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(54) Title: PROCESS FOR PRODUCING WATER RESISTANT PLASTERBOARDS WITH FIBER CEMENT POWDER

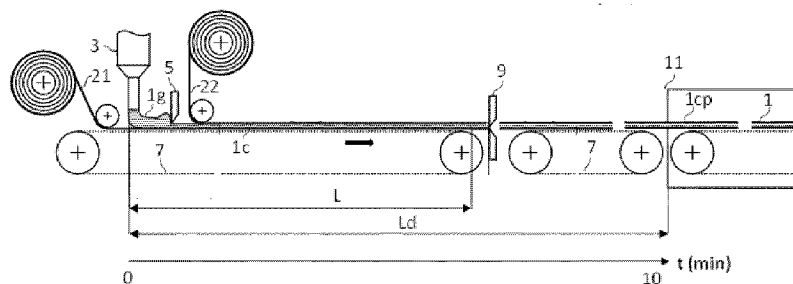


Fig.1

(57) Abstract: Process for producing water resistant plasterboard using the combination of polyhydrogenomethylsiloxane and the waste of fiber cement board.



PROCESS FOR PRODUCING WATER RESISTANT PLASTERBOARDS WITH FIBER CEMENT POWDER

TECHNICAL FIELD

- 5 **[0001]** The present invention concerns a process for producing water resistance plasterboards formed of a gypsum core sandwiched between two facers. The plasterboards obtained by the process of the present invention have enhanced water resistance and use cured fiber cement powder. The invention also concerns the use of fiber cement powder with poly methyl hydrogen siloxane.

BACKGROUND OF THE INVENTION

- 10 **[0002]** Fiber cement (FC) material is a composite material typically comprising cement, cellulose fibers, and at least one of silica sand, synthetic fibers and fillers. It is widely used in construction and can take the form of a plurality of products, such as for example corrugated sheets for roofs, small sheets for tiles (slates), sheets for sidings, cladding, boards, etc.

- 15 **[0003]** Cured fiber cement waste material has a chemical composition similar to the corresponding fresh fiber cement products from which the waste is derived, and therefore could, and ideally should, be recycled and reused. However, recycling waste material from cured fiber cement products, e.g. finished products being out of spec, demolition and/or construction waste and alike, remains a major challenge. Reusing fiber cement waste for various new purposes remains however the main, if not the only, approach to avoid disposal of large fiber cement waste streams.

- 20 **[0004]** Plasterboards are plates particularly useful for albeit not restrict to the building industry, comprising a core made of gypsum with various additives in minor amounts, sandwiched between bottom and top facers, generally made of paper and / or glass mat. They can be produced continuously on long production lines and cut to the desired lengths and dried in a drier.

- 25 **[0005]** A slurry of calcined calcium sulphate hemihydrate (or stucco) in water with the desired additives, such as accelerators, fibrous reinforcements, and the like, is continuously dispensed onto a bottom facer moving on a conveyor. The thickness of the layer of slurry on the bottom facer is controlled and a top facer is laid on top of a free surface of the slurry, such as to form a sandwich structure with a core formed by the slurry sandwiched between bottom and top facers. The calcined stucco in the core is allowed to undergo a hydration reaction to form a setting plasterboard with calcium sulphate hemihydrate ($\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$) being progressively replaced by calcium sulphate dihydrate ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) as the hydration reaction proceeds.
- 30

- [0006]** Once the core has set to a reasonably hard structure, the continuous setting plasterboard is cut to a desired length, prior to being moved into a drier to complete the hydration reaction and remove any excess water present in the core.

[0007] According to the final application, some plasterboard requires enhanced water resistance as it is known that regular gypsum plasterboard can lose its strength when exposed to high amount of moisture.

[0008] In order to improve the moisture resistance of a gypsum board, silicone and in particular poly methyl hydrogen siloxane (PHMS) is well known as water repellent agent. Increasing the pH of the slurry enhances the PHMS effect.

[0009] EP1112986 indeed describes the use PHMS and Portland cement to produce water resistant plasterboard. The hydrogen methyl siloxane used is cross-linked to form a silicone network in the material. The polymerization of siloxane to silicone requires water as a reagent. During the drying of a board, free water is removed, thereby reducing the water available for the reaction. Therefore, unreacted siloxane will not continue the cross-linking reaction after the board is completely dried. If the cross linking reaction is too slow, and the processing time of the board too short, the siloxane will not polymerize. Difficulties will also arise if the reaction occurs too quickly. If the siloxane begins to cross-link during its feeding into the slurry, a gel type silicone will form which is difficult to further uniformly disperse into the whole slurry, and hence the matrix of material. Additionally, if the polymerization reaction is completed before the rehydration of the hemihydrate is completed, the surface of the hemihydrate crystals will be blocked from contacting water needed for continuation of rehydration, causing a decrease in the strength of the material. Control of the cross-linking time to match the process time is an important factor. The amount of Portland cement as catalyst to the curing is however limited to 2 % relative to stucco because higher amount would be dangerous specifically in the case of a plasterboard produced not by a cake filtering but by an online process wherein the plasterboards enter in a dryer after few minutes.

[0010] US2014/0178 624 describes a method for making a water-resistant gypsum board comprising a hydroxide with siloxane. In order to minimise the production of hydrogen, part of the hydroxide is replaced by silicate.

[0011] Looking for recycling waste of FC, it has been surprisingly found that the use of powder of fiber cement board could be used in a large amount with a limited amount of hydrogen produced and an increase of the water repellence resulting not only from an increase of pH was observed.

[0012] As the correct dosage of fresh cement is very delicate to reach, most of the time the use of cement brings more disadvantages than advantages with regard to the expected benefits. Cement is so reactive with silicone that a slight overdosing releases a very high amount of gas (H₂) in a short period of time. This has two main negative consequences: one is potential safety issue related to H₂ gas and the other is a quality issue as the released gas can destroy the bond between the paper and the gypsum core. Also, the quick reaction of silicone may have also at least one negative consequence which is the too quick hydrofugation of the board in the process. Water is then expelled from the board, interfering with the hydration process and humidity balance of the core in the wet end part of the process where water is critical.

[0013] The use of fiber cement powder circumvents these negative consequences as the dosage is more easily controlled. Overdosing cured fiber cement powder is much less critical than it is for cement. In addition, the hydrophobization of the boards produced is higher than the one expected with the solely an increase of pH.

5 SUMMARY OF THE INVENTION

[0014] The present invention is defined in the appended independent claims. Preferred embodiments are defined in the dependent claims. In particular, the present invention concerns a process for producing water resistant plasterboards comprising the following steps,

- 10 • feeding a gypsum slurry (1g) comprising stucco, water, alkyl hydrogen polysiloxane, cured fiber cement powder and additives onto a bottom facer (21) laid on a conveyor (7) in motion,
- applying a top facer (22) onto a free surface of the gypsum slurry to form a sandwich structure with a core (1c) made of the gypsum slurry sandwiched between the top and bottom facers (21, 22),
- 15 • setting by hydration the gypsum slurry (1g) forming the core (1c) to form a setting plasterboard on the conveyor,
- cutting the setting plasterboard to desired dimensions to yield cut plasterboards (1cp),
- drying the cut plasterboards in a drying station (11) to yield the plasterboards (1),

wherein the cured fiber cement powder in an amount from 0.5 to 10 wt%, preferably 2 to 8, most preferably 3 to 5 wt.% relative to stucco.

- 20 **[0015]** In a preferred embodiment, the gypsum slurry comprises 100 parts of stucco and between 60 and 90 parts of water.

[0016] In a preferred embodiment the alkyl hydrogen polysiloxane is polymethylhydrogensiloxane (PMHS), the amount of PHMS is between 0.05 and 2 wt.%, most preferably between 0.1 and 1 wt. % relative to stucco.

- 25 **[0017]** The fiber cement can be air cured or autoclaved cured. In a preferred embodiment the cured fiber cement powder is autoclaved.

[0018] In a preferred embodiment, the pH of the slurry is between 9 and 11.5. The pH is measured according the testing method ME-A5-005.

- 30 **[0019]** The cured fiber cement can have different granulometry according the waste it comes from. In a preferred embodiment, the fiber cement powder has a d50 below 100 μm , preferably below 50 μm , most preferably below 40 μm .

[0020] In a preferred embodiment, the slurry does not comprise fly ash nor added clay.

[0021] The invention also relates plasterboards obtained by the process described here above.

[0022] The invention also relates to the use of cured fiber cement with PHMS to provide water resistant plasterboard having a water absorption according to the Norm EN520-2009 below 5%

BRIEF DESCRIPTION OF THE FIGURE

Figure 1 shows a side view of an example of production line configured for carrying out the process of the present invention.

DETAILED DESCRIPTION OF THE INVENTION

The present invention concerns a process for producing water resistant plasterboards (1) comprising the following steps,

- feeding a plaster slurry (1g) comprising stucco, water, alkyl hydrogen polysiloxane, cured fiber cement powder and additives onto a bottom facer (21) laid on a conveyor (7) in motion,
- applying a top facer (22) onto a free surface of the gypsum slurry to form a sandwich structure with a core (1c) made of the gypsum slurry sandwiched between the top and bottom facers (21, 22),
- setting by hydration the gypsum slurry (1g) forming the core (1c) to form a setting plasterboard on the conveyor,
- cutting the setting plasterboard to desired dimensions to yield cut plasterboards (1cp),
- drying the cut plasterboards in a drying station (11) to yield the plasterboards (1),

The cured fiber cement powder is added in an amount from 0.5 to 10 wt%, preferably 2 to 8, most preferably 3 to 5 wt.% relative to stucco.

[0023] The expression "water resistant" plasterboard should be understood to mean the ability of a plasterboard to limit the uptake of water by the plasterboard, while still retaining the dimensional stability and mechanical integrity of the structural element in question.

[0024] According to the norm NF EN520+A1,2009 the total water absorption of a plasterboard should be below 5%.

[0025] Gypsum slurry comprises stucco or calcined gypsum in the form of calcium sulphate hemihydrate ($\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$), water, and additives, including an accelerator, retarder, fibre reinforcement, fluidizer, starch. In order to obtain stucco, natural or synthetic gypsum may be used. Natural gypsum may be obtained from gypsum rock or gypsum sand. Synthetic gypsum typically originates from flue gas desulfurization (FGD) or phosphoric acid production. The stucco comprises more than 70 wt% of β calcium sulphate hemihydrate, preferably more than 80%, most preferably more than 90 wt% relative to the stucco weight.

[0026] The water repellent additive is polysiloxane hydrogen methyl (PHMS), in the form of an oligomer (several tens or more of siloxane units), in the form of an oil or of an aqueous emulsion.

