CONVENTION APPLICATION FOR A PATENT

We hereby apply for the grant of a Patent for an invention entitled:

WATER AND FIRE RESISTANT BUILDING MATERIAL.

which is described in the accompanying complete specification. This application is a Convention application and is based on the application numbered (1)

860066 and 864501

for a patent or similar protection made in (4) Norway

on 10th January 1986 and 12th November 1986

Our address for service is Messrs. Edwd. Waters & Sons, Patent Attorneys,

50 Queen Street, Melbourne, Victoria, Australia.

DATED this 8th day of January 1987

NORSK PROCO A/S

by

Ian A. Scott

Registered Patent Attorney
COMMONWEALTH OF AUSTRALIA
Patents Act 1952-1969

DECLARATION IN SUPPORT OF A CONVENTION APPLICATION FOR A PATENT OR PATENT OF ADDITION

In support of the Convention Application made by:

Norsk Proco A/S

(hereinafter referred to as the applicant) for a Patent

for an invention entitled: Water and Fire Resistant Building Material.

I, Johannes G. Berg

of Fagerstrandv. 58, 1322 HØVIK, Norway

do solemnly and sincerely declare as follows.

1. I am authorised by the applicant for the patent to make this declaration on its behalf.

2. The basic application as defined by Section 141 of the Act was made in

Norway

on the 10th day of January 1986, by Norsk Proco A/S

on the 12th day of November 1986, by Norsk Proco A/S


are the actual inventors of the invention and the facts upon which the applicant is entitled to make the application are as follow:

The applicant is the assignee of the said actual inventors.

4. The basic application referred to in paragraph 2 of this Declaration was the first application made in a Convention country in respect of the invention the subject of the application.

DECLARED at Hovik

this twenty-sixth day of January, 1987.

JOHANNES G. BERG - for and on behalf of NORSK PROCO
Claim

1. Water and fire resistant building material, characterized by composition built up in layers and then shaped and hardened, consisting of one or more absorbant components, magnesia cement, one or more lignin sulphonates, chemically active fumed silica, alkali silicate, possibly one or more inorganic filler additives, and possibly ethyl silicate, whereby the final product includes two binder systems, one consisting of water resistant magnesia cement and lignin sulphonate, the other consisting of the reaction product between chemically reactive fumed silica, alkali silicate and possibly magnesium oxide.

15. Procedure for making a building material according to claims 1 - 14, characterized by thoroughly mixing a composition consisting of one or more absorbant materials, magnesium oxide, lignin sulphonate, a magnesium salt, water, alkali silicate, reactive fumed silica, and possibly ethyl silicate and inorganic filler additives, after which the completed composition is shaped and cured under elevated pressure and temperature until the free water in the mixture is converted to a hydrate, and the material is shaped and hardened into the desired building article.
COMMONWEALTH OF AUSTRALIA
PATENTS ACT 1952-49

COMPLETE SPECIFICATION

(ORIGINAL)

Application Number: 67446/87

Complete Specification lodged:

NORSK PROCO A/S

Address of Applicant: c/o Johannes G. Berg, Fagerstrandv. 58, 1322 Hovik, Norway

Actual Inventor: JOHANNES G. BERG and ROBERT SMITH-JOHANSEN

Address for Service: EDWD. WATERS & Sons, 50 Queen Street, Melbourne, Australia, 3000.

Complete Specification for the invention entitled:

WATER AND FIRE RESISTANT BUILDING MATERIAL

The following statement is a full description of this invention, including the best method of performing it known to:

US
This invention concerns water and fire resistant building materials based on a combination of magnesia cement, one or more absorbant materials, lignin sulphonate, chemically active fumed silica, alkali silicate, water as well as one or more additives. The invention also concerns the method of producing these materials.

Magnesia or Sorel cement has been known for about 100 years. It has the advantage over Portland cement in that it cures faster and gets harder. On the other hand magnesia cement is slightly water soluble, which results in a reduced water resistance for products made from magnesia cement. However, according to Norwegian patent No-PS 151035 its water resistance can be greatly improved by the addition of 0.5-2.0% by weight of ethyl silicate.

