COMMONWEALTH of AUSTRALIA Patents Act 1952

APPLICATION FOR A STANDARD PATENT 625698

I/We

Atochem

of

La Defense 10, 4 & 8 Cours Michelet, Puteaux, 92800, France

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

Process for the manufacture of insulating panels

which is described in the accompanying complete specification.

Details of basic application(s):-

Number	Convention Country	<u>Date</u>	
88 05619	France	27 April 1988	

The address for service is care of DAVIES & COLLISON, Patent Attorneys, of 1 Little Collins Street, Melbourne, in the State of Victoria, Commonwealth of Australia.

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DATED this TWENTY SEVENTH day of APRIL 1989

To: THE COMMISSIONER OF PATENTS

a member of the firm of DAVIES & COLLISON for and on behalf of the applicant(s)

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GOMMONWEALTH OF AUSTRALIA

PATENTS ACT 1952-1973

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

In support of the Application made for a patent whited: PROCESS FOR THE MANUFACTURE OF INSULATING PANELS

I Michel ROCHET

of: ATOCHEM, a French Body Corporate cf:

La Defense 10, 4 & 8 Cours Michelet, 92800 PUTEAUX, France.

do solemnly and sincerely declare as follows :-

or (b) I am authorized by

ATOCHEM,

the applicant.⁵⁵ for the patent to make this declaration on its behalf.

2. (a) lam the actual inventor we are invention

or(b) LUCIEN BOURSON: YVES BONNAUD and JEAN-PAUL GUILLAUME, all citizens of France of: 15 rue Raspail, 92270 BOIS-COLOMBES, France: 2 Place de l'Auditoire, 95160 MONTMORENCY, France and 3 Allee Pablo Picasso, Le Plessis Bouchard, 95130 FRANCONVILLE, France, respectively.

is the actual inventor...... of the invention and the facts upon which the applicantX $x \to x^{15}$ is entitled to make the application are as follows :--

"The applicant would, if a patent were granted upon an application made by the inventors be entitled to have the patent assigned to it".

by



19th day of April 1989h. Michel ROCHET.

Insert title of invention

Insert full name(s) and addrem(cs) of declarant(s) being the applicant(s) or person(s) authorized to sign on behalf of an applicant company.

Cross out whichever of paragraphs 1(a) or 1(b) does not apply 1(a) relates to application made by individual(s) 1(b) relates to application made

by company; insert name of pplicant company.

' Cross out whichever of paragraphs ' 2(a) or 2(b) does not apply

2(a) relates to application made by invantor(s)

2(b) relates to application made by company(s) or person(s) who are not inventor(s); insert full - name(s) and address(es) of inventors.

* ate manner in which applicant(s) erive title from inventor(s)

Cross, out paragraphs 3 and 4 for non-convention applications. For convention applications, insert basic country(s) followed by data(s) and basic applicant(s).

Insert place and date of signature.

Signature of declarant(a) (no attestation required)

Note: Initial all elterations.

(12) PATENT ABRIDGMENT (11) Document No. AU-B-33720/89 (19) AUSTRALIAN PATENT OFFICE (10) Acceptance No. 625898 (54) Title PROCESS FOR THE MANUFACTURE OF INSULATING PANELS International Patent Classification(s) (51)⁴ D21H 003/28 D21J 001/20 D21H 003/68 E04B 001/80 (21) Application No. : 33720/89 (22) Application Date : 27.04.89 (30) Priority Data Number (31) (32) Date (33) Country 8805619 27.04.88 **FR FRANCE** (43) Publication Date : 02.11.89 (44) Publication Date of Accepted Application : 16.07.92 (71) Applicant(s) ATOCHEM (72) Inventor(s) LUCIEN BOURSON; YVES BONNAUD; JEAN-PAUL GUILLAUME (74) Attorney or Agent DAVIES COLLISON CAVE, 1 Little Collins Street, MELBOURNE VIC 3000 **Prior Art Documents** (56) EP 342071 US 3929666 (57) Claim 1. Process for the manufacture of a panel which

comprises forming a moist plaque by filtering a suspension containing at least one fibrous material, an inorganic filler, starch, and a metal polysalt; and drying the moist plaque to form a panel.