[0027] The cured fiber cement material powder is typically a waste material which is either originated from comminuted cured fiber cement waste or directly produced in the process of fiber cement board when the board is sanded for example.

[0028] Cured fiber cement powder for use in plasterboard manufacturing process, preferably, though not necessarily, is autoclaved-cured fiber cement powder. In the alternative, the cured fiber cement product may be air-cured fiber cement powder.

[0029] The cured fiber cement powder particles of the invention have a particle size distribution, which is similar to the particle size distribution of a cementitious binder material (e.g. cement) or a siliceous source (e.g. sand or quartz) or a filler material (e.g. CaCO₃). In particular embodiments, the produced cured fiber cement powder particles are characterized by a particle size distribution, which is similar to the particle size distribution of cement. In particular embodiments, the produced cured fiber cement powder particles are characterized by a particle size distribution, which is similar to the particle size distribution of a siliceous source.

[0030] An example of a chemical composition in w% of one of the autoclaved cured fiber cement is the following (in (m-%))

LOI ^{1000°C}	17.2
C organic	4.5% corresponding to 10.1 cellulose content
CO ₂	2.46
SiO ₂	45.71
CaO	25.96
MgO	0.73
Al ₂ O ₃	5.8
Mn ₂ O ₃	3.86
K ₂ O	0.24
SO ₃	0.46
Fe ₂ O ₃	3.86
Na ₂ O	0.19
P ₂ O ₅	0.16
TiO ₂	0.18

Table1

[0031] The particle size can be expressed by d50. The term "d50" in the sense of the present invention is a measure for the average particle size and is defined as follows: 50% (by number) of the particles (e.g. grains) in the corresponding sample have a size which is equal or smaller than the given d50 value. The term "particle size" especially represents the diameter of a sphere whose volume is identical to that of the particle under consideration having an arbitrary shape. The particle sizes referred to herein can be measured using Laser Diffraction Spectrometry (LOS). The particle size distribution was measured via laser beam diffraction in dry dispersion at 3 bar with a Malvern MasterSizer 2000.

CONTINUOUS PRODUCTION LINE

[0032] A production line for the continuous production of plasterboards suitable for the present invention is schematically illustrated in Figure 1. It comprises a roll of bottom facer (21) configured for continuously feeding a conveyor (7) with the bottom facer (21). A slurry dispensing unit (3) is positioned above the bottom facer (21) as it is transported by the conveyor (7) and is configured for pouring a gypsum slurry (1g) onto an inner face of the bottom facer (21), which faces upwards. A levelling blade (5) or roller is arranged downstream of the slurry feeding unit (3) to control the thickness of the layer of slurry deposited on the bottom facer (21). A roll of top facer is positioned above the conveyor, downstream of the levelling blade (5) and is configured for applying the top facer (22) on the free surface of the gypsum slurry (1g) to form a sandwich structure with a core (1c) made of the gypsum slurry sandwiched between the top and bottom facers (21, 22).

[0033] A cutting unit (9) is positioned at a cutter distance (L) from the slurry dispensing unit (3) and is configured for cutting the continuous setting plasterboard into panels of specific length referred to as cut plasterboards (1cp). The cut plasterboards (1cp) are then driven into a drying station (11)

[0034] The hydration reaction transforming the calcium hemihydrate to gypsum according to the reaction $\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O} + 1\frac{1}{2}\text{H}_2\text{O} \rightarrow \text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ is almost finished (around 97 % of hydration) when the board get to the drying station (11).

[0035] The drying station (11) is positioned downstream of the cutting unit (9). It is configured for drying the cut plasterboards (1cp) during a drying period sufficient for gently evaporating any excess water, which was required to form the initial slurry. After the plasterboards (1) have dried, they are trimmed and stacked to form pallets ready for use (not shown).

[0036] The water-repellence of PHMS is enhanced when the silicon compound cures or cross linked. However, this accompanied by significant evolution of hydrogen, which is particularly dangerous if this hydrogen is confined in the dryer.

[0037] The invention is further described in and illustrated by the following Examples. The Examples are not to be construed as limiting the invention in anyway.

[0038] EXAMPLES

[0039] A series of experiments were carried out using stucco and FC powders.

Stucco	pH measured (water solution at 5% at 20°C)
Stucco A ref	7.1
Stucco B ref	7.7
Stucco C	8.6
FC powder A	11.7
FC powder B	11.1

Table 2

PHMS used in the examples is Wacker SILRES® BS 94.

- 5 All the wt % are expressed relative to the stucco weight.

[0040] Production of HYDROGEN

In order to assess the potential release of hydrogen when using fiber cement, a test was conducted using a calcimeter instrument. The cured FC powder and PHMS were brought into contact with each other, in water at 20°C in the reactor, in the same relative proportions than in recipe of Table 5. The evolution of hydrogen is monitored over time, at a controlled temperature of 20° C.

The total volume of hydrogen evolved after three hours is measured.

This volume is then converted into an equivalent gas volume released for 1 sqm of 12.5mm board.

	weight	Equivalent in a plaster composition wt%	PHMS	H2 (L)
FC powder A	0.26g	2	0.1g	1.8
CaO	0.1g	0.8	0.1g	4.5

Table3

The H2 volume produced is lower than the one obtained with a mixture with CaO being an major component of cement.

[0041] Water intake on 4x 4 x 16 Prism samples;

First tests were carried out by preparing prism samples using two different kinds of stucco, same amount of PHMS (0.74wt%) and different amounts of FC powder. When qualifying a plasterboard water resistant, the target value for water uptake after 2hr immersion of water resistant plasterboard is below 5%. The immersion tests were performed according to the standard EN520+A1; said standard is followed unless specified otherwise.

Stucco	FC	wt% of FC powder	Water uptake after 2 H	pH (in the slurry)
A ref	A	0	6.03	6.9
A	A	1.4	1.55	9.8
A	A	4.3	1.49	10.3
B ref	B	0	26.31	6.7
B	B	1	10.17	7.8
B	B	3	4.85	9.3
B	B	5	2.84	9.6
B	B	7	1.95	9.8

Table 4

[0042] The FC powder has a positive impact on the hydrophobization of silicone in gypsum prism formulations, irrespective to the gypsum origin. Depending on the gypsum source, the performance of PHMS alone varies greatly.

5 [0043] Water uptake on lab minibboards (316mm x 316 mm x 12.5 mm)

Lab minibboards having a thickness of 12,5mm are made by mixing the ingredients as set out in table 5 into a gypsum slurry. The slurry was mixed using a suitable mixer such as a propeller type mixer and was poured into a mould and allowed to hydrate and set between two facers. After setting the mould was removed and the miniboard was dried in a heated air circulating drying kiln. In this preparation, the time sequence for the mixing, pouring and drying mimics the one of the industrial process. The sample surface size is 1/10 sqm.

Component	g
Stucco	1200
accelerator	8
starch	7.25
retarder	0.03
FC dust	25.9
water	880
foam water	110
foaming agent	0.4
fluidizer	4.54
water repeller BS94	9.67

Table5

[0044] The water uptake done according to the norm NF EN520-A+ 2009 has also been measured after 24 and 48 hrs of full immersion in order to get more insight on the long-term behaviour of the formulations tested.

Stucco	PHMS Wt%	FC powder	FC powder Wt%	Water uptake after 2 H	Water uptake after 24 H	Water uptake after 48 H	pH (in the slurry)
C Ref	0.74	A	0	6.6	14.9	15.3	7.4
C	0.74	A	2	2.3	5.6	6.1	9.5
B	0.74	B	2	3.29	6.34	8.18	9.3
B	0.74	B	5	3.2	7.4	9.43	9.1

Table 6

The results confirm the positive effect of PHMS/FC powder observed on the prism samples.

[0045] Table 7 demonstrates that an increase of pH is not solely responsible of the improvement of the water resistance of the plasterboard.

	Stucco	pH slurry	PHMS(%)	FC powder(%)	Ca(OH) ₂	Water uptake 2H
Ref 1	D	6.3	0.74	0	0	6.3
Ref 2	D	10.7	0.74	0	0.1	4.7
1	D	9.7	0.74	2	0	4

Table7

[0046] Ref 2 shows an improvement of the water resistance of the plasterboard obtained by increasing the pH. According to the invention sample 1 shows better water resistance than ref2 with a lower pH. The inventor believes that in addition to the pH effect, the FC powder provides surface adhesion allowing a better spread of the PHMS in the slurry.

[0047]

REF	DESCRIPTION
1	plasterboard
1c	core
1cp	Cut plasterboard
1g	Gypsum slurry
1cp	cut plasterboards
3	Slurry dispensing unit
5	Levelling blade
7	conveyor
9	Cutting unit
11	Drying station
21	Bottom facer
22	Top facer
30	Cooling station

CLAIMS

1. Process for producing **water resistant** plasterboards (1) comprising the following steps,

- 5 • feeding a gypsum slurry (1g) comprising stucco, water, alkyl hydrogen polysiloxane, cured fiber cement powder and additives onto a bottom facer (21) laid on a conveyor (7) in motion,
- applying a top facer (22) onto a free surface of the gypsum slurry to form a sandwich structure with a core (1c) made of the gypsum slurry sandwiched between the top and bottom facers (21, 22),
- 10 • setting by hydration the gypsum slurry (1g) forming the core (1c) to form a setting plasterboard on the conveyor,
- cutting the setting plasterboard to desired dimensions to yield cut plasterboards (1cp),
- drying the cut plasterboards in a drying station (11) to yield the plasterboards (1),

characterized in that cured fiber cement powder in an amount from 0.5 to 10 wt%, preferably 2 to 8, most preferably 3 to 5 wt.% relative to stucco.

15 2. Process according to claim 1 wherein the gypsum slurry comprises 100 parts of stucco and between 60 and 90 parts of water.

3. Process according to anyone of claims 1 to 2 wherein the alkyl hydrogen polysiloxane is polymethylhydrogensiloxane (PHMS).

20 4. Process according to anyone of claims 1 to 3 wherein the amount of PHMS is between 0.05 and 2 wt.%, most preferably between 0.1 and 1 wt. % relative to stucco.

5. Process according to anyone of claims 1 to 4 wherein the cured fiber cement waste is autoclaved.

6. Process according to anyone of claims 1 to 5 wherein the stucco comprises more than 70 wt% of β calcium sulphate hemihydrate, preferably more than 80%, most preferably more than 90 wt% relative to the stucco weight.

25 6. Process according to anyone of claims 1 to 5 wherein the pH of the slurry is between 9 and 11.5 said pH being measured according to the testing method ME-A5-005.

