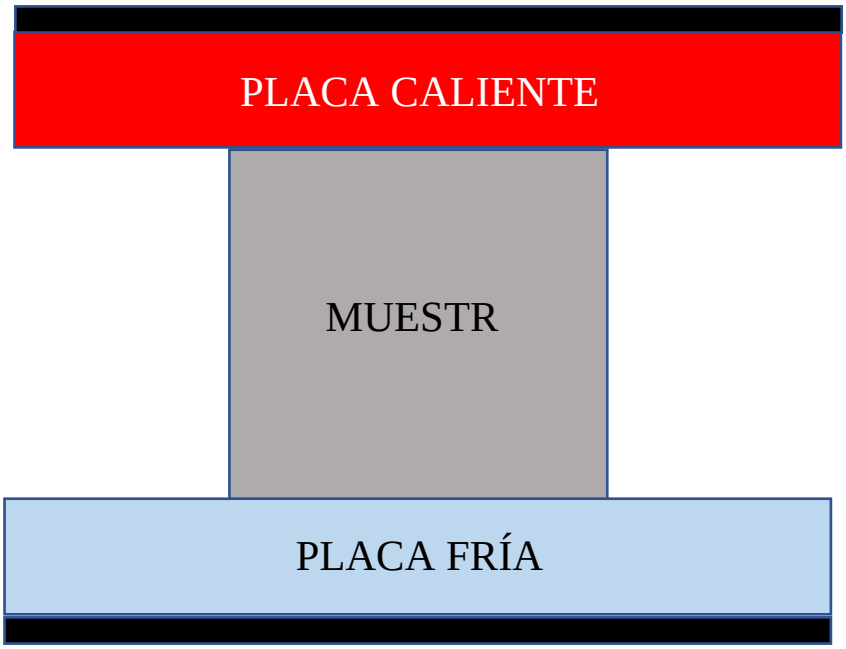


Ilustración 3 Esquema del método de ensayo.

Nota: Fuente: Autor

CAPÍTULO II

2. METODOLOGÍA DEL TRABAJO DE INVESTIGACIÓN

El siguiente trabajo de investigación se lo ha dividido en los siguientes capítulos:

1. Introducción

2. Marco teórico

3. Metodología del trabajo de investigación

4. Elaboración de las muestras

5. Ensayos y resultados

6. Conclusiones y recomendaciones

Se recalca que el trabajo se centrará en analizar el comportamiento térmico del mortero reforzado con fibra de abacá previamente tratada con Hidróxido de Sodio (NaOH) versus el mortero convencional. Se elabora una metodología para este trabajo de investigación con el fin de cumplir los objetivos planteados y confirmar la hipótesis planteada previamente es correcta.

2.1. Primer Período (Revisión Bibliográfica)

Todo trabajo de investigación pasa por una previa revisión bibliográfica que sirven para aclarar posibles dudas con respecto al objetivo principal del trabajo, por ello en esta primera etapa se investigó la mejor metodología para determinar el coeficiente térmico de nuestro mortero reforzado con fibra donde se encontró que el mejor método es el ensayo de Placa Caliente (ASTM-C-177) debido que este ensayo es recomendado para mortero, hormigón o materiales que estén conformados por diferentes componentes.

El proceso de este trabajo se realizó gracias al aporte económico de la facultad de ingeniería civil de la Universidad Católica Santiago de Guayaquil ,y a Holcim Ecuador S.a. en el departamento de Centro de Innovación Holcim(CIH) brindándonos con las instalaciones y equipos necesarios para la elaboración de las muestras para el ensayo de conductividad térmica, adicionalmente con el material necesario para la investigación e información necesaria para la realización del ensayo Placa Caliente(ASTM-C-177).

Continuando con investigaciones previas nos enfocaremos en la metodología propuesta por (Calle, 2019) donde determinó que el porcentaje óptimo de fibra para el refuerzo del mortero para mampostería es del 0,2% de la masa total del cemento y arena, también determinó la longitud óptima que debe tener la fibra para que tenga un mejor comportamiento que es de 3 cm.

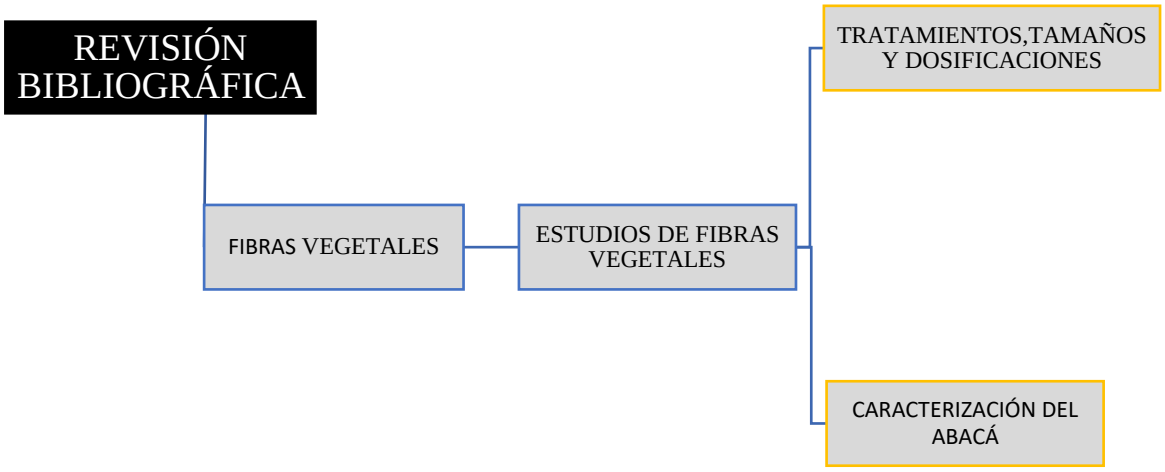


Ilustración 4 Metodología de la revisión bibliográfica.

Nota: Fuente: Autor

2.2.Segundo Período (Experimental)

Este segundo período que corresponde la parte experimental, la cual se subdivide en fases de la siguiente manera:

Fase I: Se tomó como guía la investigación de (Calle, 2019) donde se obtuvo la dosificación del mortero reforzado con fibra de abacá, debido que presentó un comportamiento mecánico óptimo. Con el trabajo de (Freire, 2019) se procedió al realizar los tres tratamientos a la fibra de abacá donde se les realizó ensayos TGA.

Fase II: En esta fase se realizaron las muestras de morteros reforzados con fibra de abacá, pero de cada tratamiento planteado, adicionalmente se realizó una muestra de mortero simple. Se procedió a realizar ensayos de flujo, densidad para cada muestra y finalmente se realiza el ensayo de conductividad térmica a las muestras.

Este proyecto de investigación se enfoca en la segunda etapa que es experimental, por ello se debe seguir un proceso ordenado para obtener resultados de los ensayos de conductividad térmica de buena calidad. En la ilustración 5 se detalla el proceso de este período:

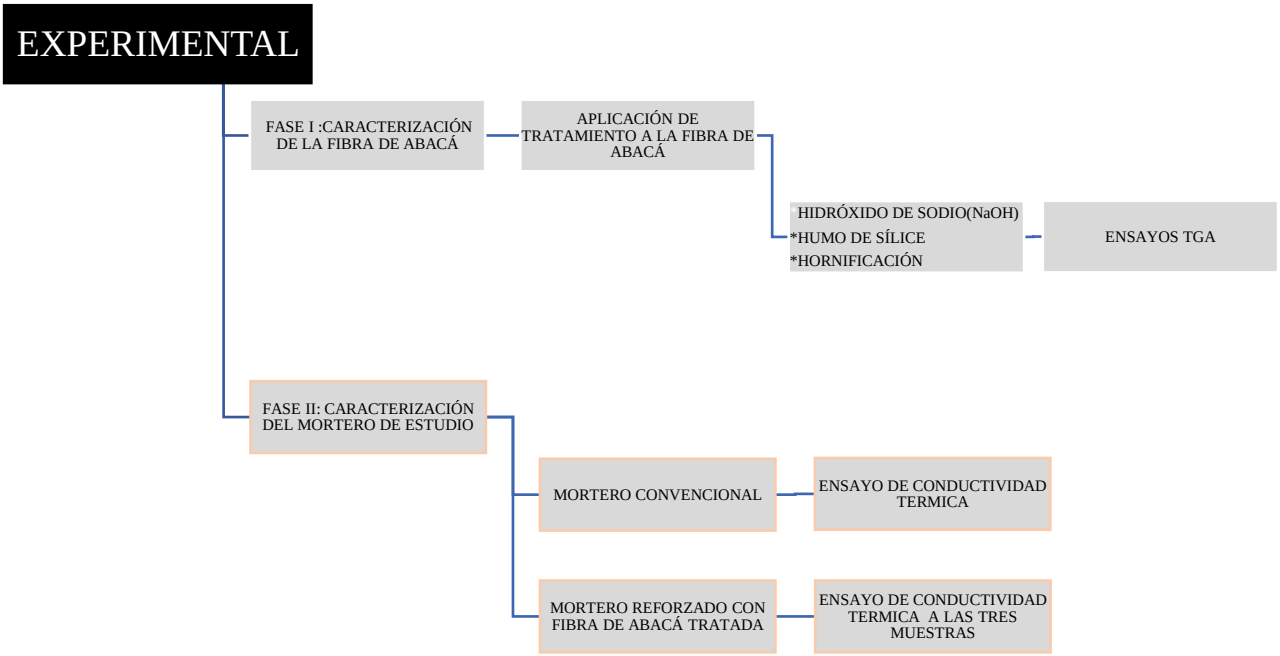


Ilustración 5 Metodología de trabajo para la fase experimental.

Nota: Fuente: Autor

2.3.Fase I

Tomando como guía el trabajo de investigación de (Freire, 2019) para la caracterización de la fibra de abacá, luego se aplicó los tres tratamientos planteados para la investigación de manera minuciosa para obtener los mejores resultados, el tratamiento de hidróxido de sodio (NaOH) es el proceso más complejo debido que es peligroso, por ello se pidió la ayuda de un ingeniero del departamento de Centro de Innovación de Holcim Ecuador S.a. para la manipulación de este compuesto, se procedió con el tratamiento de hornificación cumpliendo los ciclos secado a la perfección, y finalmente se concluyó con el tratamiento de Humo de sílice que es el más sencillo de todos pero el que más tiempo toma debido que se trata fibra por fibra

para obtener mejores resultados. Al finalizar los tratamientos a la fibra de abacá se entregaron para que se le realicen los ensayos TGA, con esto se finalizó la primera fase.

2.4.Fase II

La caracterización del mortero reforzado con fibra de abacá se obtuvo de la investigación realizada por (Cubillo & Chasiguasín, 2020) , con la dosificación ya planteada, se preparó el material necesario para la elaboración de las muestras. Con los moldes ya listos se procedió con la fundición del mortero simple, luego se realizaron las fundiciones de los morteros reforzados con fibra de los tres tratamientos planteados, en total se elaboraron cuatro muestras. Para tener un control de calidad en cada fundición se realizaron ensayos de densidad, y flujo que según NTE INEN 1806 en un rango de 110% ± 5% previamente establecida por (Cubillo & Chasiguasín, 2020). Las muestras se desmoldan un día después e inmediatamente se sumergen en las piscinas de curado por 28 días y luego entregadas al INSTITUTO DE INVESTIGACIÓN GEOLÓGICO Y ENERGÉTICO para que se realicen los ensayos de conductividad térmica aplicando el método de la Placa Caliente (ASTM-C-177).

CAPÍTULO III

3. ELABORACIÓN DE MUESTRAS

Durante el desarrollo de este capítulo se explicará de manera detallada el procedimiento de la elaboración de las muestras de mortero a las que posteriormente se les realizará el ensayo de conductividad térmica.

Este trabajo de investigación desea realizar una comparación en el comportamiento térmico del mortero simple versus el mortero reforzado con fibra tratada, se planteó tres tratamientos de fibra y se realizarán muestras para cada uno, la investigación se enfocará en la fibra tratada con hidróxido de sodio(NaOH) al 3% debido que por estudios previos este tratamiento es el que mejora las propiedades mecánicas de la fibra dando mejores resultados en su comportamiento con el tiempo ya que es el más efectivo en evitar la degradación de la misma.

- **Muestra de mortero reforzado:** este mortero esta reforzado con fibra de abacá previamente tratada, con una dosificación establecida en la investigación de (Cubillo & Chasiguasín, 2020), se realizará una muestra para cada tratamiento planteado.
- **Muestra de mortero simple(patrón):** se realiza esta muestra para tener un control y realizar una comparativa, este mortero debe cumplir la norma NTE INEN 1806 y no agregar ningún componente que mejore sus propiedades o su trabajabilidad.

3.1. Preparación de fibra de abacá

La fibra que se utiliza en toda la investigación es de segundo orden que representa una fibra de buena calidad y es de exportación, esta fibra es utilizada en investigaciones previas como (Calle, 2019) (Cubillo & Chasiguasín, 2020).

3.2. Corte de fibra

Gracias a la investigación de (Calle, 2019) se determinó el tamaño óptimo de la fibra de abacá que debe tener una longitud de 3 cm, las muestras tienen este corte ya establecido.

3.3. Tratamientos a la fibra de abacá

Obteniendo la información necesaria del trabajo de investigación de (Freire, 2019) que planteó los tratamientos de hornificación, humo de sílice, y por último el de hidróxido de sodio (NaOH) que fue el que concluyó como el óptimo. En este trabajo se aplicaron los mismos tratamientos y se determinó que el mortero reforzado con fibra de abacá previamente tratada con hidróxido de sodio tuvo el mejor comportamiento en sus propiedades mecánicas.

3.3.1. Tratamiento de hidróxido de sodio (NaOH)

La fibra de abacá cortada a 3 cm para tener un comportamiento óptimo, paso por este proceso alcalino que consiste en hacer una solución de agua con hidróxido de sodio donde el hidróxido de sodio corresponde al 3% de la masa total del agua, en este caso basándonos en el trabajo de investigación de (Cubillo & Chasiguasín, 2020) se usó 5820 gr de agua y 180 gr de hidróxido de sodio para elaborar esta solución. Para el tratamiento del NaOH se realizaron en orden los siguientes puntos:

- Pesar el agua en un recipiente grande y preferiblemente resistente debido a que el NaOH es altamente volátil y podría causar daños si el recipiente es plástico con una capa muy delgada.

Ilustración 6 Pesado de agua de 5820 gr en el recipiente de la solución.

Nota: Fuente: Autor

- Con el equipo adecuado pesar y disolver el hidróxido de sodio hasta que no se aprecien las perlas de NaOH, se utilizó una mesa magnética para este paso como se muestra en la Ilustración 7.

Ilustración 7 Mesa magnética utilizada para disolución del NaOH.

Nota: Fuente: Autor

- Pesar 350 gr de fibra y colocarla en la solución hasta que esté sumergida completamente por la misma solución.

Ilustración 8 Pesado de la fibra de abacá.

Nota: Fuente: Autor

*Ilu*stración 9 Colocando la fibra de abacá en la solución.

Nota: Fuente: Autor

- Dejar la fibra sumergida durante 4 horas en un sector aislado de personas, en este caso se colocó en un horno apagado, pero completamente cerrado.

Ilustración 10 Se colocó el recipiente en un horno aislado para evitar algún accidente.

Nota: Fuente: Autor

- Luego de dejar reposar por 4 horas se retira la solución en un recipiente y luego se realizan lavados a la fibra que son alrededor de 7 lavados, luego todo el líquido se guarda en el recipiente inicial y luego se debe agregar la misma cantidad en vinagre para estabilizar el PH.

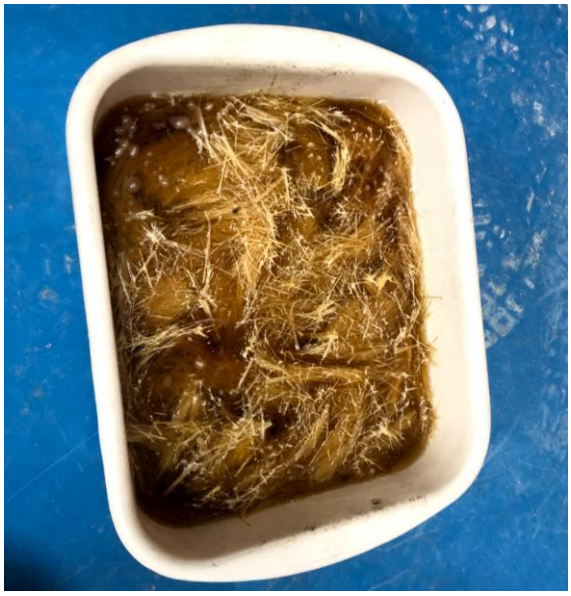


Ilustración 11 Recipiente antes de aplicar los lavados.

Nota: Fuente: Autor

Ilustración 12 Recipiente luego de aplicar los lavados.

Nota: Fuente: Autor

- Luego de extraer el agua del recipiente, colocamos la fibra en un recipiente de metal y lo ubicamos en un horno eléctrico ventilado por 24 horas a una temperatura de 85°C+-1°C.

Ilustración 13 Colocamos la fibra en un recipiente metálico para ubicarlo en el horno.

Nota: Fuente: Autor

- Finalmente dejamos en el horno apagado por 30 minutos para evitar un choque electroestático en la fibra, al terminar este tiempo colocamos la fibra en una funda con cierre hermético para que no absorba humedad.

Ilustración 14 Se coloca la fibra en una funda de cierre hermético.

Nota: Fuente: Autor

3.3.2. Tratamiento con Humo de Sílice

Este tratamiento se toma de igual manera del trabajo de investigación de (Freire, 2019) que se basa en la utilización de material adhesivo y un material puzolánico que proteja a la fibra contra el ataque alcalino. Originalmente la solución adherente fue látex natural en polvo con agua, pero fue reemplazado por goma porque era muy complicada su manipulación y retrasaba el tratamiento. Para este tratamiento se realizaron en orden los siguientes puntos:

- Se peso 208,45 gr de agua y 235 gr de goma que equivale al 53% de la mezcla total, mezclamos los dos componentes para crear nuestra solución adherente.

Ilustración 15 Se pesan los dos componentes necesarios para mezclarlos y tener la solución adherente.

Nota: Fuente: Autor

- Luego se debe meter en el horno por 2 horas la fibra de abacá cortada a 3cm a una temperatura de 100°C.

Ilustración 16 Se deja la fibra en el horno a 100°C por 2 horas.

Nota: Fuente: Autor

- Se procede a remojar la fibra en la solución adherente por un minuto y luego cubrirlas con el humo de sílice para finalmente guardarlas en una funda con cierre hermético.

Ilustración 17 Se cubre de humo de sílice la fibra previamente sumergida en el adhesivo.

Nota: Fuente: Autor

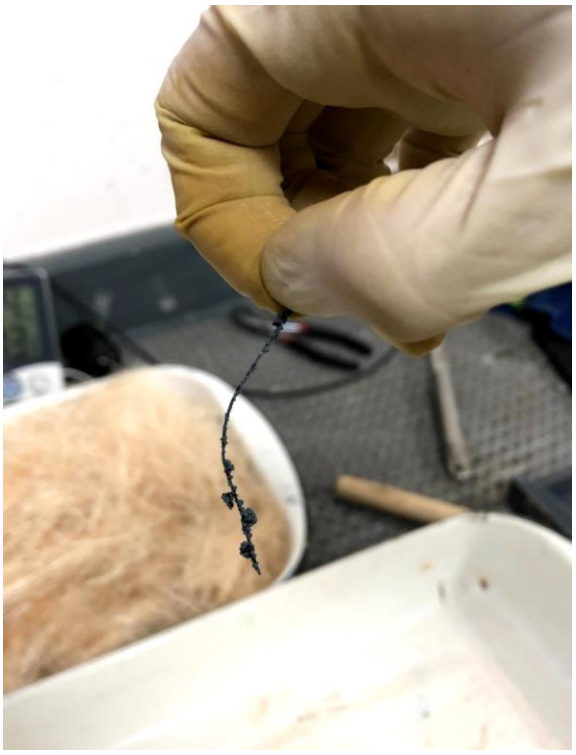


Ilustración 18 El procedimiento se debe hacer fibra por fibra y así es el resultado.

Nota: Fuente: Autor

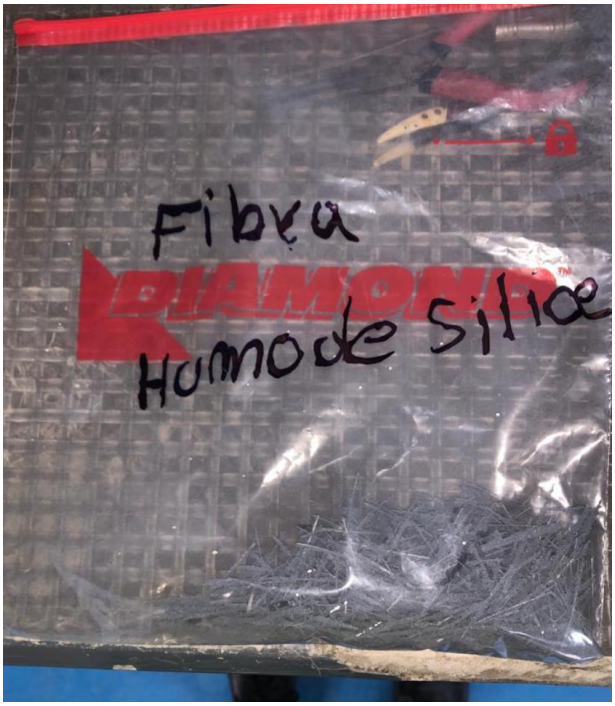


Ilustración 19 Se coloca la fibra en una funda con cierre hermético.

