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(54) Title: SOUND-ABSORBING, RESISTANT PANELS AND PROCESS FOR MAKING SAME

(57) Abstract: The present invention relates to sound- absorbing panels and to a process for making the same, as well as to a composition useful for making the abovementioned sound- absorbing panels.

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SOUND-ABSORBING, RESISTANT PANELS
AND PROCESS FOR MAKING SAME

5 The present invention relates to sound-absorbing panels and to a process for making the same, as well as to a composition useful for making the abovementioned sound-absorbing panels.

10 Compositions of set hydraulic binders have been increasingly used in combination with other materials to form composite structures. These structures typically comprise layers of differing thicknesses of compositions of set hydraulic binders and a wide variety of fibrous
15 materials. When the fibrous materials are formed into a sheet material and placed on the outer surface of the set hydraulic binder composition it is referred to as a facer. When the fibrous material surrounds the composition of set hydraulic binder, the interior element is referred to as a
20 core.

 Panels (or boards), generally comprise of two sheets of a facer material having a certain tensile strength, like paper, covering a core based on a set hydraulic binder, notably gypsum plaster. Other types of panels do not
25 necessarily have a facer in particular those used for acoustic insulation which are moulded into shaped articles.

 In both cases, it is desirable to lighten the core by for example incorporating air into the slurry used for making the core. The incorporated air in the core could
30 appear in the form of bubbles. Increasing the amount of air until percolation is known to improve the acoustic absorption properties of the boards, but also to deteriorate the mechanical resistance.

 Therefore, it is difficult to produce a board having
35 both sound-absorbing and mechanical satisfactory characteristics.

 Consequently, there is a need for boards, and a method of producing boards, in particular gypsum boards, which

have satisfactory properties both in terms of acoustics and mechanics.

There is thus still a need for boards based on a set hydraulic binder which are both sound absorbing and resistant to compression.

SUMMARY OF THE INVENTION

The invention also provides panels and slurries comprising the said composition.

It is a first object of the invention to provide a composition comprising at least

- a set hydraulic binder; and
- a foaming agent

and having

- a porosity ≥ 0.55 ;
- a flow resistivity between 10000 and 200000 N.s.m⁻⁴;
- a tortuosity between 1.2 and 3.4;
- a viscous characteristic length between 10 μm and 60 μm ;
- a thermal characteristic length between 60 μm and 1000 μm .

and having

- a compressive strength of at least 0.30 MPa as measured by modified ASTM C472.

The invention also relates to a sound-absorbing panel comprising a core, wherein said core comprises at least a set hydraulic binder and a glycoside compound.

The invention also relates to a process of making a sound-absorbing panel, comprising the steps of:

- a) pre-mixing the set hydraulic binder with water;
- b) adding a glycoside compound;
- c) injecting air into the obtained slurry of the step b) and mixing.
- d) casting the aerated slurry;
- e) setting the cast slurry.

And finally, the invention relates to an assembly comprising at least the panel according to the invention, a coating and a fixing system.

5 The present invention enables one to overcome the inconveniences cited-above. In particular the invention provides boards based on a set hydraulic binder and being both sound absorbing and resistant to compression.

According to particular embodiments, the invention also has the following advantageous features:

- 10 - Use could be made of foaming agent derived from renewable raw materials (for example namely glucoside derived for example from corn or potato starch and natural fatty alcohols derived for example from coconut or palm kernel oil);
- 15 - The good sound absorbing performance of the objects according to the invention make them useful for acoustic application;
- Besides the compressive strength in the core of the panels according to the invention results in an improved handling. The compressive strength is also important during transportation and mounting. The facer covering the core of panels (if present) provides most of the flexural strength as long as the core does not collapse. The high compressive strength of the acoustic core of the invention provides panels that have an improved rigidity during handling and that are more resistant when supported on conventional screws.
- 20
- 25
- 30 - Surprisingly, the composition of the invention does not enhance mould growth on the surface even after inoculation. Thus, the invention substantially avoids the specific addition of fungicides, which are often expensive and which may present health concerns.
- 35 - The compressive strength in the core of the panels according to the invention results in an improved resistance to surface imperfections. In particular, the acoustic ceiling application is frequently

illuminated by low angle light. Impressions in the face panels create unsightly shadows in the ceiling that are unsatisfactory for architects and interior design specialists. Such defects may be reduced owing to the invention.

By the expression "sound-absorbing panel", it must be understood an acoustically absorbent porous panel. That means a panel able to dissipate the sound energy inside the porous structure of the panel.

DESCRIPTION OF EMBODIMENTS

The invention will now be described in more details without limitation in the following description.

In a first embodiment, the invention is a composition comprising at least

- a set hydraulic binder; and
- a foaming agent

and having

- a porosity ≥ 0.55 ;
- a flow resistivity between 10000 and 200000 N.s.m⁻⁴;
- a tortuosity between 1.2 and 3.4;
- a viscous characteristic length between 10 μm and 60 μm ;
- a thermal characteristic length between 60 μm and 1000 μm .

and having

- a compressive strength of at least 0.30 MPa as measured by modified ASTM C472.

In a second embodiment, the invention is a slurry comprising the above cited composition.

Ingredients used in the invention

The composition or the slurry according to the present invention comprises a set hydraulic binder. The set

hydraulic binder is a material that will set with water. Preferably, it is a hydratable calcined gypsum, referred to as plaster, stucco, calcium sulfate hemi hydrate, or calcium sulfate semi-hydrate (or alternatively anhydrite).

5 The source of the gypsum before it is calcined by any method known to one skilled in the art may be natural or synthetic production of gypsum, synthetic production of gypsum is preferred.

Hydratable calcined gypsum material is generally a
10 fine-grained powder with a median particle size in the range of 5 to 100 μm . Specific embodiments of the invention are particularly designed for quick-setting hydraulic binder, having a setting time of less than 30 min, preferably less than 20 min, more preferably less than 10
15 min. One of the most preferred set hydraulic binder for use in the present composition of the invention is hydratable flue gas desulfurization (FGD) plaster.

