



Office de la Propriété
Intellectuelle
du Canada

Un organisme
d'Industrie Canada

Canadian
Intellectual Property
Office

An agency of
Industry Canada

CA 2192724 C 2001/07/31

(11)(21) **2 192 724**

(12) **BREVET CANADIEN
CANADIAN PATENT**

(13) **C**

(22) Date de dépôt/Filing Date: 1996/12/12

(41) Mise à la disp. pub./Open to Public Insp.: 1998/03/13

(45) Date de délivrance/Issue Date: 2001/07/31

(30) Priorité/Priority: 1996/09/13 (08/710,264) US

(51) Cl.Int.⁶/Int.Cl.⁶ C04B 28/14, C04B 14/38

(72) Inventeurs/Inventors:

Mudd, Patrick J., US;
Metz, Lambert J., US;
Winkowski, Daniel A., US;
Finkelstein, Ronald S., US;
Burkard, Edward A., US;
Stav, Elisha, US

(73) Propriétaire/Owner:

NATIONAL GYPSUM PROPERTIES, LLC, US

(74) Agent: OSLER, HOSKIN & HARCOURT LLP

(54) Titre : COMPOSITIONS CIMENTAIRES CONTENANT DU PLATRE ET DES FIBRES ET MATERIAUX FABRIQUES
A PARTIR DE CES COMPOSITIONS CIMENTAIRES

(54) Title: CEMENTITIOUS GYPSUM-CONTAINING FIBER-CONTAINING COMPOSITIONS AND MATERIALS MADE
THEREFROM

(57) Abrégé/Abstract:

A cementitious composition useful for water-resistant construction materials includes about 10 wt.% to about 35 wt.% fiber and about 65 wt.% to about 90 wt.% of a cementitious binder. The binder further includes about 30 wt.% to about 75 wt.% calcium sulfate beta-hemihydrate, about 10 wt.% to about 50 wt.% Portland cement, and about 4 wt.% to about 20 wt.% silica fume.



2192724

CEMENTITIOUS GYPSUM AND FIBER-CONTAINING
COMPOSITIONS AND MATERIALS MADE THEREFROM

ABSTRACT OF THE DISCLOSURE

A cementitious composition useful for water-resistant construction materials includes about 10 wt.% to about 35 wt.% fiber and about 65 wt.% to about 90 wt.% of a cementitious binder. The binder further includes about 30 wt.% to about 75 wt.% calcium sulfate beta-hemihydrate, about 10 wt.% to about 50 wt.% Portland cement, and about 4 wt.% to about 20 wt.% silica fume.

CEMENTITIOUS GYPSUM AND FIBER-CONTAINING
COMPOSITIONS AND MATERIALS MADE THEREFROM

5

BACKGROUND OF THE INVENTION

Field of the Invention

10 The invention relates to cementitious
compositions and in particular to cementitious
construction materials such as fiber boards, roofing
tiles, shingles, and floor underlayment made from a
composition comprising gypsum, Portland cement,
15 silica fume, and fiber.

Description of Related Technology

 Construction materials, such as backer
boards for showers and exterior applications, such
as sheeting and shingles, typically do not contain
20 gypsum because gypsum-containing materials are
usually not water resistant. However, gypsum is a
desirable component in construction materials due to
its rapid cure and early strength characteristics.
Attempts to improve the water-resistance of gypsum
25 boards by mixing Portland cement and gypsum (calcium
sulfate hemihydrate) have met with limited success
because such a mixture can result in the formation
of ettringite, which may cause expansion of the
gypsum/Portland cement product and thus lead to its
30 deterioration. Ettringites are formed when
tricalcium aluminate ($3\text{CaO}\cdot\text{Al}_2\text{O}_3$) in the Portland
cement reacts with sulfate.

 A cementitious composition useful as a
pavement patching compound which contains Portland

cement and alpha gypsum is disclosed in Harris, U.S. Patent No. 4,494,990. The composition also includes a pozzolan source, such as, for example, silica fume, fly ash or blast furnace slag. The Harris
5 patent discloses that the pozzolan blocks the interaction between the tricalcium aluminate and the sulfate from gypsum. The Harris patent discloses mixing a three-component blend of Type I Portland cement, alpha gypsum and silica fume with a fine
10 aggregate to prepare a mortar used to cast mortar cubes for evaluating the strength of the resulting composition.