Building materials in sheet or strip form made by hot pressing and curing of a mixture consisting mainly of wood fibers, inorganic fillers, finely divided light calcined magnesia (MgO) and an aqueous magnesium chloride solution, and a method for producing them is described in Norwegian patent NO-PS 141889.

Such a building product according to NO-PS 141889 is characterized by the cured mixture having:

(a) a weight ratio of MgO to wood fiber of 1:3-1:4,
(b) a weight ratio of MgCl₂ to wood fiber material 1:9-1:12,
(c) chemically inactive silica of 2-15% based on the wood fiber content
(d) possibly up to 5%, preferably 2-4% waterglass, based on the wood fiber content.
The procedure according to NO-PS 141889 is characterized by 30-40 parts by weight finely divided light calcined magnesia being mixed with 3-4 times its weight of a wood fiber filler which consists mainly of splinters with a length of at the most 20 mm, 2-8 parts by weight of inactive silica, at least one aqueous liquid containing 9-12 parts by weight MgCl₂ and 30-60 parts by weight of water, with possibly 1-5 parts water-glass, and where this mixture is formed and cured from 3-20 minutes at a pressure of 15-50 bar and a temperature of 120-220°C. Production of wood fiber boards according to NO-PS 141889 can be continuous by extrusion or piece by piece in a flat press.

Wood fiber boards made according to NO-PS 141889 are claimed to be dimensionally stable and "showed, even after long submersion in water no noticeable swelling" (NO-PS 141889), page 7 lines 1-4.

The present invention concerns a water and fire resistant building material which is characterized by being a shaped and cured structure consisting of a layered combination of one or more absorbant components, magnesia cement, one or more lignin sulphonates, chemically active fumed silica, alkali silicate, possibly one or more inorganic additives (fillers) and possibly ethyl silicate. The final product thereby consists of two binder systems, one consisting of water resistant magnesia cement, lignin sulphonate, preferably magnesium lignin sulphonate, and the other consisting of the reaction product of the chemically active fumed silica, alkali silicate and possibly magnesium oxide.

The invention further concerns the method of producing the above mentioned building material, and is characterized by the thorough mixing of one or more absorbant materials with a dispersion of fumed silica in alkali silicate, possibly with ethyl silicate, and then with magnesium oxide and inorganic fillers, followed by a solution of magnesium salt containing lignin sulphonate in which also may be dispersed inorganic fillers,
after which the finished composition is formed and cured under suitable pressure and temperature conditions until the free water in the mixture is bound as hydrate, and the material is shaped and hardened to form the desired building material.

The new and essential about this invention is that the final product is built up of a combination of two binder systems. One of them, system A consists of water resistant magnesia cement with a lignin sulphonate salt, while the other, system B, consists of the reaction product of chemically active fumed silica, alkali silicate and possibly magnesium oxide. By the layered structuring of the final product the two binder systems are separated from one another during the initial stages of curing, at which time they are incompatible with one another, and the curing reactions are assumed to take place independently of one another.

System A gives good early strength and short press time and contributes to high water- and fire resistance.

System B develops and increases strength over a longer period of time as well as also having a beneficial effect on both water and fire resistance. In addition the use of the lignin sulphonate in combination with system A increases both strength and water resistance, especially when the absorbant base material consists of wood fibers.

The short press time is of vital importance in a commercial operation. According to this invention the cure time is in the order of 2 minutes at a temperature of between 150-175°C. In contrast to this the Sorel cement described in US Patent 2,466,145 apparently requires much longer cure times. Usual practice with Sorel bonded wood fiber systems requires cure times up to 20 minutes.