The advantages of FGD gypsum plaster are among others higher purity, more uniform and finer particles size,
20 lighter color and no abrasive particles. Higher purity results in more set hydraulic binder per unit weight for generally improved strength over natural gypsum plaster of lower purity. Uniform fine particles are more uniformly and completely calcined for a more uniform setting time.
25 Lighter color is more aesthetically pleasing. The lack of abrasive particles in the gypsum plaster causes less wear on conveying equipment and mixer parts.

The slurry according to the present invention comprises water. The final water-to-plaster (W/P) ratio in
30 the slurry prior to setting is preferably comprised between 0.3 and 0.9, more preferably between 0.45 and 0.75 and most preferably between 0.55 and 0.65.

The composition or the slurry of the present invention comprises a foaming agent.

35 By foaming agent according to the invention, it must be understood any suitable compound or any surfactant able to foam a composition comprising a hydraulic binder.

Suitable foaming agents according to the invention are preferably non-ionic foaming agents having a hydrophilic / lipophilic balance, namely HLB, from 5 to 18, preferably from 7 to 15, more preferably from 9 to 13.

5 Suitable foaming agents according to the invention are preferably alkylpolysaccharides.

Alkylpolysaccharides as foaming agents suitable for the invention, are those having a hydrophobic group containing from 8 to 22 carbon atoms, preferably from about
10 10 to about 16 carbon atoms, most preferably from 12 to 14 carbon atoms, and a polysaccharide hydrophilic group containing from 1 to 10, saccharide units (e.g., galactoside, glucoside, fructoside, glucosyl, fructosyl and/or galactosyl units).

15 Preferably, alkylpolysaccharides as foaming agents suitable for the invention, are alkylpolyglucosides having from 4 to 22, preferably from 4 to 16, more preferably from 8 to 12 carbon atoms.

The composition or the slurry of the present invention
20 comprises, as preferred foaming agent, glycoside compound (which is also meant to cover mixtures of several glycoside compounds).

The term glycoside compound according to the invention means any chemical compound comprising a sugar part
25 (glycone) bound to a non-sugar part (aglycone). The glycone may comprise one or more sugar units. If more than one unit is present on average, the glycoside compound may be termed polyglycoside. The glycoside compound may be a (poly)fructoside compound (if the glycone is based on
30 fructose), a (poly)galactoside compound (if the glycone is based on galactose), a (poly)glucoronide compound (if the glycone is based on glucuronic acid) etc. Preferably, the glycoside compound is a glucoside or polyglucoside, i.e. a glycoside based on glucose.

35 The glycoside compound may also be an alkylpolysaccharide having the general formula $RO(R^1O)_tZ_x$ as defined in US 4,565,647 on col.1, 1.36-55, and more particularly an alkylpolysaccharide as described in

Foaming agent sold by Huntsman, like alkylethersulfates, in particular the MILLIFOAM family, are also particularly well-suited, notably Millifoam C. The composition or the slurry of the present invention comprises preferably from 0.1 to 2.0 wt% (% by weight of the weight of the hydraulic binder) of alkylethersulfates (which is also meant to cover mixtures of several alkylethersulfates compounds), particularly from 0.25 to 0.8 wt% alkylethersulfates, more particularly from 0.30 to

0.60 wt% alkylethersulfates, and preferably from 0.35 to 0.50 wt% alkylethersulfates (% by weight of the weight of the set hydraulic binder).

Advantageously, the glycoside compound mentioned above
5 is the sole foaming agent or surfactant used in the composition or slurry according to the invention. In other terms, the composition or the slurry preferably does not substantially comprise any other foaming agent or surfactant. In one embodiment, the glycoside compound
10 represents more than 90% of any surfactant composition, advantageously more than 95%, by weight. In one embodiment, there is no other surfactant or foaming agent. It is to be understood that the absence of any other foaming agent or surfactant also covers the case where there are less than
15 0.01 wt% (preferably less than 0.001 wt%) of other foaming agent(s) or surfactant(s) in the composition.

The composition or the slurry of the present invention comprises preferably from 0.1 to 2.0 wt% (% by weight of the weight of the hydraulic binder) of the glycoside
20 compound (which is also meant to cover mixtures of several glycoside compounds), particularly from 0.25 to 0.8 wt% glycoside compound, more particularly from 0.30 to 0.60 wt% glycoside compound, and preferably from 0.35 to 0.50 wt% glycoside compound (% by weight of the weight of the set
25 hydraulic binder).

The composition or the slurry of the present invention may also comprise aggregates and/or fillers and / or others inorganic materials. Examples of fillers are fumed silica, fly ash, blast furnace slag, micro-silica and fine
30 limestone. Examples of likely aggregates are lightweight vermiculite, silica, limestone sand, perlite, micro-spheres, and expanded shale.

Additives influencing the behavior of the composition or slurry like retarders/accelerators pairs are
35 advantageously used in the present invention. An example of a retarder/accelerator pair is conventional protein plaster retarder/ball milled accelerator (BMA).

It should be understood that any additive classically used in the art could also be used in the instant composition or slurry, in particular additives like thickeners or viscosity modifiers or fluidizers but not limited to them. The range of additives is very wide as will be appreciated by the skilled person.

Resins for the improvement of the mechanical and/or aesthetic properties, known in the art can be added to the composition or slurry of the present invention. Examples of resins beneficial alone or in combination are: polyacrylic, polyvinylalcohol, fluoropolymer and mixtures thereof. These types of resins can be combined in copolymers or other combinations, e.g. as styrene-butadiene copolymers, styrene-acrylate copolymers, vinyl-acetate-ethylene copolymers and acrylate copolymers.

The composition or slurry of the present invention may also comprise a thickener (also named stabilizing agent). The thickener may be effective by increasing the viscosity of the water in the matrix, or by stabilizing the bubble formation by the foaming agent. One skilled in the art will appreciate that polyvinyl alcohol is a suitable bubble stabilizing agent.

The composition or slurry of the present invention may also comprise a viscosity modifier like for example a water-soluble viscosity modifier. Examples are polymers (cellulosic, polyalcohol, polyurethane, polyester, polyether, polyacrylic, co- and terpolymers thereof), clay (modified/natural), fumed silica, hydrophobically-modified or surface-modified additives.