Ortega et al., U.S. Patent No. 4,661,159 discloses a floor underlayment composition that
15 includes alpha gypsum, beta gypsum, fly ash and Portland cement. The patent also discloses that the floor underlayment material can be used with water and sand or other aggregate to produce a fluid mixture which may be applied to a substrate.
20 Neither the Harris patent nor the Ortega et al. patent discloses fiber-containing compositions.

Sattler et al., U.S. Patent No. 5,030,289 discloses a first group of molded construction parts made from waste paper or cellulose fibers and a
25 binder made from (1) Portland cements, alumina cements, belite cements, or mixtures thereof; and (2) a pozzolan such as amorphous silicic acid, powdered trass, fly ash, or mixtures thereof. Sattler et al. also discloses a second group of
30 molded construction parts made from fiber and a binder of (1) a latently hydraulic component such as blast sand or blast slag; (2) hemihydrate gypsum; and (3) Portland cement. However, the Sattler et al. patent does not disclose combining gypsum with

- 3 -

the cement/pozzolan-containing mixtures used to make the first group of molded construction parts.

SUMMARY OF THE INVENTION

It is an object of the invention to
5 overcome one or more of the problems described above.

According to the invention, a cementitious composition includes about 10 wt.% to about 35 wt.% fiber and about 65 wt.% to about 90 wt.% of a
10 cementitious binder. The binder composition further includes about 30 wt.% to about 75 wt.% calcium sulfate beta-hemihydrate, about 10 wt.% to about 50 wt.% Portland cement, and about 4 wt.% to about 20 wt.% silica fume.

15 The invention further includes construction compositions and materials made from the inventive cementitious composition and methods for making the same.

Other objects and advantages of the
20 invention will be apparent to those skilled in the art from the following detailed description taken in conjunction with the appended claims.

DETAILED DESCRIPTION OF THE INVENTION

According to the invention, a composition
25 for use in construction materials is provided which is particularly useful in areas where water resistance is an important consideration for both interior and exterior applications, such as floor underlayment, fiber board, sheeting, roofing tile
30 and shingles.

Compositions according to the invention include a binder made from about 30 wt.% to about 75 wt.% calcium sulfate beta-hemihydrate (i.e., beta-

gypsum), about 10 wt.% to about 50 wt.% Portland cement, and about 4 wt.% to about 20 wt.% silica fume. Alternatively, the binder may include about 1 wt.% to about 40 wt.% pozzolanic aggregate. About
5 65 wt.% to about 90 wt.% of the binder is then mixed with about 10 wt.% to about 35 wt.% of a fiber component to result in a gypsum- and fiber-containing composition according to the invention.

The beta-gypsum component of the binder of
10 a composition according to the invention is calcium sulfate beta hemihydrate, commonly referred to as stucco. Beta-gypsum is traditionally less expensive than alpha-gypsum. Alpha-hemihydrate powder has a higher apparent density and smaller related surface
15 area than beta-hemihydrate, resulting in a lower water requirement for the same workability and a higher compressive strength of the set material. However, boards made from the inventive composition exhibit more than adequate strength for interior
20 applications such fiberboards and boards used as backer boards, and exterior applications, such as exterior sheeting, roof tiles and shingles.

The Portland cement component of the composition according to the invention may be any of
25 Types I, II, III, IV, or IV (or mixtures thereof) as set forth according to ASTM standards. However, Type III Portland cement is preferred. Type III Portland cement cures faster than Type I and Type II Portland cement and exhibits an early high strength.

30 Blended cements also may be used in compositions according to the invention. Blended cements are blends of Portland cement with one or more pozzolanic materials such as fly ash and blast-furnace slag.

The silica fume component of the binder of a composition according to the invention is an extremely active pozzolan and prevents the formation of ettringite. Silica fume is very fine (particle
5 average diameter of between about 0.1 microns and about 0.3 microns), has a high surface area (between about 20 meter²/gram and about 30 meter²/gram), and is highly amorphous (between about 98 wt.% and about 100 wt.% amorphous SiO₂ (glassy material)).