7. Process according to anyone of claims 1 to 6 wherein the fiber cement powder has a d50 below 100 μm , preferably below 50, most preferably below 40 μm , the particle sizes being measured using Laser Diffraction Spectrometry (LOS).

30 8. Process according to anyone of claims 1 to 7 wherein the stucco does not comprise fly ash nor added clay.

9. Plasterboard obtained by the process of anyone of claim 1 to 8.

10. Use of cured fiber cement with PHMS to provide water resistant plasterboard having a total water absorption according to the Norm EN520-2009 below 5%.

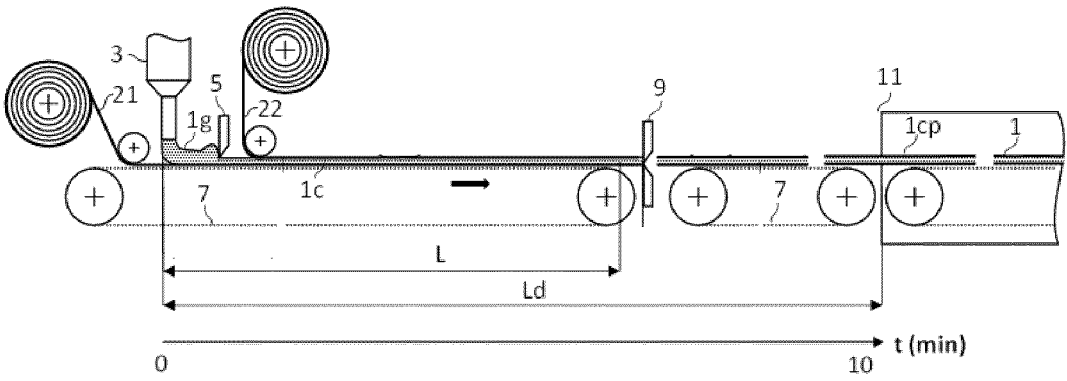


Fig.1

INTERNATIONAL SEARCH REPORT

International application No

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A. CLASSIFICATION OF SUBJECT MATTER

INV. C04B28/14 C04B18/16 C04B24/42
 ADD. C04B111/27

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C04B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	JP 3 284756 B2 (MITSUBISHI CHEM CORP; YOSHINO GYPSUM CO) 20 May 2002 (2002-05-20)	1-10
A	paragraphs [0004], [0013], [0017]; claims; examples -----	11
Y	EP 3 305 742 A1 (ETEX SERVICES NV) 11 April 2018 (2018-04-11)	1-10
A	paragraphs [0006], [0008], [0035], [0037], [0042], [0043]; figures 1,2 -----	11
A	US 5 154 874 A (KOSLOWSKI THOMAS [DE]) 13 October 1992 (1992-10-13) claims; figure -----	1-11
A	DE 297 05 438 U1 (FELS WERKE GMBH [DE]) 26 June 1997 (1997-06-26) the whole document -----	1-11
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INTERNATIONAL SEARCH REPORT

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C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	MÜLLER A ET AL: "Material utilization of fibre cement waste", ZKG INTERNATIONAL - ZEMENT - KALK - GIPS INTERNATIONAL, BAUVERLAG BV GMBH, DE, vol. 64, no. 3, 1 March 2011 (2011-03-01), pages 60-72, XP001561555, ISSN: 0949-0205 the whole document -----	1-11

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/EP2022/087559

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
JP 3284756 B2	20-05-2002	JP 3284756 B2	20-05-2002
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PROCESS FOR PRODUCING WATER RESISTANT PLASTERBOARDS WITH FIBER CEMENT POWDER

TECHNICAL FIELD

- 5 **[0001]** The present invention concerns a process for producing water resistance plasterboards formed of a gypsum core sandwiched between two facers. The plasterboards obtained by the process of the present invention have enhanced water resistance and use cured fiber cement powder. The invention also concerns the use of fiber cement powder with poly methyl hydrogen siloxane.

BACKGROUND OF THE INVENTION

- 10 **[0002]** Fiber cement (FC) material is a composite material typically comprising cement, cellulose fibers, and at least one of silica sand, synthetic fibers and fillers. It is widely used in construction and can take the form of a plurality of products, such as for example corrugated sheets for roofs, small sheets for tiles (slates), sheets for sidings, cladding, boards, etc.

- 15 **[0003]** Cured fiber cement waste material has a chemical composition similar to the corresponding fresh fiber cement products from which the waste is derived, and therefore could, and ideally should, be recycled and reused. However, recycling waste material from cured fiber cement products, e.g. finished products being out of spec, demolition and/or construction waste and alike, remains a major challenge. Reusing fiber cement waste for various new purposes remains however the main, if not the only, approach to avoid disposal of large fiber cement waste streams.

- 20 **[0004]** Plasterboards are plates particularly useful for albeit not restrict to the building industry, comprising a core made of gypsum with various additives in minor amounts, sandwiched between bottom and top facers, generally made of paper and / or glass mat. They can be produced continuously on long production lines and cut to the desired lengths and dried in a drier.

- 25 **[0005]** A slurry of calcined calcium sulphate hemihydrate (or stucco) in water with the desired additives, such as accelerators, fibrous reinforcements, and the like, is continuously dispensed onto a bottom facer moving on a conveyor. The thickness of the layer of slurry on the bottom facer is controlled and a top facer is laid on top of a free surface of the slurry, such as to form a sandwich structure with a core formed by the slurry sandwiched between bottom and top facers. The calcined stucco in the core is allowed to undergo a hydration reaction to form a setting plasterboard with calcium sulphate hemihydrate ($\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$) being progressively replaced by calcium sulphate dihydrate ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) as the hydration reaction proceeds.
- 30

- [0006]** Once the core has set to a reasonably hard structure, the continuous setting plasterboard is cut to a desired length, prior to being moved into a drier to complete the hydration reaction and remove any excess water present in the core.

[0007] According to the final application, some plasterboard requires enhanced water resistance as it is known that regular gypsum plasterboard can lose its strength when exposed to high amount of moisture.

[0008] In order to improve the moisture resistance of a gypsum board, silicone and in particular poly methyl hydrogen siloxane (PHMS) is well known as water repellent agent. Increasing the pH of the slurry enhances the PHMS effect.

[0009] EP1112986 indeed describes the use PHMS and Portland cement to produce water resistant plasterboard. The hydrogen methyl siloxane used is cross-linked to form a silicone network in the material. The polymerization of siloxane to silicone requires water as a reagent. During the drying of a board, free water is removed, thereby reducing the water available for the reaction. Therefore, unreacted siloxane will not continue the cross-linking reaction after the board is completely dried. If the cross linking reaction is too slow, and the processing time of the board too short, the siloxane will not polymerize. Difficulties will also arise if the reaction occurs too quickly. If the siloxane begins to cross-link during its feeding into the slurry, a gel type silicone will form which is difficult to further uniformly disperse into the whole slurry, and hence the matrix of material. Additionally, if the polymerization reaction is completed before the rehydration of the hemihydrate is completed, the surface of the hemihydrate crystals will be blocked from contacting water needed for continuation of rehydration, causing a decrease in the strength of the material. Control of the cross-linking time to match the process time is an important factor. The amount of Portland cement as catalyst to the curing is however limited to 2 % relative to stucco because higher amount would be dangerous specifically in the case of a plasterboard produced not by a cake filtering but by an online process wherein the plasterboards enter in a dryer after few minutes.

[0010] US2014/0178 624 describes a method for making a water-resistant gypsum board comprising a hydroxide with siloxane. In order to minimise the production of hydrogen, part of the hydroxide is replaced by silicate.

[0011] Looking for recycling waste of FC, it has been surprisingly found that the use of powder of fiber cement board could be used in a large amount with a limited amount of hydrogen produced and an increase of the water repellence resulting not only from an increase of pH was observed.

[0012] As the correct dosage of fresh cement is very delicate to reach, most of the time the use of cement brings more disadvantages than advantages with regard to the expected benefits. Cement is so reactive with silicone that a slight overdosing releases a very high amount of gas (H₂) in a short period of time. This has two main negative consequences: one is potential safety issue related to H₂ gas and the other is a quality issue as the released gas can destroy the bond between the paper and the gypsum core. Also, the quick reaction of silicone may have also at least one negative consequence which is the too quick hydrofugation of the board in the process. Water is then expelled from the board, interfering with the hydration process and humidity balance of the core in the wet end part of the process where water is critical.

[0013] The use of fiber cement powder circumvents these negative consequences as the dosage is more easily controlled. Overdosing cured fiber cement powder is much less critical than it is for cement. In addition, the hydrophobization of the boards produced is higher than the one expected with the solely an increase of pH.

5 SUMMARY OF THE INVENTION

[0014] The present invention is defined in the appended independent claims. Preferred embodiments are defined in the dependent claims. In particular, the present invention concerns a process for producing water resistant plasterboards comprising the following steps,

- 10 • feeding a gypsum slurry (1g) comprising stucco, water, alkyl hydrogen polysiloxane, cured fiber cement powder and additives onto a bottom facer (21) laid on a conveyor (7) in motion,
- applying a top facer (22) onto a free surface of the gypsum slurry to form a sandwich structure with a core (1c) made of the gypsum slurry sandwiched between the top and bottom facers (21, 22),
- 15 • setting by hydration the gypsum slurry (1g) forming the core (1c) to form a setting plasterboard on the conveyor,
- cutting the setting plasterboard to desired dimensions to yield cut plasterboards (1cp),
- drying the cut plasterboards in a drying station (11) to yield the plasterboards (1),

wherein the cured fiber cement powder in an amount from 0.5 to 10 wt%, preferably 2 to 8, most preferably 3 to 5 wt.% relative to stucco.

- 20 **[0015]** In a preferred embodiment, the gypsum slurry comprises 100 parts of stucco and between 60 and 90 parts of water.

[0016] In a preferred embodiment the alkyl hydrogen polysiloxane is polymethylhydrogensiloxane (PMHS), the amount of PHMS is between 0.05 and 2 wt.%, most preferably between 0.1 and 1 wt. % relative to stucco.

- 25 **[0017]** The fiber cement can be air cured or autoclaved cured. In a preferred embodiment the cured fiber cement powder is autoclaved.

[0018] In a preferred embodiment, the pH of the slurry is between 9 and 11.5. The pH is measured according the testing method ME-A5-005.

- 30 **[0019]** The cured fiber cement can have different granulometry according the waste it comes from. In a preferred embodiment, the fiber cement powder has a d50 below 100 μm , preferably below 50 μm , most preferably below 40 μm .

