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TRADUCCION OFICIAL No. 05/599 de un documento escrito en ingles, el cual para su identificaciôn se sella con el sello del traductor at tiempo con la presente. Esta traducciôn ha sido preparada par Juan Pablo Piedrahita Aguilera, Traductor e IntØrprete Oficial debidamente reconocido de conformidad con el Certificado de Idoneidad Profesionat No. 0045 expedido par la Universidad Nacional de Colombia, par el Ministerio de Relaciones Exteriores y autorizado par el Ministerlo del Interior y de Justicia, par resoluciOn
nümero 718 de 2003.

(Se traduce un Cerhficado notarial, cerhficado de autorizaciOn del Estado de Hinos y Apostilla No. C05GL23962, escritos en inglØs, adjuntos a un poder otorgado por la compaæia UNITED STATES GYPSUM COMPANY, firmado par Michel M. Geoffrey, con fecha 12 de octubre, 2005, escrito en inglØs y esoaæol.)

(En espaæol e ingles, poder otorgado por la compaæia UNITED STATES

GYPSUM COMPANY, firmado por Michel M. Geoffrey, con fecha 12 de octubre,

2005, una (1) hoja).

CERTIFICADO NOTARIAL (Cuando el solicitante es una SOCIEDAD)

(Poderes otorgados en El Salvador, Mexico, Estados Unidos de America y

Venezuela)

Expedido hoy dIa 12 de octubre, 2005, el suscrito Notario CERTIFICA que:

1). Michael M. Goifrey, (nombre de quien firma el poder), mayor de edad y

domiciliado en Chicago, Illinois, suscribió el anterior documento.

2) El otorgante le es conocido personalmente y tiene capacidad para firmar el

documento.

3) El otorgante ha suscrito el anterior documento en su calidad de Secretario

Asistente de la persona juridica UNITED STATES GYPSUM COMPANY.

4) La mencionada persona jurIdica con domicilio comercial en Chicago, Illinois, tjmente constituida, tiene actualmente existencia legal y elfin para el
 :

cI:aeste poder se encuentra dentro de su objeto social.

T 5itpfnte tlene en efecto la capacidad refenda y su representacion es

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legal y estÆautorizado para otorgar este poder.

6) Fundamento las certificaciones 3), 4) y 5) en los documentos autØnticos que

para tal fin me fueron exhibidos.

(Firmado) AIDA BLEKHMAN, Notario Piiblico

(Sello Notarial a tinta: SELLO OFICIAL, AIDA BLEKHMAN, NOTARIO PUBLICO - ESTADO DE ILLINOIS, MI COMISION VENCE SEPT. 30, 2006’ Nota: El Consulado de Colombia debe autenticar Ia firma del Notario PUblico.

Para todos los demªs palses el SeæorConsul de Colombia o ante quien se otorgue, debe expedir una certificación dejando constancia de: 1) Ia existencia legal de la sociedad, 2) que curnple actualmente con su objeto social, y 3) que Ia persona o personas que otorgan este poder estÆn facultadas para hacerlo. El Consul tambiØn certificarª que los tuvo a Ia vista los documentos que prueban y acreditan tales circunstancias. LEGALIZACION POR EL CONSUL DE COLOMBIA A 0 POR APOSTILLA

(Sello Pre-impreso del Condado de Cook, Illinois)
Estado de Illinois
Condado de Cook)

CERT!F!CADO DE AUTOR!ZACION

Yo, DAVID ORR, Escribano del Condado de Cook en el Estado de Illinois certifico
BLEKHMAN AIDA

iLa mbrada en el sello y firma del documento adjunto es un Notario rA14 EL Estado de Illinois, y estaba autorizado para obrar como tal

IRADUCCION OFICAL No / 5 99
2 JUAN PABLO PIEDRAHITA AGUILERA
TRADUCTOR E INTERPRETE OFKJAL

RES. 78 OF MAYO 2003 - L MN. INTERIOR Y LA JUSTICl

en el momento de la presentación en Notarla de dicho documento.

Para verificar este Certificado de Autorización para un Acto Notarial, he estampado mi firma y seUo oficial hoy dIa 26 de Octubre, 2005.
(Firmado) David Orr
Condado de Cook, Estado de Illinois

(Firmado) Maria Canalu

(Sello en Relieve, Condado de Cook, Estado de Illinois)

ESTADO DE ILLINOIS

SECRETARIA DE: ESTADO

SPRINGFIELD, ILLINOIS
APOS TI LLA
(Convención de la Haya de Octubre 5 de 1961)
1. Pals: Estados Unidos de America
Este documento pæblico
2. Ha sido firmado por DAVID ORR
3. Actuando en calidad de Escribano del Condado, Condado
de Cook.
4. Lleva el sello/estampilla del ESTADO DE ILLINOIS
Certi ficada
5. En Chicago, Illinois
6. Hoy 26 de octubre, 2005
7. Por El Secretario de Estado del Estado de Illinois,
8. No. C05GL23962

9. Sello / Estampilla (Sello Redondo Dorado de Oblea del
Estado de Illinois)
rrna (Firmado)
SE WHITE

retario de Estado, Estado de Illinois___

. : ION FIEL Y COMPLETA de la cual se deja copia en los archivos de esta oficina para su
lY on, y a la cual me remito.
ogo4 q I bre 10, 2005.

 ; .. DUCCION OFICIAL No, _
"JUAN PABLO PEDAHITA AGUiLEA
TRADUGOR E INTERPETE QH(JAL
: : RL.;a E MAYO 23
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COLOMBIA
Can-era 11 n°86-53, Piso6 Bogota Colombia Tel: (57 1)6181088 Fax: (57 1)6350824Email: info@clarkemodet.com.co

PODER

Yo (nosotros), abajo firmante(s)

en ml (nuestro) carÆcter derepresentante(s) legates de

UNITED STATES GYPSUM COMPANY

una sociedad organizada y existente de acuerdo con las ieyes de

y en actual cumplimiento de so objeto social, con domicilio en

por ci presente instrumento confiero (conferimos) poder especial a favor de DILIA MARIA RODRIGUEZ DALEMAN, con CØdula de Ciudada-nia No. 41.654.882 de Bogota, y tp. 20824 y CLAUDIA LUCIA CARO RAMIREZ, con CØdula de Ciudadania No. 40.017.374 de Tunja, y tp. 36448, con domicilio en Bogota, Colombia, para que actuando en su orden en nombre del (de Ia) otorgante le (la) representen ante cuaiquier entidad, autondad y/o persona, especialmente ante las del orden Adminis-trativo, Contencioso Administrativo, Judicial, Tribunales de Arbitramento y/o cualquier otro en lo referente a los siguientes asuntos:
Solicitud de Patente en Colombia, presentada bajo el No. 05087218 correspondiente a PCT/US2004/083 146

I) Solicitar, tramitar, obtener y/o registrar cualquiera de las modalidades de Propiedad Industrial, tales como: patentes de invención, modelos de utilidad; diseflos industriales, variedades vegetales; marcas, lemas, nombres y ensefias comerciales y denominaciones de origen; asi como sus renovaciones, pron-ogas, transferencias, cambios de nombre y/o domici-lio, contratos y licencias.
2) Solicitar, tramitar, obtener y/o registrar derechos de autor, en cualquie-ra de sus presentaciones 551 como la protección de secretos industriales y
know-how.

3) Solicitar, tramitar, obtener y/o registrar registros sanitarios, permisoa y/o licencias de funcionamiento; asi como sus renovaciones o prØrrogas, transferencias, cambios de nombre y/o domicilio, contratos y licencias.
4) Ejercer acciones de oposición, observación, reclamos, reconsideración, reposicion, apelacion, cancelación administrativa o judicial, nulidad, protección at consumidor, concesión de licencias, proteccion de secretos industriales y demÆs acciones y recursos relacionados con derechos derivados de los asuntos arriba indicados y en contra de la competcncia desleal, acciones derivadas de la obtención o explotación de licencias, acciones de indemnización de perjuicios. TambiØn podra(n) intervenir en juicios civiles, comerciales, penales, contencioso-administrativos, tutelas y de poiicia; en acciones o juicios promovidos por el (la) mandante o en contra el (ella).
5) Registrar nombres de dominio ante la entidad competente.
6) En ci ejercicio de este mandato los mandatarios an-lbs mencionados quedan facultados para: conciliar, cancelar, transigir, comprometer en Ærbitros, desistir,recibir y tomar posesión de la cosa objeto de Iitigio y renunciar o desistir a derechos concedidos o en curao de concesión, admitir los hechoa del proceso.
7) Ratificar o no la agencia oficiosa.
8) Asi mismo faculto (facultamos) a los mencionados apoderados para recibir notificaciones y designar apoderados judiciales o extrajudiciales, austituir el presente poder y revocar las sustituciones que hicieren.

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POWER OF ATTORNEY

I (we), the undersigned

Michael M. Geoffrey,
legal representative(s) of

UNITED STATES GYPSUM COMPANY

an organization existing under the laws of
State of Delaware

and in present execution ofits business activity, domiciled at
125 South Franklin Street, Chicago, Illinois, U.S.A.

do hereby grant POWER OF ATTORNEY to DILIA MARIA RODRIGUEZ D’ALEMAN, identification card No. 41.654.882 ofBogota, and tp. 20824 and CLAUDIA LUCIA CARO RAMIREZ, tp. 36448, identification card No. 40.017.374 of Tunja, residing in BogotØ, Colombia, to act in the order oftheir appointment in behalf of grantor before any entity, authority and/or natural or juridical person, specially before those Administrative, Administrative-Contentious, Judicial, arbitration courts, and/or any other, in all matters concerning the following:
Patent Aplication in Colombia, file No.