10 The alternative pozzolanic aggregate or filler component of the binder of a composition according to the invention may be a natural or man-made aggregate that contains a high percentage of amorphous silica. Natural pozzolanic aggregates are
15 of volcanic origin and include trass, pumice, and perlite. Man-made pozzolanic aggregates include fly ash and FILLITETM (hollow silicate spheres produced by Fillite Division of Boliden Intertrade, Inc. Atlanta, Georgia).

20 The fiber component of a composition according to the invention is selected from wood and paper fibers, including recycled waste paper fibers, other ligneous materials such as flax and cotton, and mixtures of such fibers. Wood fiber is a
25 preferred fiber component for a composition according to the invention.

Most preferably, the fiber is obtained from debarked wood which is refined to long thin flakes having a thickness of about 0.008 inches
30 (about 0.2 mm) to about 0.013 inches (about 0.33 mm) and a length of up to about 1.18 inches (about 30 mm). The flaked wood is then milled and screened and possibly further refined using known processes in order to provide fibers or fiber flakes of
35 substantially constant geometry.

If the wood fiber material used in a composition according to the invention is waste paper, such paper must first be processed to remove foreign material such as plastic, dirt and metals.

5 The paper is then further processed by shredding, preferably with a heavy hammermill. The shredded paper is then preferably dry-refined to result in fibers of substantially constant geometry.

The binder for compositions according to

10 the invention preferably includes about 30 wt.% to about 75 wt.% calcium sulfate beta-hemihydrate (about 36 wt.% to about 47 wt.% is preferred for fiberboard), about 10 wt.% to about 50 wt.% Portland cement (about 40 wt.% to about 50 wt.% is

15 preferred), and about 4 wt.% to about 20 wt.% silica fume (about 10 wt.% to about 15 wt.% is preferred for fiberboard).

About 65 wt.% to about 90 wt.% (preferably about 70 wt.% to about 85 wt.%) of the binder is

20 then mixed with about 10 wt.% to about 35 wt.% wood fiber (preferably about 15 wt.% to about 30 wt.% wood fiber) to form a fiberboard.

Most preferably, a binder according to the invention for use in a fiberboard includes about 40

25 wt.% calcium sulfate beta-hemihydrate, about 46 wt.% Portland cement, and about 14 wt.% silica fume.

A construction material, such as a fiberboard according to the invention may be manufactured by the following process:

30 Raw gypsum may be calcined at about 160°C (320°F) to about 175°C (347°F) to form calcium sulfate hemihydrate. The calcined gypsum can be post-ground to a finer particle size if, for example, certain strengths, water requirements, and

35 working properties are desired.

All components of the composition, including gypsum, cement, silica fume, water, wood fiber, and any other additives preferably are weighted batch-wise. Moisture in the wood fiber
5 also is measured.

The gypsum powder is fed to a mixer, such as a large batch or continuous mixer, and blended with Portland cement and silica fume.

In a second mixer, the fiber is mixed with
10 water to allow the fiber/water mixture to loosen. The gypsum/cement/silica fume binder is then added to the fiber/water mixture and intensively mixed with the humid fiber. Although water may be added to the binder/fiber mixture (or to the binder prior
15 to mixing with the fiber), preferably, the water is added to the fiber and then the binder is added to the water/fiber mixture.

Most preferably, the water addition to the fiber and the subsequent binder addition to the
20 wetted fiber are performed with the aid of computer control so that it is possible to add to the fiber the total quantity of water required for the process (i.e. a slight stoichiometric excess amount of water required for hydration), and then vigorously mix the
25 wetted fiber with the binder.

Other ingredients, such as set control additives (e.g. accelerators), water reducing agents, water repellent additives, retarders, and latex or polymer modifiers may be added to the
30 fiber/binder mixture. Some additives may be added to the dry binder mixture prior to mixture with the wet fiber. Preferably, the composition includes about .01 wt.% to about 1.5 wt.% retarder, based upon the total weight of the composition.