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The present invention relates to a process for the manufacture of insulating panels.

These panels consist of a mixture of rock wool or glass wool, clay, waste paper and starch and can be used to 5 insulate structures thermally and against sound. They can be painted or a decorative coating can be bonded adhesively thereto; they can be employed as false ceilings.

Patent Application DE OS 2,617,601 describes the manufacture of insulating panels comprising glass fibre 10 fabrics. The disadvantage of this is that the solids making up the panel have to be handled when it is being moulded.

The present invention relates more particularly to processes in which the panel is formed by filtering and then drying a suspension.

A process for the manufacture of these panels consists in suspending in water glass wool or rock wool fibres, clay or another inorganic filler, ground waste paper or waste cardboard and starch. This suspension is then filtered through a cloth and a kind of moist plaque or board 20 several centimetros in thickness is obtained, which is dried in a tunnel oven. Rigid plaques which can be used as a construction material are thus obtained. One of the purposes of the starch is to endow the panel with cohesion and rigidity. The disadvantage of a process of this kind is 25 that the starch is only partly retained in the moist plaque, the remaining part leaving in suspension with the

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filtering water under the cloth. A new process has now been found which makes it possible to fix the starch better in the moist plaque during the filtration such that under the filter cloth, the water contains very little starch and 5 generally very little suspended matter.

The process according to the invention comprises adding a metal polysalt before filtration to a suspension containing:

- at least one fibrous material,

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- an inorganic filler, and

- starch.

A water suspension of the above products in a more or less divided state can be produced by any known means and a metal polysalt is then added to this suspension and this 15 suspension is filtered, for example on a continuous band filter, so that a moist plaque a few centimetres in thickness (the filter cake) is obtained which can then be dried.

Any fibrous material, for example vegetable fibres, 20 glass fibres or waste textile fibres may be employed. However, rock wool or glass wool is preferred.

Clay in any of its forms, kaolin or calcium carbonate may, for example, be chosen as an inorganic filler. A mixture of two or more of these inorganic fillers 25 may, of course, be employed. Clay or a product consisting essentially of clay is preferably employed.

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All kinds of starch may be employed, such as, for example, natural, so-called "unmodified" corn starch, or modified starch, such as anionic or cationic starch.

All these components of the suspension may be 5 introduced in the dry state or in aqueous suspension, in one or more portions and in any order.

The metal polysalt is generally a product of the formula:

 $[M_n(OH)_m Cl_{3n-m}]_z$

10 in which M is a trivalent metal, m and n are positive integers, 3n-m is > 0 and z is greater than or equal to 1. Such products are known and are employed as flocculants in water treatment.

The product in which M denotes aluminium is 15 preferred.

It is also possible to employ a product of formula:

 $M_n (OH)_m X_{3n-m}$

in which M is a metal which may be a tri- or polyvalent metal, X is an anion which may be Cl, NO₃ or CH₃COO, 3n is 20 greater than m with a basicity (m/3n x 100) of approximately from 30 to 83%, a polyvalent anion (called Y) having been chemically introduced into the said basic salt, the quantity of anion Y being such that the ratio Y/M is 0.015 - 0.4.

This product is described in French Patent 25 2,036,685. The product in which M is aluminium, X is chlorine and Y is a sulphate is preferably employed. It is

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also possible to employ the product of the formula: $Al_m (OH)_n X_{3m-n-2k} (SO_4)_k$

in which X denotes Cl or NO_3 , k, m and n are positive numbers respectively, 3m > n+2k, the basicity (n x 100)/3m is from 30 to 70% and k/m is from 0.01 to 0.3.