I) To apply, process, obtain and/or register any of the modalities of Industrial Property, such as: Patents of Invention, Utility Models; Designs, plant varieties; Marks, Slogans, Trade Names and emblems and appellations of Origin; as well as their renewals, assignments, changes of name and/or domicile, agreements and licenses.
2) To apply, process, obtain and/or register copyrights of any kind as well as protection ofindustrial secrets or know-how.
3) To apply, process, obtain and/or register sanitary registrations, operating authorizations and/or licenses; as well as their renewals or assignments, changes of name and/or domicile, agreements and licenses.
4) To bring actions of opposition, observations, claims, reconsideration, appeal or legal remedies, cancellations, nullity, protection to the consumer, granting of licenses, protection of trade secrets and other actions related with rights derived from the above- mentioned matters and against the unfair competition, actions resulting from the obtaining or exploitation of licenses, recovering damages actions. They can also intervene or participate in Civil, Commercial, Criminal or Contentious-Administrative Trials; the actions for writ mandamus, police actions; in actions, lawsuits or litigations instituted by the grantor or against him.
5) To register domain names before the competent entity.
6) To the effect of this mandate the above-named proxy are empowered to: conciliate, cancel, settle, submit to arbitration, withdraw and to receive and take possession of the litigation related objet and waive or renounce the rights granted or in process ofgranting, admit the facts ofthe case.
7) To ratify or not the officious agency.
8)1 (we) hereby appoint the afore mentioned attorneys with powers to receive notifications and to designate judicial and extrajudicial attorneys, substitute
this Power of Attorney and to revoke such sustitutions.

Granted and signed at Chicago, Illinois

ay of October

Firma / signatura

Michael M. Geoffrey

of 2005.

Ir LINO

APOSTILLE

(Convention de La Haye du 5 Octobre 1961)

1. Country: United States of America

This public document

2. has been signed by DAVID D. ORR

3. acting in the capacity of COUNTY CLERK, COOK COUNTY

4. bears the seal/stamp of STATE OF ILLINOIS

Certified

5. Chicago, Illinois

6. OCTOBER 26, 2005

7. by the Secretary of State, State of Illinois

3962

tamp: 10. Signature:

- 168.2

JESSE WHITE
SECRETARY OF STATE
STATE OF ILLINOIS
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CERTIFICATE OF AUTHORITY

I, DAVID ORR, County Clerk of Cook County in the State ofIllinois, certify that

BLEKHMAN, AIDA

the person named in the seal and signature on the attached document, is a Notary Public for the State of Illinois and was authorized to act as such at the time ofthe document’s notarization.

To verify this Certificate of Authority for a Notarial Act, I have affixed my signature and seal of office this 26 day of October, 2005

>-

D,?
Cook County Clerk, State of Illinois
0 > >

Deputy

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CERTIFICADO NOTARIAL

(Cuando el solicitante es SOCIEDAO)
(Poderes otorgados en: El Salvador, Estados Unidos, Mexico y Venezuela)

A los dias del mes de
ci Notario que suscribe DA FE de que:

(nombre de la persona que firma ci poder)

mayor de edad y domiciliado

NOTARIAL CERTIFICATE

(When the applicant is a CORPORATION)
(Powers of attorney granted in: El Salvador, Mexico, U.S.A. and Venezuela)

de This day of October, 2005,
the undersigned Notary CERTIFIES that:

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y tetra ci documento que antecede.
2. Conoce at (a Is) otorgante y que este (a) estÆ legalmentecapacitado pars ci otorgamiento.
3. El otorgante ha fimsado ci referido documento en so carÆcter
de______________________________________
de la persona jurIdica UNITED STATES GYPSUM COMPANY

4. La citada persona juridica con sede en
estÆ legalmente
constituida, que tiene actualmente
existencia legal y que ci acto para ci coal se ha otorgado este poder estÆ comprendido entre los que constituyen so objeto social.
5. El otorgante tiene efectivamente la representación que ostenta, que esta es legitima y que estÆ facultadopars otorgar este poder.
6. his anteriores certificaciones de los puntos 3, 4 y 5, me constan porque he tenido a is vista ion documentos autØnticos.

Notario Püblico

NOTA: A continuacidn de 10 anterior, ci Consul de Colombia debe autenticar la firma del Notario POblico.

CERTIFICADO NOTARIAL (Cuando el solicitante es INDIVIDUO)

A los dias del mes de de ci Notario que suscribe DA FE de que:

(nombre de la persona que firma el poder)

mayor de edad y domiciliado en
fume en so presencia
ci documento que antecede, conoce at otorgante y que este estÆ legalmente cspacitado pars ci otorgamiento.

Notario POblico
NOTA: A continuación de to anterior, ci COnsul de Colombia debe autenticar la firma del Notario POblico.

Para todos los demÆs paises ci Seæor Consul de Colombia o ante quien se otorgue, debe expedir una certificación dejando constancia

_y

1) de Ia existencia legal de Ia sociedad, 2) de que euta ejerce actual-mente su objeto social, y 3) de que Ia persona o personas que otorgan ci poder estÆnfacultadas para hacerlo, dejando
constancia asi mismo de que tuvo a Ia vista las pruebas que acrediten tales circuns.taa ’ s...

DE COLOMBIA

1. _Michael M. Geoffrey
(name ofthe person who signs the power of attorney)

of legal age and domiciled in Chicago, Illinois set his hand on the foregoing document.
2. The grantor is to him personally known and that he/she has legal capacity to execute the document.

3. The grantor has executed the foregoing document in his capacity of Assistant Secretary.
ofthe juridical person_ UNITED STATES GYPSUM COMPANY

4. The mentioned juridical person with place of business in Chicago, Illinois
is duly organized, has actual legal existence and that the purposes for which
the power of attorney is granted are within the scope ofits corporate purposes.
5. The grantor has in f act the referred quality and that his/her representation is legal and that he/she is empowered to grant this power
of attorney.

6. I have based the above certifications 3, 4 andJIM dullie’0MCWTEAL which for such purpose were exhibited to me. ENOYTARYUCSTATE AIDA PUB OF! BLEKHN

Notary Public COMMISSION P.SEPT.WNO!S

NOTA: Then the Colombian Consul must legalize the signature of the Notary Public.

NOTARIAL CERTIFICATE (When the applicant is an INDIVIDUALS)

On this day of the undersigned Notary CERTIFIES that:

(name ofthe person who signs the Power of Attorney)

of legal age and domiciled at
signed in his presence the foregoing
document, the grantor is to him personally known and that he/she has legal capacity to execute the document.

Notary Public
NOTA: Then the Colombian Consul must legalize the signature of the Notary Public.

For all other countries the Colombian Consul or before who it is granted must issue a certification stating 1) the legal existence of the corporation,
2) that the corporate purposes are actually being accomplished, 3) that the person or persons who grant this Power of Attorney are empowered to do it. The Consul will certify also that the documents evidencing those circumstances were exhibited to him.

LEGALIZATION BY THE COLOMBIAN CONSUL OR BY APOSTILLE.

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(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization _________
International Bureau I iiH iiiiii ii DII IiII HIlI 11111111011 III

(43) International Publication Date (10) International Publication Number

15 February 2007 (15.02.2007) PCT WO 2007/018705 A2

(51) International Patent Classification:
C04B 11/02 (2006.01) C04B 24/00(2006.01)
C04B 28/00 (2006.01) C04B 24/42(2006.01)

(21) International Application Number:
PCT/US2006/021793

(22) International Filing Date: 5 June 2006 (05.06.2006)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
11/192,652 29 July 2005 (29.07.2005) US

(71) Applicant: UNITED STATES GYPSUM COMPANY [USIUS]; 125 South Franklin Street, Chicago, IL 60606
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- Vernon Hills, IL 60061 (ITS). REED, Paul; 1430 W. Van Meter Street, Kankakee, IL 60901 (US). YU, Qiang; 1252 Sandpiper Court, Grayslake, IL 60030 (US).

- (74) Agents: JANCI, David, F. et al.; USG Corporation, 125 South Franklin Street, Chicago, IL 60606 (US).

(81) Designated States (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, H, GB, GD, GE, Gil, GM, HR, RU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LC, LK, LR, LS, IT, LU, LV,
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(84) Designated States (unless otherwise indicated for every kind of regional protection available): ARIPO (BW, Gil, GM, KB, LS, MW, MZ, NA, SD, SL, SZ, YZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, BE, ES, F!,
FR, GB, GR, RU, TB, IS, IT, IT, LU, IN, MC, NL, PT,, PT,
RO, SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Published:

before the expiration of the time limit for amending the claims and to be republished in the event of receipt of
amendments
- without international search report and to be republished upon receipt ofthat report

For two-letter codes and other abbreviations, refer to the "Guid-
ance Notes on Codes andAbbreviations" appearing at the begin-
ning ofeach regular issue ofthe PCT Gazette.