The composition or slurry of the present invention may comprise a fluidizer which is usefully incorporated into the gypsum slurry in order to minimize the water to calcined gypsum ratio. A fluidizer (also referred to as a water-reducing agent or a plasticizer) may be added to the aqueous gypsum slurry (e.g., via a pump) to increase the flow of the slurry. Some examples of such fluidizer are carboxylate compounds such as polycarboxylate ethers. Preferred additives are polycarboxylate ethers or the like.

The composition or slurry of the present invention may comprise a blocking agent which is preferably incorporated into the slurry in order to stop the setting of the set hydraulic binder with water. A blocking agent is also referred to as a calcium sequestering agent and may also serve as a water-reducing agent in the aqueous gypsum slurry to increase the flow of the slurry. Any suitable product with a calcium sequestering functionality can be used. Blocking agents are typically used with and unblocking agent in a pair. Examples of typical blocking unblocking pairs are sodium polyacrylate/aluminum sulfate and sodium phosphonate/zinc sulfate.

The present invention may be practiced in the absence of fibers. In the absence of fibers means that the amount could be less than 0.01% by weight (% by weight of the weight of the set hydraulic binder), preferably less than 0.001% (only unintended impurities) and preferably no fiber will be present at all. A fiber is any fiber typically used in the art. "In the absence of fibers" does not exclude the presence of cellulosic material, especially originating from reclaim material, as is typically used in the instant field.

Advantageously, in a particular embodiment, the composition or the slurry of the present invention does not comprise fibers.

The composition of the present invention may have a volume of air voids incorporated into the composition in order to absorb sound. The quantity of air voids may come from two sources, water voids created when water of convenience is dried out of the composition and air voids that may be created when air is injected into the slurry.

The water voids may be smaller than the air voids and may have a slight contribution to sound absorption at higher frequencies and at higher water-to-plaster ratio.

The air voids may be larger than the water voids, and may tend to be interconnected which may allow the composition to absorb sound.

According to the invention, the term « porosity » means the total of the porosity induced by the presence of air voids and the porosity resulting from the evaporation of water (water voids). Consequently the porosity values
5 take into account the air voids and the water voids.

According to another aspect of the invention, the composition of the present invention may have

- a porosity ≥ 0.55 ;
- a flow resistivity between 10000 and 200000
10 N.s.m⁻⁴;
- a tortuosity between 1.2 and 3.4;
- a viscous characteristic length between 10 μm
and 60 μm ;
- a thermal characteristic length between 60 μm
15 and 1000 μm .

Preferably, according to the composition of the present invention

- the porosity is ≥ 0.70 ;
- the flow resistivity is between 50000 and
20 180000 N.s.m⁻⁴;
- the tortuosity is between 1.3 and 2.5;
- the viscous characteristic length is between
15 μm and 50 μm ;
- the thermal characteristic length is between
25 70 μm and 500 μm .

More preferably, according to the composition of the present invention

- the porosity is ≥ 0.76 ;
- the flow resistivity is between 100000 and
30 160000 N.s.m⁻⁴;
- the tortuosity is between 1.4 and 2.3;
- the viscous characteristic length is between
15 μm and 40 μm ;
- the thermal characteristic length is between
35 80 μm and 300 μm .

These 5 parameters correspond to those which have been well described by the model of Biot-Johnson-Allard in the

following book: J.F.Allard, Propagation of Sound in Porous Media, Elsevier Applied Science, 1993):

- **porosity** is as defined above; the porosity can be easily measured by picnometry;

5 - **flow resistivity** means the velocity of air volume displacement which is the material quantity that flows through an area in a time period. The resistance to air flow is equal to the pressure loss measured between the two sides of the porous sample when it is crossed by a constant
10 laminar air flow. Flow resistivity is thus equal to the ratio of the air pressure over the flow rate times the sample surface divided by the sample thickness.

- **tortuosity** means the complexity of the internal structure of the material;

15 - **thermal characteristic length** characterizes the heat exchange between the air and the rigid frame. This is a measure of the effective size of the pores involved in the heat exchange. For gypsum cores, this is directly related to the bubble size.

20 - **viscous characteristic length** characterizes the viscous interaction of the air with the stiff frame. This is a measure of the effective size of the pores involved in the viscous interaction. For gypsum cores, this is directly related to the size of the interconnections between the air
25 voids.

Several methods exist for the measurement of characteristic lengths and tortuosity. The most accurate way to achieve measurements consists in an analytic inversion of acoustic models. References of scientific
30 articles describing these methods are:

- X. Olny, R. Panneton, and J. Tran-van, An indirect acoustical method for determining intrinsic parameters of porous materials. In Poromechanics II, Actes de la 2nde conférence de BIOT, 2002.

35 According to another aspect of the invention, the composition of the present invention may have a density comprised between 250 and 450 kg/m³, particularly between 320 and 420 kg/m³, more particularly between 340 and 380

kg/m³, preferably 350 and 360 kg/m³, more equal to 360 kg/m³.

The composition of the present invention has sound-absorbing properties.

5 In another embodiment, the invention is a slurry comprising the composition of the present invention.

The composition or the slurry of the present invention may be used to make the core of sound-absorbing panels, in particular gypsum boards. Thus, the invention provides a
10 panel comprising a core, the said core comprising a composition according to the invention. This panel is a sound-absorbing panel, having a sound absorption average (SAA) of at least 0.3, preferably at least 0.5, most
15 preferably at least 0.6 as measured by modified ASTM E1050 - 98 (see example 3 below). In particular, this panel has a compressive strength of at least 0.30 MPa as measured by modified ASTM C472. More particularly, this panel has a compressive strength of at least 0.40 MPa, preferably of at
20 least 0.50 MPa, and more preferably of at least 0.60 MPa.

The invention provides also in a third embodiment a sound-absorbing panel which comprises a core wherein the core comprises at least a set hydraulic binder, and a
25 glycoside compound.

The invention provides also as an improvement a sound-absorbing panel or board which comprises a core wherein the core comprises at least a set hydraulic binder, water, and a glycoside compound.