An essential feature of the present invention is the layered addition of the reactive components. In contrast to this the cited US patent 1,175,427 states that it is immaterial how the different ingredients are mixed. The layered concept of the present invention is required because of the nature of the dual binder system.
A preferred embodiment of the procedure according to this invention requires the absorbant component to be covered as much as possible by a continuous layer of a dispersion of fumed silica in alkali silicate (water glass). The layer is rendered insoluble by the following addition of a layer or coating of magnesium oxide. After this the magnesium chloride or sulphate solution is added along with the lignin sulphonate, which may also contain dispersed inorganic filler materials. Under high pressure the absorbant components and binders are consolidated and at elevated temperature the magnesium cement hydrate is formed removing free water from the mix and storing it in the form of crystalline water.

When exposed to high temperature, as in a fire, the alkali silicate in the product expands and helps to shut off the supply of air (oxygen). The magnesia cement also under these conditions, gives off water over a wide temperature range, and in this way acts to limit the temperature and to smother the fire. The lignin sulphonate acts as a bridge between the magnesia cement structure and the cellulose in the wood fiber base material, and has the further advantage that it surprisingly improves the water resistance. Of the inorganic filler additives dolomite is especially attractive because under fire conditions it gives off carbon dioxide gas.

The following examples show that a building material made according to this invention has high bending strength and tensile strength, little swelling and low water absorption under submersion in water, and good fire resistance. The components in the following examples were mixed in the indicated order and proportions.
### Example 1 (Sample 989-28). Made in laboratory.

1. Coarse wood fiber 18.9%
2. Fine wood fiber 16.7%
3. 29.8% fumed silica dispersed 5.1%
   - Water glass 4.7%
4. Fumed silica ("Micropoz") 5.6%
5. Premix containing:
   - 60.6% MgCl₂·6H₂O
   - 14.7% Lignin sulphonate (Calcium)
   - 24.7% water
6. Dolomite 5.6%
7. Recycled surface grindings 7.0%
8. Magnesium Oxide 15.6%
9. Premix as in 5 10.4%
10. 100.0%

Press time: 2.2 minutes
Press temperature: 160/170°C

### Example 2 (Sample 989-14). Made in laboratory.

1. Coarse wood fiber 20.8%
2. Fine wood fiber 18.4%
3. Water glass 5.2%
4. Fumed silica 29.8% dispersion 4.7%
5. Dolomite 6.1%
6. Fumed silica ("Micropoz") 6.1%
7. Magnesium oxide 17.2%
8. Premix containing:
   - 60.6% MgCl₂·6H₂O
   - 14.7% Lignin sulphonate (calcium)
   - 24.7% water
9. 100.0%

Press time: 2.2 minutes
Press temperature: 160/170°C.
Example 3 (Sample 989). Full Scale Test.

1. Coarse wood fiber 20.4%
2. Fine wood fiber 18.2%
3. Magnesium Oxide 17.4% Premixed
4. Dolomite 6.0%
5. Fumed silica ("Micropoz") 6.0%
6. Water 7.2% Premixed
7. Water glass 3.7%
8. Lignin sulphonate 5.4% Premixed
9. 33% MgCl₂ solution 15.7%
10. 100.0%

Press time 2.5 minutes.
Press temperature 163/170°C.

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Example 4 (Sample 498). Full Scale Test.

1. Coarse wood fiber 23.3%
2. Fine wood fiber 23.3%
3. Water glass 2.8% Premixed
4. Water 5.5%
5. Magnesium Oxide 20.6%
6. Fumed silica ("Micropoz") 5.5%
7. Ethyl Silicate 0.5%
8. 33% MgCl₂ Solution 18.5%
9. 100.0%

Press time 3 minutes
Press temperature 120/136°C
Example 5 (Sample 989-60). Made in laboratory

1. Coarse wood fiber 18.7%
2. Premix containing: 19.9%
   25.9% water
   7.4% lignin sulphonate (calcium)
   30.3% MgCl₂·6H₂O
   36.4% Fumed silica ("Micropoz")
3. Fine wood fiber 16.5%
4. Recycled surface grindings 6.1%
5. Magnesium oxide 15.4%
6. Premix as in 2 19.9%
7. Water glass 3.5%
   100.0%

Press time: 2.2 minutes.
Press temperature 170/170°C.