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(54) Title: SILOXANE POLYMERIZATION IN WALLBOARD

(57) Abstract: Polymerization of siloxane is improved using a gypsum-based slurry that includes stucco, Class C fly ash, magne-sium oxide and an emulsion of siloxane and water. This slurry is used in a method of making water-resistant gypsum articles that ' includes making an emulsion of siloxane and water, then combining the slurry with a dry mixture of stucco, magnesium oxide and Class C fly ash. The slurry is then shaped as desired and the stucco is allowed to set and the siloxane polymerizes. The resulting product is useful for making a water-resistant gypsum panel having a core that includes interwoven matrices of calcium sulfate dihy-drate crystals and a silicone resin, where the interwoven matrices have dispersed throughout them a catalyst comprising magnesium oxide and components from a Class C fly ash.

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SILOXANE POLYMERIZATION IN WALLBOARD

BACKGROUND OF THE INVENTION

This invention relates to a method for making water resistant
gypsum products that include siloxane. More specifically, the present
invention relates to the addition of a novel catalyst for curing of the
5 siloxane in a gypsum product.
Gypsum-based building products are commonly used in
construction. Wallboard made of gypsum is fire retardant and can be
used in the construction of walls of almost any shape. It is used primarily
as an interior wall and ceiling product. Gypsum has sound-deadening
10 properties. It is relatively easily patched or replaced if it becomes
damaged. There are a variety of decorative finishes that can be applied
to the wallboard, including paint and wallpaper. Even with all of these advantages, it is still a relatively inexpensive building material.
Gypsum is also known as calcium sulfate dihydrate, terra
15 alba or landplaster. Plaster of Paris is also known as calcined gypsum,
stucco, calcium sulfate semihydrate, calcium sulfate half-hydrate or
calcium sulfate hemihydrate. Synthetic gypsum, which is a byproduct of
flue gas desulfurization processes from power plants, may also be used. When it is mined, raw gypsum is generally found in the dihydrate form. In
20 this form, there are approximately two water molecules of water
associated with each molecule of calcium sulfate.
In order to produce the hemihydrate form, the gypsum can
be calcined to drive off some of the water of hydration by the following equation:
25 CaS0492H20*CaSO4.1/2H20 3/2H20
A number of useful gypsum products can be made by
mixing the stucco with water and permitting it to set by allowing the
calcium sulfate hemihydrate to react with water to convert the
hemihydrate into a matrix of interlocking calcium sulfate dihydrate
30 crystals. As the matrix forms, the product slurry becomes firm and holds

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a desired shape. Excess water must then be removed from the product
by drying.
In the absence of additives to prevent it, set gypsum
absorbs up to 50% of its weight when immersed in water. Boards or
5 panels that absorb water swell, become deformed and lose strength. This
property is undesirable in products that are likely to be exposed to water.
In areas such as bathrooms or kitchens, high temperature and humidity
are common, and walls are likely to be splashed. In such areas, it is preferable to use a gypsum board that exhibits water resistancy, thus
10 maintaining strength and dimensional stability.
Many attempts have been made to improve the water
resistance of gypsum products. Various hydrocarbons, including wax,
resins and asphalt have been added to the slurry in order to impart water resistance to the set product. The use of siloxanes, which form silicone
15 resins in gypsum products, to impart water resistance is well known.
Although the use of siloxanes in gypsum slurries is a useful
means of imparting water resistance to the finished product, there are drawbacks associated with it. When added to a gypsum slurry to form silicone resins in situ, siloxane can be slow to cure. The siloxane forms a
20 reactive silanol intermediate to yield polymethylsilicic acid, which cross
links to form the silicone resin. The reaction proceeds slowly, often continuing after the gypsum is set and requiring one to two weeks to fully develop water-resistance. Wallboard made using this method must be
stored for a time sufficient for the water-resistance to develop before the
25 board can be shipped. In some cases, the siloxane may not cure within a
reasonable time or it may not cure fully. In such cases, the water
resistance does not develop in the gypsum board to a satisfactory level. Additionally, failure to cure fully leads to using a larger dose of the
siloxane, increasing the cost of the raw materials.
30 Catalysts, such as alkaline earth oxides and hydroxides, are known to accelerate the curing reaction of siloxane in a stucco slurry.
These catalysts are relatively water soluble and elevate the pH of the
slurry. High pH can interfere with the rehydration of the stucco, and can

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negatively react with some preferred wallboard additives. Thus, while the siloxane polymerization is promoted, other considerations make the use
of these catalysts undesirable.
Magnesium oxide ("MgO") is known to catalyze siloxane
5 reactions, but where the catalyst reactivity is high enough to fully cure the
siloxane, undesirable cracking results. Light-burned MgO has the activity needed to cure siloxane quickly, but the activity leads to unwanted side reactions. These side reactions generate hydrogen, which cause
expansion of the product and cracking of set gypsum. Hard-burned or
10 dead-burned MgO has lower reactivity, but results in a less water-
resistant product. Thus, when MgO is used alone, it is very difficult to balance catalyst activity with the desired extent of siloxane
polymerization.
There are also certain stucco sources for which it is very
15 difficult to drive the polymerization of siloxane. Gypsum is a complex
mixture of calcium sulfate in various forms, salts and a variety of
aluminates, silicates and aluminosilicates. Apparently some gypsum
sources include one or more components that suppress the formation of
the silicone resin. When used with these stuccos, known catalysts fall
20 short of the desired level of water-resistance of less than 5% water
absorbance.

Thus there is a need in the art for a catalyst and a method of
producing water-resistant gypsum articles with improved water-resistance
at reasonable cost. The catalyst should be relatively inexpensive, having
25 good activity for siloxane polymerization with a minimum of unwanted side
reactions. There should be little interference between the catalyst and
other common gypsum additives.

SUMMARY OF THE INVENTION

These and other needs are met or exceeded by the present
30 invention which accelerates the polymerization of siloxane and in some
cases reduces the amount of siloxane needed to meet the specifications
of ASTM 1398.

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More specifically, polymerization of siloxane is improved
using a slurry that includes stucco, Class C fly ash, magnesium oxide and
an emulsion of siloxane and water. This slurry is used in a method of
making water-resistant gypsum articles that includes making an emulsion
5 of siloxane and water, then combining the slurry with a dry mixture of
stucco, magnesium oxide and Class C fly ash. The slurry is then shaped
as desired and the stucco is allowed to set and the siloxane polymerizes.
The resulting product is useful for making a water-resistant
gypsum panel having a core that includes interwoven matrices of calcium
10 sulfate dihydrate crystals and a silicone resin, where the interwoven
matrices have dispersed throughout them a catalyst comprising
magnesium oxide and components from a Class C fly ash.
The mixture of magnesium oxide and Class C fly ash
catalyzes the polymerization of siloxane to accelerates development of
15 water-resistance in product made from the slurry. Water-resistant
products such as wallboard need not be stored for lengthy periods of time awaiting completion of the polymerization reactions of the siloxane.
Use of this catalyst also increases the extent of the reaction,
leading to improved water-resistance. Water absorption of less than 5%
20 by weight was attainable using the fly ash and magnesia combination,
where it had not been achieved with either catalyst alone. Thus, in
addition to causing the polymerization reaction to accelerate, this catalyst also allows the siloxane to polymerize more completely allowing the
amount of siloxane to be reduced in some cases. Since the siloxane is
25 one of the more expensive wallboard additives, reduction in the dosage
leads to a savings in the cost of the raw materials.
Another advantage of the present invention is the
dimensional stability of the product. Some compounds used to catalyze this reaction result in significant expansion as the product dries. As the
30 board interior expands, it causes cracking in the exterior board surface,
damaging it. Use of fly ash and magnesium oxide results in very little expansion and very little cracking in the finished product.

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This combined fly ash and magnesia catalyst also allows for
satisfactory polymerization using a wider range of magnesium oxide
grades. While the prior art discloses only that dead-burned magnesia is suitable to act as a catalyst for siloxane polymerization, when combined
5 with fly ash, even hard-burned or light-burned magnesium oxide may be
used. This feature allows manufacturers of gypsum products additional freedom in selection sources of magnesium oxide to be used in the slurry.

DETAILED DESCRIPTION OF THE INVENTION

10 The present invention broadly contemplates improving the water resistance of gypsum based articles by adding a polymerizable siloxane to the slurry used to make the gypsum based articles.
Preferably, the siloxane is added in the form of an emulsion. The slurry is then shaped and dried under conditions which promote the polymerization
15 of the siloxane to form a highly cross-linked silicone resin. A catalyst
which promotes the polymerization of the siloxane to form a highly cross-linked silicone resin is added to the gypsum slurry.
Preferably, the siloxane is generally a fluid linear hydrogen-
modified siloxane, but can also be a cyclic hydrogen-modified siloxane.
20 Such siloxanes are capable of forming highly cross-linked silicone resins.
Such fluids are well known to those of ordinary skill in the art and are commercially available and are described in the patent literature.
Typically, the linear hydrogen modified siloxanes useful in the practice of
the present invention comprise those having a repeating unit of the
25 general formula:

wherein R represents a saturated or unsaturated mono-
valent hydrocarbon radical. In the preferred embodiments, R represents
an alkyl group and most preferably R is a methyl group. During
30 polymerization, the terminal groups are removed by condensation and
siloxane groups are linked together to form the silicone resin. Cross-
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linking of the chains also occurs. The resulting silicone resin imparts
water resistance to the gypsum matrix as it forms.
The gypsum-based water resistant articles of the present
invention are preferably made with a solventless methyl hydrogen
5 siloxane fluid sold under the name SILRES BS 94 by Wacker-Chemie
GmbH (Munich, Germany) as the siloxane. The manufacturer indicates
this product is a siloxane fluid containing no water or solvents. It is contemplated that about 0.3 to 1.0% of the BS 94 siloxane may be used, based on the weight of the dry ingredients. It is preferred to use from
10 about 0.4 to about 0.8% of the siloxane based on the dry stucco weight.
After the slurry is formed, the siloxane is formed into an
emulsion or a stable suspension with water. A number of siloxane
emulsions are contemplated for use in this slurry. Emulsions of siloxane
in water are also available for purchase, but they may include emulsifying
15 agents that tend to modify properties of the gypsum articles, such as the
paper bond in wallboard products. Emulsions or stable suspensions prepared without the use of emulsifiers are therefore preferred.
Preferably, a suspension is formed in situ by mixing the siloxane fluid with water. It is essential that the siloxane suspension be stable until it
20 reaches the pin mixer and that it remain well dispersed under the
conditions of the slurry. The siloxane suspension or emulsion must
remain well dispersed in the presence of the optional additives, such as
set accelerators, that are present in the slurry. The siloxane suspension
or emulsion must also remain stable through the steps in which the
25 gypsum based articles are formed as well. Preferably, the suspension
remains stable for more than 40 minutes. More preferably, it remains
stable for at least one hour. In the discussion and claims that follow, the term "emulsion" is intended to include true emulsions and suspensions
that are stable at least until the stucco is 50% set.
30 In a preferred embodiment, at least a portion of the gauging water is continuously fed to the high shear mixer. Siloxane fluid is
metered into the high shear mixer with the water to form the emulsion in
1-2 seconds. The proportion of water to siloxane is not critical and a
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mixture of 25 parts water to one part siloxane is known to be useful. This emulsion is stable for several minutes without the addition of an
emulsifier, long enough to mix the slurry, form the article and allow it to
start to set. In the alternative, use of a portion of the gauging water to
5 form the emulsion is also contemplated. A slip stream of the gauging
water is combined with the siloxane in the high shear mixer. The siloxane emulsion is then preferably added to the gauging water before the slurry
is formed to provide sufficient time for the siloxane emulsion to thoroughly mix with water used to form the slurry and be uniformly dispersed
10 throughout the resulting articles.
While not wishing to be bound by theory, it is believed that
water resistance develops when the siloxane cures within the formed wallboard. The polymerization reaction proceeds slowly on its own,
requiring that the wallboard be stored for a time sufficient to develop
15 water-resistance prior to shipping. Catalysts are known to accelerate the
polymerization reaction, reducing or eliminating the time needed to store wallboard product as the water-resistance develops. Use of dead-burned magnesium oxide for siloxane polymerization is described in co-pending
U.S. Serial No. 10/917,177, entitled "Method of Making Water-Resistant
20 Gypsum-Based Article", herein incorporated by reference. Dead-burned
magnesium oxide is water-insoluble and interacts less with other
components of the slurry. It accelerates curing of the siloxane and, in
some cases, causes the siloxane to cure more completely. It is
commercially available with a consistent composition. A particularly
25 preferred source of dead-burned magnesium oxide is BAYMAG 96. It has
a BET surface area of at least 0.3 m2/g. The loss on ignition is less than
0.1% by weight. The magnesium oxide is preferably used in amounts of about 0.1 to about 0.5% based on the dry stucco weight.
There are at least three grades of magnesium oxide on the
30 market, depending on the calcination temperature. "Dead-burned"
magnesium oxide is calcined between 1500°C and 2000°C, eliminating most, if not all, of the reactivity. MagChem P98-PV (Martin Marietta Magnesia Specialties, Bethesda, MD) is an example of a "dead burned"

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magnesium oxide. BayMag 96 (Baymag, Inc. of Calgary, Alberta,
Canada) and MagChem 10 (Martin Marietta Magnesia Specialties,
Bethesda, MD) are examples of "hard-burned" magnesia. "Hard-burned" magnesium oxide is calcined at temperatures from 1000°C to about 1500
5 C. It has a narrow range of reactivity, a high density, and is normally
used in application where slow degradation or chemical reactivity is
required, such as in animal feed and fertilizer. The third grade is "light-
burn" or "caustic" magnesia, produced by calcining at temperatures of
about 700°C to about 1000°C. This type of magnesia is used in a wide
10 range of applications, including plastics, rubber, paper and pulp
processing, steel boiler additives, adhesives and acid neutralization. Examples of light burned magnesia include BayMag 30, BayMag 40, and BayMag 30 (-325 Mesh) (BayMag, Inc. of Calgary, Alberta, Canada).
It has been discovered that preferred catalysts are made of
15 a mixture of magnesium oxide and Class C fly ash. When combined in
this manner, any of the grades of magnesium oxide are useful. However, dead-burned and hard-burned magnesium oxides are preferred due to reduced reactivity. The relatively high reactivity of magnesium oxides,
can lead to cracking reactions which can produce hydrogen. As the
20 hydrogen is generated, the product expands, causing cracks where the
stucco has set. Expansion also causes breakdown of molds into which
the stucco is poured, resulting in loss of detail and deformation of the
product in one or more dimensions. Preferably, BayMag 96, MagChem P98-PV and MagChem 10 are the preferred sources of magnesium oxide.
25 Preferably, the magnesium oxide and fly ash are added to the stucco prior
to their addition to the gauging water. Dry components such as these are often added to the stucco as it moves along a conveyer to the mixer.
A preferred fly ash is a Class C fly ash. Class C hydraulic
fly ash, or its equivalent, is the most preferred fly ash component. A
30 typical composition of a Class C fly ash is shown in Table 1. High lime
content fly ash, greater than 20% lime by weight, which is obtained from
the processing of certain coals. ASTM designation C-618, herein incorporated by reference, describes the characteristics of Class C fly

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ash. A preferred Class C fly ash is supplied by Bayou Ash Inc., Big
Cajun,ll, LA. Preferably, fly ash is used in amounts of about 0.1% to
about 5% based on the dry stucco weight. More preferably, the fly ash is
used in amounts of about 0.2% to 1.5% based on the dry stucco weight.
TABLE I

Typical Type C Fly Ash Composition
Composition Amount, wt
Si02 25-59
A1203 14-22
Fe203 5-13
CaO 8-32
MgO 3.2-12.5
K20 0.3-1.6
Na20 0.8-6.0
S03 0.4-5.0
Ti02 <1.0

Loss On Ignition 0.1-2.3

5 Catalysis of the siloxane results in faster and more complete polymerization and cross-linking of siloxane to form the silicone resin. Hydration of the stucco forms an interlocking matrix of calcium sulfate dihydrate crystals. While the gypsum matrix is forming, the siloxane molecules are also forming a silicone resin matrix. Since these are
10 formed simultaneously, at least in part, the two matrices become intertwined in each other. Excess water and additives to the slurry, including the fly ash, magnesium oxide and additives described below, which were dispersed throughout the slurry, become dispersed
throughout the matrices in the interstitial spaces.
15 When used to make gypsum board, a number of additives are useful to improve the properties of the finished article. Traditional amounts of additives are used. Except as noted, there are no known interactions of the catalyst or polysiloxane that interferes with the

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additives. Amounts of several additives are reported as "lbs/MSF," which stands for pounds of additive per one thousand square feet of board.
Some embodiments of the invention employ a foaming
agent to yield voids in the set gypsum-containing product to provide
5 lighter weight. In these embodiments, any of the conventional foaming
agents known to be useful in preparing foamed set gypsum products can
be employed. Many such foaming agents are well known and readily available commercially, e.g. the HYONIC line of soaps from GEO
Specialty Chemicals, Ambler, PA. Foams and a preferred method for
10 preparing foamed gypsum products are disclosed in U.S. Patent No.
5,683,635, herein incorporated by reference.
Dispersants are used to improve the flowability of the slurry
and reduce the amount of water used to make the slurry. Any known dispersant is useful, including polycarboxylates, sulfonated melamines or
15 naphthalene sulfonate. Naphthalene sulfonate is the most preferred
dispersant, and is used in amounts of about 0 lb/MSF to 18 lb/MSF, preferably from about 4 lb/MSF to about 121b/MSF. A preferred
naphthalene sulfonate dispersant is DAXAD Dispersant (Dow Chemical, Midland, Ml)
20 Water is added to the slurry in any amount that makes a flowable slurry. The amount of water to be used varies greatly according
to the application with which it is being used, the exact dispersant being used, the properties of the stucco and the additives being used. The
water to stucco ratio ("WSR") for wallboard is preferably about 0.2 to
25 about 1.2 based on the dry weight of the stucco. Commonly, a WSR of
about 0.4 to about 0.9 is preferred. Water used to make the slurry should
be as pure as practical for best control of the properties of both the slurry
and the set plaster. Salts and organic compounds are well known to
modify the set time of the slurry, varying widely from accelerators to set
30 inhibitors. Some impurities lead to irregularities in the structure as the
interlocking matrix of dihydrate crystals forms, reducing the strength of the
set product. Product strength and consistency is thus enhanced by the
use of water that is as contaminant-free as practical.