It can be manufactured by a process which consists in mixing a water-soluble sulphate or a solution containing SO₄ ions, a solution containing Al and X and a solution of an alkali metal aluminate at a temperature below 40°C so as 10 to produce a gel, and then maintaining the whole mixture at from 50 to 80°C so as to allow the gel to dissolve and thus to obtain a solution of a basic aluminium salt.

This product is described in French patent 2,226,361.

The product of formula $Al_2(CH)_x Cl_y$, in which x is from 1 to 3.5 and y from 5 to 2.5, may also be employed; this product is described in US Patent 3,909,439.

It is also possible to employ basic aluminium polychlorosulphates corresponding to the general formula:

 $[Al_n(OH)_{3n-m-2p}(SO_4)_p]_zCl_m$

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in which:

(3n-m-2p)/3n is from 0.4 to 0.7

p is from 0.04 n to 0.25 n

m/p is from 8 to 35, and

z is greater than or equal to 1.

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This product may be prepared by the action of basic

aluminium chloride on basic aluminium sulphate.

This process is described in French Patent 2,534,897.

It is also possible to employ the basic aluminium 5 polychlorosulphates described in European Patent Application EP 218,487:

 $Al_n OH_m (SO_4)_k Cl_{3n-m-2k}$

in which the basicity or the ratio (m x 100)/3n is from approximately 40% to approximately 65% and which has an Al 10 equivalent/Cl equivalent ratio of from 2.8 to 5, an apparent molecular mass AM, measured by conventional light scattering and apparent hydrodynamic diameters \$\$\$ Z and \$\$\$ W, measured by quasielastic light scattering, with the following values:

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AM = 7,000 - 35,000 $\phi Z (A) = 350 - 2,500$ $\phi W (A) = 200 - 1,200$

or such that:

MW		=	10,000 -300,000		,000
φZ	(Å)	1 2	90	-	450
φW	(Å)	æ	50	-	300

or else basic aluminium polychlorosulphate comprising the product of general formula:

 $Al_n OH_m (SO_4)_k Cl_{3n-m-2k}$

which may, for example, be obtained by a process of the type 25 comprising a step of preparation of a slurry of calcium chloride and of calcium carbonate (chlorocarbonate slurry),

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a step or bringing the chlorocarbonate slurry into contact with aluminium sulphate, then a step of separation of the reaction mixture thus obtained, enabling a calcium sulphate cake to be separated from a filtrate containing basic

5 aluminium chlorosulphate, the quantities of chlorocarbonate slurry and of aluminium sulphate being such that in the case of the basic aluminium chlorosulphate obtained there is a ratio (m x 100)/3n of from approximately 40% to approximately 65% and an Al equivalent/Cl equivalent ratio 10 of from 2.8 to 5.

It is also possible to employ basic aluminium polychlorosulphates comprising products having a certain degree of polymerization and of composition represented by the expression:

 $Al_n Cl_m (OH)_{3n+2k-m-2p} (SO_4)_p X_k$

in which:

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X denotes an alkaline-earth element, preferably calcium

n, m, p and k denote the molar concentrations 20 (mol/1) of the components in solution

fewer than 20% of the SO_4^{2-} ions can be precipitated by the reaction with barium chloride at ambient temperature,

whose basicity: $(3n + 2k - m - 2p) \times 100/3n$ is from 45 to 25 70%.

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These products may be manufactured according to a process which consists in preparing an aqueous solution containing the aluminium ion, the chloride ion and the sulphate ion, and in then bringing this solution into contact with an 5 alkaline-earth metal compound and, lastly, in removing the alkaline-earth metal sulphate formed.

One or more of these metal salts may be employed.

It is preferable to add the metal polysalt to the suspension just before the filtration. It may also be added 10 in several portions.

Although the suspension may contain the fibrous material, the inorganic filler and starch in any proportions, advantageously the fibrous material represents from 50 to 80% by weight of the solids content of the 15 suspension.

With regard to the inorganic filler, this is introduced in a proportion of 10 to 20% relative to the weight of the solids content of the suspension.