Nota: Fuente: Autor

3.3.3. Tratamiento de Hornificación

De igual manera este tratamiento es extraído del proyecto de investigación de (Freire, 2019), es el tratamiento más sencillo pero el de mayor duración debido que se deben realizar 3 ciclos de hidratación y secado. Para este tratamiento se realizaron en orden los siguientes puntos:

- Sumergir la fibra en un recipiente con agua durante 3 horas que debe estar a una temperatura de 22°C.

Ilustración 20 Fibra sumergida en agua que tiene una temperatura de 22°C.

Nota: Fuente: Autor

- Luego se retira la fibra en un recipiente de metal para posteriormente meter a un horno con una temperatura de 80°C durante 16 horas.

Ilustración 21 Se coloca la fibra en el horno a una temperatura de 80°C.

Nota: Fuente: Autor

- Los dos puntos anteriores se deben repetir 2 veces más y luego guardar la fibra en una funda hermética.

Ilustración 22 Se guarda la fibra tratada en una funda hermética.

Nota: Fuente: Autor

3.4.Preparación de las muestras

3.4.1. Materiales

Para la elaboración de las muestras se utilizaron las siguientes cantidades basándonos en la dosificación del trabajo de investigación de (Cubillo & Chasiguasín, 2020)

- Arena =5.47kg, donde su caracterización se presenta en la siguiente tabla:

Tabla 4 Caracterización de la arena para la muestra.

TAMIZ	MASA (gr)
8 (2,36 mm)	229,5
16 (1,18 mm)	831
30 (600 μm)	1650,5
50 (300 μm)	1896,5
100 (150 μm)	858
TOTAL	5465,5

Nota: Fuente: Autor

- Cemento =1.8kg
- Agua =1.1kg
- Fibra de abacá tratada = 14.53 gr

Se recuerda que se elaborarán 3 muestras con el tratamiento a la fibra abacá que son: de hidróxido de sodio, de humo de sílice y de hornificación. Las 3 muestras respetaran la dosificación que se planteó y adicionalmente se realizará una muestra que servirá como patrón respetando la dosificación planteada, pero sin fibra de abacá.

3.4.2. Amasado

Al tener preparado los pesos de los materiales que se utilizaran, se procede a realizar el amasado de la muestra que está explicado en orden en los siguientes puntos:

- Humedecer el recipiente que se utilizará para realizar el amasado de la muestra.

Ilustración 23 Se humedece con agua el recipiente donde se realizará el amasado.

Nota: Fuente: Autor

- Se vierte la arena en el recipiente y procede a mezclar por 30 segundos.

Ilustración 24 Se mezcla la arena para que sea uniforme la caracterización.

Nota: Fuente: Autor

- Verter el cemento en el recipiente y mezclar con la arena por 30 segundos más.

Ilustración 25 Se vierte el cemento en el recipiente.

Nota: Fuente: Autor

- Verter la mitad del agua pesada y mientras mezclamos arrojamos la fibra, este procedimiento tomará 30 segundos.

Ilustración 26 Se arroja la fibra a la mezcla.

Nota: Fuente: Autor

- Paramos durante 30 segundos para retirar el mortero adherido en las paredes laterales y al final vertemos el agua faltante para continuar con la mezcla.

Ilustración 27 Se vierte la otra mitad del agua en la mezcla.

Nota: Fuente: Autor

- Mezclamos por un minuto y estaría lista la muestra para los ensayos de flujo y densidad y su fundición.

Ilustración 28 Mezclamos durante un minuto y estará lista la muestra.

Nota: Fuente: Autor

3.4.3. Molde

Para el ensayo de la conductividad térmica, las muestras deben ser entregadas con las medidas de un cubo de 15cmX15cmX15cm, donde el Centro de Innovación Holcim proveyó con estos moldes de acero que cumplen con las medidas requeridas para el ensayo.

Ilustración 29 Moldes para ensayo de conductividad térmica.

Nota: Fuente: Autor

3.4.4. Fundición de muestras

La elaboración de las muestras es similar al procedimiento de la toma de cilindros 2 a 1, después del amasado se procede a la fundición del mortero en los moldes donde se siguió en orden los siguientes puntos:

- Armar el molde.
- Engrasar el molde con aceite.
- Se funde una capa a la mitad del molde, con 25 golpes en la capa con la varilla y 8 golpes laterales en espiral con el chapulín.

Ilustración 30 Proceso de elaboración de muestras, primera capa.

Nota: Fuente: Autor

- Se lo ubica al molde en la mesa de vibración para extraer el aire, este proceso se realiza en 45 segundos a máxima potencia de esta mesa.
- Se repite este proceso en la última capa.
- Con el bailejo se enrasa el material en la superficie.

Ilustración 31 Muestras enrasadas y terminadas.

Nota: Fuente: Autor

- Desmoldar a las 24 horas, se limpian los moldes y se repite el proceso hasta obtener todas las muestras.

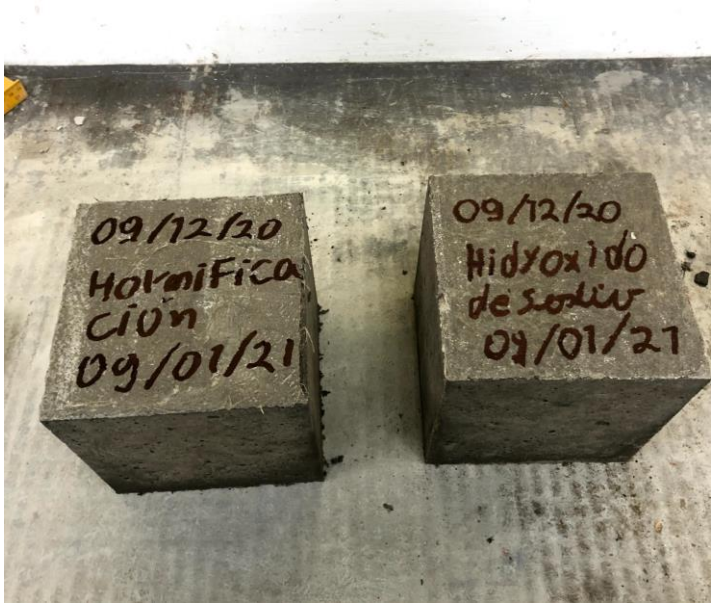


Ilustración 32 Se desmoldan las muestras y se limpian los moldes.

Nota: Fuente: Autor

Todas las muestras después de ser desmoldadas se colocan inmediatamente en la piscina de curado hasta que cumplan 28 días.

Ilustración 33 Muestras sumergidas en piscina de curado.

Nota: Fuente: Autor

CAPÍTULO IV

4. ENSAYOS Y RESULTADOS

4.1. Nomenclatura de los resultados

En esta investigación se utilizará la siguiente nomenclatura para una mejor comprensión:

- Tratamiento
- Dosificación
- Muestras para ensayo de conductividad térmica

Tabla 5 Nomenclatura - Tratamientos

TRATAMIENTO	NOMENCLATURA
HIDRÓXIDO DE SODIO	THS
GOMA + HUMO DE SÍLICE	TGH
HORNIFICACIÓN	THR

Nota: Fuente: Autor

Tabla 6 Nomenclatura - Dosificación

DOSIFICACIÓN	NOMENCLATURA
3 CM LONGITUD DE FIBRA,0,2% (14,5 GR)	3/0,2

Nota: Fuente: Autor

Tabla 7 Nomenclatura – Muestras analizadas

MUESTRAS	NOMENCLATURA
MORTERO REFORZADO CON FIBRA DE ABACÁ PREVIAMENTE TRATADA CON HIDRÓXIDO DE SODIO(NAOH).	MTHS
MORTERO REFORZADO CON FIBRA DE ABACÁ PREVIAMENTE TRATADA CON GOMA + HUMO DE SÍLICE.	MTGH
MORTERO REFORZADO CON FIBRA DE ABACÁ PREVIAMENTE TRATADA CON EL PROCESO DE HORNIFICACIÓN.	MTHR
MORTERO SIMPLE O PATRÓN.	MP

Nota: Fuente: Autor

4.2.Fase 1: Ensayos previos a la elaboración de las muestras

4.2.1. Ensayo de TGA

(Dacoat-Chemicals , 2020) , nos dice que el análisis termogravimétrico (TGA) es una técnica, donde se somete a un material a un proceso de descomposición en función de la temperatura y el tiempo, el ensayo consta en calentar el material en un horno y se va midiendo su peso a medida que es calentado. Al terminar este proceso de descomposición obtendremos diferentes perfiles del proceso que sufrió el material al ser calentado. Gracias a la ayuda de Holcim Ecuador S.A. se realizó el ensayo de termogravimétrico a la fibra de abacá tratada por Hornificación y Humo de sílice, se agregaron los resultados de la fibra de abacá en estado natural y tratada por hidróxido de sodio (NaOH) del trabajo de investigación de (Calle, 2019) para realizar una comparación completa de la variación de cada tratamiento. Para el tratamiento de Humo de sílice y de Hornificación se sometieron 2 muestras de cada una, y para la comparación se agregaron 2 muestras de Hidróxido de sodio y 2 muestras en estado natural. Se analizarán dos curvas, de temperatura y la pérdida de peso en porcentaje.

Al analizar la gráfica de tiempo versus temperara, se puede observar que la fibra natural, la fibra TGH y THR van subiendo de manera gradual, pero en el caso de la fibra natural llega a la máxima temperatura 4 horas después, mientras que el otro caso la fibra THS sufre una subida abrupta de temperatura en la mitad del tiempo que la fibra TGH Y THR, lo que demuestra que las fibras TGH y THR mantienen en menor proporción sus componentes naturales como: la lignina, celulosa, hemicelulosa. En cambio, en la fibra THS es afectada principalmente por el hidróxido de sodio al ser un álcali fuerte, debido a esto, pierde por completo estos componentes.

En la curva de pérdida de peso (%), nos indica la descomposición de la fibra, la fibra THS presenta una gráfica gradual y en los 33 minutos se descompone por completo, en cambio la curva THR es irregular y similar a la fibra en estado natural, pero se descompone por completo a los 30 minutos del ensayo y la fibra natural 2 horas después de esta. La fibra THR es la más llamativa debido que su comportamiento es similar al de la fibra en estado natural. Lo que demuestran estos resultados es que el tratamiento de hidróxido de sodio elimina por completo los componentes de la fibra natural que ayudan a la degradación de esta en el paso del tiempo, los tratamientos de

hornificación y humo sílice no eliminan por completo estos componentes por lo que no son tratamientos completamente efectivos para evitar la degradación de la fibra.

4.2.1.1. Fibra de abacá (natural)

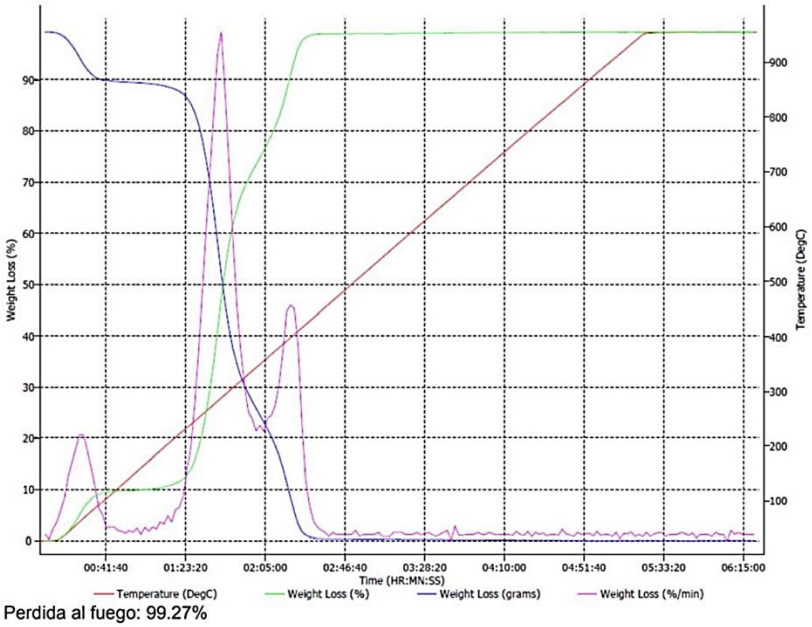


Ilustración 34 Resultado de ensayo TGA, fibra de abacá en estado natural (fibra 1).

Nota: Fuente: (Holcim Ecuador S.A., 2018)

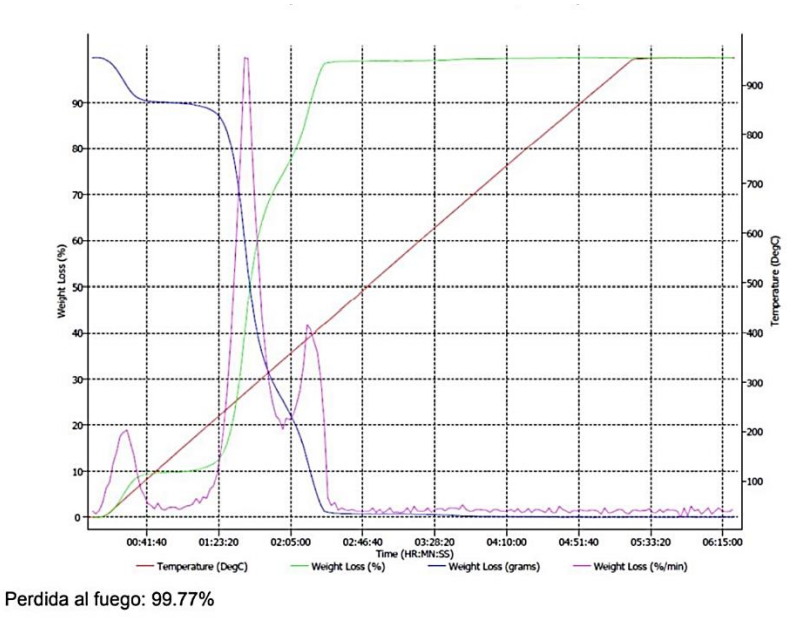


Ilustración 35 Resultado de ensayo TGA, fibra de abacá en estado natural (fibra 2).

Nota: Fuente: (Holcim Ecuador S.A., 2018)

4.2.1.2. Fibra de abacá tratada con Hidróxido de Sodio (NaOH)

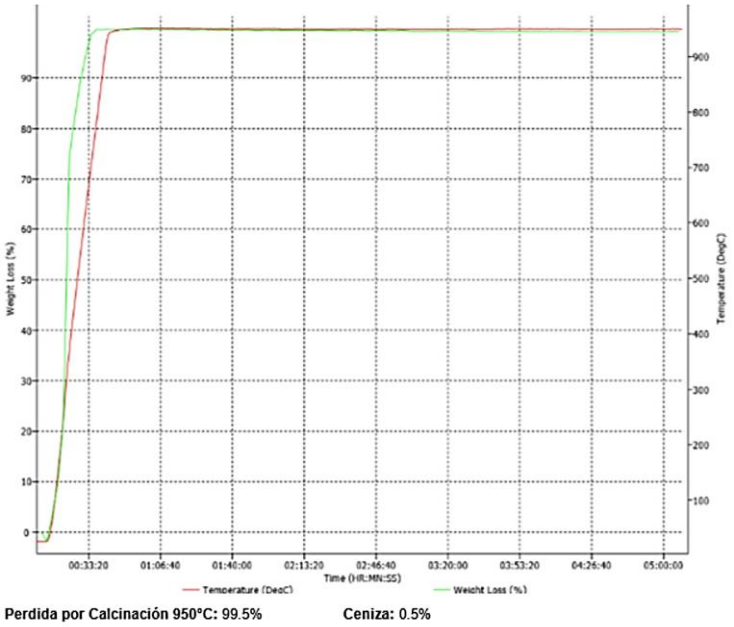


Ilustración 36 Resultado de ensayo TGA, fibra de abacá tratada con NaOH (fibra 3).

Nota: Fuente: (Holcim Ecuador S.A., 2018)

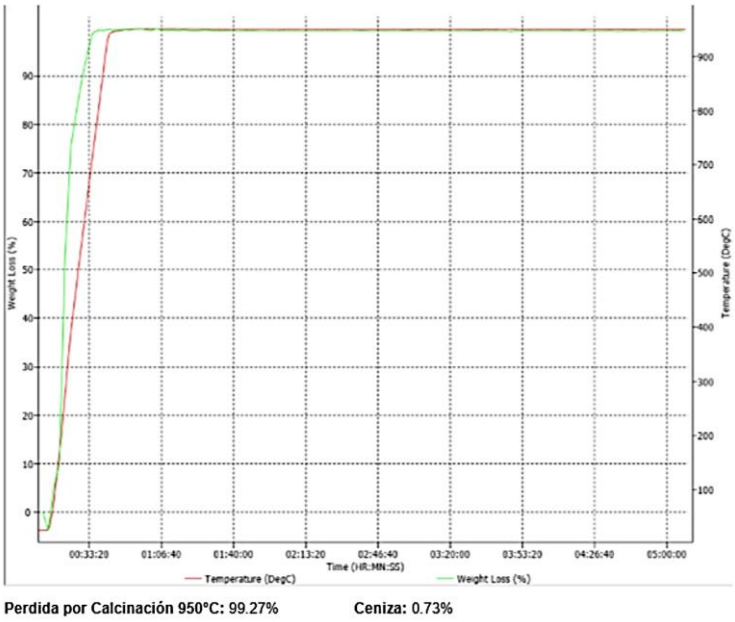


Ilustración 37 Resultado de ensayo TGA, fibra de abacá tratada con NaOH (fibra 4).

Nota: Fuente: (Holcim Ecuador S.A., 2018)

4.2.1.3. Fibra tratada con Hornificación

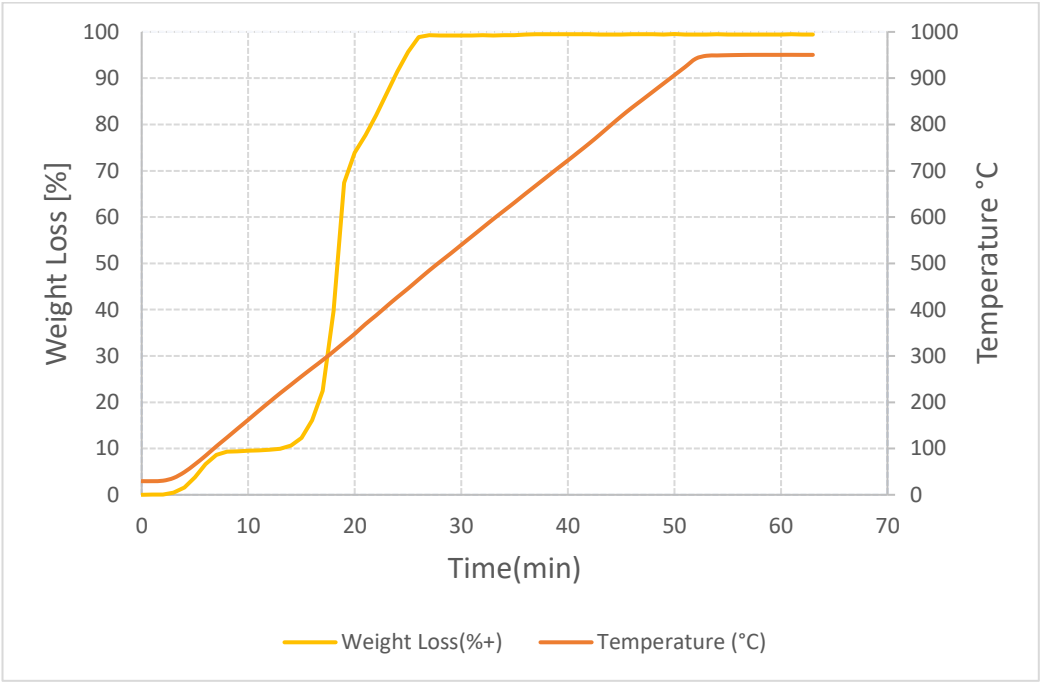


Ilustración 38 Resultado de ensayo TGA, fibra de abacá tratada con hornificación (fibra 5).

Nota: Fuente: (Holcim Ecuador S.A, 2021)

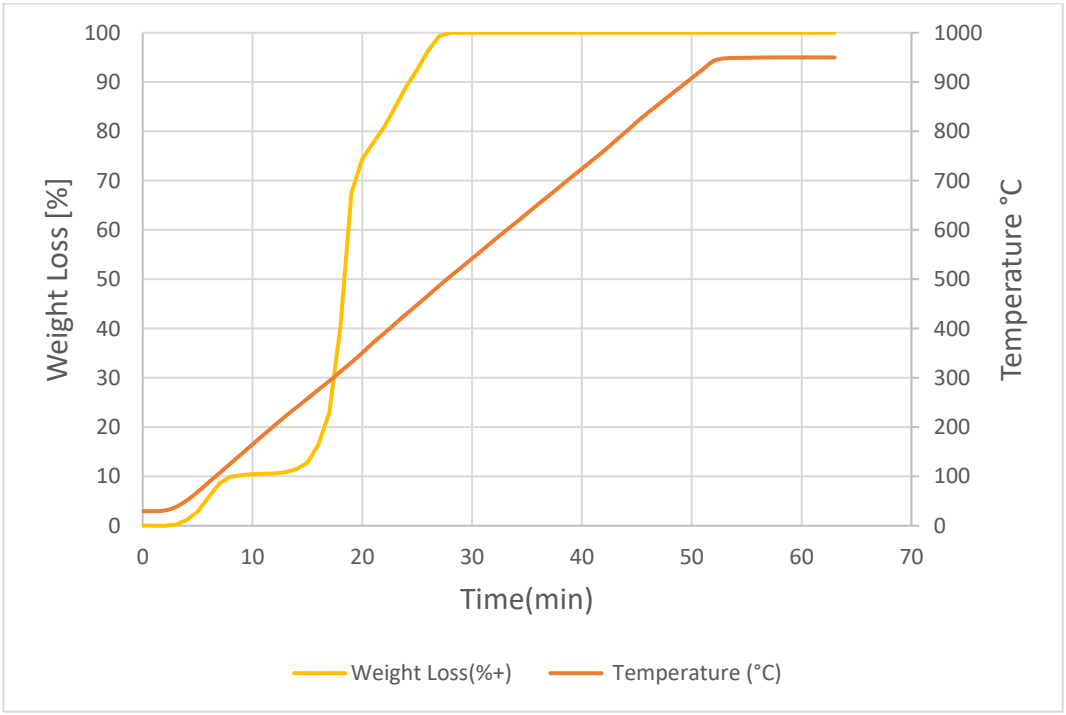


Ilustración 39 Resultado de ensayo TGA, fibra de abacá tratada con hornificación (fibra 6).