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The stucco, also known as calcium sulfate hemihydrate or
calcined gypsum, is present in amounts of at least 50% of the dry
materials. Preferably, the amount of stucco is at least 80%. In many wallboard formulations, the dry component material is more than 90% or
5 even 95% calcium sulfate hemihydrate. The method of calcination is not
important, and either alpha or beta-calcined stucco is suitable. Use of calcium sulfate anhydrite is also contemplated, although it is preferably
used in small amounts of less than 20%.
A trimetaphosphate compound is added to the gypsum
10 slurry in some embodiments to enhance the strength of the product and to
improve sag resistance of the set gypsum. Preferably the concentration
of the trimetaphosphate compound is from about 0.07% to about 2.0%
* based on the weight of the calcined gypsum. Gypsum compositions
including trimetaphosphate compounds are disclosed in U.S. Patent No.
15 6,342,284 and 6,632,550, both herein incorporated by reference.
Exemplary trimetaphosphate salts include sodium, potassium or lithium
salts of trimetaphosphate, such as those available from Astaris, LLC., St.
Louis, MO. Care must be exercised when using trimetaphosphate with
lime or other modifiers that raise the pH of the slurry. Above a pH of
20 about 9.5, the trimetaphosphate looses its ability to strengthen the product
and the slurry becomes severely retardive.
Other additives are also added to the slurry as are typical for
the particular application to which the gypsum slurry will be put. Set
retarders (up to about 2 Ib./MSF (9.8g/m2)) or dry accelerators (up to
25 about 35 lb./MSF (170 g/m2)) are added to modify the rate at which the
hydration reactions take place. "CSA" is a set accelerator comprising
95% calcium sulfate dihydrate co-ground with 5% sugar and heated to
250°F (121°C) to caramelize the sugar. CSA is available from USG Corporation, Southard, OK plant, and is made according to U.S. Patent
30 No. 3,573,947, herein incorporated by reference. Potassium sulfate is
another preferred accelerator. HRA is calcium sulfate dihydrate freshly ground with sugar at a ratio of about 5 to 25 pounds of sugar per 100
pounds of calcium sulfate dihydrate. It is further described in U.S. Patent

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No. 2,078,199, herein incorporated by reference. Both of these are
preferred accelerators.
Another accelerator, known as wet gypsum accelerator or
WGA, is also a preferred accelerator. A description of the use of and a
5 method for making wet gypsum accelerator are disclosed in U.S. Patent
No. 6,409,825, herein incorporated by reference. This accelerator
includes at least one additive selected from the group consisting of an organic phosphonic compound, a phosphate-containing compound or mixtures thereof. This particular accelerator exhibits substantial longevity
10 and maintains its effectiveness over time such that the wet gypsum
accelerator can be made, stored, and even transported over long
distances prior to use. The wet gypsum accelerator is used in amounts ranging from about 5 to about 80 pounds per thousand square feet (24.3
to 390 g/m2) of board product.
15 Other potential additives to the wallboard are biocides to reduce growth of mold, mildew or fungi. Depending on the biocide
selected and the intended use for the wallboard, the biocide can be added
to the covering, the gypsum core or both. Examples of biocides include
boric acid, pyrithione salts and copper salts. Biocides can be added to
20 either the covering or the gypsum core. When used, biocides are used in
the coverings in amounts of less than 500 ppm. Pyrithione is known by several names, including 2-mercaptopyridine-N-oxide; 2-pyridinethiol-1 -
oxide (CAS Registry No. 1121-31-9); 1-hydroxypyridine-2-thione and I hydroxy-2(1 H)-pyridinethione (GAS Registry No. 1121-30-8). The sodium
25 derivative (C5H4NOSNa), known as sodium pyrithione (GAS Registry No.
3811-73-2), is one embodiment of this salt that is particularly useful. Pyrithione salts are commercially available from Arch Chemicals, Inc. of
Norwalk, CT, such as Sodium OMADINE or Zinc OMADINE.
In addition, the gypsum composition optionally can include a
30 starch, such as a pregelatinized starch or an acid-modified starch.
Starches are used in amounts of from about 3 to about 20 lbs/ MSF (14.6
to 97.6 g/m2) to increase paper bond and strengthen product. The
inclusion of the pregelatinized starch increases the strength of the set and N
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dried gypsum cast and minimizes or avoids the risk of paper delamination under conditions of increased moisture (e.g., with regard to elevated
ratios of water to calcined gypsum). One of ordinary skill in the art will appreciate methods of pregelatinizing raw starch, such as, for example,
5 cooking raw starch in water at temperatures of at least about 185°F
(85°C) or other methods. Suitable examples of pregelatinized starch
include, but are not limited to, PCF 1000 Starch, commercially available
from Lauhoff Grain Company and AMERIKOR 818 and HQM PREGEL starches, both commercially available from Archer Daniels Midland
10 Company. If included, the pregelatinized starch is present in any suitable
amount. For example, if included, the pregelatinized starch can be added
to the mixture used to form the set gypsum composition such that it is present in an amount of from about 0.5% to about 10% percent by weight
of the set gypsum composition. Starches such as USG95 (United States
15 Gypsum Company, Chicago, IL) are also optionally added for core
strength.
Other known additives may be used as needed to modify
specific properties of the product. Sugars, such as dextrose, are used to improve the paper bond at the ends of the boards. Wax emulsions or
20 siloxanes are used for water resistance. If stiffness is needed, boric acid
is commonly added. Fire retardancy can be improved by the addition of vermiculite. These and other known additives are useful in the present slurry and wallboard formulations. Glass fibers are optionally added to
the slurry in amounts of up to 11 lb./MSF (54 g/m2). Up to 15 lb./MSF
25 (73.2 g/m2) of paper fibers are also added to the slurry. Wax emulsions
are added to the gypsum slurry in amounts up to 90 lb./MSF (.439 kg/M2)
to improve the water-resistency of the finished gypsum board panel.
In operation, a slip stream is taken from the gauging water
and combined with siloxane and water in a high shear mixer to form the
30 siloxane emulsion. The two components are mixed for several minutes
until a stabile emulsion is formed. From the high shear mixer, the
emulsion goes directly to the slurry mixer where it is combined with the remainder of the gauging water.
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Meanwhile, the stucco is moved toward a slurry mixer. Prior
to entry into the mixer, dry additives, such as starches, or set
accelerators, are added to the powdered stucco. Some additives are
added directly to the mixer via a separate line. For most additives, there
5 is no criticality regarding placing the additives in the slurry, and they may
be added using whatever equipment or method is convenient.
After mixing, wallboard optionally has foam added to
decrease the product density. Foam is generated by combining soap and water. The foam is then injected into the moving gypsum slurry after it
10 exits from the mixer through a hose or chute. The foam ring is an
apparatus having multiple ports that are arranged in a ring perpendicular
to the axis of the hose so that foam is forced under pressure into the
gypsum slurry as it passes by the foam ring.
When the foam and the slurry have been brought together,
15 the resulting slurry moves toward and is poured onto a conveyor lined
with one piece of facing material. Another piece of facing material is
placed on top of the slurry, forming a sandwich with the slurry between
the two facing materials. The sandwich is fed to a forming plate, the
height of which determines the thickness of the board. Next the
20 continuous sandwich is cut into appropriate lengths at the cutting knife,
usually eight feet to twelve feet.
The boards are then moved to a kiln for drying.
Temperatures in the kiln typically range to 450°F to 500°F maximum. Preferably there are three or more temperature zones in the kiln. In the
25 first zone contacted by the wet board, the temperature increases to the
maximum temperature, while the temperature slowly decreases in the last
two zones. The blower for the first zone is positioned at the exit of the
zone, blowing the air countercurrent to the direction of board travel. In the second and third zones, the blowers are located at the entrance to the
30 zone, directing the hot air co-current with board travel. Heating that is
less severe in the last zone prevents calcination of dry areas of the board, causing poor paper bond. A typical residence time in the kiln is about

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forty minutes, but the time will vary depending on the tine capacity, the wetness of the board and other factors.
EXAMPLE I

Two grams of BAYMAG 30 dead-burned magnesium oxide
5 and 4 grams of Class C fly ash were added to 500 grams of stucco from
each of several sources specified in Table II. These dry components
were placed in a plastic bag and shaken to mix them. An emulsion was prepared by mixing 3.2 grams of BS 94 siloxane and 550 grams of water
for 2.5 minutes in a high shear Silverson Mixer (Silverson Machines, East
10 Longmeadow, MA). The emulsion was transferred to a I liter Waring
blender at 7500 rpm (Waring Products, Inc., Torrington, CT) for 10
seconds. The slurry was then cast into a 2" x 2" x 2" cube mold. After
set, the cubes were unmolded and placed in an oven for drying to a
constant weight at 110°F. Dried cubes were soaked in water for two
15 hours for the water absorption test as specified in ASTM Cl 396, herein
incorporated by reference. The weight gain during the soaking was used
to calculate the water absorption.
TABLE II
Stucco
Source

Dry
Density

Dry
Weight

Wet

Weight

Water

Pickup Absorption

Empire 43.0 90.27 93.44 3.17 3.5 it

_______ 42.6 89.5 92.8 3.31 3.7
42.8 89.8 93.1 3.29 3.7
Montreal 43.4 91.26 93.71 2.45 2.7
______ 43.5 91.38 93.97 2.59 2.8
43.3 91.1 93.5 2.46 2.7

Sperry 42.8 89.89 93.89 4.0 4.4 11 42.5 89.4 92.9 3.55 4.0 It 42.6 89.6 93.3 3.74 4.2

Sweet
Water

43.8 92.09 96.51 4.42 4.8

43.5 91.4 195.6 14.12 14.5
43.7 _FF-.81 96.1 j 4.31 14.7

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This example demonstrates the ability of this catalyst
combination to reduce water absorption to less than 5% in a variety of

stuccos.