30 Preferably, the set hydraulic binder of sound-absorbing panel of the invention is set gypsum plaster.

More preferably, the set gypsum plaster of sound-absorbing panel of the invention is a set flue gas desulphurization gypsum plaster (FGD).

35 As mentioned above for the composition and the slurry, the sound absorbing panels comprise a glycoside compound, in particular an alkylpolyglycoside or alkylpolyglucoside.

In a preferred embodiment, the sound absorbing panels comprise a C4-C16 alkylpolyglycoside or C4-C16 alkylpolyglucoside.

Preferably, the sound-absorbing panel comprises from
5 0.1 to 2.0 wt% glycoside compound, particularly from 0.25 to 0.8 wt% glycoside compound, more particularly from 0.30 to 0.60 wt% glycoside compound, and preferably from 0.35 to 0.50 wt% glycoside compound (% by weight of the weight of the set hydraulic binder).

10 By sound absorbing panels, as described above according to any embodiments of the invention, is meant in particular boards having a sound absorption average (SAA) of at least 0.3, preferably at least 0.5, most preferably at least 0.6 as measured by modified ASTM E1050 - 98 (see
15 example 3 below).

In particular, the sound-absorbing panel of the invention has a compressive strength of at least 0.30 MPa as measured by modified ASTM C472. More particularly, the sound-absorbing panel of the invention has a compressive
20 strength of at least 0.35 MPa, preferably of at least 0.40 MPa, and more preferably of at least 0.50 MPa.

In a particular embodiment, the sound-absorbing panel of the invention has a compressive strength of at least 0.30 MPa as measured by modified ASTM C472, and a sound
25 absorption average of at least 0.65 as measured by modified ASTM E1050 - 98.

In a particular embodiment, the sound-absorbing panel of the invention has a compressive strength of at least 0.40 MPa as measured by modified ASTM C472, and a sound
30 absorption average of at least 0.65 as measured by modified ASTM E1050 - 98.

In a particular embodiment, the sound-absorbing panel of the invention has a compressive strength of at least 0.50 MPa as measured by modified ASTM C472, and a sound
35 absorption average of at least 0.65 as measured by modified ASTM E1050 - 98.

The composition of the invention could be used to produce the core of sound-absorbing panels. The thus obtained core of the sound-absorbing panels could be made either with the composition of the invention or partially
5 with the composition of the invention and the rest of the core is made with conventional plaster core composition.

In another embodiment the sound-absorbing panels comprising the composition of the invention could include paper, reinforced joint or abutted joint. By paper it means
10 paper used to form the facing or backing of the panel.

Another object of the invention is an acoustic insulated system comprising at least a composition according to the invention. The advantages of this system
15 is to improve insulating systems without the necessary use of any absorbent material inside the cavity.

By the term "system", it is understood according to the invention an assembly of at least one panel, at least
20 one frame and at least one fixing component.

The acoustic insulated system may comprise a panel comprising the composition according to the invention.

According to a first embodiment, the acoustic insulated system of the invention is a partition comprising
25 at least one frame, at least one any known gypsum board and at least one panel comprising the composition according to the invention.

The system according to the first embodiment provides a improvement of at least 5dB expressed in R_{w+c} .

30 The R_{w+c} is the Weighted Sound Reduction Index, showing the ability of a wall or other building structure to provide sound insulation. The R_{w+c} has been measured in an acoustic testing laboratory according to the following method *EN ISO 140 part3 - Standard Test Method for*
35 *Laboratory Measurement of Airborne Sound Transmission Loss of Building Partitions and Elements* and calculated in an acoustic testing laboratory according *EN ISO 717 Part1 -*

Rating of sound insulation in buildings and in building elements.

The figure 2 provides one embodiment of the acoustic insulated system according to the invention, where C represents a stud, A represents any known gypsum board and B represents a panel comprising the composition according to the invention.

According to a second embodiment, the acoustic insulated system of the invention is a partition comprising at least one frame, at least one board, the said board having at least a layer comprising the composition according to the invention.

According to a third embodiment, the acoustic insulated system of the invention is a partition comprising at least one stud, at least one board, the said board having at least a layer comprising the composition according to the invention and the said board being airtight on one side.

A board being airtight suitable according to the invention is board having a skimming coat. Such skimming coat reduces the porosity of the surface of the board and finishes the surface rendering. The said skimming coat can be made for instance with a standard readymix compound applied at 300gr/m², like for instance the P852 from Lafarge.

By the expression "airtight", it is understood according to the invention having a flow resistivity higher than 200 001 N.s.m⁻⁴.

The frame suitable for the acoustic insulated system of the invention are all frame or studs available for partitions.

The known gypsum boards suitable for the acoustic insulated system of the invention are all known gypsum board available for partitions.

Another object of the invention is an assembly comprising at least the panel according to the invention, a coating and a fixing system.

5 Process for making compositions and panels

The invention provides a process of making a sound-absorbing panel, comprising the steps of:

- 10 a) pre-mixing a set hydraulic binder with water;
- b) adding a glycoside compound;
- c) injecting air into the obtained slurry of step
 b) and mixing;
- d) casting the aerated slurry;
- e) setting the cast slurry.

15 According to the process of the invention, the set hydraulic binder could be gypsum plaster.

 The glycoside compound incorporated during the step b) could be an alkylpolyglycoside or alkylpolyglucoside, in particular a C4-C16 alkylpolyglycoside or a C4-C16
20 alkylpolyglucoside.

 The process of the invention may comprise further step(s). In particular the step a) could comprise a step of pre-mixing the set hydraulic binder with water and a blocking agent. The process of the invention could further
25 comprise a step of introducing an unblocking agent into the aerated slurry. Alternatively, the step a) could comprise pre-mixing the set hydraulic binder with water and a retarding agent. And also, the step d) (casting of the aerated slurry) of the process according to the invention
30 could comprise a step of depositing the aerated slurry onto a facer, said process also comprising:

- covering the deposited slurry with another facer,
- forming a ribbon from the deposited slurry,
35 and
- cutting the ribbon into panels.

In particular, the process of the invention may comprise a step of depositing the aerated slurry into moulds.

By casting, it must be understood according to the
5 invention, to give shape, by any means.