Nota: Fuente: (Holcim Ecuador S.A, 2021)

4.2.1.4. Fibra tratada con Humo de sílice

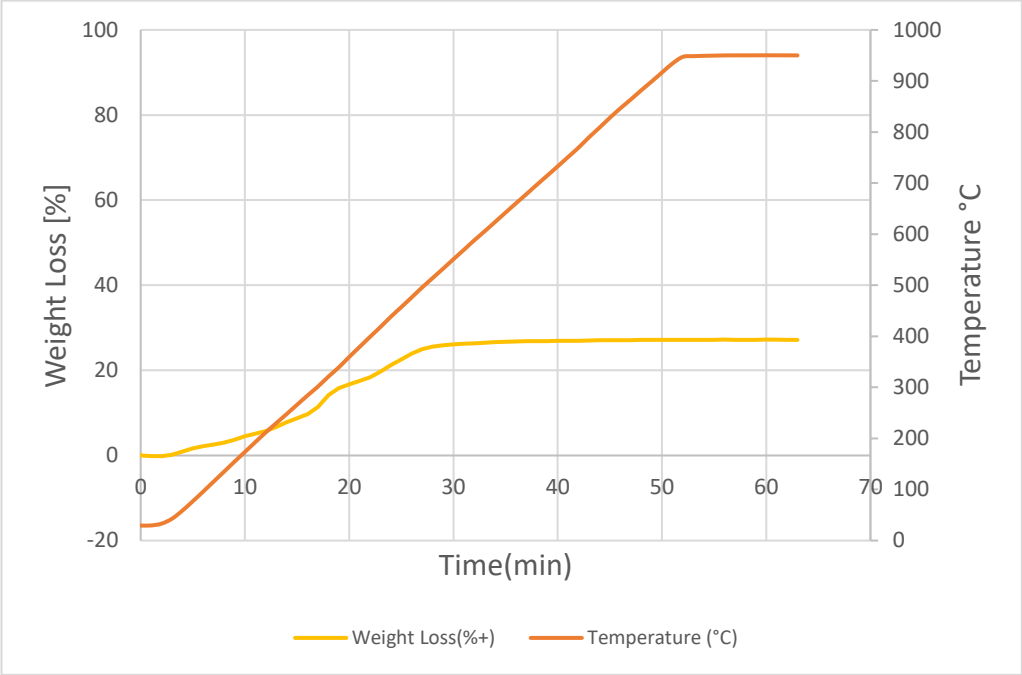


Ilustración 40 Resultado de ensayo TGA, fibra de abacá tratada con humo de sílice (fibra 7).

Nota: Fuente: (Holcim Ecuador S.A, 2021)

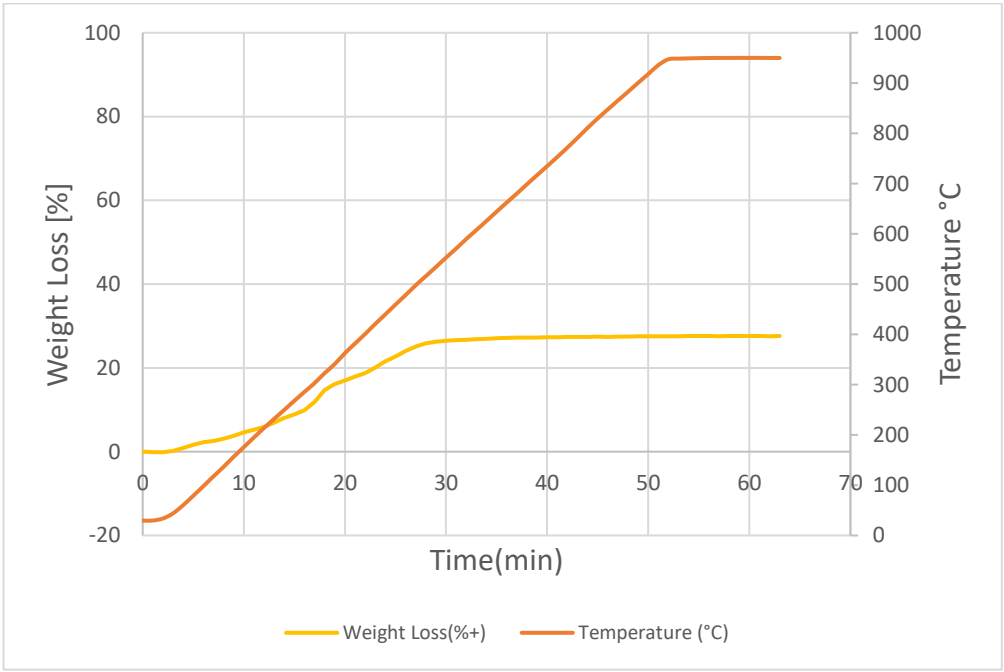


Ilustración 41 Resultado de ensayo TGA, fibra de abacá tratada con humo de sílice (fibra 8).

Nota: Fuente: (Holcim Ecuador S.A, 2021)

4.3. Fase 2: Ensayos realizados a las muestras de inicio y al final de su elaboración

4.3.1. Flujo

Este ensayo se realizó durante la mezcla de cada muestra debido que el mortero debe estar en estado fresco, se determina la cantidad de agua que contiene el mortero para así definir su consistencia. El flujo que se busca obtener para cada una de las muestras será de 110% ± 5% según lo indica el (INSTITUTO ECUATORIANO DE NORMALIZACIÓN, 2010). Este ensayo se basó en la norma (UNE-EN 1015-3, 2000) la cual indica el procedimiento del ensayo y los materiales necesarios en esta prueba.

Los materiales necesarios para el ensayo son los siguientes:

- Batidora eléctrica
- Paleta
- Cuchara
- Molde troncocónico donde (Cubillo & Chasiguasín, 2020),nos dice que "acero inoxidable o de latón de 60 mm ± 0,5 mm de altura, con un diámetro interior de 100 mm ± 0,5 mm en la base y de 70 mm ± 0,5 mm en la parte superior. La superficie interior y los bordes del molde están pulidos" (p.52). El espesor mínimo de la pared del molde es de 2,0 mm (INSTITUTO ECUATORIANO DE NORMALIZACIÓN, 2010).
- Pisón no absorbente con una masa ideal entre 0,250 kg ± 0,015 kg.
- Mesa de golpes
- Un calibre capaz de medir diámetros de hasta 300 mm.

Las cantidades de arena, cemento y fibra son las mismas que se utilizan en la elaboración de las muestras.

El proceso es el siguiente:

Se recomienda que la mesa de golpes no debe estar en funcionamiento dentro de un lapso de 24 horas y adicionalmente se deben realizar 10 sacudidas previo al inicio del ensayo.

- Tener lista la mezcla de la muestra mediante la batidora eléctrica.

Ilustración 42 Mezcla elaborada con batidora eléctrica.

Nota: Fuente: Autor

- Limpiar la mesa de golpes y el molde troncocónico con un pañuelo húmedo.

Ilustración 43 Limpieza de mesa de golpes y el molde troncocónico.

Nota: Fuente: Autor

- Se aplican dos capas de mortero acompañada de 10 golpes cada capa.

Ilustración 44 Aplicación de golpes a la primera capa de mortero.

Nota: Fuente: Autor

- Se enrasa el molde y se quita cualquier sobrante.

Ilustración 45 Enrasado de molde para ensayo de flujo.

Nota: Fuente: Autor

- Se retira el molde e inmediatamente se procede a aplicar 25 golpes de la mesa.

Ilustración 46 Aplicación de 25 golpes para ensayo de flujo.

Nota: Fuente: Autor

- Finalmente se procede a tomar las 4 medidas con el calibre.

Ilustración 47 Toma de mediciones para determinar el flujo de la mezcla.

Nota: Fuente: Autor

Tabla 8 Resultados de flujo de las muestras de mortero.

RESULTADOS DE FLUJO

MORTERO	CEMENTO	ARENA	AGUA	FIBRA	D1	D2	D3	D4	FLUJO
MP	1,8 KG	5,47 KG	1,10KG	0 gr	28	29	29	29	115
MTHS	1,8 KG	5,47 KG	1,11KG	14,53 gr	29	29	29	29	116
MTGH	1,8 KG	5,47 KG	1,11KG	14,53 gr	29	28	29	29	115
MTHR	1,8 KG	5,47 KG	1,11KG	14,53 gr	29	29	30	28	116

Nota: Fuente: Autor

4.3.2. Densidad del mortero en estado fresco

Este ensayo se lo realiza al instante que se termine el mezclado de la muestra para determinar como referencia el contenido de aire del mortero, ya que a menor densidad tiene más contenido de aire en otras palabras, el mortero tendrá una mayor cantidad de poros. Este ensayo está basado en la norma a NTE INEN 195 (INSTITUTO ECUATORIANO DE NORMALIZACIÓN, 2016) y este proceso se explica en los siguientes puntos:

- Se realiza el mezclado del mortero basado a la NTE INEN 155 (INSTITUTO ECUATORIANO DE NORMALIZACIÓN, 2010).

Ilustración 48 Mezclado de mortero respetando la norma.

Nota: Fuente: Autor

- Usando una cuchara se procede a llenar el recipiente de 400 cm3 en tres capas iguales, compactar cada capa 20 veces alrededor de la superficie interior del recipiente. Al terminar se debe llenar con un exceso de aproximadamente 20 mm para luego eliminar el aire atrapado por el proceso de llenado con el mazo dando ligeros golpes en 5 puntos diferentes con espaciado aproximadamente igual. (Cubillo & Chasiguasín, 2020,p.55)

Ilustración 49 Aplicación de golpes para eliminar el aire atrapado.

Nota: Fuente: Autor

- Enrasar el recipiente y luego determinar la masa del mortero

Ilustración 50 Determinación de la masa del mortero.

Nota: Fuente: Autor

Para determinar la densidad del mortero en estado fresco se basará en la expresión que utilizo (Cubillo & Chasiguasín, 2020) en su trabajo de investigación:

$$\rho = \frac{M}{V}$$

Donde:

- ρ= densidad del mortero(fresco), kg/m³.
- M= masa del mortero(fresco), kg.
- V= Volumen del recipiente, m³.

Tabla 9 Resultados de densidad de las muestras analizadas.

RESULTADOS DE DENSIDAD DE CADA BLOQUE

MORTERO	CEMENTO	ARENA	AGUA	FIBRA	DENSIDAD (kg/m3)
MP	1,8 KG	5,47 KG	1,10KG	0 gr	2189,13
MTHS	1,8 KG	5,47 KG	1,11KG	14,53 gr	2158,23
MTGH	1,8 KG	5,47 KG	1,11KG	14,53 gr	2175,43
MTHR	1,8 KG	5,47 KG	1,11KG	14,53 gr	2185,55

Nota: Fuente: Autor

4.3.3. Ensayo de conductividad térmica por método de Placa Caliente (ASTM-C-177).

En la Ilustración 51 se observa gráficamente los resultados de conductividad térmica realizados a las 4 muestras planteadas. El ensayo consistió en aplicar calor mediante una placa en un extremo de la muestra, y luego medir la temperatura en el otro extremo con una placa fría que detecta la variación de la temperatura en un periodo de tiempo establecido. La temperatura media del ensayo fue de 23°C y la diferencia de temperatura fue de 30°C.

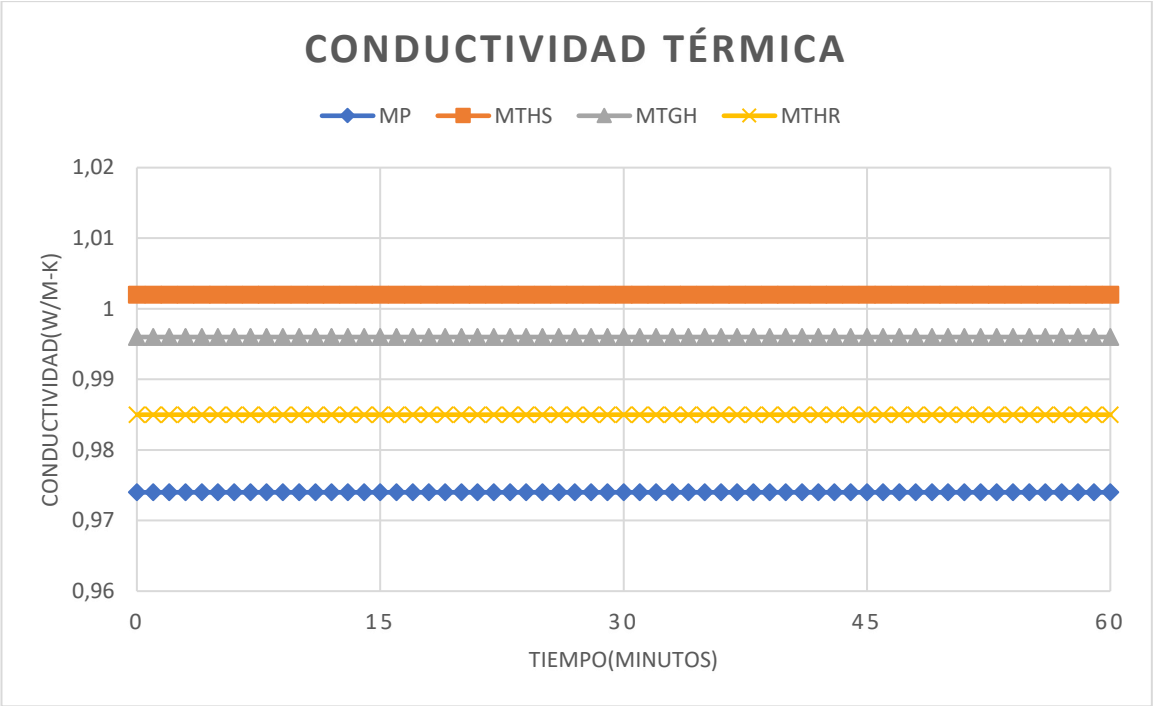


Ilustración 51 Resultados de la conductividad térmica de las muestras.

Nota: Fuente: Autor

Se puede observar Tabla 10 los resultados finales del ensayo y mediante un registro aportado por el (INSTITUTO DE INVESTIGACIÓN GEOLÓGICO Y ENERGÉTICO, 2020) para comparar el resultado de la muestra MP con morteros de similar densidad, siendo muy cercanos los resultados se puede decir, que el ensayo y la preparación de las muestras se realizaron de manera correcta.

Tabla 10 Resultados de conductividad térmica.

Muestras	Conductividad Térmica (W/(m-K))
MP	0,974
MTHS	1,002
MTGH	0,996
MTHR	0,985

Nota: Fuente: Autor

4.3.4. Análisis de muestras MP y MTHS

Estas dos muestras son las principales de la investigación y de donde se basarán las conclusiones.Principalmente se determinó su densidad en estado endurecido, ambas son cercanas pero la muestra A1(MP) es ligeramente superior que la muestra A2(MTHS) y de igual manera se refleja en su peso. En la Ilustración 52 podemos observar gráficamente los resultados de conductividad térmica y podemos determinar que la muestra A1(MP) es menor por ello tiene una mayor resistencia al calor, por lo que se considera un material más aislante que la muestra A2(MTHS).

Tabla 11 Condiciones y descripción de las muestras.

Código	Largo (mm)	Ancho (mm)	Espesor (mm)	Peso(g)	Densidad (kg/m3)	Fecha de recepción	Otros (composición)
2102001	152	153	151,5	7561,1	2141,3	5/2/2021	A1. Convencional
2102002	152	154	152	7541,6	2128,9	5/2/2021	A2. con fibra

Nota: Fuente: (INSTITUTO DE INVESTIGACIÓN GEOLÓGICO Y ENERGÉTICO, 2020)

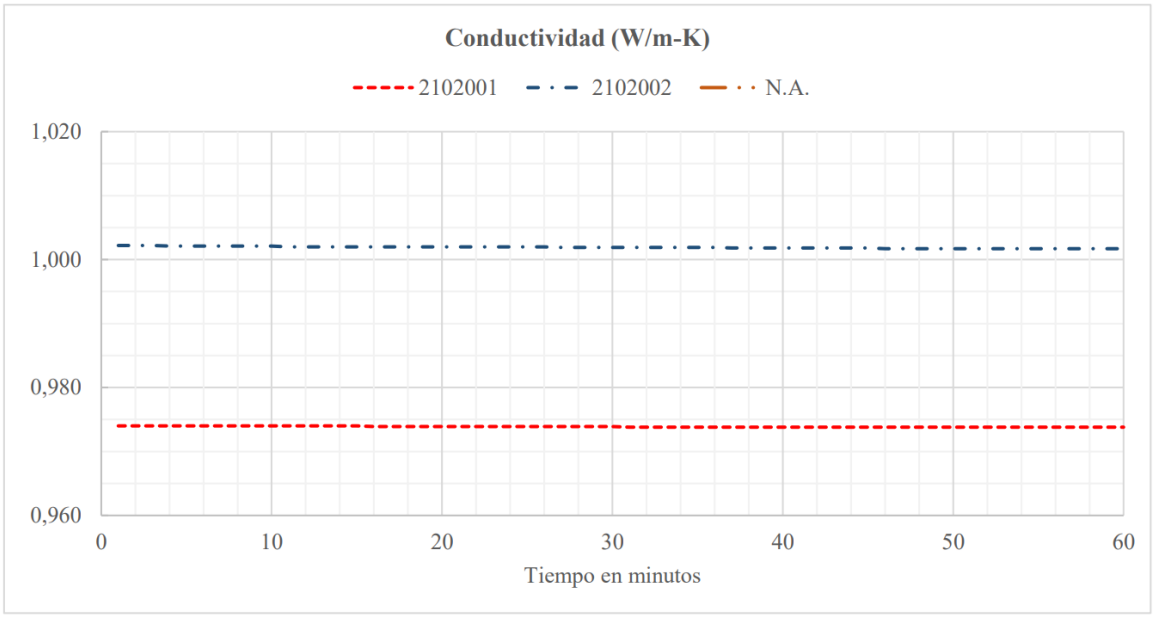


Ilustración 52 Resultados de muestras MP y MTHS.

Nota: Fuente: (INSTITUTO DE INVESTIGACIÓN GEOLÓGICO Y ENERGÉTICO, 2020)

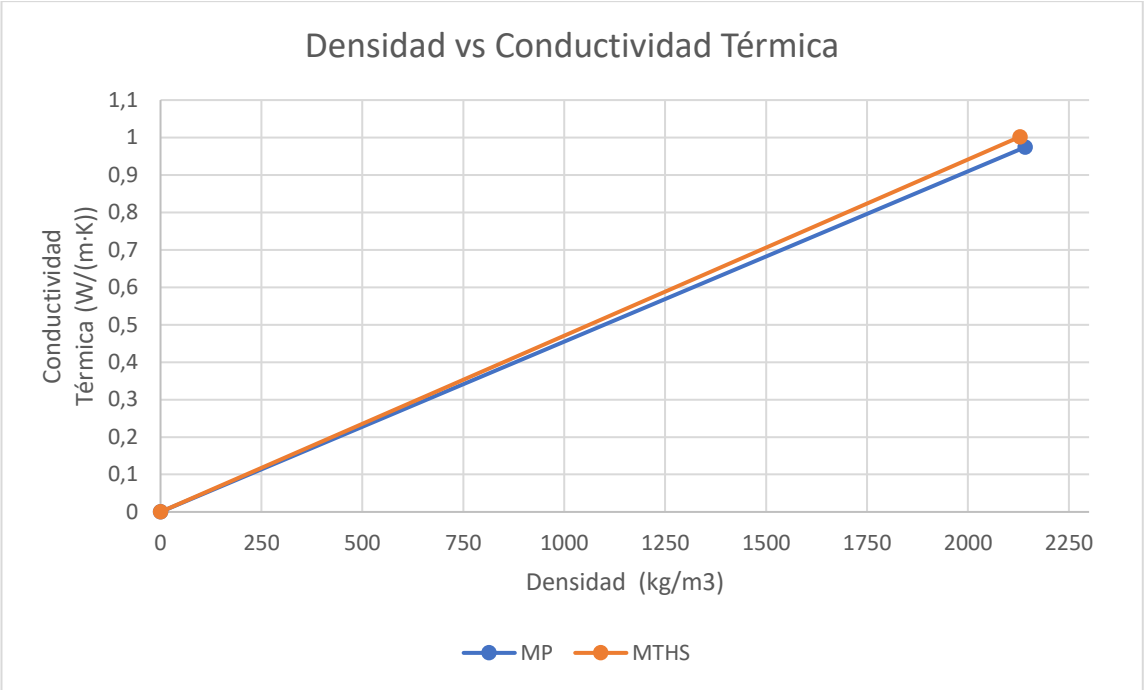


Ilustración 53 Gráfica Densidad vs Conductividad Térmica.