5 EXAMPLE 2

Commercial scale trials where held to test the behavior of a

slurry including 12 IbIMSF siloxane, and having magnesium oxide and fly ash as indicated in Table Ill. The fly ash and magnesium oxide were
10 added to the stucco prior to entering the mixer. The brand and type of calcined magnesium oxide is also shown in Table Ill.
Siloxane was added to the gauging water and mixed in a
high shear mixer to form an emulsion. This emulsion and the dry components were combined in the stucco mixer until a homogeneous
15 slurry was formed, and the slurry was deposited on a face paper on a conveyor. Backing paper was placed atop the slurry and the sandwich
was fed to a forming roller that flattened the sandwich to a uniform ‰inch (1.2 cm) thickness. When the slurry was sufficiently set to hold its shape, the continuous board was cut into 8-foot lengths.

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TABLE Ill
Trial Condition Trial Board
MgO Source MgO Amount Flyash Amount 2 Hr. Absorption
Baymag 96 4 0 5.3%
91 4 0 59%
4 0 4.4%
U 4 0 5.4%
4 0 5.8%
MagChem 10 4 0 5.2%
4 0 5.2%
4 0 5.5%
4 0 5.7%
4 0 5.7%
4 10 4.3%
4 10 46%
4 10 4.3%
4 10 3.9%
4 10 4.5%
Baymag 30 3 10 4.4%
It 3 10 44%
3 10 46%
3 10 46%
3 10 44%
3 0 57%
3 0 5.8%
U 4 0 5.6%
4 0 62%

4 0 58%

4 0 7.1%

When fly ash was added to MagChem 10 dead-burned magnesium oxide, the water resistance improved more than 25%. The

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same comparison holds when the same amount of fly ash was added to 3 lb/MSF of BayMag 30. The combination of fly ash with 3 lb/MSF BayMag
30 also performs better than 4 lb/MSF of BayMag 30 alone.
EXAMPLE 3

5 Cubes were made according to Example I using Shoals stucco and 0.6 wt% BS 94 siloxane. Either Baymag 30 magnesium oxide, fly ash or both was added to the slurry as indicated in Table IV. The
target water adsorption was 6%.
TABLE IV

Baymag 30 Fly Ash Water Adsorption
0.4% 0 21.5%
0 0.8% 24%
0.4% 0.8% 2.0%

When magnesium oxide and fly ash are used together,
10 water reduction improved by an order of magnitude in the above example. EXAMPLE 4

Gypsum cubes were made according to the method of
Example I using the siloxane dosage and catalyst composition shown in Table IV. Results of water absorption tests are also shown in Table V. TABLE V
Stucco
Source

Siloxane
Dosage

MgO Fly Ash Water
Absorption
Empire 4.2g 1.2g 0 6.1%
It 4.2g 0 6.0g 32%
3.1 g 1.2g 6.0g 3.7%
Montreal 4.2 g 1.2 g 0 7%
4.2g 0 6.0g 40%
it 3.1 g 1.2 g 6.0 g 2.9%

These tests were preformed on stuccos with which it is
15 particularly difficult to obtain satisfactory water resistance. Neither fly ash nor MgO alone were able to produce the desired standard of less than 5%
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water absorption. However, when both catalysts were used together,
absorption well below the standard was achieved, even with a lower
dosage of siloxane.
EXAMPLE 5

5 A plant trial was held testing this catalyst in wallboard on a
commercial scale. The composition of the wallboard is shown in Table VI. TABLE VI

Component Amount, lbs/MSF
Stucco 1324
Gauging Water 546
Siloxane Water 119
Foam Water 75
Siloxane 10.5
MgO 4
Fly Ash 10.85
Soap 0.4
HRA Set Accelerator 16.6
Trimetaphosphate 0.8
LC-211 3.0
USG 95 Starch 3.5
Thickener 0.49
Daxad Dispersant 5.5
Foam Air 17ft3/MSF

HRA, Trimetaphosphate, USG95, thickener, Daxad, LC-21 1,
Fly ash and MgO were added to the dry stucco. The siloxane water and siloxane were mixed in a high speed mixer at high speed for less than I
10 minute to make a stable suspension of siloxane in water. The suspension was then pumped to the slurry mixer and combined with the gauging
water, the catalyst/stucco blend. Residence in the mixer was less than 15 seconds. As the slurry was discharged from the mixer, foam, made of the soap, foam air and foam water was inserted into the slurry to reduce the
15 product density.
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TABLE VII

Sample Water Absorption
IA 4.11%
1 4.36%
2A 4.34%
2B 4.37%
3A 4.19%
4B 4.06%

Cubes were made from a slurry sample according to ASTM
Cl 396. Results of the soak tests are shown in Table VII. These tests confirm that wallboard having less than 5% water absorption are producable in a commercial setting using the catalyst, slurry and method
5 of this invention.
While a particular embodiment of the fly ash and
magnesium oxide catalyst for siloxane polymerization has been shown and described, it will be appreciated by those skilled in the art that changes and modifications may be made thereto without departing from 10 the invention in its broader aspects and as set forth in the following claims.

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WHAT IS CLAIMED IS:

1. A slurry comprising:
stucco;
5 Class Cfly ash;
magnesium oxide; and
an emulsion of siloxane and water.
2. The slurry of claim 1 wherein the ratio of said fly ash to said
magnesium oxide is from about 2:1 to about 3:1.
10 3. The slurry of claim 2 wherein said fly ash is present in the
slurry in amounts of about 0.1% to about 5% based on the dry stucco weight.
4. The slurry of claim I wherein said fly ash is present in
amounts ranging from about 0.1 % to about 5 % based on the dry stucco
15 weight.
5. The slurry of claim I wherein said magnesium oxide is
present in amounts of about 0.1 to about 0.5 % based on the dry stucco weight.
6. The slurry of claim I wherein said magnesium oxide is a
20 hard-burned or a dead-burned magnesium oxide.
7. The slurry of claim I wherein said emulsion comprises a
polysiloxane fluid and water.
8. The slurry of claim I wherein said siloxane is a fluid, linear,
hydrogen-modified siloxane.
25 9. The slurry of claim I further comprising at least one of the
group consisting of starches, foaming agents, set accelerators, set
retarders, biocides, dispersants, fibers or strength enhancers.
10. The slurry of claim I wherein said emulsion comprises a
stable suspension.
30 II. The slurry of claim 1 wherein said suspension is emulsifier-
free.

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12. A method of making a water-resistant gypsum article
comprising:
making a siloxane emulsion with siloxane and water;
mixing magnesium oxide and Class C fly ash with stucco;
5 combining the siloxane emulsion with the stucco/catalyst mixture; shaping the slurry;
allowing the gypsum slurry to set, forming the wallboard core; and polymerizing the siloxane.
13. The method of claim 12 wherein said making step
10 comprises mixing the siloxane and water in a high shear mixer.
14. The method of claim 12 wherein said shaping step
comprises sandwiching the slurry between two pieces of facing material to form a wallboard panel.
15. The method of claim 12 therein said mixing step takes place
15 before said combining step.
16. The method of claim 11 further comprising taking a portion
of a metered amount of gauging water for use as the water.
17. A water-resistant gypsum panel having a core comprising
interwoven matrices of calcium sulfate dihydrate crystals and a silicone
20 resin, said interwoven matrices having dispersed throughout them a
catalyst comprising magnesium oxide and components from a Class C fly ash.

18. The panel of claim 17 further comprising at least one of the
group consisting of starches, foaming agents, set accelerators, set
25 retarders, biocides, dispersants, fibers or strength enhancers dispersed
throughout said interwoven matrices.
19. The panel of claim 17 wherein said core is sandwiched
- between two pieces of facing material.

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POLIMERIZACION DE SILOXANO EN PANELES PARA PAREDES ANTECEDENTES DE LA INVENCION

La presente invención se refiere a un mØtodo para producir productos do yeso resistentes al agua que incluyen siloxano. MÆs especIficamente, la presente invenciOn se refiere al agregado de un novedoso catalizador para curar el siloxano en un producto do yeso.
En la construcciOn se usan comünmente productos de edificación basados en yeso. El tabique preformado elaborado con YSO es retardador del fuego y puede utilizarse en la construcciOn de paredes casi do cualquier forma. Se utiliza principalmente como un producto de paredes interiores y cielorrasos. El yeso tiene propiedades amortiguadoras del sonido. Cuando se dana, resulta relativamente fÆcil de emparchar o reemplazar. Existen una variedad de terminaciones decorativas, incluso pintura y empapelado, quo pueden aplicarse al tabique preformado. Mn contando con todas estas ventajas, do todos modos se trata de un material de construcción relativamente económico.
El yeso se conoce tambiØn como sulfato de calcio dihidratado, terra alba
o yeso natural.
El yeso Paris se conoce tambiØn como yeso calcinado, estuco, sulfato
do calcio semihidratado, hemihidrato do sulfato do calcio o sulfato de calcio hemihidratado.

TambiØn so puedo usar yeso sintØtico, quo es un subproducto de los procesos de desulfurizaciOn do los gases de escape de centrales goneradoras. Cuando se extrae, el yeso en bruto en general se encuentra en forma de dihidrato. En dicha forma, hay aproximadamente dos molØculas de agua de agua asociadas con cada molØcula de sulfato de calcio.