The invention provides two embodiments for the manufacture of compositions and boards, but is not limited thereto.

10 According to an embodiment hereafter referred to as the semi-batch embodiment, the panel of the present invention may be produced by mixing together, in a primary mixer, gypsum plaster, water, a fluidizer, an accelerator and a blocking agent (preferably dispersed in part or all
15 of the gauging water). A blocked and unfoamed gypsum slurry is obtained. The slurry is continuously pumped to a secondary mixer. Prior to entry in the secondary mixer, the foaming agent (glycoside compound solution) is continuously injected into the slurry. The secondary mixer namely an air
20 mixer (e.g. a rotating, vibrating agitator) achieves aeration and foaming of the slurry. The size of the air bubbles produced is adjusted by the speed of agitation of the secondary mixer. After the secondary mixer, the foamed slurry is passed to yet a tertiary mixer, where an
25 unblocking agent is added at an inlet. The tertiary mixer achieves mixing of the slurry with the unblocking agent and thus the foamed slurry becomes unblocked (relative to setting). The tertiary mixer can be for example one or more static mixers, optionally with a slow rotating motion. The
30 foamed and unblocked slurry is then deposited on a facer. A second facer may be applied to the exposed surface of the slurry and the edges glued to the first facer. The cross section of the board is adjusted by a forming plate and transported by a conveyor belt until the slurry is set. The
35 continuous board is then cut to length and dried in a hot, forced air wallboard dryer that is known in the art of making gypsum wallboard to provide the final board.

Alternatively, according to an embodiment hereafter referred to as the continuous embodiment, the panel may be produced by mixing together in a primary mixer, gypsum plaster, water, a fluidizer, a retarder and an accelerator
5 dosed in order to retard setting of the plaster. The primary mixer is a continuous mixer. The slurry thus produced is then continuously poured into a tank from which it is pumped to a secondary mixer. The time the slurry resides in the tank is preferably small. The foaming agent
10 (glycoside compound solution) is injected into the slurry prior to entry in the secondary mixer. The secondary mixer is an air mixer similar to the one mentioned in relation with the above semi-batch embodiment, which achieves aeration and foaming of the slurry. The foamed slurry is
15 then directly deposited on a facer without any need for a tertiary mixer and an unblocking agent. The rest of the process is as described above in relation with the semi-batch process.

Preferably, the amount of air injected during the
20 aeration step (according to either the first or second embodiment) is such that it permits to obtain a density of about 310 kg/m^3 for the dried panel.

In either of the two processes of the invention, direct air injection can be used, as is disclosed in
25 document US 2005/0219938, the content thereof being incorporated herein by reference.

The facer materials also named liner or paper, used to produce the panels are those that are used in the art in a conventional manner. The facer may be paper. Alternatively,
30 the facer material may be a non-woven mat, preferably a glass mat or a mat formed of other fibers (e.g. synthetic fibers or a mixture of cellulosic fibers and synthetic fibers). The cementitious slurry may penetrate partly in the facer, fully, or the facer may even be embedded in the
35 cementitious core.

The following steps in the process are those commonly used in the field and easily identified and implemented by the skilled person: covering the slurry with a second

facers, forming a preform (by passing under a conventional wallboard forming plate), allowing the gypsum slurry to set (supported on the conveyor belt), cutting boards from the continuous ribbon of set material, inverting the boards to
5 expose the underside of the boards, drying the boards in a wallboard dryer, optionally coating with a reinforcing binder.

Alternatively, the foamed slurry could be moulded into tiles, as is commonly practiced in the art of acoustic tile
10 manufacturing.

The invention concerns also the product obtainable by the process according to the invention.

15 The figure 1 provides a picture of the composition according to the invention, in particular the microstructure of the gypsum core (SEM pictures).

20 The figure 2 provides one embodiment of the acoustic insulation system according to the invention.

The following examples illustrate without limiting the
25 invention.

30 EXAMPLES

Example 1 - production of panels according to the first embodiment of the process

In a stirred tank (primary mixer), a slurry was
35 prepared using the following ingredients:

- 15 g of Ball milled accelerator (BMA, from the Carpentras factory, France) which comprises 50% gypsum, 40% starch and 10% calcium lignosulfonate ;

This BMA is prepared by co-grinding and the particule size distribution is $D_{10}=3\pm 1\mu\text{m}$, $D_{50}=15\pm 3\mu\text{m}$ and $D_{90}=60\pm 5\mu\text{m}$;

- 9.45 kg of water;
- 5 - 24 g of Coatex TP169 (polyacrylate blocking agent, obtained from Coatex);
- 15 g of Ball milled accelerator (BMA, from the Ottmarsheim factory, France) which comprises gypsum, starch and lignosulfonate;
- 10 - 35.7 g of Optima 100 (phosphonate fluidizer obtained from Chryso);
- 363 g of Vinnapas CEF52W (vinyl acetate resin obtained from Wacker).

The primary slurry thus obtained was pumped in a tube
15 at a flow rate of 1 L/min. A solution of Glucopon 215 CS UP (containing 64% by weight of alkylpolyglycoside surfactant) obtained from Cognis, prepared with 61.1 g of Glucopon diluted in water (400g Glucopon and 600g of water) was continuously injected into the circulation tube of the
20 primary slurry by an injection pump (flow rate: 10 g/min). The mean concentration of the active material contained in the slurry is thus 0.11%.

The primary slurry then entered a Mondomix[®] air mixer, [type of machine: minimondo H1776, capacity 5-50kg/hr
25 distributed by Haas Mondomix (secondary mixer)] rotating at 450 rpm, where air was introduced at a flow rate of 1.5 L/min to 2.5 L/min so that foaming took place.

The foamed slurry was then transported to a tertiary mixer where a solution of aluminum sulfate (unblocking agent) was added and continuously mixed to the foamed
30 slurry. The solution of aluminum sulfate was prepared with 85 g of aluminum sulfate powder, at an active content of 150 g/kg. The injection rate was 27 g/min. The tertiary mixer was a 30-cm long vertical static mixer having a 20-mm
35 diameter, based on a Kenics[®] geometry. At the outlet of the mixer, the slurry was directly deposited on a liner and a second liner was applied on top of the slurry and it was

allowed to set. The distance between the tertiary mixer and the liner was 10 cm.