The mixed composition is then conveyed directly to a forming machine which spreads an endless mat onto an elongated belt of a continuous press. The mat enters the press on the conveyor belt, is pressed and may be cut into sections, and exits on a conveyor belt in the form of an endless board-ribbon or panel sections. A pressing machine which can be used for this purpose is the Bison-Hydro-Dyn-PressTM (Bison GmbH, Springe, Germany). In such a press, the hydration of the board occurs quickly and may be hastened by warming the board in the press up to an optimal hydration temperature. Preferred processing conditions include pressing at room temperature (about 25°C) at pressures up to about thirty (30) kg/cm² for a press or clamping time of about three (3) to about eight (8) hours.

The board-ribbon (or panel sections) leaving the press has sufficient green strength so that it can be transferred onto a conveyor which will carry the board forward to a cutting station. Hydration may continue as the board-ribbon or panel sections are conveyed to the board cutter. The board-ribbon is then cut or sawed to a desired panel length. If necessary, the panels are then dried to a final moisture content.

Finally the board panels are trimmed and, if desired, split lengthwise to a final dimension. Boards are typically cut into 3 ft. (0.9 meter) x 5 ft. (1.5 meter) sheets, and have a thickness between about 1/2 inch (about 1.3 cm) and about 5/8 inch (about 1.6 cm).

Compositions according to the invention produce building materials which set up quickly, exhibit high strength and durability, and are water resistant. Because compositions according to the

invention set up quickly, building materials made from such compositions can be handled (e.g. sheets can be cut into smaller sheets or boards) much faster than products made from Portland cement and fiber alone. For example, a benefit of a composition and process according to the invention is that a fiberboard made from the inventive process undergoes a total pressing/curing time of under seven hours (and as little as three hours) as compared to the seven to twelve-hour pressing/curing time required for some other Portland cement/fiber board processes. Furthermore, unlike traditional gypsum board, boards or other products made from a composition according to the invention do not require kiln drying, and in fact, kiln drying should be avoided.

EXAMPLE 1

A cementitious composition according to the invention was prepared with the binder components set forth in the amounts stated in Table I below:

TABLE I

	<u>Material</u>	<u>Weight Percent</u>
	Beta-gypsum (Stucco)	40
25	Type III Portland Cement	46
	Silica Fume	14

About 75% by weight of the binder materials identified in Table I were mixed with about 25 wt.% (dry weight) of fiber that had been mixed with water (slight stoichiometric excess). The wetted fiber and binder were vigorously mixed, formed into mats and pressed into sample boards

using a Bison laboratory press (Bison GmbH, Springe, Germany). The pressing conditions included 30 kg/cm² pressure; press temperature of about 25°C; and a press time of three hours.

5 The samples exhibited excellent dry and wet durability even when subjected to a continuous water spray. The final products had an extremely smooth surface.

10 Certain physical properties of boards made according to Example 1 were tested, including percent linear variation (ASTM D 1037), percent water absorption (ASTM D 1037), Mor's 3-Point Loading (ASTM C 947; and nail pull (ASTM C 473). For each of these tests, gypsum/cement/silica fume
15 (GCSF) boards made according to Example 1 were compared to cement fiber boards made of about 82 wt.% Portland cement and about 18 wt.% wood fiber. The press time required for the cement fiber boards ranged between seven to ten hours. The
20 results of the tests are set forth in the following tables:

TABLE II
LINEAR VARIATION (%) (ASTM D 1037)

	70°F/ 50% RH ³ to 90°F/ 90% RH ³		70°F/ 50% RH ³ to 109°F (Bone Dry)	
	Long Direction	Short Direction	Long Direction	Short Direction
GCSF ¹	+0.125	+0.125	-0.155	-0.155
25 Cement ²	+0.177	+0.175	-0.195	-0.192

¹Gypsum/cement/silica fume fiber board.

²Cement fiber board.

³Relative humidity.

TABLE III
WATER ABSORPTION (%) (ASTM D 1037)

	Percent by Weight		Percent Caliper Swell	
	2 Hours	24 Hours	2 Hours	24 Hours
GCSF ¹	4.5	8.4	0.47	1.1
Cement ²	12.4	21.6	0.64	1.1

5

¹Gypsum/cement/silica fume fiber board.²Cement fiber board.