Nota: Fuente: Autor

CAPÍTULO V

5. CONCLUSIONES Y RECOMENDACIONES

5.1. Conclusiones

El propósito principal de esta investigación es analizar el comportamiento térmico del mortero reforzado con fibra previamente tratada al 3% de solución de hidróxido de sodio, determinar la variación térmica que influye la fibra al mortero. Para ello, se realizó este ensayo a cuatro tipos de muestras, y gracias a trabajos similares a este como el de (Gómez, 2017) tenemos las siguientes conclusiones:

- Al reforzar el mortero simple con fibra, en el campo de la conductividad térmica tenemos una pequeña variación que provoca que el material aumente su capacidad de conducir el calor, debido que la fibra es un componente orgánico y se encuentra en el mortero con un 0.2% del peso entre el cemento y la arena disminuye despreciablemente la resistencia al calor, si el porcentaje de fibra aumentara, posiblemente se notaría una variación mayor en la conductividad térmica.
- Revisando las densidades de las muestras MP y MTHS, podemos observar que en estado fresco y endurecido hay una diferencia del 1.3% en ambos casos, la muestra MP tiene menor cantidad de vacíos que la muestra MTHS y con esta premisa se analizó la gráfica de Densidad vs Conductividad térmica y se determinó que la variación es apenas del 3%, se puede decir que; la conductividad térmica podría ser afectada por su densidad ya que la muestra MTHS al tener una mayor cantidad de vacíos la transmisión de calor se acelera por ser un material menos denso que la muestra MP.
- Al comprar las muestras principales podemos confirmar que la hipótesis planteada fue acertada, debido que existe una variación en la resistencia al calor cuando reforzamos el mortero con fibra de abacá previamente tratada con hidróxido de sodio, pero también se debe recalcar que la variación es mínima por lo que la fibra no aporta un cambio significativo a su conductividad térmica.
- Después de analizar el trabajo de investigación de (Gómez, 2017) donde se aplicó el ensayo de conductividad térmica a morteros reforzados con fibra, pero con diferentes dosificaciones para la fibra entre 5% y 15% del peso entre el cemento y la arena. Esta investigación determinó que la fibra aporta una resistencia al calor cuando la dosificación es mayor al 5% pero las propiedades

mecánicas del mortero disminuyen al aumentar este porcentaje. A lo que queremos llegar con todo esto es que la dosificación del 0.2% de la fibra tratada por hidróxido de sodio mejora las propiedades mecánicas del mortero de una manera óptima donde se puede revisar en la investigación de (Cubillo & Chasiguasín, 2020) pero se pierde un poco la resistencia al calor que debería tener un mortero sin fibra. Si la intención es mejorar la resistencia al calor del mortero se debe aumentar el porcentaje de la fibra al 5%, pero sacrificaría el rendimiento óptimo que tenía en sus propiedades mecánicas.

5.2.Recomendaciones

Si se desea obtener un mortero que aporte mayor resistencia al calor que el convencional, se recomienda reforzar con fibra de abacá previamente tratada con hidróxido de sodio debido que este tratamiento alcalino es sencillo de realizar y es que mejor comportamiento tiene a largo plazo evitando la degradación de la fibra, se recomienda realizar ensayos de conductividad térmica con diferentes dosificaciones superiores al 5%.

Para realizar los ensayos de conductividad térmica, se recomienda hacer el tratamiento de hidróxido de sodio a la fibra de abacá en las instalaciones de Holcim Ecuador S.A, y el proceso de la elaboración de las muestras debido que tienen los equipos y los moldes necesarios para respetar los requerimientos que deben cumplir las muestras.

Si se quiere priorizar el comportamiento mecánico del mortero, se recomienda mantener la dosificación del 0.2% de la fibra e ignorar su comportamiento térmico.

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ANEXOS

	LABORATORIO DE ENSAYOS TÉRMICOS Y EFICIENCIA ENERGÉTICA FORMATO TÉCNICO REPORTE DE CONDUCTIVIDAD	**LABET FT 05**
Edición 3	Fecha de revisión: 2019/08/31 / Fecha de aprobación: 2019/08/31	Pag 1 de 3

Guayaquil, 1 de marzo de 2021

Código de Solicitud S21-003

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Requerimiento:
Ensayo para la determinación de conductividad térmica en 1 muestra compuesta de mortero de cemento convencional (150x150x150) mm [A1]; y 1 muestra conformada de mortero reforzado de cemento con fibra de abacá. (150x150x150) mm [A2].

Métodología:
El ensayo para la determinación de conductividad térmica es fundamentado en el estándar ISO 8302 por el método de placa caliente resguardada. El procedimiento permite medir conductividad térmica desde 0,002 hasta 2,500 W/m-K. Los ensayos pueden ser ejecutados en un rango de temperatura desde 10°C hasta 40°C, con un diferencial de temperatura de 15°C (entre placas). El método muestra limitación sobre materiales heterogéneos o no isotrópicos. Las modificaciones al procedimiento estándar se listan en la hoja 2 del presente informe.

Equipamiento:

Equipo:	Medidor de conductividad térmica de placa caliente
Modelo:	λ-Meter EP500e, Version C.
Muestra de Verificación:	Etal 210
Verificación:	2021-01-21
Accesorios:	Apliacación de almohadillas de relleno y sensores tipo película.

Declaración:
* Los Resultados del presente informe son atribuibles únicamente a la(s) muestra(s) ensayada(s).
* El LABET no realiza procedimientos de muestreo.
* Este informe no debe ser reproducido parcialmente sin autorización manifiesta del LABET.

Analista Ténico

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INSTITUTO DE INVESTIGACIÓN GEOLÓGICO Y ENERGÉTICO
LABORATORIO DE ENSAYOS TÉRMICOS

Código de Solicitud: S21-003

Condiciones y descripción de la(s) Muestra(s):

	Código	Largo (mm)	Ancho (mm)	Espesor (mm)	Peso (g)	Densidad (kg/m^3)	Fecha de recepción	Otros (Color, composición)
1	2102001	152	153	151,5	7561,10	2141,3	2021-02-05	A1. Convencional
2	2102002	152	154	152,0	7541,6	2128,9	2021-02-05	A2. Con fibra
3	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.
4	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.	N.A.

Acondicionamiento de la(s) Muestra(s):

1.- 2102001, equilibrio con el sitio de la prueba (24 h).
2.- 2102002, equilibrio con el sitio de la prueba (24 h).
3.- No Aplica.
4.- No Aplica.

Modificaciones al procedimiento o a las muestras:

1.- 2102001, aplicación de almohadillas de relleno y sensores adicionales tipo película.
2.- 2102002, aplicación de almohadillas de relleno y sensores adicionales tipo película.
3.- No Aplica.
4.- No Aplica.

Condiciones ambientales de ensayo:

Código de Muestra:	2102001		**Fecha de ensayo:**	2021-02-19
Temperatura Ambiente Promedio:	17,9	°C		
Humedad Relativa:	61,7	%		

Código de Muestra:	2102002		**Fecha de ensayo:**	2021-02-21
Temperatura Ambiente Promedio:	17,9	°C		
Humedad Relativa:	61,7	%		

Código de Muestra:	N.A.		**Fecha de ensayo:**	N.A.
Temperatura Ambiente Promedio:	N.A.	°C		
Humedad Relativa:	N.A.	%		

Código de Muestra:	N.A.		**Fecha de ensayo:**	N.A.
Temperatura Ambiente Promedio:	N.A.	°C		
Humedad Relativa:	N.A.	%		

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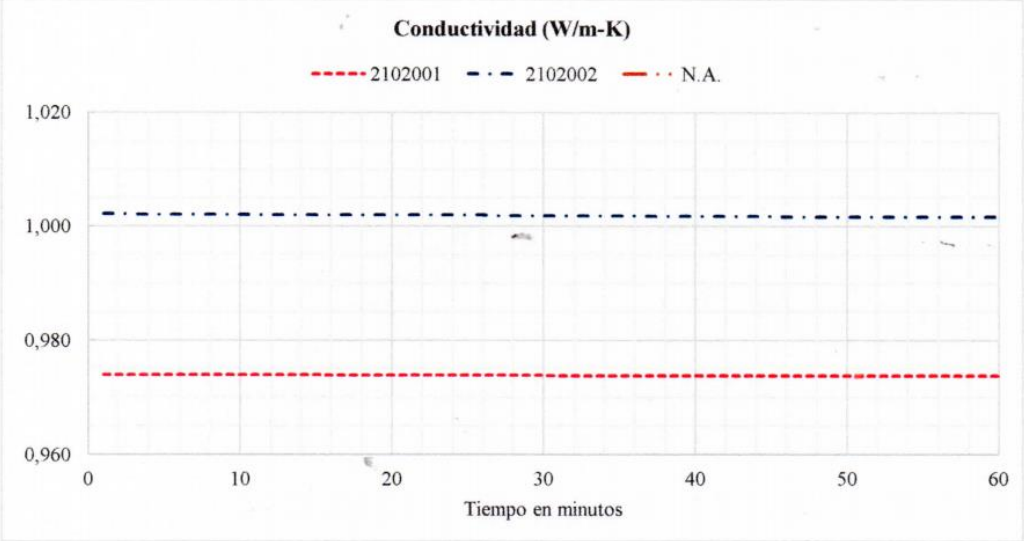
Resultado **Código de Solicitud:** S21-003



Temperatura media de ensayo: 23 °C
Diferencia de Temperatura: 30 °C

			Id. Muestra
Conductividad Térmica:	0,974	**W/m-K**	2102001
	1,002	**W/m-K**	2102002
	-	**W/m-K**	N.A.
	-	**W/m-K**	N.A.

Observaciones:

* El procedimiento de ensayo es una variación al método en proceso de designación SAE.

Responsable Técnico

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Designation: C177 – 13

Standard Test Method for Steady-State Heat Flux Measurements and Thermal Transmission Properties by Means of the Guarded-Hot-Plate Apparatus[1]

This standard is issued under the fixed designation C177; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This test method establishes the criteria for the laboratory measurement of the steady-state heat flux through flat, homogeneous specimen(s) when their surfaces are in contact with solid, parallel boundaries held at constant temperatures using the guarded-hot-plate apparatus.

1.2 The test apparatus designed for this purpose is known as a guarded-hot-plate apparatus and is a primary (or absolute) method. This test method is comparable, but not identical, to ISO 8302.

1.3 This test method sets forth the general design requirements necessary to construct and operate a satisfactory guarded-hot-plate apparatus. It covers a wide variety of apparatus constructions, test conditions, and operating conditions. Detailed designs conforming to this test method are not given but must be developed within the constraints of the general requirements. Examples of analysis tools, concepts and procedures used in the design, construction, calibration and operation of a guarded-hot-plate apparatus are given in Refs (**1-41**).[2]

1.4 This test method encompasses both the single-sided and the double-sided modes of measurement. Both distributed and line source guarded heating plate designs are permitted. The user should consult the standard practices on the single-sided mode of operation, Practice C1044, and on the line source apparatus, Practice C1043, for further details on these heater designs.

1.5 The guarded-hot-plate apparatus can be operated with either vertical or horizontal heat flow. The user is cautioned however, since the test results from the two orientations may be different if convective heat flow occurs within the specimens.

1.6 Although no definitive upper limit can be given for the magnitude of specimen conductance that is measurable on a guarded-hot-plate, for practical reasons the specimen conductance should be less than 16 W/(m^{2}K).

1.7 This test method is applicable to the measurement of a wide variety of specimens, ranging from opaque solids to porous or transparent materials, and a wide range of environmental conditions including measurements conducted at extremes of temperature and with various gases and pressures.

1.8 Inhomogeneities normal to the heat flux direction, such as layered structures, can be successfully evaluated using this test method. However, testing specimens with inhomogeneities in the heat flux direction, such as an insulation system with thermal bridges, can yield results that are location specific and shall not be attempted with this type of apparatus. See Test Method C1363 for guidance in testing these systems.

1.9 Calculations of thermal transmission properties based upon measurements using this method shall be performed in conformance with Practice C1045.

1.10 In order to ensure the level of precision and accuracy expected, persons applying this standard must possess a knowledge of the requirements of thermal measurements and testing practice and of the practical application of heat transfer theory relating to thermal insulation materials and systems. Detailed operating procedures, including design schematics and electrical drawings, should be available for each apparatus to ensure that tests are in accordance with this test method. In addition, automated data collecting and handling systems connected to the apparatus must be verified as to their accuracy. This can be done by calibration and inputting data sets, which have known results associated with them, into computer programs.

1.11 It is not practical for a test method of this type to establish details of design and construction and the procedures to cover all contingencies that might offer difficulties to a person without technical knowledge concerning theory of heat flow, temperature measurements and general testing practices. The user may also find it necessary, when repairing or

[1] This test method is under the jurisdiction of ASTM Committee C16 on Thermal Insulation and is the direct responsibility of Subcommittee C16.30 on Thermal Measurement.

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[2] The boldface numbers given in parentheses refer to the list of references at the end of this standard.

modifying the apparatus, to become a designer or builder, or both, on whom the demands for fundamental understanding and careful experimental technique are even greater. Standardization of this test method is not intended to restrict in any way the future development of new or improved apparatus or procedures.

1.12 This test method does not specify all details necessary for the operation of the apparatus. Decisions on sampling, specimen selection, preconditioning, specimen mounting and positioning, the choice of test conditions, and the evaluation of test data shall follow applicable ASTM Test Methods, Guides, Practices or Product Specifications or governmental regulations. If no applicable standard exists, sound engineering judgment that reflects accepted heat transfer principles must be used and documented.

1.13 This test method allows a wide range of apparatus design and design accuracy to be used in order to satisfy the requirements of specific measurement problems. Compliance with this test method requires a statement of the uncertainty of each reported variable in the report. A discussion of the significant error factors involved is included.

1.14 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.15 *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.* Specific precautionary statements are given in Note 21.

1.16 Major sections within this test method are arranged as follows:

2. Referenced Documents

2.1 *ASTM Standards:*[3]

C168 Terminology Relating to Thermal Insulation

C518 Test Method for Steady-State Thermal Transmission Properties by Means of the Heat Flow Meter Apparatus

C687 Practice for Determination of Thermal Resistance of Loose-Fill Building Insulation

C1043 Practice for Guarded-Hot-Plate Design Using Circular Line-Heat Sources

C1044 Practice for Using a Guarded-Hot-Plate Apparatus or Thin-Heater Apparatus in the Single-Sided Mode

C1045 Practice for Calculating Thermal Transmission Properties Under Steady-State Conditions

C1058 Practice for Selecting Temperatures for Evaluating and Reporting Thermal Properties of Thermal Insulation

C1363 Test Method for Thermal Performance of Building Materials and Envelope Assemblies by Means of a Hot Box Apparatus

E230 Specification and Temperature-Electromotive Force (EMF) Tables for Standardized Thermocouples

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 *ISO Standard:*

ISO 8302 Thermal Insulation—Determination of Steady-State Areal Thermal Resistance and Related Properties—Guarded-Hot-Plate Apparatus[4]

2.3 *ASTM Adjuncts:*ASTM

Table of Theoretical Maximum Thickness of Specimens and Associated Errors[5]

Descriptions of Three Guarded-Hot-Plate Designs[5]

Line-Heat-Source Guarded Hot-Plate Apparatus[6]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms and symbols used in this test method, refer to Terminology C168 and the following subsections.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *auxiliary cold surface assembly, n*—the plate that provides an isothermal boundary at the outside surface of the auxiliary insulation.

3.2.2 *auxiliary insulation, n*—insulation placed on the back side of the hot-surface assembly, in place of a second test specimen, when the single sided mode of operation is used. (*Synonym*—backflow specimen.)

3.2.3 *cold surface assembly, n*—the plates that provide an isothermal boundary at the cold surfaces of the test specimen.

[3] 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.

[4] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

[5] Available from ASTM Headquarters, Order Adjunct: ADJC0177.

[6] Available from ASTM Headquarters, Order Adjunct: ADJC1043.

3.2.4 *controlled environment, n*—the environment in which an apparatus operates.

3.2.5 *guard, n*—promotes one-dimensional heat flow. Primary guards are planar, additional coplanar guards can be used and secondary or edge guards are axial.

3.2.6 *guarded-hot-plate apparatus, n*—an assembly, consisting of a hot surface assembly and two isothermal cold surface assemblies.

3.2.7 *guarded-hot-plate, n*—the inner (rectangular or circular) plate of the hot surface assembly, that provides the heat input to the metered section of the specimen(s).

3.2.8 *hot surface/assembly, n*—the complete center assembly providing heat to the specimen(s) and guarding for the meter section.

3.2.9 *metered section, n*—the portion of the test specimen (or auxiliary insulation) through which the heat input to the guarded-hot-plate flows under ideal guarding conditions.

3.2.10 *mode, double-sided, n*—operation of the guarded-hot-plate apparatus for testing two specimens, each specimen placed on either side of the hot surface assembly.

3.2.11 *mode, single-sided, n*—operation of the guarded-hot-plate apparatus for testing one specimen, placed on one side of the hot-surface assembly.

3.2.12 *thermal transmission properties, n*—those properties of a material or system that define the ability of a material or system to transfer heat such as thermal resistance, thermal conductance, thermal conductivity and thermal resistivity, as defined by Terminology C168.

3.3 *Symbols*—The symbols used in this test method have the following significance:

3.3.1 ρ_m—specimen metered section density, kg/m^3.

3.3.2 ρ_s—specimen density, kg/m^3.

3.3.3 λ—thermal conductivity, W/(m K).

3.3.4 σ—Stefan-Boltzmann constant, W/m^2 K^4.

3.3.5 A—metered section area normal to heat flow, m^2.

3.3.6 A_g—area of the gap between the metered section and the primary guard, m^2.

3.3.7 A_m—area of the actual metered section, m^2.

3.3.8 A_s—area of the total specimen, m^2.

3.3.9 C—thermal conductance, W/(m^2 K).

3.3.10 C_i—the specific heat of the ith component of the metered section, J/(kg K).

3.3.11 dT/dt—potential or actual drift rate of the metered section, K/s.

3.3.12 λ_g—thermal conductivity of the material in the primary guard region, W/(m K).

3.3.13 L—in-situ specimen thickness, m.

3.3.14 m—mass of the specimen in the metered section, kg.

3.3.15 m_i—the mass of the ith component, kg.

3.3.16 m_s—mass of the specimen, kg.

3.3.17 Q—heat flow rate in the metered section, W.

3.3.18 q—heat flux (heat flow rate per unit area), Q, through area, A, W/m^2.

3.3.19 Q_{ge}—lateral edge heat flow rate between primary Guard and Controlled Environment, W.

3.3.20 Q_{gp}—lateral heat flow rate across the gap, W.

3.3.21 Q_{grd}—guard heat flow through Specimen, W.

3.3.22 Q_{se}—edge heat flow between Specimen and Controlled Environment, W.

3.3.23 R—thermal resistance, m^2 K/W.

3.3.24 ΔT—temperature difference across the specimen, $T_h - T_c$.

3.3.25 T_c—cold surface temperature, K.

3.3.26 T_h—hot surface temperature, K.

3.3.27 T_m—mean temperature, K, $(T_h + T_c)/2$.

3.3.27.1 *Discussion*— The Guarded-Hot-Plate Apparatus provides a means for measurement of steady state heat flux through insulation materials, that consists of a guarded heater unit, comprised of a center metering area and concentric separately heated guards, and an opposite, similarly sized cooling plate. Specimens are placed in the space between the heater plate and the cooling plate for testing. The guarded-hot-plate is operated as a single or double sided apparatus. Insulation thermal properties are calculated from measurements of metering area, energy input, temperatures, and thickness. The guarded-hot-plate, which provides an absolute measurement of heat flux, has been shown to be applicable for most insulating materials over a wide range of temperature conditions.

4. Summary of Test Method

4.1 Fig. 1 illustrates the main components of the idealized system: two isothermal cold surface assemblies and a guarded-hot-plate. It is possible that some apparatuses will have more than one guard. The guarded-hot-plate is composed of a metered section thermally isolated from a concentric primary guard by a definite separation or gap. Some apparatus may have more than one guard. The test specimen is sandwiched between these three units as shown in Fig. 1. In the double-sided mode of measurement, the specimen is actually composed of two pieces. The measurement in this case produces a result that is the average of the two pieces and therefore it is important that the two pieces be closely identical. For guidance in the use of the one-sided mode of measurement, the user is directed to Practice C1044. For guidance in the use of a guarded-hot-plate incorporating the use of a line source heater, refer to Practice C1043.

4.1.1 The guarded-hot-plate provides the power (heat flow per unit time) for the measurement and defines the actual test volume, that is, that portion of the specimen that is actually being measured. The function of the primary guard, and additional coplanar guard where applicable, of the guarded-hot-plate apparatus is to provide the proper thermal conditions within the test volume to reduce lateral heat flow within the apparatus. The proper (idealized) conditions are illustrated in Fig. 1 by the configuration of the isothermal surfaces and lines of constant heat flux within the specimen.

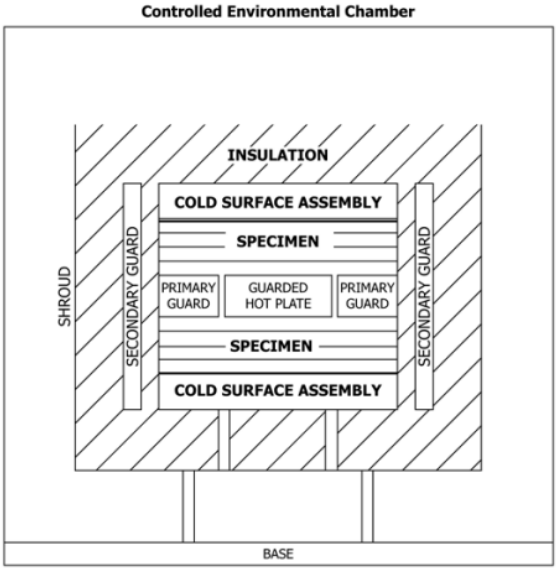

FIG. 1 General Arrangement of the Mechanical Components of the Guarded-Hot-Plate Apparatus

4.1.2 Deviations from the idealized configuration are caused by: specimen inhomogeneities, temperature differences between the metered section and the guard (gap imbalance), and temperature differences between the outer edge of the assembly and the surrounding controlled environment (edge imbalance). These experimental realities lead to heat flow measurements that are too small or too large because the power supplied to the metered section is not exactly equal to that which flows through the specimen in the metered section. The resulting qualitative heat flows are depicted in Fig. 2.