The liner used was FF 0.55/6 supplied by Johns Manville. This liner is a non-woven glass mat of 8 μm fibers adhered together by 20 g/m^2 of a blend of acrylic and polyvinyl alcohol polymers. A coating of 15 to 30 g/m^2 of an acrylic resin binder was applied to the boards after the dryer.

10 Example 2 - production of panels according to the second embodiment of the process

In a primary mixer, a powder premix was continuously introduced at a flow rate of 1 kg/min . The powder premix was composed of:

- 15 - gypsum hemi-hydrate from the Ottmarsheim factory (France) so that the water to plaster ratio is 0.58; and
 - 1 g Ball milled accelerator (BMA, see above) per kg of gypsum hemihydrate.

20 The following liquid ingredients were also introduced into the mixer:

- 410 g/min of water;
 - 57 g/min of a solution of sodium polyacrylate (Coatex TP1431EXP: fluidizing agent obtained from Coatex), the solution being a $1/10^{\text{th}}$ dilution of the commercial solution, so that the weight concentration in active material is 0.3% relative to the gypsum hemihydrate;
25 - 50 g/min of a blend of natural product with proteins in solution (Plastretard L: retarder supplied by Sicit, Vincenza Chiampo, Italy) containing 6 g per kg of the initial commercial solution;
30 - 50 g/min of a solution of K_2SO_4 obtained from Riedal de Haën containing 100 g per kg of active material.
35 The K_2SO_4 aims at providing acceleration of the final set and hardening.

The W/P ratio was 0.58. A strength reinforcement solution could be added if necessary. When a slump test was performed (with a 50-mm height and a 60-mm width ring), the diameter of the slump was between 205 and 240 mm. The setting time was comprised between 6.5 minutes and 7.5 minutes (based on the knife test), otherwise it could be adjusted by varying the quantity of Plastretard. If the Gilmore test indicated a setting time of less than 9 minutes, K_2SO_4 could be adjusted accordingly.

The slurry thus prepared exited the primary mixer and was continuously transferred to the top of a cylindrical tank having a 20 mm diameter and a 200 mm height. The slurry was then continuously pumped at the bottom of the tank at a flow rate of 1 L/min in a tube so that the amount of material in the tank remained constant. The average passing time in the tank was less than 5 seconds and the residence time distribution was narrow (95% of the slurry leaved the tank less than 10 seconds after entering the tank), as was measured by dyes concentration evolution techniques.

A solution of Glucocon 215 CS UP (containing 64% of alkylpolyglycoside surfactant) obtained from Cognis, prepared with 400 g of Glucocon and 600 g of water, was continuously injected into the circulation tube of the primary slurry by an injection pump (flow rate: 13 g/min). The concentration in active material is thus about 0.3% by weight relatively to the gypsum hemihydrate.

The primary slurry then entered a Mondomix® air mixer (secondary mixer) rotating at 300-500 rpm (400-450 rpm being preferred), where air was introduced at a flow rate of 2.5 L/min so that foaming took place. After the exit of the air mixer, the foamed slurry was deposited on a liner where it was allowed to set after the addition of a second liner on the top. The distance between the air mixer and the liner was less than 10 cm and the direction of the deposition was horizontal.

The liner used was FF 0.55/6 supplied by Johns Manville. This liner is a non-woven glass mat of 8 μm

fibers adhered together by 20 g/m² of a blend of acrylic and polyvinyl alcohol polymers.

Example 3 - Analytical tests

5

Samples of the foamed slurry produced according to examples 1 and 2 were taken at the outlet of the last mixer (i.e. the secondary mixer in the continuous process and the tertiary mixer in the semi-batch process).

10

Some samples were placed in a 20-mm high and 75-mm wide cup where they were allowed to set. These samples were used for the analysis of mechanical performances (compression test). However, it is also possible to make the analysis directly on the boards. Force-displacement

15 curves were plotted and the strength before breaking was measured and converted to a corresponding pressure in MPa.

15

Other samples were placed in a 100-mm high and 45-mm wide cup where they were allowed to set. 2-cm high slices were subsequently extracted and used for the analysis of the acoustic performances of the gypsum core. Acoustics was evaluated by Kundt tube measurements, which allow to vary the frequency of the incident waves crossing a sample perpendicularly. The normal incidence measurements made in the Kundt tube were then converted to an estimation of the

20 the acoustic performances of the gypsum core. Acoustics was evaluated by Kundt tube measurements, which allow to vary the frequency of the incident waves crossing a sample perpendicularly. The normal incidence measurements made in the Kundt tube were then converted to an estimation of the

25 Sound Absorption Average (SAA) as defined in ASTM C-423, by assuming that a), the effects of 200-mm plenum between the material and the ceiling can be taken into account, and b), calculations allows to predict the real diffuse field behavior of the material from the transformation of the

30 normal incidence results. Practical means to achieve this prediction correction can be found for instance in:

25

30

A. London, The determination of reverberant sound absorption coefficients from acoustic impedance measurements, J. Acoust. Soc. Am. 22(2), pp. 263-269, 1950.

35

or in:

L. L. Beranek LL. Noise Reduction. NewYork: McGraw-Hill; 1971 (Chapter 13).

The present method was thus close to ASTM E1050-98 and is therefore termed in the present application "modified ASTM E1050-98".