From 5 to 10% by weight of starch is usually 20 sufficient.

The quantity of metal polysalt is immaterial, but it is usually sufficient to introduce a quantity of less than 1%, and preferably from 0.1 to 0.6% by weight of the solids content of the suspension, the sum of these 25 proportions adding up to 100%.

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A cellulose-based material, such as waste timber, shavings, sawdust, waste paper or cardboard may also be added to this suspension.

It is preferred to add waste paper or waste 5 cardboard to this suspension. Advantageously, this paper and cardboard is milled or shredded before being suspended, and it can also be put into the suspension partially milled and the suspension may be milled.

Advantageously, the quantity of paper or of 10 cardboard or a mixture of paper and cardboard does not exceed 10% by weight of the solids content of the suspension, and is preferably from 2 to 7%.

It would not be a departure from the scope of the invention to add to the suspension expanded materials such 15 as vermiculite or perlite. It is also possible to add either hydrocarbon products such as bitumen or tars, which may be optionally dispersed in the suspension by means of surfactants. These expanded materials are used as a partial substitute for the fibrous material. Advantageously, up to 20 30% by weight of fibrous material may be replaced by perlite or vermiculite.

It would not be a departure from the scope of the invention to combine the metal polysalts with one or more synthetic flocculating agents.

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These are, for example, poly(alkylene polyamines), poly(hydroxyalkylene polyamines) and polyacrylamides.

It would not be a departure from the scope of the invention to add to the suspension dyes, flame retardants and, in general, any products employed for making panels of this kind.

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The following Examples further illustrate the invention.

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EXAMPLE 1

An aqueous suspension containing the following is prepared: rock wool : 59%

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clay	:	13%
starch	:	78
waste paper	:	48
scraps from previous panels (broken)	:	17%

These percentages are by weight of the solids content of the suspension. This suspension is an aqueous solution containing between 25 and 30 g/l of solids content.

Before filtration, a basic aluminium polychlorosulphate is added to the suspension, its formula being such as described in FR 2,036,685, in which M is aluminium, X is chlorine, the basicity is $50\% \pm 5$, and the anion Y is SO_4 , such that Y/M = 0.16 in a proportion of 0.5% relative to the fibrous composition, on a machine of the papermaking machine type. A moist panel 15 mm in thickness is obtained by filtration and is dried for 2 hours in a tunnel at $115^{\circ}C$.

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The fire rating of the panels obtained according to French Standards CSTB 92501 and 92510 is MO.

The suspended matter (SM) content of the water collected under the filter cloth is 200 mg/1.

EXAMPLE 2 (Comparative)

When operating as in Example 1, but employing a flocculant of an anionic polyacrylamide type without an aluminium salt instead of the above aluminium salt, the

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same panels are obtained; the SM content of the water under the clcth is 3,400 mg/l.

EXAMPLE 3

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 The operation is as in Example 1, but with 25% of the rockwool replaced by perlite. The same results are obtained.

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The claims defining the invention are as follows:

 Process for the manufacture of a panel which comprises forming a moist plaque by filtering a suspension containing at least one fibrous material, an inorganic
filler, starch, and a metal polysalt; and drying the moist plaque to form a panel.

2. Process according to claim 1 in which the metal polysalt is a metal polychloride.

Process according to claim 1 or 2 in which the
fibrous material is glass wool or rock wool.

 Process according to any one of claims 1 to 3, in which the inorganic filler consists essentially of clay.

 5. Process according to any one of claims 1 to 4, in which the suspension also comprises a cellulose-based
15 material.

6. Process according to any one of claims 2 to 5 in which the metal polychloride is a basic aluminium polychlorosulphate.

Process according to claim 1 substantially as
described in either of Examples 1 and 3.

8. A panel manufactured by a process as claimed in any one of claims 1 to 7.

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Dated this 29th day of April, 1992 ATOCHEM, By its Patent Attorneys DAVIES COLLISON CAVE



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