4.2 The three heating/cooling assemblies are designed to create isothermal surfaces on the faces of the specimens within the metered section. The two surfaces designated as the cold surface assemblies are adjusted to the same temperature for the double-sided mode of operation. In practice, because the plates and specimens are of finite dimensions, and because the external controlled environment is often at a temperature different from the edge of the metered section, some lateral heat flow occurs. The primary guard for the guarded hot plate limits the magnitude of the lateral heat flow in the metered section. The effectiveness of the primary guard is determined, in part, by the ratio of its lateral dimension to that of the metered section and to the specimen thickness (**6,7,8,20,31**).

4.3 Compliance with this test method requires: the establishment of steady-state conditions, and the measurement of the unidirectional heat flow Q in the metered section, the metered section area A, the temperature gradient across the specimen, in terms of the temperature T_h of the hot surface and the temperature T_c of the cold surface, (or equivalently, the temperature T between the two surfaces), the thickness' L_1 and L_2 of each specimen, and guard balance between the metered section and primary guard.

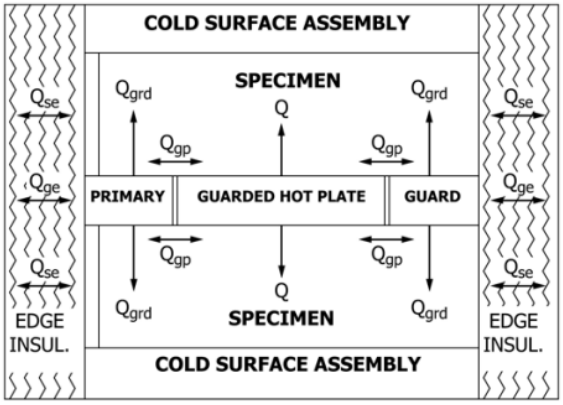

FIG. 2 Illustration of Idealized Heat Flow in a Guarded-Hot-Plate Apparatus

5. Significance and Use

5.1 This test method covers the measurement of heat flux and associated test conditions for flat specimens. The guarded-hot-plate apparatus is generally used to measure steady-state heat flux through materials having a "low" thermal conductivity and commonly denoted as "thermal insulators." Acceptable measurement accuracy requires a specimen geometry with a large ratio of area to thickness.

5.2 Two specimens are selected with their thickness, areas, and densities as identical as possible, and one specimen is placed on each side of the guarded-hot-plate. The faces of the specimens opposite the guarded-hot-plate and primary guard are placed in contact with the surfaces of the cold surface assemblies.

5.3 Steady-state heat transmission through thermal insulators is not easily measured, even at room temperature. This is due to the fact heat transmission through a specimen occurs by any or all of three separate modes of heat transfer (radiation, conduction, and convection). It is possible that any inhomogeneity or anisotropy in the specimen will require special experimental precautions to measure that flow of heat. In some cases it is possible that hours or even days will be required to achieve the thermal steady-state. No guarding system can be constructed to force the metered heat to pass only through the test area of insulation specimen being measured. It is possible that moisture content within the material will cause transient behavior. It is also possible that and physical or chemical change in the material with time or environmental condition will permanently alter the specimen.

5.4 Application of this test method on different test insulations requires that the designer make choices in the design selection of materials of construction and measurement and control systems. Thus it is possible that there will be different designs for the guarded-hot-plate apparatus when used at ambient versus cryogenic or high temperatures. Test thickness, temperature range, temperature difference range, ambient conditions and other system parameters must also be selected

during the design phase. Annex A1 is referenced to the user, which addresses such issues as limitations of the apparatus, thickness measurement considerations and measurement uncertainties, all of which must be considered in the design and operation of the apparatus.

5.5 Apparatus constructed and operated in accordance with this test method should be capable of accurate measurements for its design range of application. Since this test method is applicable to a wide range of specimen characteristics, test conditions, and apparatus design, it is impractical to give an all-inclusive statement of precision and bias for the test method. Analysis of the specific apparatus used is required to specify a precision and bias for the reported results. For this reason, conformance with the test method requires that the user must estimate and report the uncertainty of the results under the reported test conditions.

5.6 Qualification of a new apparatus. When a new or modified design is developed, tests shall be conducted on at least two materials of known thermal stability and having verified or calibrated properties traceable to a national standards laboratory. Tests shall be conducted for at least two sets of temperature conditions that cover the operating range for the apparatus. If the differences between the test results and the national standards laboratory characterization are determined to be significant, then the source of the error shall, if possible, be identified. Only after successful comparison with the certified samples, can the apparatus claim conformance with this test method. It is recommended that checks be continued on a periodic basis to confirm continued conformance of the apparatus.

5.7 The thermal transmission properties of a specimen of material have the potential to be affected due to the following factors: *(a)* composition of the material *(b)* moisture or other environmental conditions *(c)* time or temperature exposure *(d)* thickness *(e)* temperature difference across the specimen *(f)* mean temperature. It must be recognized, therefore, that the selection of a representative value of thermal transmission properties for a material must be based upon a consideration of these factors and an adequate amount of test information.

5.8 Since both heat flux and its uncertainty may be dependent upon environmental and apparatus test conditions, as well as intrinsic characteristics of the specimen, the report for this test method shall include a thorough description of the specimen and of the test conditions.

5.9 The results of comparative test methods such as Test Method C518 depend on the quality of the heat flux reference standards. The apparatus in this test method is one of the absolute methods used for generation of the reference standards. The accuracy of any comparative method can be no better than that of the referenced procedure. While it is possible that the precision of a comparative method such as Test Method C518 will be comparable with that of this test method, Test Method C518 cannot be more accurate. In cases of dispute, this test method is the recommended procedure.

6. Apparatus

6.1 A general arrangement of the mechanical components of such a guarded-hot-plate apparatus is illustrated in Fig. 1. This consists of a hot surface assembly comprised of a metered section and a primary guard, two cold surface assemblies, and secondary guarding in the form of edge insulation, a temperature-controlled secondary guard(s), and often an environmental chamber. Some of the components illustrated in Fig. 1 are omitted in systems designed for ambient conditions, although a controlled laboratory environment is still required; edge insulation and the secondary guard are typically used only at temperatures that are more than ± 10°C from ambient. At ambient conditions, the environmental chamber is recommended to help eliminate the effects of air movement within the laboratory and to help ensure that a dry environment is maintained.

6.1.1 The purpose of the hot surface assembly is to produce a steady-state, one-dimensional heat flux through the specimens. The purpose of the edge insulation, secondary guard, and environmental chamber is to restrict heat losses from the outer edge of the primary guard. The cold surface assemblies are isothermal heat sinks for removing the energy generated by the heating units; the cold surface assemblies are adjusted so they are at the same temperature.

6.2 *Design Criteria*—Establish specifications for the following specifications prior to the design. Various parameters influence the design of the apparatus and shall be considered throughout the design process, maximum specimen thickness; range of specimen thermal conductances; range of hot surface and cold surface temperatures; characteristics of the specimens (that is, rigidity, density, hardness); orientation of the apparatus (vertical or horizontal heat flow); and required accuracy.

6.3 *Hot Surface Assembly*—The hot surface assembly consists of a central metered section and a primary guard. The metered section consists of a metered section heater sandwiched between metered section surface plates. The primary guard is comprised of one or more guard heaters sandwiched between primary guard surface plates. The metered section and primary guard shall be thermally isolated from each other by means of a physical space or gap located between the sections. The hot surface assembly using a line-heat-source is covered in Practice C1043.

NOTE 1—The primary guard, in some cases, is further divided into two concentric sections (double guard) with a gap separator to improve the guard effectiveness.

6.3.1 *Requirements*—The hot surface assembly shall be designed and constructed to satisfy the following minimum requirements during operation.

6.3.1.1 The maximum departure from a plane for any surface plate shall not exceed 0.025 % of the linear dimension of the metered section during operation.

NOTE 2—Planeness of the surface can be checked with a metal straightedge held against the surface and viewed at grazing incidence with a light source behind the straightedge. Departures as small as 2.5 µm are readily visible, and large departures can be measured using shim-stock, thickness gages or thin paper.

6.3.1.2 The average temperature difference between the metered section surface plate and the primary guard surface

plate shall not exceed 0.2 K. In addition, the temperature difference across any surface plate in the lateral direction shall be less than 2 % of the temperature difference imposed across the specimen.

NOTE 3—When qualifying the apparatus, additional temperature sensors shall be applied to the surface plates of the metered section and primary guards that verify that the requirements of 6.3.1.2 are satisfied.

6.3.1.3 The surfaces of the metered and primary guard surface plates that are in contact with the test specimen shall be treated to maintain a total hemispherical emittance greater than 0.8 over the entire range of operating conditions.

NOTE 4—At high temperatures the importance of high emittance of the surfaces adjacent to the specimens cannot be stressed too strongly since radiative heat transfer predominates in many materials as the temperature increases.

6.3.1.4 The metered section and primary guard surface plates shall remain planar during the operation of the apparatus. See 6.3.1.1.

6.3.2 *Materials*—The materials used in the construction of the hot surface assembly shall be carefully chosen after considering the following material property criteria.

6.3.2.1 *Temperature Stability*—Materials are selected for the heaters and surface plates that are dimensionally and chemically stable and suitably strong to withstand warpage and distortion when a clamping force is applied. For modest temperatures, electric resistance heaters embedded in silicone have been successfully employed; at higher temperatures, heating elements sandwiched between mica sheets or inserted into a ceramic core have been used. Surface plates for hot surface assemblies used at modest temperatures have been fabricated from copper and aluminum. High purity nickel alloys have been used for higher temperature applications.

6.3.2.2 *Thermal Conductivity*—To reduce the lateral temperature differences across the metered and primary guard surface plates, fabricate these plates from materials that possess a high thermal conductivity for the temperature and environmental conditions of operation. Copper and aluminum are excellent choices for modest temperature applications; at higher temperatures consider using nickel, high purity alumina or aluminum nitride. These are examples of materials used and the operator must fully understand the thermal conductivity versus temperature dependency of the materials selected.

6.3.2.3 *Emittance*—To obtain a uniform and durable high surface emittance in the desired range, select a surface plate material or suitable surface treatment, or both. For modest temperature applications, high emittance paints may be employed. Aluminum can be anodized to provide the necessary high emittance. For high temperature applications, most ceramics will inherently satisfy this requirement while nickel surface plates can be treated with an oxide coating.

6.3.2.4 *Temperature Uniformity*—Select a heating element design that will supply the necessary heat flux density for the range of specimen thermal conductances to be investigated. The design of the heating element shall also consider the heat flux distribution of the surface of the heating element. Most apparatus incorporate the use of a distributed electric resistance heating element dispersed uniformly across the metered section and the primary guard. The surface plates and heating elements shall be clamped or bolted together in a uniform manner such that the temperature difference requirements specified in 6.3.1.2 are satisfied. Bolting the composite constructions together has been found satisfactory.

6.3.2.5 The insertion of insulating sheets between the heating elements and surface plates (that is, to mount a gap temperature imbalance detector) is allowed. To satisfy the requirements of 6.3.1.2, similar sheets shall be mounted between the heating element and the opposing surface plate.

6.3.2.6 *Hot Surface Assembly Size*—Design criteria established in 6.2 will determine the size of the apparatus. The size of the metered section shall be large enough so that the amount of specimen material in contact with the metered section (and therefore being measured) can be considered representative of the material being tested.

6.3.2.7 After determining the maximum specimen thickness that will be tested by this design, refer to Adjunct, Table of Theoretical Maximum Thickness of Specimens and Associated Errors, regarding associated errors attributable to combinations of metered section size, primary guard width, and specimen thickness.

NOTE 5—Typically the width of the primary guard equal to approximately one-half of the linear dimension of the metered section has been found to reduce edge heat loss to acceptable levels.

6.3.2.8 *Heat Capacitance*—The heat capacity of the hot surface assembly will impact the time required to achieve thermal equilibrium. Selecting materials with a low specific heat will increase the responsiveness of the apparatus. The thickness of the surface plates needs to be carefully considered; thick plates assist in reducing lateral temperature distributions but reduce responsiveness. A balance between these requirements is needed.

6.4 *The Gap*—The metered section and the primary guard shall be physically separated by a gap. The gap provides a lateral thermal resistance between these sections of the hot surface assembly. The area of the gap in the plane of the surface plates shall not be more than 5 % of the metered section area.

6.4.1 The heater windings from the metered section and primary guard heating elements shall be designed to create a uniform temperature along the gap perimeter.

6.4.2 The metered section area shall be determined by measurements to the center of the gap that surrounds this area, unless detailed calculations or tests are used to define this area more precisely.

6.4.3 Any connections between the metered section and the primary guard shall be designed to minimize heat flow across the gap. If a mechanical means is used to satisfy the requirements of 6.3.1.4, these connections shall be fabricated with materials having a high thermal resistance. Instrumentation or heater leads that cross the gap should be fabricated with fine-gage wire and traverse the gap at an oblique angle.

6.4.4 The gap may be filled with a fibrous insulation. Packing the gap with this insulation has been found to maintain the metered section and primary guard surface plates planar. An additional benefit of this practice for high temperature applications is that the densely packed insulation reduces the amount of heat conducted across the gap spacing.

6.5 *Cold Surface Assembly*—The cold surface assembly consists of a single temperature controlled section and is comprised of a cold surface heater sandwiched between cold surface plates and a heat sink. It is recommended that the size of the cold surface assembly be identical to the hot surface assembly, including the primary guard. It is acceptable to construct cold surface assemblies with a gap where operation of the apparatus is susceptible to edge loss effects. This design is the ideal design, however, this assembly has traditionally been constructed without a gap with great success.

NOTE 6—The temperature of the cold surface assembly may be maintained through the use of a temperature-controlled bath; in this instance, there is no need to install a cold surface heater. Care must be taken in this instance; the flow rate of the bath must be sufficient to satisfy the temperature uniformity requirements specified in 6.3.1.2 and 6.5.1.

6.5.1 *Requirements*—The cold surface assemblies shall be designed and constructed to satisfy all of the requirements of 6.3.1 except that, since only one surface plate of each cold surface assembly is in contact with the test specimens, the requirement that specifies the temperature difference between the surface plates shall not apply.

6.5.2 *Materials*—The criteria to select materials that will be used in the construction of the cold surface assemblies are identical to the hot surface assembly and are listed in 6.3.2.

6.5.3 *High Temperature Operation*—When the cold surface assemblies will be operated at high temperatures, it is acceptable to insert several thin sheets of insulation between the heat sink and cold surface heater. The addition of these insulation sheets will reduce the energy requirements to the cold surface heater and extend service life.

6.6 *Additional Edge Loss Protection*—Deviation from one-dimensional heat flow in the test specimen is due to non-adiabatic conditions at the edges of the hot surface assembly and the specimens. This deviation is greatly increased when the apparatus is used at temperatures other than ambient. When the guarded-hot-plate apparatus is operated at temperatures that deviate from ambient by more than 10°C, the apparatus shall be outfitted with additional components to reduce edge losses. These components are described in the following sections and shall be used if edge losses cannot be minimized.

NOTE 7—Another means of assessing whether edge insulation is required is to attach a temperature sensor to the mid-height of the exterior edge of the specimen. Sufficient edge insulation is present if the edge temperature, T_e, satisfies the following requirement.

$$(T_e - T_m)/\Delta T < 0.05 \quad (1)$$

6.6.1 *Secondary Guard*—To reduce heat exchange between the edges of the guarded-hot-plate and the environment, the guarded-hot-plate shall be outfitted with a co-axial temperature-controlled container referred to as the secondary guard. The secondary guard will be employed to adjust the ambient temperature to approximate the mean temperature of the test specimen.

6.6.1.1 *Size*—The secondary guard should have an inner dimension that is at least twice the dimension of the hot surface heater and the height should be equal to the thickness of the hot surface heater plus twice the thickness of the thickest specimen that will be tested.

6.6.1.2 *Materials*—The materials used in the construction of the secondary guard are not as critical as those selected for the hot and cold surface assemblies. However, the materials used in the design of the secondary guard shall be selected so that they are thermally stable over the intended temperature range, the heating element shall be capable of producing the necessary heat flux density to adjust the ambient temperature, and a means of cooling the secondary guard is required if the apparatus is intended for use at temperatures below the laboratory ambient. The use of high thermal conductivity metals is recommended for the construction since the secondary guard should be isothermal.

NOTE 8—Successful secondary guard designs consist of a sheathed heater wire or cable wrapped around an adequately-sized metal tube and pressed against the metal tube with another sheet of metal. For low-temperature operation, a cooling coil has been wrapped around the exterior surface of the secondary guard.

6.6.1.3 *Location*—The secondary guard shall be positioned around the hot surface assembly such that a uniform spacing is created between the components. The height of the secondary guard shall be adjusted such that the mid-height of the secondary guard is aligned with the center of the hot surface assembly thickness.

6.6.2 *Edge Insulation*—The interspace between the hot and cold surface assemblies, specimens and the secondary guard shall be filled with an insulating material. Due to the complex shapes of this interspace, a powder or fibrous insulation is recommended.

6.6.2.1 The selection of an edge insulation material will depend on the test conditions. Vermiculite is easy to use but should not be employed at temperatures above 540°C because it's thermal conductivity increases dramatically with temperature.

NOTE 9—Avoid the use of vermiculite when the guarded-hot-plate is used to evaluate specimens in different gaseous environments; vermiculite is extremely hygroscopic and the system is difficult to evacuate when it is used.

NOTE 10—Care shall be taken to ensure that there are no voids, pockets, or other extraneous sources of radiative heat transfer occurring at or near the guarded-hot-plate.

6.6.3 *Enclosure*—The guarded-hot-plate shall be placed inside an enclosure when the apparatus is used in to maintain a gaseous environment that is different than the laboratory ambient.

6.6.3.1 For low-temperature operation, a dry gas environment shall be used to prevent condensation from occurring on the cold surface assemblies and specimens.

6.6.3.2 For high temperature operation, it will often be desirable to protect the apparatus from severe degradation by using a non-oxidizing gas.

6.6.3.3 The enclosure can also be used for substituting different gaseous environments and control of the ambient pressure.

6.7 *Clamping Force*—A means shall be provided for imposing a reproducible constant clamping force on the guarded-hot-plate to promote good thermal contact between the hot and cold surface assemblies and the specimens and to maintain accurate spacing between the hot and cold surface assemblies. It is

unlikely that a force greater than 2.5 kPa will be required for the majority of insulating materials. In the case of compressible materials, a constant pressure arrangement is not needed and it is possible that spacers between the plates will be necessary to maintain constant thickness.

6.7.1 A steady force, that will thrust the cold surface assemblies toward each other can be imposed by using constant-force springs or an equivalent method.

6.7.2 For compressible specimens, spacers are required if the test thickness can not be measured by other means. The spacers shall be small in cross-section and located near the exterior perimeter of the primary guard. Avoid placing spacers on surfaces where underlying sensors are being used to measure plate conditions.

NOTE 11—Because of the changes of specimen thickness possible as a result of temperature exposure, or compression by the plates, it is recommended that, when possible, specimen thickness be measured in the apparatus at the existing test temperature and compression conditions. Gaging points, or measuring studs along the outer perimeter of the cold surface assemblies, will serve for these measurements. The effective combined specimen thickness is determined by the average difference in the distance between the gaging points when the specimen is in place in the apparatus and when it is not in place.

6.8 *Temperature Measurements:*

6.8.1 *Imbalance Detectors*—A suitable means shall be provided to detect the average temperature imbalance between surface plates of the metering section and the primary guard.

6.8.1.1 *Sensors*—The gap region shall be instrumented with temperature sensors to monitor and control the average temperature imbalance across the gap. Fine-gage thermocouples connected as thermopiles are often used for this purpose, although other temperature control sensors, such as thermistors, have been used. Highly alloyed thermocouples, rather than pure metals, should be used to maximize the thermal resistance across the gap. Because of nonuniform heat flux within the surface plates, temperature imbalance is not always constant along the gap perimeter. It has been found that with proper design the thermal conductance of the wires crossing the gap can be made relatively small and, therefore, a large number of thermocouples can be used to increase the gap imbalance sensitivity. It is not uncommon to use ten or more sensing elements.

6.8.1.2 *Sensitivity*—The detection system shall be sufficiently sensitive to ensure that variation in measured properties due to gap temperature imbalance shall be restricted to not more than 0.5 % of the metered section power, as determined experimentally or analytically.

NOTE 12—The sensitivity of many temperature sensors is reduced drastically at temperatures below the laboratory ambient. Particular care must be used in designing thermopile measurement systems to operate under these conditions.

6.8.1.3 *Location*—When using only a minimum number of sensing elements along the gap, the most representative positions to detect the average balance for a square plate are those at a distance from the corners equal to one-fourth of the side of the metering area. The corners and the axes should be avoided. For a round plate, the sensors should be spaced equally around the gap.

6.8.1.4 Electrically isolated gap imbalance sensors should be placed on both surface plates of the guarded heating unit to average the imbalance on both faces of the heating unit.

6.8.1.5 Thermal junctions or other sensitive elements should each be located in similar areas of the hot surface assembly. It is suggested that all junctions should be located at points directly adjacent to the centers of the areas between heater windings. Any leads crossing the gap should be thermally anchored to the primary guard to provide a heat sink from external thermal variations. In some instances it may be desirable to provide a heat sink for these leads outside the primary guard to minimize any radial heat flow.

6.8.2 *Temperature Sensors*—Methods possessing adequate accuracy, such as thermistors, thermocouples, diodes and precision resistance thermometers may be used for the measurement of temperatures in the apparatus. Thermocouples are the most widely used detector due to their wide range of applicability and accuracy. The goal is to measure the temperature gradient within the specimen, and the method chosen (sensors mounted on the specimen surface, in grooves, or between interior layers) should be that which yields the highest accuracy in the measurement of the temperature gradient. A discussion of these alternatives is provided in 6.8.2.3 and 6.8.2.4.