[0020] In a preferred embodiment, the slurry does not comprise fly ash nor added clay.

[0021] The invention also relates plasterboards obtained by the process described here above.

[0022] The invention also relates to the use of cured fiber cement with PHMS to provide water resistant plasterboard having a water absorption according to the Norm EN520-2009 below 5%

BRIEF DESCRIPTION OF THE FIGURE

Figure 1 shows a side view of an example of production line configured for carrying out the process of the present invention.

DETAILED DESCRIPTION OF THE INVENTION

The present invention concerns a process for producing water resistant plasterboards (1) comprising the following steps,

- feeding a plaster slurry (1g) comprising stucco, water, alkyl hydrogen polysiloxane, cured fiber cement powder and additives onto a bottom facer (21) laid on a conveyor (7) in motion,
- applying a top facer (22) onto a free surface of the gypsum slurry to form a sandwich structure with a core (1c) made of the gypsum slurry sandwiched between the top and bottom facers (21, 22),
- setting by hydration the gypsum slurry (1g) forming the core (1c) to form a setting plasterboard on the conveyor,
- cutting the setting plasterboard to desired dimensions to yield cut plasterboards (1cp),
- drying the cut plasterboards in a drying station (11) to yield the plasterboards (1),

The cured fiber cement powder is added in an amount from 0.5 to 10 wt%, preferably 2 to 8, most preferably 3 to 5 wt.% relative to stucco.

[0023] The expression "water resistant" plasterboard should be understood to mean the ability of a plasterboard to limit the uptake of water by the plasterboard, while still retaining the dimensional stability and mechanical integrity of the structural element in question.

[0024] According to the norm NF EN520+A1,2009 the total water absorption of a plasterboard should be below 5%.

[0025] Gypsum slurry comprises stucco or calcined gypsum in the form of calcium sulphate hemihydrate ($\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$), water, and additives, including an accelerator, retarder, fibre reinforcement, fluidizer, starch. In order to obtain stucco, natural or synthetic gypsum may be used. Natural gypsum may be obtained from gypsum rock or gypsum sand. Synthetic gypsum typically originates from flue gas desulfurization (FGD) or phosphoric acid production. The stucco comprises more than 70 wt% of β calcium sulphate hemihydrate, preferably more than 80%, most preferably more than 90 wt% relative to the stucco weight.

[0026] The water repellent additive is polysiloxane hydrogen methyl (PHMS), in the form of an oligomer (several tens or more of siloxane units), in the form of an oil or of an aqueous emulsion.

[0027] The cured fiber cement material powder is typically a waste material which is either originated from comminuted cured fiber cement waste or directly produced in the process of fiber cement board when the board is sanded for example.

[0028] Cured fiber cement powder for use in plasterboard manufacturing process, preferably, though not necessarily, is autoclaved-cured fiber cement powder. In the alternative, the cured fiber cement product may be air-cured fiber cement powder.

[0029] The cured fiber cement powder particles of the invention have a particle size distribution, which is similar to the particle size distribution of a cementitious binder material (e.g. cement) or a siliceous source (e.g. sand or quartz) or a filler material (e.g. CaCO_3). In particular embodiments, the produced cured fiber cement powder particles are characterized by a particle size distribution, which is similar to the particle size distribution of cement. In particular embodiments, the produced cured fiber cement powder particles are characterized by a particle size distribution, which is similar to the particle size distribution of a siliceous source.

[0030] An example of a chemical composition in w% of one of the autoclaved cured fiber cement is the following (in (m-%))

LOI $^{1000^\circ\text{C}}$	17.2
C organic	4.5% corresponding to 10.1 cellulose content
CO_2	2.46
SiO_2	45.71
CaO	25.96
MgO	0.73
Al_2O_3	5.8
Mn_2O_3	3.86
K_2O	0.24
SO_3	0.46
Fe_2O_3	3.86
Na_2O	0.19
P_2O_5	0.16
TiO_2	0.18

Table1

[0031] The particle size can be expressed by d50. The term "d50" in the sense of the present invention is a measure for the average particle size and is defined as follows: 50% (by number) of the particles (e.g. grains) in the corresponding sample have a size which is equal or smaller than the given d50 value. The term "particle size" especially represents the diameter of a sphere whose volume is identical to that of the particle under consideration having an arbitrary shape. The particle sizes referred to herein can be measured using Laser Diffraction Spectrometry (LOS). The particle size distribution was measured via laser beam diffraction in dry dispersion at 3 bar with a Malvern MasterSizer 2000.

CONTINUOUS PRODUCTION LINE

[0032] A production line for the continuous production of plasterboards suitable for the present invention is schematically illustrated in Figure 1. It comprises a roll of bottom facer (21) configured for continuously feeding a conveyor (7) with the bottom facer (21). A slurry dispensing unit (3) is positioned above the bottom facer (21) as it is transported by the conveyor (7) and is configured for pouring a gypsum slurry (1g) onto an inner face of the bottom facer (21), which faces upwards. A levelling blade (5) or roller is arranged downstream of the slurry feeding unit (3) to control the thickness of the layer of slurry deposited on the bottom facer (21). A roll of top facer is positioned above the conveyor, downstream of the levelling blade (5) and is configured for applying the top facer (22) on the free surface of the gypsum slurry (1g) to form a sandwich structure with a core (1c) made of the gypsum slurry sandwiched between the top and bottom facers (21, 22).

[0033] A cutting unit (9) is positioned at a cutter distance (L) from the slurry dispensing unit (3) and is configured for cutting the continuous setting plasterboard into panels of specific length referred to as cut plasterboards (1cp). The cut plasterboards (1cp) are then driven into a drying station (11).

[0034] The hydration reaction transforming the calcium hemihydrate to gypsum according to the reaction $\text{CaSO}_4 \cdot \frac{1}{2} \text{H}_2\text{O} + 1\frac{1}{2} \text{H}_2\text{O} \rightarrow \text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ is almost finished (around 97 % of hydration) when the board get to the drying station (11).

[0035] The drying station (11) is positioned downstream of the cutting unit (9). It is configured for drying the cut plasterboards (1cp) during a drying period sufficient for gently evaporating any excess water, which was required to form the initial slurry. After the plasterboards (1) have dried, they are trimmed and stacked to form pallets ready for use (not shown).

[0036] The water-repellence of PHMS is enhanced when the silicon compound cures or cross linked. However, this accompanied by significant evolution of hydrogen, which is particularly dangerous if this hydrogen is confined in the dryer.

[0037] The invention is further described in and illustrated by the following Examples. The Examples are not to be construed as limiting the invention in anyway.

[0038] EXAMPLES

[0039] A series of experiments were carried out using stucco and FC powders.

Stucco	pH measured (water solution at 5% at 20°C)
Stucco A ref	7.1
Stucco B ref	7.7
Stucco C	8.6
FC powder A	11.7
FC powder B	11.1

Table 2

PHMS used in the examples is Wacker SILRES® BS 94.

- 5 All the wt % are expressed relative to the stucco weight.

[0040] *Production of HYDROGEN*

In order to assess the potential release of hydrogen when using fiber cement, a test was conducted using a calcimeter instrument. The cured FC powder and PHMS were brought into contact with each other, in water at 20°C in the reactor, in the same relative proportions than in recipe of Table 5. The evolution of hydrogen is monitored over time, at a controlled temperature of 20° C.

The total volume of hydrogen evolved after three hours is measured.

This volume is then converted into an equivalent gas volume released for 1 sqm of 12.5mm board.

	weight	Equivalent in a plaster composition wt%	PHMS	H2 (L)
FC powder A	0.26g	2	0.1g	1.8
CaO	0.1g	0.8	0.1g	4.5

Table3

The H2 volume produced is lower than the one obtained with a mixture with CaO being an major component of cement.

[0041] *Water intake on 4x 4 x 16 Prism samples;*

First tests were carried out by preparing prism samples using two different kinds of stucco, same amount of PHMS (0.74wt%) and different amounts of FC powder. When qualifying a plasterboard water resistant, the target value for water uptake after 2hr immersion of water resistant plasterboard is below 5%. The immersion tests were performed according to the standard EN520+A1; said standard is followed unless specified otherwise.

Stucco	FC	wt% of FC powder	Water uptake after 2 H	pH (in the slurry)
A ref	A	0	6.03	6.9
A	A	1.4	1.55	9.8
A	A	4.3	1.49	10.3
B ref	B	0	26.31	6.7
B	B	1	10.17	7.8
B	B	3	4.85	9.3
B	B	5	2.84	9.6
B	B	7	1.95	9.8

Table 4

[0042] The FC powder has a positive impact on the hydrophobization of silicone in gypsum prism formulations, irrespective to the gypsum origin. Depending on the gypsum source, the performance of PHMS alone varies greatly.

5 **[0043] Water uptake on lab minibboards (316mm x 316 mm x 12.5 mm)**

Lab minibboards having a thickness of 12,5mm are made by mixing the ingredients as set out in table 5 into a gypsum slurry. The slurry was mixed using a suitable mixer such as a propeller type mixer and was poured into a mould and allowed to hydrate and set between two facers. After setting the mould was removed and the miniboard was dried in a heated air circulating drying kiln. In this preparation, the time sequence for the mixing, pouring and drying mimics the one of the industrial process. The sample surface size is 1/10 sqm.

Component	g
Stucco	1200
accelerator	8
starch	7.25
retarder	0.03
FC dust	25.9
water	880
foam water	110
foaming agent	0.4
fluidizer	4.54
water repeller BS94	9.67

Table5

[0044] The water uptake done according to the norm NF EN520-A+ 2009 has also been measured after 24 and 48 hrs of full immersion in order to get more insight on the long-term behaviour of the formulations tested.

Stucco	PHMS Wt%	FC powder	FC powder Wt%	Water uptake after 2 H	Water uptake after 24 H	Water uptake after 48 H	pH (in the slurry)
C Ref	0.74	A	0	6.6	14.9	15.3	7.4
C	0.74	A	2	2.3	5.6	6.1	9.5
B	0.74	B	2	3.29	6.34	8.18	9.3
B	0.74	B	5	3.2	7.4	9.43	9.1

Table 6

The results confirm the positive effect of PHMS/FC powder observed on the prism samples.

[0045] Table 7 demonstrates that an increase of pH is not solely responsible of the improvement of the water resistance of the plasterboard.