TABLE IV
MOR'S 3 POINT LOADING (DRY) (ASTM C 947)

	MOR ³		PEL ⁴	
	Long Direction	Short Direction	Long Direction	Short Direction
GCSF ¹	2146.5	2297.9	1269.5	1986.2
Cement ²	2123	1928.9	1458.8	1146.2

10

¹Gypsum/cement/silica fume fiber board.²Cement fiber board.

15

³Modules of Rupture (lb/in²).⁴Proportional Elastic Limit (lb/in²).

TABLE V
MOR'S 3 POINT LOADING (WET) (ASTM C 947)

	MOR ³		PEL ⁴	
	Long Direction	Short Direction	Long Direction	Short Direction
GCSF ¹	1141.4	1332	509.2	474.3
Cement ²	1402.7	1385.8	760.7	462.3

20

¹Gypsum/cement/silica fume fiber board.²Cement fiber board.³Modules of Rupture (lb/in²).

25

⁴Proportional Elastic Limit (lb/in²).

TABLE VI
NAIL PULL (ASTM C 473)

	Pounds Force (dry) ³	Pounds Force (wet) ³
GCSF ¹	688	356
Cement ²	615	439

5

¹Gypsum/cement/silica fume fiber board.

²Cement fiber board.

³Testing parameters included:

10 0.400 inch nail head diameter;
 0.121 inch shank diameter; and
 a loading rate of one foot/minute.

15 As shown in the tables above, boards made
according to the invention exhibited comparable or
improved physical properties as compared to cement
fiber boards which did not include gypsum or silica
fume. Furthermore, the boards according to the
invention advantageously took much less time to
process (three hour press time) as compared to the
cement fiber boards (seven to ten hour press time).

20

 The foregoing detailed description is
given for clearness of understanding only, and no
unnecessary limitations should be understood
therefrom, as modifications within the scope of the
invention will be apparent to those skilled in the
art.

25

CLAIMS

1. A cementitious composition comprising:
 - (a) 10 wt.% to 35 wt.% of a fiber component; and
 - (b) 65 wt.% to 90 wt.% of a binder, the binder further comprising:
 - i) 30 wt.% to 75 wt.% calcium sulfate beta-hemihydrate;
 - ii) 10 wt.% to 50 wt.% Portland cement; and
 - iii) 4 wt.% to 20 wt.% silica fume.
2. The composition of claim 1 comprising 15 wt.% to 30 wt.% of the fiber component and 85 wt.% to 70 wt.% binder.
3. The composition of claim 1 wherein the fiber component is selected from the group consisting of wood fibers, paper fibers, and mixtures thereof.
4. The composition of claim 1 wherein the calcium sulfate beta-hemihydrate is 36 wt.% to 47 wt.% of the binder.
5. The composition of claim 1 wherein the Portland cement is 40 wt.% to 50 wt.% of the binder.
6. The composition of claim 1 wherein the silica fume is 10 wt.% to 15 wt.% of the binder.
7. The composition of claim 1 wherein the binder further comprises a retarder.

8. A water resistant construction material prepared by combining 10 wt.% to 35 wt.% fiber, 65 wt.% to 90 wt.% of a binder, and a slight stoichiometric excess of water, the binder further comprising:

(a) 30 wt.% to 75 wt.% calcium sulfate beta-hemihydrate;

(b) 10 wt.% to 50 wt.% Portland cement; and

(c) 4 wt.% to 20 wt.% silica fume.

9. The construction material of claim 8 comprising 15 wt.% to 30 wt.% of the fiber component and 85 wt.% to 70 wt.% binder.

10. The construction material of claim 8 wherein the fiber is selected from the group consisting of wood fibers, paper fibers, and mixtures thereof.

11. The construction material of claim 8 wherein the calcium sulfate beta-hemihydrate is 36 wt.% to 47 wt.% of the binder.

12. The construction material of claim 8 wherein the Portland cement is 40 wt.% to 50 wt.% of the binder.

13. The construction material of claim 8 wherein the silica fume is 10 wt.% to 15 wt.% of the binder.

14. The construction material of claim 8 wherein the composition further comprises a retarder.

15. The construction material of claim 8 wherein said construction material is selected from the group consisting of fiberboard, roofing tile, shingles and exterior sheeting.