6.8.2.1 *Use of Thermocouples*—Precautions should be used to minimize spurious voltages in temperature control and measuring circuits. Spurious voltages, due to wire inhomogeneities, generally increase as the temperature gradients within the measuring leads increase. For the same reason, junctions between dissimilar metal leads should not be made in the regions of appreciable temperature gradients. Low thermal emf switches should be used in the temperature measurement circuits. An insulated, isothermal box of heavy sheet metal can be used when joining leads of dissimilar metals in the thermocouple circuit. It is recommended that all connections of thermocouple wire to copper wire be accomplished within the isothermal box in order that the junctions are at the same temperature; then the copper, not the thermocouple, leads are connected to the needed switching devices and/or voltmeters.

6.8.2.2 *Accuracy*—Thermocouples whose outputs are used to calculate thermal transmission properties shall be fabricated from either calibrated thermocouple wire or wire that has been certified by the supplier, and shall have a standard limit of error equal to or less than the specifications of Tables E230. The resulting error in temperature differences due to distortion of the heat flow around the sensor, to sensor drift, and other sensor characteristics shall be less than 1 %.

6.8.2.3 *Methods of Attachment*—The surface temperatures of the specimens are most often measured by means of permanently mounted thermocouples placed in grooves cut into the surface plates. Precautions shall be taken to ensure that the thermocouple is thermally anchored to the surface being measured. This method of instrumentation is employed when the contact resistance between the specimen and the surface plates is a small fraction of the specimen thermal resistance. The hot- and cold-surface assembly plate sensors on each side are sometimes connected differentially. Thermocouples mounted in this manner shall be made of wire not larger than

0.6 mm in diameter for large apparatus and preferably not larger than 0.2 mm for small apparatus.

NOTE 13—This method of deploying thermocouples is traditionally used for compressible specimens and for rigid specimens possessing flat surfaces that have a thermal resistance of greater than 0.2 m^2 K/W at ambient conditions.

NOTE 14—For rigid specimens not satisfying the requirements of 6.8.2.2, two techniques for attaching temperature sensors are recommended. Small grooves may be cut into the surfaces of the specimens and thermocouples can be affixed into these grooves. As an alternative, thermocouples may be installed onto the surfaces of the specimen and thin sheets of a compressible homogeneous material interposed between the specimen and surface plates. In this latter case, an applied force should be used as indicated in 6.7 to ensure sufficient surface contact. For either of these applications, thermocouples shall be made of wire not larger than 0.2 mm in diameter.

6.8.2.4 *Electrical Isolation*—Temperature sensors can be either completely insulated electrically from the surface plates or grounded to the surface plate at one location. Consequently, thermocouples connected differentially can only have a single junction ground. Computations or experimental verifications, or both, shall be performed to confirm that other circuits do not affect the accuracy of the temperature measurements.

6.8.2.5 *Number of Sensors*—The number of temperature sensors on each side of the specimen in the metering area shall not be less than $10 \times \sqrt{A}$, or 2, whichever is greater.

NOTE 15—It is recommended that one temperature sensor be placed in the center of the metered section and that additional sensor be uniformly distributed radially.

6.9 *Thickness Measurements*—A means shall be provided for measuring the thickness of the specimen, preferably in the apparatus, to within 0.5 %.

6.10 *Metered Section Power Measurement*—Dc power is highly recommended for the metered section. Ac power may be used but the user should note that ac power determinations are more prone to error than dc measurements. The power to the metered section is determined with a wattmeter or from voltage and current measurements across the heater in the metered section. The voltage taps for this measurement should be placed to measure the voltage from the mid-point of the gap. The current can be determined from the voltage drop across a precision resistor placed in series with the metered section heater.

6.11 *Electrical Measurement System*—A measuring system having a sensitivity and accuracy of at least ±0.1 K shall be used for measurement of the output of all temperature and temperature difference detectors. The system shall have sufficient sensitivity to measure the gap imbalance to a level equal to 1 % of the imbalance detector output that satisfies the requirement of 6.8.1.2. Measurement of the power to the metered section shall be made to within 0.2 % over the entire operating range.

6.12 *Performance Checks*—When a new apparatus is commissioned or an apparatus has undergone significant refurbishment, a series of careful checks shall be performed before initiating routine testing.

6.12.1 *Planeness*—The planeness of each surface plate shall be measured. See 6.3.1.1.

6.12.2 *Temperature Measurements*—With specimens installed in the apparatus, the coolant supply to the cold surface assembly shut off, and no electrical power being supplied to any of the heaters, mount the apparatus inside the enclosure. Allow the system sufficient time to come to thermal equilibrium. With no energy being supplied to the apparatus, note the output of all of the temperature sensors. The temperature sensors shall have an output that agrees to within the uncertainty prescribed in 6.8.2.2. The output of the imbalance detection circuit shall be within the noise level of the electrical measurement system.

6.12.3 *Imbalance Detection*—Determine the maximum imbalance that can be allowed that satisfies the requirements in 6.8.2.2. With the apparatus energized and operating normally, note the thermal resistance of a specimen and the imbalance detector output at equilibrium. Repeat the test at various levels of imbalance. Linearly fit the thermal resistance data as a function of bias. The slope of this relationship will define the maximum imbalance detector output that can be allowed during routine operation.

NOTE 16—The number of bias levels that need to be analyzed will depend on the quality of the curve fit; the scatter within the data set, as defined by twice the standard deviation, shall be less than the noise level of the electrical measurement system as defined in 6.11.

6.12.4 *Edge Heat Losses*—Edge heat losses give rise to the greatest measurement errors when the specimens approach the maximum specified thickness and thermal resistance. This series of experiments will determine which edge loss strategies must be employed to maintain edge losses to levels prescribed by this method.

6.12.4.1 Install specimens in the apparatus that approach the apparatus limits described above and instrument these specimens with the edge temperature sensors described in 6.6. Do not install any components described in 6.6 to reduce edge heat loss. While performing a test, verify that the difference between the specimen mean temperature and edge temperature satisfy the requirements of 6.6. Add additional edge loss apparatus components (edge insulation, secondary guard, enclosure) until the requirements of 6.6 are satisfied. These experiments will define the required levels of edge loss that shall be incorporated into the routine testing. In extreme cases, it is possible that the secondary guard will have to be biased to satisfy these requirements; include these biases as part of the routine test procedure.

6.12.5 *Emittance of Surface Plates*—The emittance of the surfaces can be experimentally verified by testing an air gap, where the thickness of the air gap is limited to prevent the onset of convection. The heat flow rate per unit temperature difference is the sum of the thermal conductance of air and $4\sigma\, T_m^3$ (2/ε-1). A best fit of the plot of the heat flow rate per unit temperature difference and the inverse of the air space thickness supplies both the thermal conductivity of the air and ${}_4T_m^3$ (2/ε-1). From this plot, the plate emittance can be verified (**42**).

6.12.6 *Overall Design Verification*—When all of the other checks have been successfully completed, tests shall be performed on specimens that are traceable to a national standards organization. These tests shall cover the range of temperatures for which the apparatus has been designed. It is possible that

verification of the apparatus will be limited by the temperature range of available standards. See 5.7.

7. Specimen Preparation and Conditioning

7.1 *Specimen Selection*—Only those specimen selection factors important to the performance of the apparatus are considered here. Factors related to the specimens' thermal properties are typically described in material specifications. When two specimens are required, the specimens should be selected to be as similar in thickness and thermal characteristics as possible. The use of Test Method C518 can be used to check the similarity of the specimens' thermal characteristics.

7.1.1 *Thickness*—The maximum specimen thickness that can be measured to a given accuracy depends on several parameters, including the size of the apparatus, thermal resistance of the specimen, and the accuracy desired. To maintain edge heat losses below approximately 0.5 %, for a guard width that is about one-half the linear dimension of the metered section, the recommended maximum thickness of the specimen is one-third the maximum linear dimension of the metered section. For more specific quantitative information on this limitation see Refs **(1,5,7,8)** and adjunct material given in this test method.

7.1.2 *Size*—The specimen shall be sized to cover the entire metered section and guard area when possible. It is desirable to cover the gap between the guarded-hot-plate and the primary guard when sample size is limited. The guard portion of the volume between the heating and cooling plates should be filled with material having similar thermal conductance characteristics as the specimen. When the specimen has a high lateral conductance such as a dense solid, a gap between the metered section and the primary guard shall be provided within the specimen. Refer to 7.2.3 for special precautions.

7.1.3 *Homogeneity*—Specimens exhibiting appreciable inhomogeneities in the heat flux direction shall not be tested with this method. There are two potential problems in attempting to determine the heat flux through highly inhomogeneous specimens. One is related to the interpretation and application of the resulting data, see Practice C1045. The other is the degradation in the performance of the apparatus. If the specimen is highly inhomogeneous, that is, the heat flux varies appreciably over the metered section, several errors can be significantly increased. The plate temperature distribution can deviate appreciably from isothermal conditions which, in turn, can cause large uncertainties in the average temperature difference across the specimen. The increased plate temperature variations can also lead to increased gap and edge heat losses. The importance of measuring the plate or specimen surface temperatures at numerous points is greatly increased under such conditions.

7.2 *Specimen Preparation*—Prepare and condition the specimens in accordance with the appropriate material specification. Use the following guidelines when the material specification is unavailable. In general, the surfaces of the specimen should be prepared to ensure that they are parallel with and have uniform thermal contact with the heating and cooling plates.

7.2.1 *Compressible Specimens*—It is possible that the surfaces of the uncompressed specimens will be comparatively uneven so long as surface undulations are removed under test compression. It will potentially be necessary to smooth the specimen surfaces to achieve better plate-to-specimen contact. If the apparent thermal conductivity of the contact void is greater than that of the specimen, compressible or otherwise, the measured heat flux will be greater than the heat flux that would be obtained if the voids were absent. This is most likely the case at higher temperatures where radiant heat transfer predominates in the void. For the measurement of compressible specimens, the temperature sensors are often mounted directly in the plate surfaces. Also, it is possible that plate spacers will be required for the measurement of compressible specimens.

7.2.2 *Rigid and High Conductance Specimens*—The measurement of rigid specimens or high conductance specimens requires careful surface preparation. First, the surfaces should be made flat and parallel to the same degree as the guarded-hot-plate. If the specimen has a thermal resistance that is sufficiently high compared to the specimen-to-plate interface resistance, temperature sensors mounted in the plates may be adequate. However, for materials such as plastics or ceramics, when the thermal conductivity of the material exceeds 0.1 W/m·K, the following techniques shall be used to ensure accurate surface temperature measurement.

7.2.2.1 In some cases it is necessary to mount the temperature sensors directly on the specimen surfaces or in grooves in the specimens. Under vacuum conditions, the slightest space between plate and specimen is essentially an infinite thermal resistance (except for radiative heat transfer). Under these conditions extreme heat flux nonuniformities will occur. In any event the user should always try to minimize the ratio of contact resistance to specimen resistance and to strive for a constant ratio over the entire surface.

7.2.2.2 Another potential solution (that must be used with caution) is to mount a compressible thin sheet (for example, a soft rubber or thin fibrous pad) between the plates and specimen to improve the uniformity of the thermal contact. When this procedure is used, temperature sensors shall be instrumented in or on the surface of the specimens to ensure accurate temperature measurement of the specimen surface. An applied force should be used as in 6.7 to ensure sufficient surface contact.

7.2.3 *Anisotropic Specimens*—Specimens that have a high lateral to axial conductance ratio require that a low conductance gap be created in the specimen directly in line with the gap between the metered section and the primary guard.

7.2.4 *Loose-Fill Specimens*—The measurement of loose-fill specimens requires special handling, conditioning, and measurement techniques. The user is directed to Practice C687 for details.

7.3 *Specimen Conditioning*—Condition the specimens either as stated in the material specification or where no guideline is given, at 22 ± 5°C and 50 ± 10 % relative humidity for a period of time until less than a 1 % mass change in 24 h is observed.

NOTE 17—Specimens can be conditioned at different conditions in order to determine the effect on the thermal properties of the specimens. Conditioning environments shall be reported with the test results.

8. Procedure

8.1 For a double sided test, select a pair of test specimens as outlined in Section 7.

8.2 Measure and record the specimen mass and dimensions. Also see 8.12.

8.3 Install the specimen into the apparatus at the desired test thickness.

8.4 Install the appropriate secondary guarding and an environmental chamber (as required).

8.5 If the test is to be conducted with gases other than air in the specimen-plate assembly, purge the environmental chamber and backfill with the desired gas. Care should be taken to limit the pressure of the fill-gas to below its condensation point at the lowest temperature expected within the chamber. Since the measured heat flux is dependent on both the type of fill gas and pressure, record both of these parameters.

8.6 Adjust the heating and cooling systems to establish the desired test conditions. For guidance in establishing test temperatures, refer to Practice C1058. The ambient temperature should be the same as or slightly above the mean temperature of the test. It is possible that this will require the use of a temperature controlled surrounding. This can be accomplished utilizing a controlled perimeter heater and insulation materials to aid in the control of the surrounding temperature.

8.7 Record the start time and date of the test. Begin data acquisition. The recorded data shall include: the date and time of data acquisition; power to the guarded-hot-plate; hot side guarded-hot-plate surface temperature; hot side guard temperatures; cold surface assembly temperatures; controlled environment ambient temperature and relative humidity; temperature difference or thermopile output across the gap between the guard and metered section; and calculated heat flux and estimated thermal property of interest.

NOTE 18—Thermal steady-state is the time required for the test apparatus to stabilize. This varies considerably with the apparatus design, specimen to be measured, and test conditions. Generally, however, the stabilization time is on the order of hours. Stabilization times generally increase with thick specimens, specimens with low thermal diffusivity and is dependent on the mass of the metered section area. Measurements in a vacuum and on microporous materials create small monotonic changes over a long period of time and may take longer to stabilize.

8.8 Thermal steady state must be achieved for this test method to be valid. To determine if steady state is achieved, the operator must document steady state by time averaging the data, computing the variation and performing the following tests on the data taken in Section 8.

8.8.1 Thermal steady state for the purpose of this test method is defined analytically as:

8.8.1.1 The temperatures of the hot and cold surfaces are stable within the capability of the equipment at the test conditions. Ideally an error analysis will determine the magnitude of the allowable differences, however the difference is usually less than 0.1 % of the temperature difference.

8.8.1.2 The power to the metering area is stable within the capability of the equipment. Ideally an error analysis will determine the magnitude of the allowable differences, however the difference is usually less than 0.2 % of the average result expected.

8.8.1.3 The required conditions above exist during at least four intervals 30 min in duration or four system time constants, whichever is longer.

NOTE 19—The thermal time constant of the system is the time required to come to within 1/*e* (37 %) of the fixed value after a step thermal disturbance of the system. The thermal time constant in the constant power mode is the time required to come to within 37 % of the final temperature. The thermal time constant in the constant temperature mode is the time required to come to within 37 % of the final power. The thermal time constant of a system can be approximated from the thermal diffusivities of the system components, but is generally determined experimentally.

8.9 After achievement of the desired steady-state as defined in 8.8.1, three successive repeat data acquisition runs shall be completed. These runs shall be conducted at intervals of at least 30 min and should not be less than the thermal time constant of the system (see Note 19). This combination of three runs shall be considered a valid test if each datum obtained for each measured variable meets the following criteria.

8.9.1 The data do not differ from the mean by no more than the uncertainty of that variable, see A1.5.

8.9.2 The data obtained does not change monotonically with time. This is determined by comparing the average result of the final three test periods to the averages of the previous four periods. Graphing of the test parameters versus time or monitoring the slope of the data are techniques for determining monotonic conditions.

8.9.3 If the data continues to drift, the test shall be considered incomplete and further data acquisition sets shall be conducted until thermal steady state is achieved. Drift, even at low levels, has the potential to indicate that either the specimen characteristics are changing or the system is not at steady-state. For further details see Refs **(3,12,13)**.

8.10 Prior to terminating the test, measure and record the pressure of the chamber.

8.11 Upon completion of the thermal test outlined above, remove the specimen and examine the system components, such as temperature sensor mounting, for proper placement and operation.

8.12 Determine the specimen thickness and weight after the test to ensure that they have not changed from the initial condition. Record any changes in the physical characteristics of the specimen.

9. Calculation

9.1 The primary data required for this test method include electrical power, surface temperatures, area, and thickness. Of these, only thickness is generally a directly measured quantity. The others are either calculated from other more fundamental measurements or are converted by an electrical device. The manner in which these variables can be obtained is discussed in 8.9 and below.

9.2 *Heat Flow*—The heat flow to be reported is that which passes through each specimen. This is equal to the power generated by the metered section heater. For the double-sided mode of operation, only one-half the power generated by the

heater flows through each specimen. Determine the power, Q, from emf, E, and current, I, and calculate as follows:

$$Q = E \times I \tag{2}$$

9.3 *Metered Section Area*—Determine the metered section area, A, from the area, A_m, of the guarded-hot-plate and the gap area, A_g. If there is no discontinuity in specimen characteristics in the gap region, the metered area is calculated as follows:

$$A = A_m + \frac{A_g}{2} \tag{3}$$

For high precision measurements, it is possible that this assumption that the gap contributes half of its area to the effective metered section area, A, will need to be verified for the particular apparatus used. If there is a discontinuity between the specimen in the metered section and the guard region, this equation is modified slightly, as in ISO 8302, to include the effect of heat flux distortion in the gap region:

$$A = A_m + \frac{A_g \lambda_g}{2\lambda} \tag{4}$$

Where significant expansion, or contraction, of the guarded-hot-plate is known during a test, appropriate corrections to the area shall be made.

9.4 *Heat Flux*—The heat flux is obtained from the ratio of the heat flow, Q, and the total metered section area, A, and is calculated as follows:

$$q = \frac{Q}{A} \tag{5}$$

9.5 *Temperature*—Electrical readings from the temperature sensors are normally converted to temperature using a mathematical equation based on either the sensor's calibration curve or an appropriate reference such as a thermocouple voltage table.

9.6 *Density*—The metered section area specimen density, ρ_m, or the sample density, ρ_s where metered section area density cannot be obtained, are to be reported as the average of the two pieces. The equation for density, is the following:

$$\rho_m = \frac{m}{A \times L} \tag{6}$$

or:

$$\rho_s = \frac{m_s}{A_s \times L}$$

9.7 *Thermal Transmission Properties*—These properties shall be reported only in accordance with the requirements and restrictions of Practice C1045.

10. Report

10.1 To be in conformance with this test method, report the following:

10.1.1 The report shall be identified with a unique numbering system to allow traceability to the individual measurements taken during each test performed,

10.1.2 The average values as obtained from the test. Standard deviation about that average. The results may be reported in a form similar to that shown in Fig. 3,

10.1.2.1 Identification of the test organization, responsible person in charge, test operator (optional) and the test sponsor,

10.1.2.2 The generic name, or other identification required to provide a complete and detailed description of the tested material. For hygroscopic materials, such as concrete and wood, the moisture content should also be given,

NOTE 20—A generic description in addition to the brand name should be reported where possible.

10.1.2.3 Information regarding the specimen preconditioning,

10.1.2.4 Variables that effect thermal transmission properties, such as fill-gas and pressure, shall be specified when applicable,

10.1.2.5 The dimensions of the metered section and guard(s) and their relationship to the overall specimen dimensions (m). The plate emittance,

10.1.2.6 Specimen orientation and the direction of heat transfer during the test,

10.1.2.7 The total area of the specimen (m^2),

10.1.2.8 The specimen density of the metered section area or sample density where metered section area density cannot be obtained (kg/m^3),

10.1.2.9 The thickness of the specimen(s) within the metered section (m),

10.1.2.10 The area averaged temperatures of both hot and cold specimen surfaces (K),

10.1.2.11 Net steady-state average heat flux through the specimen (W/m^2),

10.1.2.12 Any thermal transmission properties calculated and reported and their estimated error, and

10.1.2.13 The test date and time, the time required for steady temperature conditions, the time to reach steady-state, the data acquisition time period, frequency of data collection and the end date and time.

10.2 The following is optional information for inclusion in the report:

10.2.1 Values for guard loss, back side energy loss and other losses included in the net energy calculation (W/m^2), and

10.2.2 A full description (or references) of test procedures and data analysis techniques used.

10.3 When certification of the test results is required, include the date of the latest apparatus verification and a description of the procedures used. References for the verification report(s) shall also be included. Where applicable, include a statement of laboratory accreditation of the test facility used, including date of latest inspection.

10.3.1 Where agreed upon between the customer and the test laboratory, it is acceptable that less be reported but the remainder of the results shall be made available.

NOTE 21—**Caution:** Where this test method might be specifically referenced in published test reports and published data claims, and where deviations from the specifics of the test method existed in the tests used to obtain said data, the following statement shall be required to accompany such published information: **"This test did not fully comply with following the provisions of Test Method C177."**This statement shall be followed by a listing of specific deviations from this test method and any special test conditions that were applied.

Test Report

Date:	Test Report Number:
Operator:	Duration of Test:

Specimen Identification:	Product, name, manufacturers description.
Specimen Characteristics:	Unique characteristics such as degree of homogeneity or anisotropy, density (optional).
Specimen Conditioning:	Temperature, time, humidity.
Specimen Dimensions and Mass:	Before and after conditioning and after measurement.

Apparatus Description:	Size, shape and orientation of plates. Single or double-sided operation, description of secondary guarding, unique procedures.

Experimental Results

		Uncertainty	
Variable	**Measured Value**	**Systematic**	**Random**
Q, W			
Th, K			
Tc, K			
Tm, K			
ΔT, K			
A, m^2			
L, m			
Fill gas pressure, Pa			
Other			

Derived thermal transmission properties including the applicable range of conditions shall be in conformance with Practice C 1045.

FIG. 3 Example Test Report Form

11. Precision and Bias

11.1 This section on precision and bias for the guarded hot plate apparatus includes a discussion of; general statistical terms; statistical control; factors affecting test results; ruggedness tests; interlaboratory comparisons conducted by ASTM Committee C-16; proficiency testing conducted under the auspices of the National Voluntary Laboratory Accreditation Program (NVLAP); and error propagation formulae.