	Stucco	pH slurry	PHMS(%)	FC powder(%)	Ca(OH) ₂	Water uptake 2H
Ref 1	D	6.3	0.74	0	0	6.3
Ref 2	D	10.7	0.74	0	0.1	4.7
1	D	9.7	0.74	2	0	4

Table7

[0046] Ref 2 shows an improvement of the water resistance of the plasterboard obtained by increasing the pH. According to the invention sample 1 shows better water resistance than ref2 with a lower pH. The inventor believes that in addition to the pH effect, the FC powder provides surface adhesion allowing a better spread of the PHMS in the slurry.

[0047]

REF	DESCRIPTION
1	plasterboard
1c	core
1cp	Cut plasterboard
1g	Gypsum slurry
1cp	cut plasterboards
3	Slurry dispensing unit
5	Levelling blade
7	conveyor
9	Cutting unit
11	Drying station
21	Bottom facer
22	Top facer
30	Cooling station

CLAIMS

1. Process for producing **water resistant** plasterboards (1) comprising the following steps,

- 5 • feeding a gypsum slurry (1g) comprising stucco, water, alkyl hydrogen polysiloxane, cured fiber cement powder and additives onto a bottom facer (21) laid on a conveyor (7) in motion,
- applying a top facer (22) onto a free surface of the gypsum slurry to form a sandwich structure with a core (1c) made of the gypsum slurry sandwiched between the top and bottom facers (21, 22),
- 10 • setting by hydration the gypsum slurry (1g) forming the core (1c) to form a setting plasterboard on the conveyor,
- cutting the setting plasterboard to desired dimensions to yield cut plasterboards (1cp),
- drying the cut plasterboards in a drying station (11) to yield the plasterboards (1),

characterized in that cured fiber cement powder in an amount from 0.5 to 10 wt%, preferably 2 to 8, most preferably 3 to 5 wt.% relative to stucco.

15 2. Process according to claim 1 wherein the gypsum slurry comprises 100 parts of stucco and between 60 and 90 parts of water.

3. Process according to anyone of claims 1 to 2 wherein the alkyl hydrogen polysiloxane is polymethylhydrogensiloxane (PHMS).

20 4. Process according to anyone of claims 1 to 3 wherein the amount of PHMS is between 0.05 and 2 wt.%, most preferably between 0.1 and 1 wt. % relative to stucco.

5. Process according to anyone of claims 1 to 4 wherein the cured fiber cement waste is autoclaved.

6. Process according to anyone of claims 1 to 5 wherein the stucco comprises more than 70 wt% of β calcium sulphate hemihydrate, preferably more than 80%, most preferably more than 90 wt% relative to the stucco weight.

25 6. Process according to anyone of claims 1 to 5 wherein the pH of the slurry is between 9 and 11.5 said pH being measured according to the testing method ME-A5-005.

7. Process according to anyone of claims 1 to 6 wherein the fiber cement powder has a d50 below 100 μm , preferably below 50, most preferably below 40 μm , the particle sizes being measured using Laser Diffraction Spectrometry (LOS).

30 8. Process according to anyone of claims 1 to 7 wherein the stucco does not comprise fly ash nor added clay.

9. Plasterboard obtained by the process of anyone of claim 1 to 8.

10. Use of cured fiber cement with PHMS to provide water resistant plasterboard having a total water absorption according to the Norm EN520-2009 below 5%.

ABSTRACT

PROCESS FOR PRODUCING WATER RESISTANT PLASTERBOARDS WITH FIBER CEMENT POWDER

- 5 Process for producing water resistant plasterboard using the combination of polyhydrogenomethylsiloxane and the waste of fiber cement board.

Fig. 1

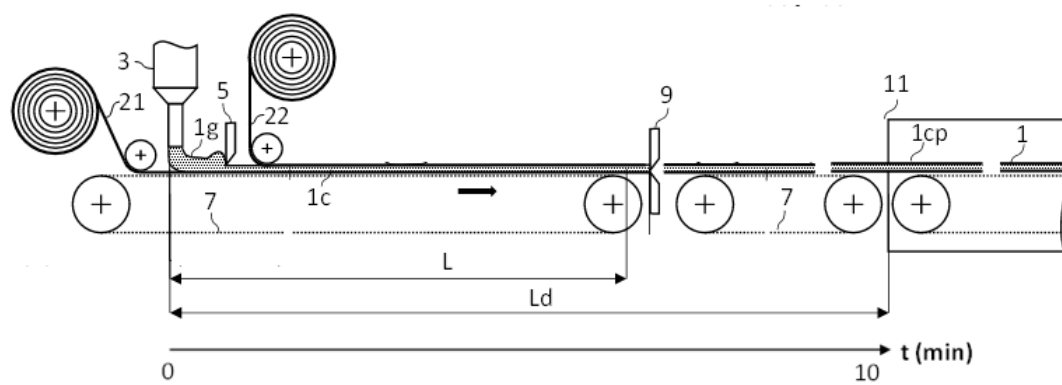


Fig.1

PATENT COOPERATION TREATY

From the INTERNATIONAL SEARCHING AUTHORITY

PCT

To

ETEX SERVICES NV
Kuermansstraat 1
1880 Kapelle-op-den-Bos
BELGIQUE

NOTIFICATION OF TRANSMITTAL OF
THE INTERNATIONAL SEARCH REPORT AND
THE WRITTEN OPINION OF THE INTERNATIONAL
SEARCHING AUTHORITY, OR THE DECLARATION

(PCT Rule 44 1)

Date of mailing
(day/month/year)

21 April 2023 (21-04-2023)

Applicant's or agent's file reference
GY2114WO

FOR FURTHER ACTION See paragraphs 1 and 4 below

International application No
PCT/EP2022/087559

International filing date
(day/month/year)
22 December 2022 (22-12-2022)

Applicant

ETEX BUILDING PERFORMANCE INTERNATIONAL SAS

- 1 ☒ The applicant is hereby notified that the international search report and the written opinion of the International Searching Authority have been established and are transmitted herewith

Filing of amendments and statement under Article 19:

The applicant is entitled, if he so wishes, to amend the claims of the international application (see Rule 46)

When? The time limit for filing such amendments is normally two months from the date of transmittal of the international search report

How? Directly to the International Bureau preferably through ePCT, or on paper to
The International Bureau of WIPO, 34 chemin des Colombettes, 1211 Geneva 20, Switzerland

For more detailed instructions, see the *PCT Applicant's Guide*, International Phase, paragraphs 9 004 - 9 011

- 2 ☐ The applicant is hereby notified that no international search report will be established and that the declaration under Article 17(2)(a) to that effect and the written opinion of the International Searching Authority are transmitted herewith

- 3 ☐ **With regard to any protest** against payment of (an) additional fee(s) under Rule 40 2, the applicant is notified that

☐ the protest together with the decision thereon has been transmitted to the International Bureau together with any request to forward the texts of both the protest and the decision thereon to the designated Offices

☐ no decision has been made yet on the protest, the applicant will be notified as soon as a decision is made

4 **Reminders**

The applicant may **submit comments on an informal basis on the written opinion of the International Searching Authority** to the International Bureau. These comments will be made available to the public after international publication. The International Bureau will send a copy of such comments to all designated Offices unless an international preliminary examination report has been or is to be established.

Shortly after the expiration of **18 months from the priority date**, the international application will be published by the International Bureau. If the applicant wishes to avoid or postpone publication, a notice of withdrawal of the international application, or of the priority claim, must reach the International Bureau before the completion of the technical preparations for international publication (Rules 90bis 1 and 90bis 3).

Within **19 months** from the priority date, but only in respect of some designated Offices, a demand for international preliminary examination must be filed if the applicant wishes to postpone the entry into the national phase **until 30 months** from the priority date (in some Offices even later), otherwise, the applicant must, **within 20 months** from the priority date, perform the prescribed acts for **entry into the national phase** before those designated Offices. In respect of other designated Offices, the time limit of **30 months** (or later) will apply even if no demand is filed within 19 months. For details about the applicable time limits, Office by Office, see www.wipo.int/pct/en/texts/time_limits.html and the *PCT Applicant's Guide*, National Chapters.

Within **22 months from the priority date**, the applicant may request that a **supplementary international search** be carried out by a different International Searching Authority that offers this service (Rule 45bis 1). The procedure for requesting supplementary international search is described in the *PCT Applicant's Guide*, International Phase, paragraphs 8 006-8 032.

Name and mailing address of the International Searching Authority



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PATENT COOPERATION TREATY

PCT

INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference GY2114WO	FOR FURTHER ACTION see Form PCT/ISA/220 as well as, where applicable, item 5 below	
International application No PCT/EP2022/087559	International filing date (<i>day/month/year</i>) 22 December 2022 (22-12-2022)	(Earliest) Priority Date (<i>day/month/year</i>) 24 December 2021 (24-12-2021)
Applicant ETEX BUILDING PERFORMANCE INTERNATIONAL SAS		

This international search report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This international search report consists of a total of 4 sheets



It is also accompanied by a copy of each prior art document cited in this report

1 Basis of the report

a With regard to the **language**, the international search was carried out on the basis of



the international application in the language in which it was filed



a translation of the international application into _____, which is the language of a translation furnished for the purposes of international search (Rules 12 3(a) and 23 1(b))

b ☐

This international search report has been established taking into account the **rectification of an obvious mistake** authorized by or notified to this Authority under Rule 91 (Rule 43 6bis(a))

c ☐

With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, see Box No. I

2 ☐

Certain claims were found unsearchable (See Box No. II)

3 ☐

Unity of invention is lacking (see Box No. III)

4 With regard to the **title**,



the text is approved as submitted by the applicant



the text has been established by this Authority to read as follows

5 With regard to the **abstract**,



the text is approved as submitted by the applicant



the text has been established, according to Rule 38 2, by this Authority as it appears in Box No. IV. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority

6 With regard to the **drawings**,

a the figure of the **drawings** to be published with the abstract is Figure No. 1



as suggested by the applicant



as selected by this Authority, because the applicant failed to suggest a figure



as selected by this Authority, because this figure better characterizes the invention

b ☐

none of the figures is to be published with the abstract

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2022/087559

A CLASSIFICATION OF SUBJECT MATTER
INV. C04B28/14 C04B18/16 C04B24/42
ADD. C04B111/27

According to International Patent Classification (IPC) or to both national classification and IPC

B FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
C04B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal, WPI Data

C DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No
Y	JP 3 284756 B2 (MITSUBISHI CHEM CORP; YOSHINO GYPSUM CO) 20 May 2002 (2002-05-20)	1-10
A	paragraphs [0004], [0013], [0017]; claims; examples	11
Y	EP 3 305 742 A1 (ETEX SERVICES NV) 11 April 2018 (2018-04-11)	1-10
A	paragraphs [0006], [0008], [0035], [0037], [0042], [0043]; figures 1,2	11
A	US 5 154 874 A (KOSLOWSKI THOMAS [DE]) 13 October 1992 (1992-10-13) claims; figure	1-11
A	DE 297 05 438 U1 (FELS WERKE GMBH [DE]) 26 June 1997 (1997-06-26) the whole document	1-11
-/-		