Para producir Ia forma hemihidratada, el yeso se puede calcinar para expulsar parte del agua de hidrataciOn segün la siguiente ecuaciOn: CaSO4.2H20*CaSO4.1/2H20 + 3/21-120
Se pueden hacer muchos productos de yeso ütiles mezclando el estuco
con agua y permitiØndole fraguar dejando que el sulfato de calcio hemihidratado reaccione con el agua para convertir el hemihidrato en una matriz de cristales entrelazados de sulfato de calcio dihidratado. A medida que se forma la matriz, la lechada del producto se vuelve firme y mantiene una forma deseada. Luego se debe extraer el agua en exceso del producto por secado.
En ausencia de aditivos para impedirlo, el yeso fraguado absorbe hasta
un 50% de su peso cuando se sumerge en agua. Los tableros o paneles que absorben agua se hinchan, se deforman y pierden resistencia. Esta propiedad no es deseable en productos que es probable que sean expuestos at agua. En areas tales como cuartos de baæo o cocinas, son comunes las altas temperaturas y humedades, y es probable que las paredes sean salpicadas. En dichas areas, es preferible usar una placa de yeso que muestre resistencia al agua, manteniendo de esa manera su rigidez y estabilidad dimensional.
Se han hecho muchos intentos de mejorar la resistencia al agua de los productos de yeso. A la lechada se le han agregado diversos hidrocarburos, incluso cera, resinas y asfalto, para conferir resistencia al agua al producto fraguado. Se conoce bien el uso de siloxanos, que forman resinas de silicona en los productos de yeso, para conferir resistencia al agua.
Aunque el uso de siloxanos en lechadas de yeso es un medio ütil para conferir resistencia al agua al producto terminado, existen desventajas

asociadas con el mismo. Cuando se agrega a una lechada de yeso para formar resinas de silicona in situ, el siloxano puede hacer que el curado se haga mÆs lento. El siloxano forma un intermediario reactivo de silanol para dar Æcido polimetilsilIcico, que se reticula para formar la resina de silicona. La reacción procede lentamente, y frecuentemente continUa despuØs de que el yeso estÆ fraguado y requiere entre una y dos semanas para desarrollar completamente su resistencia al agua. Los paneles para paredes que se hacen usando este mØtodo se deben almacenar durante un tiempo suficiente como para desarrollar resistencia al agua antes de que la placa se pueda embarcar. En algunos casos, el siloxano puede no curar dentro de un tiempo razonable o puede no curar completamente. En dichos casos, la resistencia al agua no se desarrolla en el placa de yeso hasta un nivel satisfactorio. AdemÆs, la falta de un curado completo conlieva at uso de una mayor dosis de siloxano, aumentando el costo de los materiales en bruto.
Se sabe que los catalizadores, como por ejemplo óxidos alcalino tØrreos
e hidrOxidos, aceleran Ia reacción de curado del siloxano en una lechada de estuco. Dichos catalizadores son relativamente solubles en agua y elevan el pH de la lechada. Un alto pH puede interferir con la rehidrataciOn del estuco, y puede reaccionar negativamente con algunos aditivos preferidos para paneles para paredes. Por lo tanto, aunque se promueve Ia polimerizaciOn del siloxano, otras consideraciones hacen que no sea deseable el uso de dichos catalizadores.
Se sabe que el óxido de magnesio ("MgO") cataliza [as reacciones del siloxano, pero si la reactividad del catalizador es suficientemente alta como para curar completamente el siloxano, sucede un agrietamiento que no es

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deseable. El MgO calcinado liviano tiene la actividad necesaria para curar rÆpidamenteel siloxano, pero dicha actividad causa reacciones secundarias no deseadas. Dichas reacciones secundarias generan hidrogeno, que causa la expansion del producto y grietas en el yeso fraguado. El MgO muy calcinado o calcinado a fondo tiene menor reactividad, pero da como resultado un producto menos resistente al agua. Por lo tanto, cuando se utiliza MgO es solo, es muy difIcil balancear la actividad del catalizador con el grado deseado de polimerizaciOn del siloxano.
TambiØn existen ciertas fuentes de estuco en las cuales es muy difIcil promover la polimerización del siloxano. El yeso es una mezcla compleja de sulfato de calcio en diversas formas, sales y una variedad de aluminatos, silicatos y aluminosilicatos. Aparentemente algunas fuentes de yeso incluyen UflO 0 mÆs componentes que impiden la formación de la resina de silicona. Cuando se utilizan con dichos estucos, los catalizadores conocidos no Ilegan al nivel deseado de resistencia al agua de menos del 5% de absorbancia de agua.

Por lo tanto, en el arte existe la necesidad de un catalizador y un mØtodo
para producir artIculos de yeso resistentes al agua con una mejor resistencia al agua a un costo razonable. El catalizador deberla ser relativamente barato, con buena actividad para la polimerizacion de siloxano con un mmnimo de reacciones secundarias no deseadas. Deberla haber poca interferencia entre el catalizador y otros aditivos comunes para yeso.
RESUMEN DE LA INVENCION

La presente invenciOn satisface o excede las anteriores necesidades y otras, acelerando Ia polimerizaciOn de siloxano y en algunos casos reduciendo

la cantidad de siloxano que es necesaria para satisfacer las especificaciones de ASTM 1398.

MÆs especIficamente, la polimerizaciOn de siloxano mejora al usar una lechada que incluye estuco, cenizas finas de clase C, Oxido de magnesio y una emulsiOn de siloxano y agua. Esta lechada se utiliza en un mØtodo para hacer artIculos de yeso resistentes al agua que incluye hacer una emulsion de siloxano y agua, luego combinar la lechada con una mezcla seca de estuco, óxido de magnesio y cenizas finas de clase C. Luego se forma la lechada segUn se desee y el estuco se deja fraguar y el siloxano polimeriza.
El producto que se obtiene es ütil para hacer un panel de yeso resistente
al agua con un nUcleo que incluye matrices entrelazadas de cristales de sulfato de calcio dihidrato y una resina de silicona, donde las matrices entrelazadas tienen disperso a travØs de sI un catalizador que comprende Oxido de magnesio y componentes provenientes de cenizas finas de clase C.
La mezcla de Oxido de magnesio y cenizas finas de clase C cataliza la polimerización de siloxano para acelerar el desarrollo de resistencia al agua en el producto hecho a partir de la lechada. No es necesario almacenar los productos resistentes al agua tales como paneles para paredes durante largos perlodos de tiempo a la espera de que se completen las reacciones de polimerización del siloxano.
El uso de dicho catalizador tambiØn aumenta el grado en que se completa la reacciOn, permitiendo obtener una mejor resistencia al agua. Se pudo conseguir una absorciOn de agua menor que un 5% en peso usando la combinación de ceniza fina y magnesia, que no se pudo conseguir con ninguno de los catalizadores solos. Por lo tanto, ademÆs de acelerar la reacciOn de

polimerizaciOn, dicho catalizador tambiØn permite que el siloxano polimerice mÆs completamente permitiendo reducir Ia cantidad de siloxano en algunos casos. Como el siloxano es uno de los aditivos mÆs costosos para paneles para paredes, la reducciOn de la dosificaciOn permite obtener ahorros en el costo de los materiales en bruto.

Otra ventaja de la presente invenciOn es la estabilidad dimensional del producto. Algunos compuestos que se utilizan para catalizar esta reacciOn dan como resultado una expansion significativa a medida que se seca el producto. La expansion del interior del tablero causa grietas en la superficie exterior del mismo, daæÆndola. El uso deceniza fina y óxido de magnesio da como resultado muy poca expansion y grietas muy pequenas en el producto terminado.

Este catalizador combinado de ceniza fina y magnesia tambiØn permite
una polimerización satisfactoria usando un mÆs amplio rango de grados de Oxido de magnesio. Aunque el arte anterior revela solamente grados de óxido de magnesio que la magnesia calcinada a fondo es apropiada para actuar como catalizador para la polimerización de siloxano, cuando se combinan con ceniza fina, aUn se puede utilizar Oxido de magnesio muy calcinado o calcinado liviano. Esta caracterIstica da a los fabricantes de productos de yeso una libertad adicional al seleccionar fuentes de Oxido de magnesio para utilizar en la lechada.
DESCRIPCION DETALLADA DE LA INVENCION La presente invención contempla ampliamente mejorar la resistencia al agua de los artIculos a base de yeso agregando un siloxano polimerizable a la lechada que se utiliza para hacer los artIculos a base de yeso. Preferiblemente,

el siloxano se agrega en forma de una emulsion. Luego se forma la lechada y se seca en condiciones que promueven la polimerización del siloxano para formar una resina de silicona altamente reticulada. Se agrega al lechada de yeso un catalizador que promueve la polimerización del siloxano para formar una resina de silicona altamente reticulada.
Preferiblemente, el siloxano es en general un siloxano lineal fluido modificado con hidrOgeno, pero tambiØn puede ser un siloxano cIclico modificado con hidrogeno. Dichos siloxanos son capaces de formar resinas de silicona altamente reticuladas. Dichos fluidos son bien conocidos por aquellos con una experiencia normal en el arte y se pueden obtener comercialmente y estÆndescritos en Ia literatura de patentes. Tipicamente, los siloxanos lineales modificados con hidrOgeno que son Utiles para practicar la presente invención comprenden aquellos con una unidad repetitiva con la formula general:

LRJn

donde R representa un radical hidrocarburo monovalente saturado o insaturado. En las formas de realización preferidas, R representa un grupo aiquilo y aün mÆs preferiblemente A es un grupo metilo. Durante la polimerizaciOn, los grupos terminales se eliminan por condensación y los grupos siloxano estÆn conectados juntos para formar la resina de silicona. TambiOn ocurre Ia reticulación de las cadenas. La resina de silicona que se obtiene confiere resistencia al agua a la matriz de yeso a medida que se forma. Los artIculos a base de yeso resistentes al agua de la presente invención se hacen preferiblemente con un metil hidrogeno siloxano fluido sin solvente que comercializa Wacker-Chemie GmbH (Munich, Alemania) con el

nombre SILRES BS 94 como el siloxano. El fabricante dice que este producto es un siloxano fluido que no contiene agua ni solventes. Se contempla que se puede utilizar entre aproximadamente 0,3 y 1,0% del siloxano BS 94, en base al peso de los ingredientes secos. Se prefiere usar entre aproximadamente 0,4 y aproximadamente 0,8% del siloxano en base al peso del estuco seco. DespuØs de formar la lechada, con el siloxano se forma una emulsiOn o
una suspension estable al agua. Se contempla el uso de varias emulsiones de siloxano en esta lechada. Las emulsiones de siloxano en agua tambiØn se pueden adquirir comercialmente, pero las mismas pueden incluir agentes emulsionantes que tienden a modificar las propiedades de los artIculos de yeso, como por ejemplo el pegado del papel en productos de paneles para paredes. Por lo tanto, se prefieren las emulsiones o suspensiones estables que se preparan sin usar emulsionantes. Preferiblemente, se forma una suspension in situ mezclando el siloxano fluido con agua. Es esencial que la suspension de siloxano sea estable hasta alcanzar la mezcladora de patillas y que permanezca bien dispersa en las condiciones de la lechada. La suspensiOn o emulsiOn de siloxano debe permanecer bien dispersa en presencia de los aditivos opcionales, como por ejemplo aceleradores de fraguado, que estÆn presentes en la lechada. La suspension o emulsion de siloxano tambiØn debe seguir siendo estable a travØs de los pasos donde se forman los artIculos a base de yeso. Preferiblemente, la suspension permanece estable durante mÆs de 40 minutos. MÆs preferiblemente, permanece estable durante por lo menos una hora. En la memoria descriptiva y las reivindicaciones a continuación, el tØrmino "emulsiOn" incluye verdaderas emulsiones y suspensiones que son estables por lo menos hasta que el estuco estÆ fraguadoen un 50%.

En una forma de realizaciOn preferida, por 10 menos una porcion del agua de mezcla se alimenta continuamente a Ia mezcladora de alto esfuerzo de corte. El siloxano fluido se dosifica dentro de la mezcladora de alto esfuerzo de corte junto con el agua para formar la emulsion en 1-2 segundos. La proporcion de agua a siloxano no es crItica y se sabe que es ütil una mezcla de 25 partes de agua a una parte siloxano. Esta emulsion es estable durante varios minutos sin el agregado de un emulsionante, un tiempo suficientemente largo como para mezclar la lechada, formar el artIculo y permitir que comience a fraguar. En la alternativa, tambiØn se contempla el uso de una porciOn del agua de mezcla para formar la emulsion. Se combina con el siloxano una corriente que se separa del agua de mezcla en la mezcladora de alto esfuerzo de corte. Luego, preferiblemente la emulsion de siloxano se agrega al agua de mezcla antes de formar la lechada para proveer un tiempo suficiente como para que la emulsiOn de siloxano se mezcle Intimamente con el agua que se utiliza para formar la lechada y que se disperse uniformemente a travØs de todo el artIculo que se obtiene.
Aunque sin deseos de limitarse a una teorla, se cree que la resistencia
al agua se desarrolla cuando el siloxano cura dentro de los paneles para paredes formados. La reacciOn de polimerización procede lentamente por Si misma, requiriendo que los paneles para paredes se almacenen durante un tiempo suficiente como para desarrollar resistencia al agua antes del embarque. Se sabe que los catalizadores aceleran la reacciOn de polimerización, reduciendo o eliminando el tiempo necesario para almacenar el producto de paneles para paredes mientras se desarrolla Ia resistencia al agua. El uso de Oxido de magnesio calcinado a fondo para Ia polimerización de

siloxano se describe en U.S. No. de Serie 10/917.177, co-pendiente, titulada "Method of Making Water-Resistant Gypsum-Based Article", que se incorpora aqul por referencia. El Oxido de magnesio calcinado a fondo es insoluble en agua e interactüa menos con otros componentes de la lechada. Este acelera el curado del siloxano y, en algunos casos, hace que el siloxano cure mÆs completamente. Se puede obtener comercialmente con una composiciOn consistente. Una fuente de Oxido de magnesio calcinado a fondo preferida en particular es BAYMAG 96. Este tiene un area superficial por BET de por lo menos 0,3m2/g. La pØrdida a la ignición es menor del 0,1% en peso. El Oxido de magnesio se utiliza preferiblemente en cantidades entre aproximadamente 0,1 y aproximadamente 0,5% en base al peso del estuco seco.
Existen por lo menos tres grados de Oxido de magnesio en el mercado, dependiendo de la temperatura de calcinaciOn. El Oxido de magnesio "calcinado a fondo" se calcina a entre 1500°C y 2000°C, eliminando la mayor parte, de la reactividad, si no toda. El MagChem P98-PV (Martin Marietta Magnesia Specialties, Betesda, MD) es un ejemplo de un óxido de magnesio "calcinado a fondo". BayMag 96 (Baymag, Inc. de Calgary, Alberta, Canada) y MagChem 10 (Martin Marietta Magnesia Specialties, Betesda, MD) son ejemplos de magnesia "muy calcinada". El óxido de magnesio "muy calcinado" se calcina a temperaturas entre 1000°C y aproximadamente 1500°C. Este tiene un estrecho rango de reactividad, alta densidad, y normalmente se utiliza en aplicaciones donde se requiere degradacion lenta 0 reactividad qulmica, como por ejemplo en piensos para animales y fertilizantes. El tercer grado es la magnesia "calcinado liviana" o "cÆustica", que se produce por calcinaciOn a temperaturas entre aproximadamente 700°C y aproximadamente 1000°C.

Dicho tipo de magnesia se utiliza en un amplio rango de aplicaciones, que incluyen plÆsticos, caucho, papely el proceso de pulpa, aditivos para calderas de acero, adhesivos y neutralización de Æcidos. Los ejemplos de magnesia calcinada liviana incluyen BayMag 30, BayMag 40, y BayMag 30 (-325 Mesh) (BayMag, Inc. de Calgary, Alberta, Canada).
Se ha descubierto que los catalizadores preferidos se hacen con una mezcla de óxido de magnesio y cenizas finas de clase C. Cuando se combinan en dicha manera, es Util cualquiera de los grados de Oxido de magnesio. Sin embargo, se prefieren los óxidos de magnesio calcinado a fondo y muy calcinado debido a su menor reactividad. La reactividad retativamente alta de los Oxidos de magnesio, puede causar reacciones de craqueo que pueden producir hidrOgeno. A medida que se genera el hidrOgeno, el producto se expande, causando grietas donde ha fraguado el estuco. La expansiOn tambiØn causa Ia rotura de los moldes donde se vierte el estuco, causando pØrdida de detalles y deformaciOn del producto en una o mÆs dimensiones. Preferiblemente, las fuentes de Oxido de magnesio preferidas son BayMag 96, MagChem P98-PV y MagChem 10. Preferiblemente, el Oxido de magnesio y la ceniza fina se agregan al estuco antes de agregarlos al agua de mezcla. Frecuentemente, los componentes secos tales como los anteriores, se agregan at estuco a medida que se mueve a lo largo de una cinta transportadora hacia la mezcladora.
Una ceniza fina preferida es una ceniza fina de clase C. La ceniza fina hidrÆulica de claseC, o su equivalente, es el componente de ceniza fina mÆs preferido. En la tabla 1 se muestra una composiciOn tIpica de una ceniza fina de clase C. La ceniza fina con alto contenido de cal, con un contenido de cal

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mayor del 20% en peso, se obtiene del proceso de ciertos carbones. La denominación ASTM C-618, que se incorpora aqul por referenda, describe las caracterIsticas de las cenizas finas de clase C. Una ceniza fina de ciase C preferida la provee Bayou Ash Inc., Big Cajun, II, LA. Preferiblemente, la ceniza
fina se utiliza en cantidades entre aproximadamente 0,1% y aproximadamente
5% en base al peso del estuco seco. MÆs preferiblemente, la ceniza fina se utiliza en cantidades entre aproximadamente 0,2% y 1,5% en base al peso del

estuco seco.
labia I
ComDosiciOn tIDica de la ceniza fina de tiDo C Composición Cantidad, en peso
Si02 25-59
A1203 14-22
Fe203 5-13
CaO 8-32
MgO 3,2-12,5
K20 0,3-1,6
Na20 0,8-6,0
S03 0,4-5,0
hO2 <1,0
PØrdida a la igniciOn 0,1-2,3
La catÆlisis del siloxano da como resultado una polimerizaciOn y reticulaciOn del siloxano mÆs rÆpiday mÆs completa para formar la resina de silicona. La hidrataciOn del estuco forma un matriz de cristales entrelazados de sulfato de calcio dihidrato. Mientras se estÆ formando Ia matriz de yeso, las