11.2 *General Statistical Terms*—The accuracy of a test result refers to the closeness of agreement between the observed value and an accepted reference value. When applied to a set of observed values, the accuracy includes a random component (imprecision) and a systematic component (bias). The variability associated with the set of observed values is an indication of the uncertainty of the test result. Additional information on statistical terminology is available in Terminology E456.

11.3 *Statistical Control*—The user of the guarded-hot-plate apparatus shall demonstrate that the apparatus is capable of performing in a consistent manner over time (**35**). The use of control charts (see Manual 7 (**34**)) to monitor the operation of the guarded hot plate is one recommended way to monitor the control stability of the apparatus. When possible, it is recommended that a reference material traceable to a national stand ard s laboratory be used as the control specimen. Ideally, the long-term variation should be no greater than the short-term variability.

11.4 *Factors Affecting Test Results*—Experiments and theoretical analyses have identified two principal (systematic) errors that affect the operation of an idealized guarded hot plate apparatus. These errors are edge heat flows at the periphery of the specimens; and, heat flow across the gap due to a thermal imbalance. Other errors studied include the effect of gap width on the heat flow; and, the proper determination of the metered section area. These errors and others are discussed in detail in A1.3.

11.4.1 *Edge Loss Errors*—These have been found to depend on the size (and type) of the guard, the specimen thermal conductivity and thickness, and ambient temperature **(7,18,20, 21,31,33)**. By using a sufficiently wide guard (see Section 6), appropriate levels of edge insulation, and proper selection of the ambient temperature (see Section 8), the edge loss error can be reduced to a negligible value relative to the specimen heat flow (see Annex A4.2). There is only limited experience (at room temperature) with measurement of apparent conductivity at large thickness' (above 30 cm), but experience suggests that errors are some times expected to be above 2 %, especially if the user does not reduce the problems associated with long time constants and large lateral heat flows **(31)**.

11.4.2 *Gap Imbalance Error*—These have been found to depend on several parameters including the temperature difference, the gap geometry, the structural support system, the wires crossing the gap (number, size, and type), the gap fill material (gas or insulation), the emittance of the gap surfaces, and the specimen material in the vicinity of the gap **(5,6,8,18, 22,36)**. The resulting heat flow due to a temperature imbalance can be obtained either by calculation based on the above parameters or empirical data. An empirical relationship for the gap heat flow can be determined by purposely introducing a temperature imbalance across the gap and measuring the resulting change in the specimen heat flow (see A1.4.3).

11.5 *Ruggedness Tests*—The results of one ruggedness study for a 200 mm^2 guarded hot plate and two materials having different thermal conductivity's have been reported **(37)**. Matched pairs, 85 mm thick, of polyurethane foam and silicone rubber were measured at a mean temperature of 297 K and a temperature difference of 23 K. For each specimen, the width of edge insulation was set at one of five levels (0, 12.7, 25.4, 50.8, and 76.2 mm) while the ambient temperature was varied at one of three levels. The results indicate that the edge losses are reduced with edge insulation but only become zero when the ambient temperature is at one specific value. The optimum ambient temperature appears to be a function of specimen thickness and thermal conductivity, and edge insulation thickness.

NOTE 22—As noted in Section 8, the value of the ambient temperature is set to either the same value as the mean temperature of the test or a value slightly above the mean temperature. The user should determine the optimum value for their apparatus and test conditions by using the sensitivity analysis described in A4.2. This dependence may change appreciably for different specimens or apparatus conditions and, therefore, should be done under typical test conditions.

11.6 *Interlaboratory Tests*—The results of three published interlaboratory tests for guarded-hot-plate apparatus are discussed below. The results, where appropriate, state an index of precision (between laboratory) of two-standard deviation limits (2s). Certain aspects of the interlaboratory tests were not conducted completely in accordance with the requirements of Practice E691, for example, the number of test laboratories was less than six in one study and none of the studies required replicates. Furthermore, a study involving a variety of materials is needed. Consequently, a general statement for the index of precision and bias that covers all conditions and materials is unavailable. In the interim, the user is directed to the interlaboratory tests if information on precision and bias is needed (see Practice C687 for loose-fill materials).

11.6.1 In 1951, results of an interlaboratory comparison were reported **(38)**f or 20 guarded-hot-plate apparatus from 17 laboratories. The plates ranged in size from 200 to 600 mm square. Different (numbered) pairs of corkboard (25 mm thick) were measured by each laboratory at a mean temperature from 266 to 322 K. The data from 15 of the 20 apparatus (75 %) were within ±3 % of the mean value as determined by the National Bureau of Standards (now the National Institute of Standards and Technology). The maximum deviations were + 13 and – 16 %

11.6.2 In 1985, results of a third round of interlaboratory comparisons were reported **(41)** for five large guarded-hot-plate apparatus ranging from 610 to 1219 mm^2 and 1016 mm diameter (the last apparatus mentioned being a circular line-heat-source guarded-hot-plate). The same specimens of fibrous-glass blanket (16 kg/m^3) were circulated to each laboratory. Matched pairs were tested at 297 K and thicknesses of 25.4, 50.8, 76.2, and 101.6 mm. Imprecision of the data versus a semi-empirical model for a density range of 11 to 20 kg/m^3 were 1.9, 2.3, 2.6, 2.9 % (2s level) at thicknesses of 25.4, 50.8, 76.2, 101.6 mm, respectively.

11.6.3 In 1988, results of a interlaboratory comparison were reported **(30)** for seven high-temperature guarded-hot-plate apparatus. The plates ranged in size from 203 to 406 mm in diameter and 300 to 610 mm^2. Different matched pairs of fibrous alumina-silica and calcium silicate were measured by each laboratory over a mean temperature range from 330 to 701 K. Reference equations based on NIST-Boulder corrections were fit to the data. Imprecision in the deviations from the model were 15 and 16 % (2s level) for fibrous alumina-silica and calcium silicate, respectively. It was established that a significant percentage of the standard deviation in this comparison was due to material variability and not apparatus error.

11.7 *Proficiency Tests*—In 1985, the results of a series of proficiency tests conducted for NVLAP over a four-year period were reported **(39)**for guarded-hot-plate apparatus (plate size not reported). Different specimens of four thermal insulation materials were distributed to each laboratory for testing. The materials were expanded polystyrene; foam board; low-density glass-fiber batt (8 to 16 kg/m^3); and, high-density glass-fiber batt, foil-faced (64 kg/m^3). Each laboratory reported a single test result, that is, no replicates were conducted. Results of the proficiency tests are summarized in Table 1. The index of precision (between laboratory) is expressed as a percentage for the one-standard deviation limit(s) divided by the mean of the test result, or one-coefficient of variation (CV %).

TABLE 1 NVLAP Proficiency Tests for Guarded-Hot-Plate Apparatus Ref (39)

Material	Nominal Thickness, mm	Thermal Conductivity Group Mean, W/(m K)	Number of Labs	Coefficient of Variation, %	Round
Expanded polystyrene board	25	0.037	6	1.80	10
Foam Board, rigid	25	0.040	9	2.52	4
Glass-fiber batt	25	0.040	10	2.15	5
Glass-fiber batt	25	0.040[A]	6[A]	2.26[A]	7[A]
Glass-fiber batt	25	0.039[A]	7[A]	2.82[A]	3B[A]
Glass-fiber batt	25	0.040	9	3.28	3A
Glass-fiber batt	25	0.040	7	3.43	7
Glass-fiber batt	25	0.040	9	4.66	3B
Glass-fiber batt, foil faced	25	0.032	9	0.98	6
Glass-fiber batt, foil-faced (stacked)	50	0.033	7	1.45	9
Glass-fiber batt, foil faced	25	0.032	8	1.95	8

[A] Recalculation with one or more laboratories excluded from the group statistics because their test results deviated from the pre-characterized value by more than 6 %.

11.8 *Error Propagation*—Several formulae are available (**40**) for determining the apparatus uncertainty by error propagation. For guidelines on using a standard procedure, the user is referred to ISO Guide to the Expression of Uncertainty in Measurement (**32**). Strictly speaking, determining a statement of uncertainty for a test result requires treating random and systematic errors separately. A description of random and systematic errors and possible sources of error are discussed below.

11.8.1 *Random Error,* δ_r—In a measurement, random errors (imprecision) are considered to be the sum total of all the small (negligible) independent errors that are uncontrolled, for example small fluctuations in environmental conditions or plate temperatures. Random errors are assumed normally distributed, uncorrelated, and preferably small. In general, random errors are a function of the capabilities of the control system and, to a lesser extent, the measurement system.

11.8.2 *Systematic Error,* δ_s—A systematic error (bias) is a fixed deviation that is inherent in each and every measurement. If the magnitude and direction of the systematic error are known, the user can make appropriate correction(s) to the measured value. Under such circumstances a justification for the correction should be provided. In general, the magnitude of the error, $|\delta_s|$, is estimated by experience or judgment.

11.8.3 *Statement of Uncertainty*—The statement of uncertainty requires an expression having credible limits for its inaccuracy. Different traditions and usage have resulted in different expressions of uncertainty that can be summarized as follows: both imprecision and bias negligible; imprecision negligible, bias not negligible; neither imprecision nor bias negligible; and, imprecision not negligible, bias negligible.

11.8.4 *Sources of Errors*—The uncertainty of the apparatus as determined by propagation of errors should consider the error in each of the separate measurements used to determine the test result. For a guarded-hot-plate apparatus, these errors in measurements are the uncertainty in: heat flow δQ; temperature difference, $\delta\Delta T$; metered section area, δA; and specimen thickness, δL. These errors and an example are discussed in A1.3.

12. Keywords

12.1 error analysis; guarded-hot-plate; heat flow; heat flux; steady-state; thermal conductivity; thermal resistance; thermal transmission; thermal conductance; thermal testing

ANNEX

(Mandatory Information)

A1. THICKNESS MEASUREMENT, LIMITATIONS AND MEASUREMENT UNCERTAINTY

A1.1 *Importance of the Thickness of the Insulation Specimens in Guarded-Hot-Plate Measurements*—The thickness of the specimen as installed in the apparatus determines both the density of the material and the temperature gradient applied to it during the measurement of the thermal property. If the thickness of a specimen is changed from its room-temperature value by thermal effects (thermally reversible expansion or contraction, or thermally induced irreversible shrinkage or expansion of the specimen), or by compression, then use of the room-temperature thickness outside the apparatus will lead to error in the determination of the apparent conductivity (or resistivity) of the specimen. A given relative (percentage) error in the thickness leads to an equal relative error in the determination of the conductivity. For measurements of thermal properties at mean specimen temperatures near room temperature it is possible that the error in neglecting any changes in thickness will be negligible, but this can be ascertained only by observation in the specific case at hand.

A1.2 *Suggested Ways to Measure Thickness of Incompressible Specimens*—In determining the thickness of a specimen, one assumes that it is properly shaped, so that the measured thickness is valid. However, two different situations may sometimes occur to affect the thickness measurement. It is possible that the shape of the specimen will be distorted by warping or bowing at the time it is first installed in the apparatus. In this case, either the (flexible) specimen should be compressed enough to remove the distortion when installed, (or, preferably, a specimen of better quality should be selected). Independent of this, it is possible that the specimen will

undergo a change of shape as it is subjected either to high mean temperatures or to large temperature gradients, due to chemical changes occurring in the specimen at high temperatures. In this case it is difficult to define what the thickness of the specimen actually is during the measurement. The thickness of the specimen needs to be measured both before and after the thermal transmission property is measured, to show whether such dimensional changes are occurring. Any warping or bowing of the specimen, before or during measurement of thermal properties, adds to the uncertainty in the value of thickness. Some materials such as polymers have large coefficients of expansion and the material tends to bow unless a small thickness and temperature difference across the specimen is used.

A1.2.1 The recommended procedure for measuring specimen thickness is to measure the thickness while installed in the apparatus. This is necessary if the correct temperature gradient actually applied to the specimen during the measurement of the thermal property is to be obtained. Install rigid rods securely extending laterally from the outer edges of the metered area/primary guard assembly, at two or three equally spaced locations along the circumference of the plate. The portion of the rod extending from the plate shall be smooth and parallel to the plane of the plate surface. Alternatively, the plates may be machined with flat, horizontal plates extending from the circumference. Similar rods (or plates) are likewise located on each auxiliary heater plate, at the same circumferential positions, vertically (within 5° of arc) above or below the rods on the metered area/primary guard assembly.

A1.2.1.1 With no specimens installed, with the heater plates contacting each other in their usual order, and taking care not to change the plate separation, measure the separation between each vertical pair of rods on two adjacent plates with a vernier calliper. Compute the arithmetic mean of the plate separation for each pair of adjacent plates. Then, with specimens installed between the plates in the apparatus, and with the usual mechanical loading applied, measure the separation between the pairs of rods on adjacent plates, taking care not to change the plate separation. Compute the arithmetic mean. Subtract the mean separation obtained with no specimen from the mean separation with the specimen present, for corresponding pairs of plates, to obtain the as-installed thickness of each specimen. The standard deviation about the average of values from repeated measurements of the plate separation, starting from total disassembly, gives a statistical measure of the reproducibility. If contact cannot be made between the plates, standard spacers can be inserted between the plates. Bringing the plates in contact with the spacers can determine the adjustment in specimen measured thickness required.

A1.2.1.2 The accuracy of this procedure is equal to the imprecision with which the vernier can be read. The accuracy of this test method depends on the precision with which the rods are mounted in a true horizontal orientation, and on not changing the plate separation during the measurement. The standard deviation about the average of values from repeated measurements of the plate separation, starting from total disassembly, gives a statistical measure of the reproducibility.

A1.2.2 An alternative is to place the specimen on a flat surface and measure the thickness at various points across the specimen with a thickness gage mounted above the specimen. The zero is first established by resting the foot of the gage on the flat surface. The specimen is then measured. This procedure has the advantage that specimen flatness and warp can be measured. Thickness is measured typically in at least five different locations across the full specimen and within the metered section to establish the metered thickness within the apparatus. The thickness, when applicable, is measured after the test to monitor any significant changes that have the potential to affect the results.

A1.2.2.1 The accuracy of this test method is equal to the imprecision with which the gage can be read. The accuracy and reproducibility of this test method depends on the ability of the operator to reproduce the amount of force exerted on the specimen especially in the case of compressible specimens.

A1.2.3 Another alternative is to use a micrometer or vernier calliper. This assumes that the specimen is not bowed or warped, that should of course be ascertained. During a measurement of thickness with a calliper, prevent the narrow jaws of the measuring tool from penetrating into the surface of the specimen. Cut two small pieces of flat, rigid rectangular metal sheet, about 20 by 40 mm and 0.5 to 1.0 mm thick. Measure the combined thickness of the two metal rectangles; then measure the thickness of the specimen while holding one metal piece under each jaw, between the surface of the specimen and the jaws of the micrometer or calliper. Be sure to subtract the combined thickness of the two metal plates from the total thickness of specimen plus metal pieces, to obtain the net specimen thickness. By this method measure the thickness at eight different, equally spaced locations around the outer margin of the specimen.

A1.2.3.1 The accuracy of this procedure is equal to the precision with which the vernier (or micrometer) can be read. The accuracy and reproducibility of this test method is lower than that described above in A1.2.1 and A1.2.2, due to the variable pressure used by different people in measuring the specimen between the jaws of the micrometer or calliper.

A1.3 *Limitations Due to Apparatus:*

A1.3.1 *Limitations Due to Contact Resistances*—When testing a rigid specimen of high thermal conductance (that is, specimens of a material too hard and unyielding to be appreciably altered in shape by the pressure of the heating and cooling units), even small, non-uniformities of the surface of both the specimen and the apparatus (surfaces not perfectly flat) will allow contact resistances not uniformly distributed between the specimens and the plates of the heating and cooling units.

A1.3.1.1 These will cause nonuniform heat flow-rate distribution and thermal field distortions within the specimens; moreover, accurate surface temperature measurements will be difficult. For specimens having thermal resistances less than 0.1 m^2 K/W, special techniques for measuring surface temperatures will be required. Metal surfaces should be machined or cut flat and parallel and stress-relieved.

A1.3.2 *Upper Limits for the Thermal Resistance:*

A1.3.2.1 The upper limit of thermal resistance that can be measured is limited by the stability of the power supplied to the metered section, the ability of the instrumentation to measure power level and the extent of the heat losses or gains due to temperature imbalance errors between the central and guard sections of the specimens and of the metered section.

A1.4 *Limits to Temperature Difference:*

A1.4.1 Providing uniformity and stability of the temperature of the hot and cold surfaces of the plates, the noise, resolution and temperature measurements can be maintained within the limits outlined in Section 6, temperature differences as low as 5 K, when measured differentially, can be used. Lower temperature differences shall be reported as not complying with this standard. See Practice C1058.

A1.4.2 If temperature measurements of each plate are made by means of thermocouples with independent reference junctions, it is possible that the accuracy of the calibration of each thermocouple will be the limiting factor in the accuracy of measured temperature differences. In this case, it is recommended that temperature differences of at least 10 K to 20 K are used in order to minimize temperature-difference measurement errors.

A1.4.3 Higher temperature differences are limited only by the capability of the apparatus to deliver enough power while maintaining required temperature uniformity.

A1.4.4 *Maximum Specimen Thickness:*

A1.4.4.1 The boundary conditions at the edges of the specimens due to the effects of edge insulation, of auxiliary guard heaters and of the surrounding ambient temperature will limit the maximum thickness of specimen for any one configuration, as described in Section 6. For composite or layered specimens, the mean measurable thermal conductivity of each layer should be less than twice that of any other layer.

A1.4.4.2 This is an approximation and the results do not necessarily imply the measurement of conductivity of each layer. The accuracy will remain close to that predictable for tests on homogeneous specimens. No guidelines can be supplied to assess measurement accuracy when the requirement of 2.3 is not met.

A1.4.5 *Minimum Specimen Thickness:*

A1.4.5.1 The minimum specimen thickness is limited by contact resistances given in A1.3.1. Where thermal conductivity or thermal resistivity is required, the minimum thickness is also limited by the accuracy of the instrumentation for measuring the specimen thickness.

A1.4.5.2 The metered area, that is, the area of the specimen traversed by the heat flow-rate fed by the metered section, is related to the specimen thickness and to the gap width. As the thickness tends to zero, the metered area tends to the area of the metered section, while for thick specimens the metered area is bounded by the line defining the centre of the primary guard gap. To avoid complex corrections, this definition can be retained, provided the thickness of the specimen is at least ten times the width of the gap.

A1.4.6 *Maximum Operating Temperature:*

A1.4.6.1 It is possible that the maximum operating temperature of the heating and cooling units may be limited by oxidation, thermal stress or other factors that degrade the flatness and uniformity of the surface plate and by changes of electrical resistivity of electrical insulations that affect accuracy of all electrical measurements.

A1.4.7 *Vacuum Conditions:*

A1.4.7.1 Care must be taken if a guarded hot plate is used for measurements under vacuum conditions. If a high vacuum is desired, the materials used in the design of the apparatus must be carefully selected to avoid excessive outgassing under such conditions. Under vacuum conditions, especially at lower temperatures, serious errors can arise if care is not taken when installing heater and temperature sensor leads so as to minimize extraneous heat flow-rates and temperature measurement errors.

A1.4.8 *Apparatus Size:*

A1.4.8.1 The overall size of a guarded hot plate will be governed by the specimen dimensions that typically range from 0.2 to 1 m diameter or square. Samples smaller than 0.3 m are potentially not representative of the bulk material, while specimens larger than 0.5 m have the potential to create considerable problems in maintaining the flatness of the specimens and plates, temperature uniformity, equilibrium time and total cost within acceptable limits.

A1.5 *Limitations Due to Specimen:*

A1.5.1 *Thermal Resistance or Thermal Conductance:*

A1.5.1.1 *Specimen Homogeneity*—In inhomogeneous specimens, the thermal flux density both within the specimen and over the faces of the metered section area has the potential to be neither unidirectional nor uniform. Thermal field distortions will be present within the specimen and can give rise to serious errors. The region in the specimen contiguous to the metered section area and especially near the edges of this area is most critical. It is hard to give reliable guidelines on the applicability of the method in such cases. The major risk is that the imbalance errors, edge heat loss errors, etc., now unpredictable, can vary in an unpredictable way when inhomogeneities take different relative positions within the specimen.

A1.5.1.2 One way to estimate the error is to compare the results for two specimens from the same sample, selected so that they have as widely different a structure near the edges or the metered section area. If the two extremes cannot be identified, a number of specimens may have to be tested.

A1.5.1.3 In some samples, the variation in structure may occur over small distances. This is true for many thermal insulations. In such cases, it may be possible to use a single specimen cut larger than the apparatus. This over-size specimen is tested twice, in each case with the specimen carefully positioned so that the edges of the test area are exposed to the two extremes in structure. The two results are then compared and the difference credited to distortion. The portion of the specimen(s) protruding from the apparatus should be well insulated in the two tests to reduce the possibility of the exposed section increasing edge losses. The size and thickness of the specimen affects the size of the variations in structure

that can be accommodated. The larger the test area, the smaller the effect on the results. The effect of distortion may either increase or decrease with specimen thickness.

A1.5.1.4 Direct thermal short circuits may exist between the surfaces of the specimens in contact with the plates of the heating and cooling units. The largest effect occurs when sections of material which conduct heat readily, with extended surface area on each side of the specimen, are connected by a path of low thermal resistance relative to other paths. The effect can best be identified by breaking the thermal paths, especially when the collecting surfaces can be disconnected from the rest of the path. Sheets of thermally insulating materials can be used at the critical surfaces to provide the break. Sheets made of finely ground cork, or a similar material 2 mm or more thick, work well. The surfaces must be ground to the same degree of flatness as the heating unit. The thermal resistance of these sheets can be determined in separate measurements. The net change in thermal resistance of the specimen, due to thermal shorting, can thus be determined. If greater than 1 %, another measurement should be made with thicker sheets imposed.