☒ Further documents are listed in the continuation of Box C

☒ See patent family annex

* Special categories of cited documents

"A" document defining the general state of the art which is not considered to be of particular relevance
 "E" earlier application or patent but published on or after the international filing date
 "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
 "O" document referring to an oral disclosure, use, exhibition or other means
 "P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance, the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance, the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search

14 April 2023

Date of mailing of the international search report

21/04/2023

Name and mailing address of the ISA/
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Authorized officer

Theodoridou, K

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2022/087559

C(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No
A	MÜLLER A ET AL: "Material utilization of fibre cement waste", ZKG INTERNATIONAL - ZEMENT - KALK - GIPS INTERNATIONAL, BAUVERLAG BV GMBH, DE, vol. 64, no. 3, 1 March 2011 (2011-03-01), pages 60-72, XP001561555, ISSN: 0949-0205 the whole document -----	1-11

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/EP2022/087559

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
JP 3284756	B2	20-05-2002	JP 3284756 B2	20-05-2002
			JP H07330410 A	19-12-1995

EP 3305742	A1	11-04-2018	AU 2017339075 A1	14-03-2019
			BR 112019005707 A2	09-07-2019
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DE 29705438	U1	26-06-1997	NONE	

Information on Search Strategy - Pilot phase (see OJ 2015, A86)

The type of information contained in this sheet may change during the pilot for improving the usefulness of this new service

Application Number

PCT/EP2022/087559

TITLE: PROCESS FOR PRODUCING WATER RESISTANT PLASTERBOARDS WITH FIBER CEMENT POWDER

APPLICANT: ETEX BUILDING PERFORMANCE INTERNATIONAL SAS

IPC CLASSIFICATION: C04B28/14, C04B18/16, C04B24/42, C04B111/27

EXAMINER: Theodoridou, K

CONSULTED DATABASES: WPI, NPL

CLASSIFICATION SYMBOLS DEFINING EXTENT OF THE SEARCH:

IPC:

CPC: 1- C04B28/14, C04B18/16, C04B20/008, C04B24/42, C04B40/00, C04B28/14/LOW, C04B2111/0062, C04B2111/27, C04B18/16, C04B24/42

FI/F-TERMS:

KEYWORDS OR OTHER ELEMENTS FEATURING THE INVENTION:

JP3284756B2 20020520

EPO-THOMSON

SUBJECT OF THE INVENTION

INDUSTRIAL APPLICATION

[0001] This invention relates to the water-repellent|hydrophobic plaster board which has the unprecedented outstanding performance.

In more detail, it is related with the water-repellent|hydrophobic plaster board in which water resistance and fire resistance were improved.

PRIOR ART

[0002] The present condition is that water resistance has a problem and the use range is limited by the characteristic that a plaster|gypsum has the plaster-board products using a plaster|gypsum, in spite of the remarkable characteristics, such as fire resistance, soundproofing, an intensity|strength, and a low price

In order to supplement the fault of such a plaster|gypsum, the method (US Patent 3455719) to add dimethyl polysiloxane to a plaster|gypsum and to improve water-repellent property, the method (Unexamined-Japanese-Patent No. 56-54263) of mix|blending the alkyl siliconate, slaked lime, or quicklime of an alkali metal with a plaster|gypsum, etc. are proposed,

However,

Since containing organic substance would increase when sufficient water-repellent property is not acquired or water-repellent property is satisfied in these methods, there existed a fault by which fire resistance is impaired

Moreover, even if water-repellent property was good, the fault that it could not manufacture to a plaster board bad also had adhesion|attachment of a board base paper and a plaster|gypsum.

PROBLEM TO BE SOLVED BY THE INVENTION

[0003] It aims at providing the plaster board which this invention does not impair the fire resistance which a plaster|gypsum has, and has sufficient water-repellent property.

MEANS TO SOLVE THE PROBLEM

- [0004] When the present inventors earnestly examine per elimination method of this trouble, the usage-amount of a methyl-hydrogen-polysiloxane shows water-repellent property also for a small quantity by using together a methyl-hydrogen-polysiloxane and a specific compound, Fire resistance was not impaired, either, but it found out that adhesion|attachment with a board base paper was also favorable, and resulted in this invention.
- [0005] That is, the summary of this invention consists of the plaster|gypsum composition and board base paper containing a methyl-hydrogen-polysiloxane, and at least 1 sort(s) chosen from a cement, a calcium silicate hydrate, magnesium oxide, and magnesium hydroxide and a plaster|gypsum, It exists in the water-repellent|hydrophobic plaster board characterized by the above-mentioned. Hereafter, this invention is demonstrated in detail.
- [0006] The polysiloxane used for this invention is a methyl-hydrogen-polysiloxane which has following Structural formula.
- [0007] [Image]
- [0008] Content of a methyl-hydrogen-polysiloxane is 0.2-5 weight part with respect to 100 weight part (hemihydrate gypsum conversion) of plaster|gypsums, Preferably it is 0.2-1 weight part When less than 0.2 weight part, sufficient water-repellent property will not be acquired. When more than 5 weight part, it will become disadvantageous by a fire-resistant point. In said general formula, n shows the number of 1-100
- [0009] Even if it uses it as it is, you may use a methyl-hydrogen-polysiloxane as an emulsion formed by emulsifying a methyl-hydrogen-polysiloxane in water. Since a methyl-hydrogen-polysiloxane can be uniformly add-mixed in a gypsum slurry when it is used as an emulsion, it is preferable. There is no restriction|limiting in particular in the emulsifier used in order to set it as the emulsion formed by emulsifying a methyl-hydrogen-polysiloxane in water, For example, a nonionic emulsifier, an anionic emulsifier, polyvinyl alcohol etc are mentioned.

- [0010] As a nonionic emulsifier, they are glycerol monostearate, glycerol monooleate, sorbitan mono-palmitate, sorbitan monostearate, a sorbitan tri stearate, and sorbitan monooleate, Sorbitan trioleate, a polyoxyethylene sorbitan monolaurate, polyoxyethylene sorbitan mono-palmitate, polyoxyethylene sorbitan monostearate, a polyoxyethylene sorbitan tri stearate, Polyoxyethylene sorbitan monooleate, polyoxyethylene sorbitan trioleate, polyoxyethylene lauryl ether, Polyoxyethylene cetyl ether polyoxyethylene stearyl ether, polyoxyethylene higher alcohol ether, polyoxyethylene octylphenyl ether, a polyoxyethylene nonylphenyl ether, etc. are mentioned.
- [0011] A sodium lauryl sulfate, a sodium dodecyl benzenesulfonate, an alkyl naphthalene specific sulfonate, the dialkyl sodium sulfosuccinate, the mixed fatty-acid soda soap, a stearic-acid soda soap, The sodium salt of an oleic-acid potash soap, higher alcohol sodium sulfate, and (beta)-naphthalene sulfonic-acid formalin condensate etc are mentioned as an anionic emulsifier
- [0012] As polyvinyl alcohol, polyvinyl alcohol with a saponification degree of 75 mol% or more is preferable, and 76-90 mol% polyvinyl alcohol is preferable especially. Especially these polyvinyl alcohol has [the thing of three to 50 centipoise] normally the 4-% aqueous solution viscosity preferable [moreover,] in 20 degreeC one to 80 centipoise. The usage-amount of these emulsifiers is 0.5 to 10 weight% normally with respect to a methyl-hydrogen-polysiloxane, Preferably it is 0.7 to 5 weight% When less than 0.5 weight%, a stable emulsion will not be obtained, but mechanical stability will also worsen, and when more than 10 weight%, sufficient water-repellent property will not be acquired.
- [0013] The addition amount of a cement, a calcium silicate hydrate, magnesium oxide, and magnesium hydroxide is 0.1 weight part or more with respect to 100 weight part (hemihydrate gypsum conversion) of plaster|gypsums, Preferably it is 0.2-1 weight part When less than 0.1 weight part, sufficient water-repellent property will not be acquired When there is no performance top problem even if it puts in abundantly, but a cement is put in abundantly, the condensation|coagulation of a plaster|gypsum hastens and there is a problem in workability|operativity.

- [0014]** A cement, a calcium silicate hydrate, magnesium oxide, and magnesium hydroxide may be used individually, and may mix and use 2 or more types
 An ordinary portland cement, high early strength Portland cement, an alumina cement, the low alkali cement etc. can be used as a cement.
 Powder, such as a tobermorite and a xonotlite, can be used as a calcium silicate hydrate.
 An effect does not have a thing which is not a hydrate like a wollastonite even if it is a calcium-silicate compound.
- [0015]** By using together a methyl-hydrogen-polysiloxane, a cement, a calcium silicate hydrate, magnesium oxide, or magnesium hydroxide, water-repellent property is begun and expresses
 The water-repellent property of a plaster|gypsum expresses, even if a cement etc. use slaked lime etc. instead of, but when slaked lime is used, the adhesiveness of a plaster|gypsum and a board base paper cannot become a plaster board bad.
- [0016]** Both (beta)-hemihydrate gypsum industrially used abundantly as a plaster|gypsum the (alpha)-hemihydrate gypsum and 2 type|mold calcium sulfate anhydrite can be used.
 The plaster|gypsum composition containing a methyl-hydrogen-polysiloxane, and at least 1 sort(s) chosen from a cement, a calcium silicate hydrate, magnesium oxide, and magnesium hydroxide and a plaster|gypsum,
 You may use together lightweight aggregates, such as perlite and a vermiculite, various foaming agents, the water reducing agent for plaster|gypsums, plaster|gypsum condensation|coagulation speed|rate regulator, a reinforced fiber, etc if needed.
- [0017]** In order to manufacture the plaster board of this invention, it reaches with at least 1 sort(s) chosen from the plaster|gypsum of a predetermined amount, the methylo hydrogen polysiloxane, a cement, a calcium silicate hydrate, magnesium oxide, and magnesium hydroxide, water is mixed, and it is set as a slurry,
 Or at least 1 sort(s) chosen from a methyl-hydrogen-polysiloxane, a cement, a calcium silicate hydrate, magnesium oxide, and magnesium hydroxide is added and mixed in arbitrary orders in the water slurry of a plaster|gypsum, and it is set as a slurry,
 It shape|molds by pouring in this slurry between board base papers in accordance with a conventional method, without using an adhesive agent between a plaster|gypsum composition and a board base paper,
 It dries as needed after hardening.