The estimation of SAA is achieved on small samples when only small amounts of material are available. If the material is produced in larger amounts, SAA can be directly evaluated in an acoustic room (also named reverberating room). More precisely, a floor surface of 3600 mm by 3000 mm, with a metallic frame supported over a 200 mm plenum, was covered with 19.5 mm thick panels according to the invention supported every 600 mm. The joints over the supports were taped with 50 mm wide plastic tape. An omnidirectional loudspeaker was located in one position of the reverberating room to send a continuous spectrum in the frequency band of interest. When the sound emission stops, the sound decay was measured at eight different locations while the sound source was recorded by rotating microphones. The calculation of the equivalent absorption area is calculated from international standard ISO 354 and defined using Sabine formula: $A = X (V / T_r)$ where X is an air temperature and humidity coefficient (0.16 for standard conditions), V is the volume of the room in cubic meters, and T_r is the reverberation time in seconds. The measurement of absorption area is made without (A_1) and then with the specimens in place (A_2). The absorption area of the room in square meters is then $A_1 - A_2$. For each band of frequency, the sound absorption coefficient of the test specimen is calculated. $\alpha = (A_1 - A_2) / S + \alpha_1$ where α_1 is the performance of the liner alone. In the acoustic room, the panels according to the invention are necessarily covered with liners or facers, whereas the small samples are not. It is known by the experts in acoustics that the individual characterization of both the core of the panels

and the liners is sufficient to calculate the acoustic behavior of the panels. Therefore, providing the acoustic characteristics of the liners are known, the SAA measured in the room and the evaluation of the SAA through the application of the "modified ASTM E1050-98" methods applied to the core and to the liner provide very close results. Therefore, SAA be evaluated either way.

The mechanical and acoustical results are presented below in Table 1. Each sample is labeled "C" for continuous process according to example 2). The quantity of air injected and thus the final density of the product is adjusted via the air mixer speed at a constant air flow for the continuous process, and via the air flow at a constant air mixer speed for the semi-batch process.

The compressive strength of the example specimens were measured according to a modified ASTM C472 method. The 75 mm round samples were de-moulded, weighed and the thickness and diameter were measured. The thickness must be at least 20 mm, and the average of 3 diameter measurements was calculated. The samples were dried in a ventilated oven at 45°C to constant weight, and cooled to 25°C in a desiccator. The samples were removed from the desiccator and placed in the center of the compressive strength press platen. The cross head is lowered onto the specimen until the load cell detects the sample and the load cell is zeroed. The force is applied at 4 mm per minute crosshead speed from initial loading through elastic failure and then to plastic failure and force falls. The force and displacement of the crosshead are recorded every 0.01 second until plastic failure of the specimen. The force and crosshead displacement are plotted to determine the force at elastic failure. For each of the samples the force that corresponds to the lowest point of the linear plastic region of the graph is taken as the elastic failure force. The force at elastic failure in Newton is divided by the area of the specimen in square millimeters and reported as compressive strength in MPa.

The porosity, the flow resistivity, the tortuosity, the thermal characteristic length and the viscous characteristic length were measured according to the following teaching:

- 5 - X. Olny, R. Panneton, and J. Tran-van, An indirect acoustical method for determining intrinsic parameters of porous materials. In Poromechanics II, Actes de la 2^{nde} conférence de BIOT, 2002.

- 10 The results demonstrate that the invention achieves good mechanical and acoustical results at the same time.

Table 1 - acoustical and mechanical performances of plaster board samples according to the invention made with the continuous process

Sample No.	C1	C2	C3
Air mixer speed (rpm)	450	400	350
Air flow (L/min)	2.5	2.5	2.5
Compressive strength (MPa)	0.68	0.61	0.49
Sound absorption average	0.79	0.76	0.76
Porosity	0.87	0.89	0.88
Flow resistivity (N.s.m ⁻⁴)	26 100	64 000	67 900
tortuosity	1.64	1.75	1.84
viscous characteristic length (μm)	27	20	22
thermal characteristic length (μm)	231	251	143

CLAIMS

1. A composition comprising at least
- 5 - a set hydraulic binder; and
- a foaming agent
- and having
- a porosity ≥ 0.55 ;
- a flow resistivity between 10000 and 200000
- 10 N.s.m-4;
- a tortuosity between 1.2 and 3.4;
- a viscous characteristic length between 10 μm
- and 60 μm ;
- a thermal characteristic length between 60 μm
- 15 and 1000 μm .
- and having
- a compressive strength of at least 0.30 MPa
- as measured by modified ASTM C472.
- 20 2. The composition of claim 1, wherein the set
- hydraulic binder is set gypsum plaster.
3. The composition of claim 1, wherein the foaming
- agent is a glycoside compound preferably
- 25 alkylpolyglycoside or alkylpolyglucoside.
4. The composition of claim 3, wherein the glycoside
- compound is a C4-C16 alkylpolyglycoside or C4-C16
- alkylpolyglucoside.
- 30 5. The composition of claim 3, wherein the
- composition comprises from 0.1 to 2.0 wt% of the
- glycoside compound.
- 35 6. The composition of claim 3, wherein the glycoside
- compound is the sole foaming agent.
7. The composition of claim 1, wherein

- the porosity is ≥ 0.70 ;
- the flow resistivity is between 50000 and 180000 N.s.m⁻⁴;
- the tortuosity is between 1.3 and 2.5;
- 5 - the viscous characteristic length is between 15 μm and 50 μm ;
- the thermal characteristic length is between 70 μm and 500 μm .

- 10 8. The composition of claim 1, wherein
- the porosity is ≥ 0.76 ;
 - the flow resistivity is between 100000 and 160000 N.s.m⁻⁴;
 - the tortuosity is between 1.4 and 2.3;
 - 15 - the viscous characteristic length is between 15 μm and 40 μm ;
 - the thermal characteristic length is between 80 μm and 300 μm .

- 20 9. A panel comprising a core, wherein said core comprises a composition according to any claims 1 to 8.

- 25 10. The panel of the claim 9, wherein the said panel is a sound-absorbing panel.

- 30 11. A sound-absorbing panel comprising a core, wherein said core comprises at least a set hydraulic binder and a glycoside compound.

12. The sound-absorbing panel of claim 11, wherein the set hydraulic binder is set gypsum plaster.

- 35 13. The sound-absorbing panel of claim 12, wherein the set gypsum plaster is set flue gas desulphurization gypsum plaster.