A1.5.2 *Temperature-Difference Correlation:*

A1.5.2.1 Thermal resistance or thermal conductance are often a function of temperature differences across the specimen. In the report, the range of temperature differences that apply to the reported values of the two properties must be defined, or it must be clearly stated that the reported value was determined at a single temperature difference.

A1.5.3 *Mean Measurable Thermal Conductivity of a Specimen:*

A1.5.3.1 In order to determine the mean measurable thermal conductivity (or thermal resistivity) of a specimen, the criteria of A1.3.1 shall be fulfilled. The specimen shall be homogeneous. Homogeneous porous specimens shall be such that any inhomogeneity has dimensions smaller than one-tenth of the specimen thickness. In addition, at any one mean temperature, the thermal resistance shall also be independent of the temperature difference established across the specimen.

A1.5.3.2 The thermal resistance of a material is known to depend on the relative magnitude of the heat transfer process involved. Heat conduction, radiation and convection are the primary mechanisms. However, the mechanisms can combine or couple to produce non-linear effects that are difficult to analyze or measure even though the basic mechanisms are well researched and understood.

A1.5.4 The magnitude of all heat transfer processes depends upon the temperature difference established across the specimen. For many materials, products and systems, a complex dependence may occur at temperature differences which are typical of use. In these cases, it is preferable to use a temperature difference typical of use and then to determine an approximate relationship for a range of temperature differences. The dependence can be linear for a wide range of temperature differences.

A1.5.5 Some specimens, while being homogeneous, are anisotropic in that the thermal conductivity measured in a direction parallel to the surfaces is different to that measured in a direction normal to the surfaces. For such specimens, this can result in larger imbalance and edge loss errors. If the ratio between these two measurable values is lower than two, reporting according to this method is still possible if imbalance and edge heat loss errors are determined separately with anisotropic specimens mounted in the apparatus.

A1.5.6 *Thermal Conductivity or Thermal Resistivity of a Material:*

A1.5.6.1 In order to determine the thermal conductivity or thermal resistivity of a material, the criteria of A1.3.2 shall be fulfilled. In addition, adequate sampling must be performed to ensure that the material is homogeneous or homogeneous porous, and that the measurements are representative of the whole material product or system. The thickness of the specimens must be greater than that for which the thermal conductivity of the material product or system does not change by more than 2 % with further increase in thickness.

NOTE A1.1—Results obtained on specimens where thermal conductivity is still changing with specimen thickness are only applicable at that specific test thickness.

A1.5.7 *Dependence on Specimen Thickness:*

A1.5.7.1 Of the processes involved, only conduction produces a heat flow-rate that is directly proportional to the thickness of a specimen. The others result in a more complex relationship. The thinner and less dense the material, the more likely that the resistance depends on processes other than conduction. The result is a condition that does not satisfy the requirements of the definitions for thermal conductivity and thermal resistivity, both of which are intrinsic properties, since the transfer factor shows a dependence on the specimen thickness. For such materials, it may be desirable to determine the thermal resistance at conditions applicable to their use. There is believed to be a lower limiting thickness for all materials below which such a dependence occurs. Below this thickness, the specimen may have unique thermal transmission properties, but do not relate to the material. It remains, therefore, to establish this minimum thickness by measurements.

A1.5.7.2 Determination of minimum thickness above which thermal properties of the material may be defined.

A1.5.7.3 If the minimum thickness for which the thermal conductivity and resistivity can be defined is not known, it is necessary to estimate this thickness.

A1.5.7.4 In the absence of an established method, the procedure outlined below may be used to approximate the thickness and whether it occurs in the range of thickness in which a material is likely to be used.

A1.5.7.5 It is important to differentiate between added thermal resistance in measurements caused by the placement of the temperature sensors below the surfaces of the plates, added resistance caused by poor specimen surfaces, and added resistance caused by the coupling of the conduction and radiation modes of heat transfer in the specimens. All three can affect the measurements in the same way, and often the three may be additive.

A1.5.7.6 Select a sample uniform in density distribution, with the thickness L_5, equal to the greatest thickness of the material to be characterized or equal to the maximum allowable thickness for the test apparatus.

A1.5.7.7 Cut five sets of specimens in approximately equal increments from the sample ranging in thickness from the smallest likely to be used in practice. The set of specimens shall be designated s_1 to s_5 according to their respective thickness L_1 to L_5.

A1.5.7.8 For low density materials where heat is transferred by radiation and conduction mechanisms and where the absence of convection has been verified, the slope of a plot of thermal resistance versus thickness will very frequently diminish up to 1 to 2 cm and then will remain constant as the thickness increases. The reciprocal of this constant slope is the thermal conductivity to be assigned to high thickness specimens.

A1.5.7.9 Measure the thickness and thermal resistance of s_1, s_3, and s_5 at the same mean temperature and with the same temperature difference across the specimen. Plot the thermal resistance versus thickness. If these three values differ from a straight line relationship by less than ± 1 %, the slope of the straight line shall be computed. If the three values differ by more than 1 %, then similar measurements shall be made on s_2 and s_4 to check if there is a thickness above which the thermal resistance does not differ from a straight line by more than 1 %.

A1.5.7.10 If this thickness exists, the slope of the straight line shall be determined to compute a thermal conductivity $\lambda_m = \Delta L/\Delta R$ defined as the ratio between the increments of thickness, ΔL, and increments of the thermal resistance, ΔR.

A1.5.7.11 The thickness at which this occurs will vary according to the densities, types and forms of different materials, products and systems for different mean temperatures.

A1.5.7.12 Thermal conductivity and thermal resistivity then characterizes the material, product or system for thicknesses above which the transfer factor differs by less than 2 % from λ_m.

A1.5.7.13 Allowance for experimental errors must be made in the interpretation of results. Least-square curve fitting of R versus L may also help. A larger number of specimens may be used where greater definition is required.

A1.5.7.14 Thickness dependence may be a function of temperature difference across the specimens. For the purposes of this test method, the above checks, if performed at typical operating temperature differences, shall be adequate to indicate the degree of thickness dependence.

A1.5.8 *Method of Determining Dependence on Temperature Difference*—If the temperature-difference dependence of the thermal properties is not known for a material, a minimum of three measurements shall be made. These are made with widely differing temperature differences. A second-order dependence can be revealed by these measurements. When a simple linear relationship is known to occur, only two measurements, that is, one extra, need be made. This establishes the linear dependence for that particular sample.

A1.5.9 *Warping*—Special care should be exercised with specimens with large coefficients of thermal expansion that warp excessively when subjected to a temperature gradient. The warping may damage the apparatus or may cause additional contact resistance that may lead to serious errors in the measurement. Specially designed apparatus may be necessary to measure such materials.

A1.6 *Measurement Uncertainty*—The uncertainty of the apparatus is based upon consideration of the random and systematic components of the following measurement uncertainties (**32**): uncertainty in heat flow, δQ; uncertainty in temperature difference,ty in metered area, δA; and, uncertainty in specimen thickness, δL.

A1.6.1 Other specimen characterization and test condition data may need to be reported. The precision and bias of these data are to be reported to the extent they have a direct bearing on the accuracy of the results. Prescribed precision and bias of the primary data are not mandated by this test method. However, it is required that the user assess and report the precision and bias of the data. The discussion below provides guidelines to assist the user in performing this uncertainty assessment. A variety of helpful performance checks are included in this discussion. In the following discussion both random and systematic errors are considered. The subscript $_s$ is used to denote systematic, and the subscript$_r$ is used for the random components.

A1.6.1.1 *Systematic Error, δ_s*—Systematic error, δ_s, is any component of error that remains fixed during the runs that constitute a successful test. To simplify the discussion, this does not include any components of error that are known both in magnitude and sign. Under such circumstances, the user should make appropriate corrections to the conductivity measurements and supply the justification for them. The user may check for the presence of unexpected errors by using a reference specimen or transfer standard available from appropriate sources. If errors are discovered, their source should be identified and removed. A guarded hot plate cannot be calibrated. The task of estimating the remaining systematic errors is based on judgment and experience, including an awareness of the results of interlaboratory comparisons. The implications of such estimates is often that they are the maximum possible systematic errors. In this event the total maximum systematic error is the sum of the errors. It is, however, more likely that these estimates are probabilistic in nature and do not, in fact, represent the worst possible case. The total probable systematic errors are summed in the same manner as random errors, that is, the square root of the sum of squares. In the following discussion the latter approach is taken. However, the user must decide if the bias estimates are worst cases or probabilistic in nature, and sum them accordingly.

A1.6.1.2 *Random Error, δ_r*—Random error, δ_r, is that component of error that may vary both in sign or magnitude during the runs that constitute a successful test. For simplicity, it is assumed that the variations are normally distributed and conventional statistical techniques are applicable. An estimate of random error components can be obtained by repeat measurements of each variable.

A1.6.1.3 It is important to distinguish between random and systematic errors for the following reason. The results reported in the test method are mean values derived from more than a single run. The uncertainties reported generally apply to these mean values. The uncertainty of a mean value due to the

random error component decreases approximately as $1/\sqrt{n}$ where n is the number of repeat runs. In contrast to this, the uncertainty of the mean value due to the systematic error component does not decrease with repeat runs. Thus, it is recommended that the error components be treated separately. The total uncertainty is expressed by reporting both components separately.

A1.7 *Error Components*—In the following sections, the error components of each reported variable are discussed. The total random or systematic uncertainty for each variable is taken to be the square root of the sum of squares.

A1.7.1 *Heat Flow, Q*—The objective of the test method is to establish and measure uniaxial heat flow through the metered area of the specimen. Any deviation from this objective represents error in the reported heat flow. The following sources of error should be considered:

A1.7.2 *Edge Heat Loss, $\delta_s Q_e$*—Edge heat loss, $\delta_s Q_e$ is a systematic error as the conditions surrounding the plate-specimen stack remain constant throughout the test procedure. Although tests have been reported that shed some light on the magnitude of this error, the results generally are not proven to the point where corrections based on these results are universally accepted (**1, 4, 6, 7, 18-22**). However, the results are considered sufficiently valid for the basis of defining the maximum specimen thickness. The optimum environmental temperature to minimize this error is a small fraction of T above the mean test temperature. To determine the sensitivity of this error to test conditions, the user should determine the heat flux as a function of secondary guard temperature. This dependence may change appreciably with specimen and apparatus characteristics and, therefore, should be done under typical test conditions.

A1.7.3 *Gap Heat Loss*—Gap heat loss is considered to be composed of both systematic, $\delta_s Q_g$, and random, $\delta_r Q_g$, components. The systematic component can be, in part, due to the fact that there may be a finite number of locations along the gap at which the imbalance is measured; reducing the temperature difference between a finite number of points on opposite sides of the gap to zero may not necessarily ensure that there is zero net flow of heat across the gap. Improper position of the sensors will lead to systematic error. Spurious emfs within the circuitry will result in a systematic imbalance. The random component is due to short-term control fluctuations. After estimating the probable imbalance across the gap in terms of temperature (or sensor voltage) one needs to determine the effect of this imbalance on the measured heat flow through the metered area. This can be done by measuring the dependence of metered area power on intentionally introduced gap imbalance. A typical way of addressing this is to run three tests, one with the guard balanced and one each biased positive and negative. The results are plotted, lambda versus gap balance, and the zero intercept is determined. The imbalance introduced should be large enough to yield an easily measured change in Q, but small enough to remain in the region where the dependence of Q upon imbalance is approximately linear.

A1.7.3.1 It has been found that (**3, 15, 16**) the gap heat loss, δQ_g is linearly dependent on temperature unbalance across the gap, ΔT_g, that is, $\delta Q_g = B\Delta T_g$. The proportionality constant, B, is dependent on the wires crossing the gap (number, size, and type), gap geometry (width and cross-sectional shape), the gap fill material (gas, insulation), the emittance of the gap surfaces and the material in the vicinity of the gap between the hot and cold plates. A reasonable approximation of this heat flow can be calculated from this information. It is recommended that this be done to confirm the value measured by the procedure described in the previous paragraph.

A1.7.4 *Effect of Drift of the Metered Area Heater*—A quasi-heat loss exists due to the changing heat content of the metered area heater as its temperature changes. Typical plates have a relatively high heat capacity and even for small drift rates can produce significant errors in measured heat flow. If the drift is monotonic, the error is systematic, $\delta_s Q_d$; if not, the error is exhibited as random error, $\delta_r Q_d$. Normally, the experiment is conducted so that there is no observable drift. Under this circumstance, the possible drift is determined by the detectability or control limit, dT/dt, of the system. One can compute the magnitude of this error, δQ_d in watts, from a knowledge of the maximum possible dT/dt and the specific heats and masses of the various components of the metered section of the plate as follows:

$$\delta Q_d = dT/dt\ \Sigma C_i M_i \qquad \text{(A1.1)}$$

The specimen heat capacity also contributes to the drift error, but for low-density insulations the heat capacity of the specimen is small compared to the plate. This error also can be determined by measuring the dependence of drift rate on measured heater power. Comparison of the calculated and measured results is advised to increase confidence in the reported result.

A1.7.5 Power determination error, composed of both systematic, $\delta_s Q_p$ and random, $\delta_r Q_p$, components. With high quality instrumentation these errors can be reduced to an insignificant level. The manufacturers' specifications on bias and precision will normally suffice to define these errors.

A1.7.6 *Temperature and Temperature Difference*—Temperature error is composed of systematic, $\delta_s T$, and random, $\delta_r T$, components. In addition, these errors are further subdivided according to the source of the error:

A1.7.6.1 Calibration error, $\delta_s T_c$, is entirely systematic as long as the same calibration is used. It is, however, not necessarily the same for each temperature sensor. In the case of thermocouples, calibration is frequently performed for each spool of wire, not for each piece of wire from that spool. Therefore, systematic differences can occur as one progresses through the spool. The calibration is frequently represented by an equation which approximates the experimental calibration data taken at selected temperatures. If a digital read-out device is used that yields temperature directly, the calibration formulation is built into the device and the same basis for error exists.

A1.7.6.2 Instrumentation measurement error, δT_m, occurs when the sensor output is measured. This error contains both systematic and random components. Each component should be estimated from equipment manufacturer's specifications and from estimated spurious circuit effects. In addition, temperature errors are introduced by long and short-term control

fluctuations. A helpful procedure to assess the magnitude of these errors is as follows. Place the guarded metered area and primary guard(s) in thermal contact with the adjacent cold plates (insert high conductance plates in place of the specimens if the plates cannot be placed physically together). Adjust the cold plates to the desired temperature; control this temperature until steady-state is reached. The metered area heater should be off. Periodically read the isothermal surface temperatures to detect systematic differences and random variations over an extended time.

A1.7.6.3 Sensor positioning, a potentially significant source of error in temperature measurement can be caused by improper positioning of the sensor or the disturbance caused by the presence of or finite size of the sensor itself. It is intended that the average temperature of each specimen surface be measured. If the sensor is mounted in the plate surface, thermal contact resistance between the plate and specimen is a source of error. If the sensor is mounted in the specimen surface, sensor separation (specimen thickness) is a source of error. If the specimen is inhomogeneous across the metered area, surface temperature variations exist and the indicated temperature will depend on its location on the surface. If heat flows along the sensor leads from the external environment, the measured temperature will be in error because of the presence of the sensor. For a single test on a given specimen, this source of error, $\delta_s T_p$, is systematic. A performance check that is helpful to determine the potential temperature error due to temperature nonuniformity is as follows: Assemble a multijunction thermocouple and place it between the specimen and plate in question. Establish steady-state at the desired test condition. Determine the variation in temperature across the plate from the multijunction thermocouple outputs.

A1.7.6.4 A helpful technique to estimate interface temperature errors is to mount sensors both within the plate and within the specimen surface. Then perform a test and calculate the difference between the two sets of data.

A1.7.6.5 Temperature difference error is also composed of systematic, $\delta_s \Delta T$, and random components, $\delta_r \Delta T$. Care must be exercised in estimating these components compared to the error components for temperature itself. The results can depend strongly on whether a differential measurement or two absolute measurements are performed. Because ΔT is frequently small, large percentage errors can occur if care is not observed. For example, at a mean specimen temperature of 300 K, an error of 1 K in the mean temperature, that corresponds to an error of about 0.2 % in thermal resistance for typical insulations. However, this same error of 1 K in measurement of a specimen temperature difference of 25 K corresponds to a 4 % error in both T and in the value of the thermal resistance, independent of the mean temperature. The ad hoc experiment described in A1.7.6.3 is recommended to provide estimates of these error components.

A1.7.7 Specimen thickness error, $\delta_s L$, and meter area error, $\delta_s A$, are both systematic errors. The specimen thickness error is determined by the ability to measure the plate spacing (including variations of this thickness over the metered area) or, in the case of rigid specimens, the specimen thickness and the changes due to thermal expansion. The effect of bowing or warping at operating temperatures should be given attention. At relatively large thicknesses (above 5 cm) this error can be maintained below 0.5 %. At small thicknesses (below 0.5 cm) this error may become a dominating factor in the overall accuracy. The meter area error is usually small except for the assumption about what proportion of the gap area to include. This error is difficult to estimate for very thin specimens or when a discontinuity in the specimen occurs at the gap. The specimen thickness error will contain a random component, $\delta_r L$, due to assembly and disassembly.

A1.8 *Thermal Conductance or Thermal Resistance*—The relative uncertainty in thermal conductance, C, caused by either random errors or systematic errors of indeterminate sign, may be calculated from the following error propagation formula:

$$(\delta C/C)^2 = (\delta Q/Q)^2 + (\delta \Delta T/\Delta T)^2 + (\delta A/A)^2 \qquad \text{(A1.2)}$$

where $\delta Q/Q$ and $\delta \Delta T/\Delta T$ and $\delta A/A$ are the total relative uncertainties of heat flux, temperature difference, and meter area respectively. The same equation applies to thermal resistance. Included in the total relative uncertainties are those due to the measurement as well as those discussed in Practice C1045. For example for fibrous glass insulation at 24°C mean temperature and a 22°C temperature difference across the specimen the following errors can be realized Note that the example below uses hypothetical values for $\delta Q/Q$ and $\delta \Delta T/\Delta T$. The user must determine their own values for this calculation:

$$(\delta C/C)^2 = (0.5)^2 + (0.25)^2 + (0.01)^2 = 0.31 \qquad \text{(A1.3)}$$

Therefore, the uncertainty in thermal conductance would be $\sqrt{0.31} = 0.56\,\%$.

A1.9 *Thermal Conductivity or Thermal Resistivity*—The relative uncertainty in thermal conductivity caused by either random or systematic errors may be calculated from the following error propagation formula:

$$\left(\frac{\delta\lambda}{\lambda}\right)^2 = (\delta Q/Q)^2 + (\delta \Delta T/T)^2 + (\delta A/A)^2 + (\delta L/L)^2 \qquad \text{(A1.4)}$$

where $\delta A/A$ and $\delta L/L$ are the total relative uncertainties of area and thickness, respectively. Again, the above total relative uncertainties include not only the measurement uncertainty, but also the effect of material variability and deviations from the definitions as discussed in Practice C1045. In addition, it should be noted that the temperature to which each measured property is assigned also contains a measurement error that affects the uncertainty of the final result. The effect of this error increases as the temperature dependence of the measured property increases.

A1.9.1 For example for fibrous glass insulation at 24°C mean temperature and a 22°C temperature difference across the specimen the following errors can be realized. Again the example below uses hypothetical values for these uncertainties. The user must obtain their own input values.

$$\left(\frac{\delta\lambda}{\lambda}\right)^2 = (0.5)^2 + (0.25)^2 + (0.01)^2 + (0.1)^2 = 0.32 \qquad \text{(A1.5)}$$

Therefore, the uncertainty in thermal conductivity would be $\sqrt{0.32} = 0.57\,\%$.

A1.10 It is recommended that the user periodically confirm these calculated uncertainties by measuring specimens of established standard reference materials or calibrated transfer specimens. Comparison of the measurement results with the accepted values will reveal whether the performance of the guarded hot plate is of acceptable quality. The results of such comparative measurements are not to be used to obtain an apparatus "calibration" or "correction" factor. For further information on this see Refs (**23-29**).

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DECLARACIÓN Y AUTORIZACIÓN

Yo, **Becerra Campoverde, George Anthony**, con C.C: # 0704549898 autor del trabajo de titulación: **"Influencia de los tratamientos: alcalino, hornificación y látex con humo de sílice en la conductividad térmica de los morteros reforzados con fibras de abacá"** previo a la obtención del título de **Ingeniero civil** en la Universidad Católica de Santiago de Guayaquil.

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REPOSITORIO NACIONAL EN CIENCIA Y TECNOLOGÍA			
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AUTOR(ES)	George Anthony Becerra Campoverde		
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RESUMEN/ABSTRACT (150-250 palabras): En este trabajo se tiene el propósito de analizar el comportamiento térmico del mortero reforzado con fibra de abacá con los diferentes tratamientos planteados que son: fibra tratada con hidróxido de sodio (NaOH 3%), tratada aplicando goma con humo de sílice, y pasando un proceso denominado de hornificación. Se realizará la comparativa de los tres tipos de mortero y adicionalmente con un mortero convencional para determinar el mejor comportamiento entre los tres tipos de morteros mencionados; para lo cual se realizó una muestra de cada tipo de mortero donde se aplicó a los veintiocho días el ensayo de conductividad térmica y adicionalmente se realizaron ensayos de densidad y flujo respectivamente de cada tipo de mortero. Se presentará paso a paso cada tratamiento que se realiza a la fibra de abacá y el proceso de elaboración de las muestras. El análisis de la conductividad térmica se centrará en el mortero simple versus el mortero reforzado con fibra de abacá previamente tratada con hidróxido de sodio debido que este tratamiento presenta un mejor comportamiento en el análisis termogravimétrico.

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