EXAMPLES

[0018] Next, an Example and a comparative example demonstrate this invention.

In addition, in the following examples, "part" and "%" mean a "weight part" and "weight%", respectively.

Example 1

The emulsion which emulsifies 99 parts of methyl-hydrogen-polysiloxanes (KF-99 made from a Shinetu Chemical), 1 part of polyvinyl alcohol (GL-05 made from synthetic combination [Japanese]) of 85% of saponification degree, and 135 parts of water in a homomixer, and contains a methyl-hydrogen-polysiloxane 42% was obtained.

[0019] 0.6 part, 1 part of cement, and 80 parts of water are added at 100 parts of (beta) hemihydrate gypsum (Yoshino Gypsum cherry tree mark A class|grade) by making the emulsion of said methyl-hydrogen-polysiloxane into a methyl-hydrogen-polysiloxane, and it is set as a slurry, This is poured in between plaster-board base papers, and it is set as a plaster board, It dried at 70 degreeC for 2 5 hours

The plaster|gypsum after drying and adhesion|attachment of a plaster-board base paper are enough, and peeling was not recognized.

Specific gravity of the obtained plaster board was 0.86.

The water absorption (Aw2, Aw24) after 2 hours and 24-hour immersion was measured for this board in the water of 20 degreeC+/- 1 degrees C.

[0020] EQUATION 1

A result is shown to table|surface-1

[0021] Example 2

95 parts of methyl-hydrogen-polysiloxanes (KF-99 made from a Shinetu Chemical), 5 parts of nonionic emulsifier polyoxyethylene nonylphenyl ethers (Kao emulgen 910) and water A part is emulsified in a homomixer,

The emulsion which contains a methyl-hydrogen-polysiloxane 45% was obtained.

[0022] 0.6 part, 1 part of cement, and 80 parts of water are added at 100 parts of (beta) hemihydrate gypsum (Yoshino Gypsum cherry tree mark A class|grade) by making the emulsion of said methyl-hydrogen-polysiloxane into a methyl-hydrogen-polysiloxane, and it is set as a slurry, This is poured in between plaster-board base papers, and it is set as a plaster board, It dried at 70 degreeC for 2.5 hours
The plaster|gypsum after drying and adhesion|attachment of a plaster-board base paper are enough, and peeling was not recognized
Specific gravity of the obtained plaster board was 0.75.
The water absorption (Aw2, Aw24) of this board is shown to table|surface-1.

[0023] Example 3
0.6 part of methyl-hydrogen-polysiloxane, 1 part of cement, and 80 parts of water are added to 100 parts of (beta) hemihydrate gypsum (Yoshino Gypsum cherry tree mark A class|grade), and it is set as a slurry,
This is poured in between plaster-board base papers, and it is set as a plaster board,
It dried at 70 degreeC for 2.5 hours.
The plaster|gypsum after drying and adhesion|attachment of a plaster-board base paper are enough, and peeling was not recognized
Specific gravity of the obtained plaster board was 0.88.
The water absorption (Aw2, Aw24) of this board is shown to table|surface-1.

[0024] Example 4
The plaster board was obtained like Example 1 except having made addition amount of the cement into 0.2 part.
The plaster|gypsum after drying and adhesion|attachment of a plaster-board base paper are enough, and peeling was not recognized.
Specific gravity of the obtained plaster board was 0.88.
The water absorption (Aw2, Aw24) of this board is shown to table|surface-1.

[0025] Example 5
The plaster board was obtained like Example 1 except having made addition amount of 0.3 part and a cement into 0.4 part for the addition amount of the methyl-hydrogen-polysiloxane.
The plaster|gypsum after drying and adhesion|attachment of a plaster-board base paper are enough, and peeling was not recognized
Specific gravity of the obtained plaster board was 0.87
The water absorption (Aw2, Aw24) of this board is shown to table|surface-1.

[0026] Example 6

The plaster board was obtained like Example 1 except having made into 1 part the powder of the calcium silicate hydrate which has a xonotlite as a main component instead of a cement

The plaster|gypsum after drying and adhesion|attachment of a plaster-board base paper are enough, and peeling was not recognized.

Specific gravity of the obtained plaster board was 0.87

The water absorption (Aw2, Aw24) of this board is shown to table|surface-1

[0027] Example 7

The plaster board was obtained like Example 1 except having made into 0.4 part the powder of the calcium silicate hydrate which has a xonotlite as a main component instead of a cement

The plaster|gypsum after drying and adhesion|attachment of a plaster-board base paper are enough, and peeling was not recognized

Specific gravity of the obtained plaster board was 0.88

The water absorption (Aw2, Aw24) of this board is shown to table|surface-1

[0028] Example 8

1 part is added for the oil of a methyl-hydrogen-polysiloxane, 1 part and 80 parts of water are added for magnesium hydroxide to 100 parts of (beta) hemihydrate gypsum (Yoshino Gypsum cherry tree mark A class|grade), and it is set as a slurry,

This is poured in between plaster-board base papers, and it is set as a plaster board,

It dried at 70 degreeC for 2.5 hours.

The plaster|gypsum after drying and adhesion|attachment of a plaster-board base paper are enough, and peeling was not recognized

Specific gravity of the obtained plaster board was 0.88

The water absorption (Aw2, Aw24) of this board is shown to table|surface-1

[0029] Example 9

The plaster board was obtained like Example 1 except having made magnesium hydroxide into 0.5 part for the addition amount of the methyl-hydrogen-polysiloxane instead of 0.5 part and a cement.

The plaster|gypsum after drying and adhesion|attachment of a plaster-board base paper are enough, and peeling was not recognized.

Specific gravity of the obtained plaster board was 0.86

The water absorption (Aw2, Aw24) of this board is shown to table|surface-1.

[0030] Example 10

In Example 1, the methyl-hydrogen-polysiloxane was made into 0.1 part, the cement was made into 1 part, and the plaster board was obtained like Example 1.

The plaster|gypsum after drying and adhesion|attachment of a plaster-board base paper are enough, and peeling was not recognized.

Specific gravity of the obtained plaster|gypsum molded object was 0.86.

The water absorption (Aw2, Aw24) of this board is shown to table|surface-1

[0031] Comparative example 1

80 parts of water was added to 100 parts of (beta) hemihydrate gypsum (Yoshino Gypsum cherry tree mark A class|grade), and it was set as the gypsum slurry, and this was poured in between plaster-board base papers, and it was set as the plaster board, and dried at 70 degreeC for 2 5 hours

The plaster|gypsum after drying and adhesion|attachment of a plaster-board base paper are enough, and peeling was not recognized.

Specific gravity of the obtained plaster board was 0.89.

The water absorption (Aw2, Aw24) of this board is shown to table|surface-1.

[0032] Comparative example 2

1 part and 80 parts of water are added by making into a methyl-hydrogen-polysiloxane the emulsion manufactured in Example 1 to 100 parts of (beta) hemihydrate gypsum (Yoshino Gypsum cherry tree mark A class|grade), and it is set as a gypsum slurry,

This is poured in between plaster-board base papers, and it is set as a plaster board, It dried at 70 degreeC for 2.5 hours.

The plaster|gypsum after drying and adhesion|attachment of a plaster-board base paper are enough, and peeling was not recognized.

Specific gravity of the obtained plaster|gypsum molded object was 0.91.

The water absorption (Aw2, Aw24) of this board is shown to table|surface-1.

[0033] Comparative example 3

The plaster board was obtained like Example 1 except 1 part of slaked lime having added instead of the cement.

There are not a plaster|gypsum after drying and adhesion|attachment of a plaster-board base paper, and the upper and lower sides of the plaster-board base paper had peeled.

Specific gravity of the obtained plaster|gypsum molded object was 0.86.

The water absorption (Aw2, Aw24) of this board is shown to table|surface-1.

[0034] Comparative example 4

The plaster board was obtained like Example 1 except having added 0.8 part of slaked lime for the methyl-hydrogen-polysiloxane instead of 1 part and a cement.

There are not a plaster|gypsum after drying and adhesion|attachment of a plaster-board base paper, and the upper and lower sides of the plaster-board base paper had peeled.

Specific gravity of the obtained plaster|gypsum molded object was 0.68.

The water absorption (Aw2, Aw24) of this board is shown to table|surface-1.

[0035] Comparative example 5

The plaster board was obtained like Example 1 except 1 part of calcium carbonate having added instead of the cement.

The plaster|gypsum after drying and adhesion|attachment of a plaster-board base paper are enough, and peeling was not recognized

Specific gravity of the obtained plaster|gypsum molded object was 0.86

The water absorption (Aw2, Aw24) of this board is shown to table|surface-1

[0036] Comparative example 6

The plaster board was obtained like Example 5 except having added 1 part of aluminium hydroxide instead of the cement.

The plaster|gypsum after drying and adhesion|attachment of a plaster-board base paper are enough, and peeling was not recognized

Specific gravity of the obtained plaster|gypsum molded object was 0.86

The water absorption (Aw2, Aw24) of this board is shown to table|surface-1.

[0037] Comparative example 7

1 part is added for dimethyl polysiloxane (SH-200 made from the Toray Dow Corning silicon|silicone), 1 part and 80 parts of water are added for a cement to 100 parts of (beta) hemihydrate gypsum (Yoshino Gypsum cherry tree mark A class|grade), and it is set as a slurry, This is poured in between plaster-board base papers, and it is set as a plaster board, It dried at 70 degreeC for 2.5 hours.

The plaster|gypsum after drying and adhesion|attachment of a plaster-board base paper are enough, and peeling was not recognized.

Specific gravity of the obtained plaster board was 0.88

The water absorption after 2-hour immersion of this plaster board is 47.8 %, and did not show water-repellent property at all.

[0038] Comparative example 8

80 parts of dimethyl polysiloxane (SH-200 made from the Toray Dow Corning silicon|silicone), and 20 parts of methyl-hydrogen-polysiloxanes (KF-99 made from a Shinetsu Chemical) 5 parts of emulsifier polyoxyethylene nonylphenyl ethers (Kao emulgen 910) and water made from a nonion. The emulsion which emulsifies in a homomixer and contains a polysiloxane compound 45% was manufactured.

[0039] 1 part and 80 parts of water are added by using said emulsion as a polysiloxane compound at 100 parts of (beta) hemihydrate gypsum (Yoshino Gypsum cherry tree mark A class|grade), it is set as a slurry, this is poured in between plaster-board base papers, and it is set as a plaster board, It dried at 70 degreeC for 2.5 hours

The plaster|gypsum after drying and adhesion|attachment of a plaster-board base paper are enough, and peeling was not recognized.