The completely cured building panels were then tested for the following properties:

Bending strength

Tensile strength

Per cent swelling and water absorption in water after 2 and 24 hours submersion in water. The panels were further tested for fire resistance according to Norwegian procedure NS 3903 and NT Fire 004.

Reference is made to the curves for combustion gas temperature and smoke density which were carried out at the Laboratory for Investigation of Fire Technology (Brann teknisk Lab.) SINTEF in Trondheim.
## Test Results

<table>
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<tr>
<th>Example no.</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
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<td>11.5</td>
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<td>8.1</td>
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<td>Swelling in water % after 24 hours</td>
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<td>Water absorption % after 2 hours</td>
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<td>16.0</td>
<td>14.1</td>
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<td>Average 3 samples</td>
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<td>Water absorption % after 24 hours</td>
<td>23.7</td>
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<td>Fire test: NS3903 and NT Fire 004 Limit: K1/IN1</td>
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THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. Water and fire resistant building material, characterized by composition built up in layers and then shaped and hardened, consisting of one or more absorbant components, magnesia cement, one or more lignin sulphonates, chemically active fumed silica, alkali silicate, possibly one or more inorganic filler additives, and possibly ethyl silicate, whereby the final product includes two binder systems, one consisting of water resistant magnesia cement and lignin sulphonate, the other consisting of reaction product between chemically reactive fumed silica, alkali silicate and possibly magnesium oxide.

2. Building material as in claim 1, characterized by containing 10-70 weight per cent absorbant material.

3. Building material as in claims 1-2, characterized by containing 10-70 per cent magnesia cement.

4. Building material according to claims 1-3, characterized by containing 0.1-15 weight per cent lignin sulphonate.

5. Building material according to claims 1-4, characterized by containing 1-15 per cent chemically active fumed silica.

6. Building material as in claims 1-5, characterized by containing 1-25 per cent alkali silicate.

7. Building material according to claims 1-6, characterized by containing 0-5 per cent ethyl silicate.
8. Building material according to claims 1 - 7, characterized by containing 0-30 per cent inorganic filler additives.

9. Building material according to claims 1 - 8, characterized by having the absorbant component consisting of cellulose fiber, wood fiber, perlite, fly ash, mineral wool, glass wool, diatomaceous earth, or a mixture of 2 or more of these single components.

10. Building material according to claims 1 - 9, characterized by the magnesium cement consisting of magnesiumoxychloride hydrate, magnesiumoxysulphate hydrate, or a mixture of these oxysalt hydrates.

11. Building material according to claims 1 - 10, characterized by the lignin sulphonate component consisting of calcium or magnesium salts, or a combination of them, or some other lignin sulphonate salt which can be converted to the magnesium salt.

12. Building material according to claims 1 - 11, characterized by the fine silica component being chemically active fumed silica.

13. Building material according to claims 1 - 12, characterized by the alkali silicate consisting of sodiumsilicate water glass or potassium silicate water glass or a mixture of these water glasses.

14. Building material according to claims 1 - 13, characterized by the inorganic filler additives being dolomite or feldspar or a combination of them.

15. Procedure for making a building material according to claims 1 - 14, characterized by thoroughly mixing a composition consisting of one or more absorbant materials,
magnesium oxide, lignin sulphonate, a magnesium salt, water, alkali silicate, reactive fumed silica, and possibly ethyl silicate and inorganic filler additives, after which the completed composition is shaped and cured under elevated pressure and temperature until the free water in the mixture is converted to a hydrate, and the material is shaped and hardened into the desired building article.

16. Procedure according to claim 15, characterized by thoroughly mixing a premix consisting of absorbant materials, an aqueous solution of magnesium chloride, possibly magnesium sulphate, and a lignin sulphonate, and adding to this premix magnesium oxide and eventually inorganic filler additives, after which the resultant mixture is pressed in a form at elevated temperature and pressure, until the free water is combined as hydrate water, and the material has been converted to the desired building article.

DATED this 8th day of January 1987,

NORSK PROCO A/S

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