14. The sound-absorbing panel as claimed in any one of claims 11 to 13, wherein the glycoside compound is an alkylpolyglycoside or alkylpolyglucoside.
- 5 15. The sound-absorbing panel of claim 14 wherein the glycoside compound is GLUCOPON 600 CSUP or GLUCOPON 215 CS UP.
- 10 16. The sound-absorbing panel as claimed in any one of claims 11 to 15, wherein the core comprises from 0.1 to 2.0 wt% glycoside compound.
- 15 17. The sound-absorbing panel as claimed in any one of claims 11 to 16, having a compressive strength of at least 0.30 MPa as measured by modified ASTM C472, and a sound absorption average of at least 0.65 as measured by modified ASTM E1050 - 98.
- 20 18. A process of making a sound-absorbing panel, comprising the steps of:
a) pre-mixing a set hydraulic binder with water;
b) adding a glycoside compound;
c) injecting air into the obtained slurry of the step b) and mixing.
25 d) casting the aerated slurry;
e) setting the cast slurry.
- 30 19. The process as claimed in claim 18, wherein the set hydraulic binder is gypsum plaster.
20. The process as claimed in any of claims 18 to 19, wherein the glycoside compound is an alkylpolyglycoside or alkylpolyglucoside.
- 35 21. The process of claim 20, wherein the glycoside compound is a C4-C16 alkylpolyglycoside or a C4-C16 alkylpolyglucoside.

22. The process of claims 18 to 21, wherein step a) comprises pre-mixing the set hydraulic binder with water and a blocking agent.
- 5 23. The process of claims 18 to 22, wherein the process comprises a step of introducing an unblocking agent into the aerated slurry.
- 10 24. The process of claim 18, wherein step a) comprises pre-mixing the set hydraulic binder with water and a retarding agent.
- 15 25. The process of any one of claims 18 to 24, wherein the casting of the aerated slurry comprises depositing the aerated slurry onto a facer, said process also comprising:
- covering the deposited slurry with another facer,
 - forming a ribbon from the deposited slurry, and
 - 20 - cutting the ribbon into panels.
- 25 26. The process as claimed in of any one of claims 18 to 25 wherein it comprises a step of depositing the aerated slurry into moulds.
- 30 27. An assembly comprising at least the panel according to claims 9 to 17, a coating and a fixing system.
- 35 28. A partition comprising at least one frame, at least one any known gypsum board and at least one panel comprising the composition according to claims 1 to 8.
29. An acoustic insulated system comprising at least a composition according to claims 1 to 8.

INTERNATIONAL SEARCH REPORT

International application No
PCT/IB2008/003757

A. CLASSIFICATION OF SUBJECT MATTER

INV. C04B24/10 C04B24/38 C04B38/10 E04B1/86 G10K11/168
C04B28/14

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 E04B G10K

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 practical, 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.
X	US 2004/231916 A1 (ENGLERT MARK H [US] ET AL) 25 November 2004 (2004-11-25)	1,2,9,10
Y	paragraphs [0010], [0011], [0016], [0017], [0072], [0073] -----	3-8, 11-29
Y	US 4 565 647 A (LLENADO RAMON A [US]) 21 January 1986 (1986-01-21) cited in the application column 8, lines 28-30; claims; examples -----	3-8, 11-29

☐ 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 document 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

31 March 2009

Date of mailing of the international search report

14/04/2009

Name and mailing address of the ISA/

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FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

Continuation of Box II.2

Claims Nos.: 1,7,8,17

Present claims 1, 7, 8 and 17 relate to a product defined (inter alia) by reference to the following unusual parameters: flow resistivity, tortuosity, viscous characteristic length, thermal characteristic length and compressive strength as measured by modified ASTM C472.

The use of these unusual parameters in the present context is considered to lead to a lack of clarity because the claim does not clearly identify the products encompassed by it as the parameters cannot be clearly and reliably determined by indications in the description or by objective procedures which are usual in the art. This makes it impossible to compare the claim to the prior art. As a result, the application does not comply with the requirement of clarity under Article 6 PCT.

Furthermore some of the measurement methods are not clear:

- the thermal characteristic length is measured in reference to a rigid frame (description page 12, Line 15ff) which is, however, not defined in the claim.
- it is not clear how the ASTM C472 has to be modified in order to measure the compressive strength

Doubt arises if these parameters are usual in the art. In fact the application does not contain sufficient information for the skilled person which would enable him to measure the desired parameters (Art. 5 PCT).

Claims 1, 7, 8 and 17 do not meet the requirements of Article 6 PCT in that the matter for which protection is sought is not clearly defined. The claims attempts to define the subject-matter in terms of the result to be achieved, which merely amounts to a statement of the underlying problem, without providing the technical features necessary for achieving this result.

The lack of clarity is to such an extent, that the search was performed taking into consideration the non-compliance in determining the extent of the search of claims 1, 7, 8 and 17.

The search of claims 1, 7, 8 and 17 was restricted to a composition comprising a hydraulic binder and a foaming agent.

The applicant's attention is drawn to the fact that claims relating to inventions in respect of which no international search report has been established need not be the subject of an international preliminary examination (Rule 66.1(e) PCT). The applicant is advised that the EPO policy when acting as an International Preliminary Examining Authority is normally not to carry out a preliminary examination on matter which has not been searched. This is the case irrespective of whether or not the claims are amended following receipt of the search report or during any

FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

Chapter II procedure. If the application proceeds into the regional phase before the EPO, the applicant is reminded that a search may be carried out during examination before the EPO (see EPO Guideline C-VI, 8.2), should the problems which led to the Article 17(2)PCT declaration be overcome.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/IB2008/003757

Box No. II Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:
2. ☒ Claims Nos.: 1,7,8,17
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:
see FURTHER INFORMATION sheet PCT/ISA/210
3. ☐ Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box No. III Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers allsearchable claims.
2. ☐ As all searchable claims could be searched without effort justifying an additional fees, this Authority did not invite payment of additional fees.
3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search reportcovers only those claims for which fees were paid, specifically claims Nos.:
4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

- ☐ The additional search fees were accompanied by the applicant's protest and, where applicable, the payment of a protest fee.
- ☐ The additional search fees were accompanied by the applicant's protest but the applicable protest fee was not paid within the time limit specified in the invitation.
- ☐ No protest accompanied the payment of additional search fees.

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/IB2008/003757

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
US 2004231916	A1	25-11-2004	US 2008156581 A1	03-07-2008
US 4565647	A	21-01-1986	NONE	