Specific gravity of the obtained plaster board was 0.86.

The water absorption after 2-hour immersion of this plaster board is 48.8 %, and did not show water-repellent property at all.

[0040] [Image]**ADVANTAGE OF THE INVENTION****[0041]** Compared with what uses the conventional water repellent, addition amount decreases the plaster board of this invention, it has higher water resistance, and the adhesive force of a plaster|gypsum and a plaster-board base paper is fully maintained.

It consists of the plaster|gypsum composition and board base paper containing a methyl-hydrogen-polysiloxane, and at least 1 sort(s) chosen from a calcium silicate hydrate, magnesium oxide, and magnesium hydroxide and a plaster|gypsum

The water-repellent|hydrophobic plaster board characterized by the above-mentioned

A plaster|gypsum composition contains 0.1-10 weight part of at least 1 sort(s) chosen from 0.2-5 weight part of methyl-hydrogen-polysiloxanes, a calcium silicate hydrate, magnesium oxide, and magnesium hydroxide with respect to 100 weight part of plaster|gypsums

The water-repellent|hydrophobic plaster board of Claim 1 characterized by the above-mentioned.

A plaster|gypsum composition contains 0.2-1 weight part of at least 1 sort(s) chosen from 0.2-1 weight part, a calcium silicate hydrate, magnesium oxide, and magnesium hydroxide in a methyl-hydrogen-polysiloxane with respect to 100 weight part of plaster|gypsums

The water-repellent|hydrophobic plaster board of Claim 1 characterized by the above-mentioned.

The plaster|gypsum composition which add-mixed and water-slurry-ized a plaster|gypsum, a methyl-hydrogen-polysiloxane, and at least 1 sort(s) chosen from a calcium silicate hydrate, magnesium oxide, and magnesium hydroxide is poured in between board base papers, and shaping|molding hardening is carried out.

The manufacturing method of the water-repellent|hydrophobic plaster board characterized by the above-mentioned

The plaster|gypsum composition add-mixed and water-slurry-ized is used as an emulsion formed by emulsifying a methyl-hydrogen-polysiloxane in water

The manufacturing method of the water-repellent|hydrophobic plaster board of Claim 4 characterized by the above-mentioned.

The emulsion of a methyl-hydrogen-polysiloxane uses polyvinyl alcohol as an emulsifier.

The manufacturing method of the water-repellent|hydrophobic plaster board of Claim 5 characterized by the above-mentioned.

The plaster|gypsum composition which add-mixed 0.1-10 weight part of at least 1 sort(s) chosen from 0.2-5 weight part of methyl-hydrogen-polysiloxanes, a calcium silicate hydrate, magnesium oxide, and magnesium hydroxide with respect to 100 weight part of plaster|gypsums, and water-slurry-ized it is used.

The manufacturing method of the water-repellent|hydrophobic plaster board in any one of Claim 4 thru| or 6 characterized by the above-mentioned.

The plaster|gypsum composition which add-mixed 0.1-1 weight part of at least 1 sort(s) chosen from 0.2-1 weight part of methyl-hydrogen-polysiloxanes, a calcium silicate hydrate, magnesium oxide, and magnesium hydroxide with respect to 100 weight part of plaster|gypsums, and water-slurry-ized it is used

The manufacturing method of the water-repellent|hydrophobic plaster board in any one of Claim 4 thru| or 6 characterized by the above-mentioned.

PATENT COOPERATION TREATY

From the
INTERNATIONAL SEARCHING AUTHORITY

PCT

see form PCT/ISA/220

WRITTEN OPINION OF THE INTERNATIONAL SEARCHING AUTHORITY (PCT Rule 43bis.1)

Date of mailing
(day/month/year) see form PCT/ISA/210 (second sheet)

Applicant's or agent's file reference
see form PCT/ISA/220

FOR FURTHER ACTION
See paragraph 2 below

International application No
PCT/EP2022/087559

International filing date (day/month/year)
22 12 2022

Priority date (day/month/year)
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International Patent Classification (IPC) or both national classification and IPC
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Applicant
ETEX BUILDING PERFORMANCE INTERNATIONAL SAS

1 This opinion contains indications relating to the following items:

- ☒ Box No I Basis of the opinion
- ☐ Box No II Priority
- ☐ Box No III Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- ☐ Box No IV Lack of unity of invention
- ☒ Box No V Reasoned statement under Rule 43bis 1(a)(i) with regard to novelty, inventive step and industrial applicability, citations and explanations supporting such statement
- ☐ Box No VI Certain documents cited
- ☒ Box No VII Certain defects in the international application
- ☐ Box No VIII Certain observations on the international application

2 FURTHER ACTION

If a demand for international preliminary examination is made, this opinion will usually be considered to be a written opinion of the International Preliminary Examining Authority ("IPEA") except that this does not apply where the applicant chooses an Authority other than this one to be the IPEA and the chosen IPEA has notified the International Bureau under Rule 66 1bis(b) that written opinions of this International Searching Authority will not be so considered

If this opinion is, as provided above, considered to be a written opinion of the IPEA, the applicant is invited to submit to the IPEA a written reply together, where appropriate, with amendments, before the expiration of 3 months from the date of mailing of Form PCT/ISA/220 or before the expiration of 22 months from the priority date, whichever expires later

For further options, see Form PCT/ISA/220

Name and mailing address of the ISA



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Date of completion of
this opinion

see form
PCT/ISA/210

Authorized Officer

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Box No. I Basis of the opinion

- 1 With regard to the **language**, this opinion has been established on the basis of:
- ☒ the international application in the language in which it was filed
 - ☐ a translation of the international application into , which is the language of a translation furnished for the purposes of international search (Rules 12 3(a) and 23 1 (b))
- 2 ☐ This opinion has been established taking into account the **rectification of an obvious mistake** authorized by or notified to this Authority under Rule 91 (Rule 43bis 1(a))
- 3 ☐ With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, this opinion has been established on the basis of a sequence listing
- a ☐ forming part of the international application as filed
 - b ☐ furnished subsequent to the international filing date for the purposes of international search (Rule 13ter 1(a)),
 - ☐ accompanied by a statement to the effect that the sequence listing does not go beyond the disclosure in the international application as filed
- 4 ☐ With regard to any nucleotide and/or amino acid sequence disclosed in the international application, this opinion has been established to the extent that a meaningful opinion could be formed without a WIPO Standard ST 26 compliant sequence listing
- 5 Additional comments

Box No. V Reasoned statement under Rule 43bis.1(a)(i) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1 Statement

Novelty (N)	Yes	Claims	<u>1-11</u>
	No	Claims	
Inventive step (IS)	Yes	Claims	<u>11</u>
	No	Claims	<u>1-10</u>
Industrial applicability (IA)	Yes	Claims	<u>1-11</u>
	No	Claims	

2 Citations and explanations

see separate sheet

Box No. VII Certain defects in the international application

The following defects in the form or contents of the international application have been noted

see separate sheet

Re Item VII

Certain defects in the international application

The application does not comply with the requirements of Rule 6.1(b), because there are two claims numbered "6". For the purpose of this written opinion it is assumed that the claim numbering is as corrected (second claim 6 being considered as claim 7 and all further claims also renumbered to a total number of 11 claims)

Re Item V

Reasoned statement with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

Reference is made to the following documents, the numbering will be adhered to in the rest of the procedure

- D1 JP 3 284756 B2 (MITSUBISHI CHEM CORP; YOSHINO GYPSUM CO) 20 May 2002 (2002-05-20)
- D2 EP 3 305 742 A1 (ETEX SERVICES NV) 11 April 2018 (2018-04-11)
- D3 US 5 154 874 A (KOSLOWSKI THOMAS [DE]) 13 October 1992 (1992-10-13)
- D4 DE 297 05 438 U1 (FELS WERKE GMBH [DE]) 26 June 1997 (1997-06-26)
- D5 MÜLLER A ET AL: "Material utilization of fibre cement waste", ZKG INTERNATIONAL - ZEMENT - KALK - GIPS INTERNATIONAL, BAUVERLAG BV GMBH, DE, vol 64, no. 3, 1 March 2011 (2011-03-01), pages 60-72, XP001561555, ISSN: 0949-0205

1 Inventive step

The present application does not meet the criteria of Article 33(1) PCT, because the subject-matter of claims 1-10 does not involve an inventive step in the sense of Article 33(3) PCT.

- 1.1 Document D1 discloses a water-repellent gypsum wallboard. A gypsum slurry is prepared comprising methylhydrogen polysiloxane, a calcium silicate hydrate and magnesium oxide. The slurry is poured between two paper sheets in a way conventional to making plasterboards ([004],[013],[017], examples, claims).

The subject matter of claim 1 therefore differs from this known method and plasterboard, in that it includes not just a calcium silicate hydrate but a cured fiber cement powder.

The technical effect deriving from this difference is that the amount of hydrogen produced (from the reaction of cement with the silicone) is reduced and the water repellency of the board is increased. The production method is thus safer (less H₂ gas released) and of better quality (no destruction of bond between paper and gypsum core due to the released gas) ([011]-[013] of the description).

The problem to be solved by the present invention may therefore be regarded as how to increase the water repellency of a gypsum board.

- 1.2 The solution proposed in claim 1 of the present application cannot be considered to involve an inventive step (Article 33(3) PCT) because the use of cured fiber cement powder to increase the water repellency of cement boards as well as plaster boards has been described in document D2:

Document D2 discloses a method for producing an air-cured fiber cement product, comprising the following steps [006],[008],[035],[037],[042],[043]:

providing cured fiber cement powder in an amount of preferably 5 to 15wt% based on the dry mass of the composition; [006] mentions a preferred embodiment comprising less than 5 wt% cured fiber cement powder.

providing a aqueous fiber cement slurry,

forming into a sheet.

The term cement includes various cement types but also plaster [025].

Consequently using cured fiber cement powder to improve water repellency of a plaster board is known from document D2. The skilled person would therefore regard it as a normal option to include this feature in the method described in document D1 in order to solve the problem posed.

- 1.3 The subject matter of claims 3,4,5,6,8 (see Fig 1,2 of D2),9,10 is also known from documents D1 and D2. Document D1 uses 100% beta calcium sulfate hemihydrate in examples 1-3.

The selection of the amount of water to be used (claim 2) is considered within the routine procedures of the person skilled in the art

The subject matter of claim 7 is considered as the direct result of the composition of the slurry. This feature cannot thus contribute to a claim that involves an inventive step on its own.

- 1.4 The subject matter of claim 11 is not directly derivable from the combination of documents D1 and D2 and it therefore appears to involve an Inventive step.