

# UNITED STATES PATENT OFFICE

HUBERT L. BECHER, OF TRENTON, NEW JERSEY, ASSIGNOR TO THE AGASOTE MILLBOARD COMPANY, OF FERNWOOD ROAD, EWING TOWNSHIP, MERCER COUNTY, NEW JERSEY, A CORPORATION OF NEW JERSEY

## FIRE-RESISTANT PULPBOARD AND METHOD OF MAKING THE SAME

No Drawing.

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My invention relates to a pulp board which is highly fire-resistant and to the method of making the same.

My invention is of particular advantage in the manufacture of molded pulp board, such as is usually made by running wood pulp, paper pulp or the like into a mold and by means of pressure removing the majority of the water (see, for instance, United States Letters Patent No. 971,936 dated October 4, 1910, and No. 1,272,566 dated July 16, 1918) and subsequently drying out the remainder of the water by the application of heat. After it has been dried, or simultaneously with the drying, the board may, if desired, be subjected to high pressure in order to compact the same thus giving it higher tensile and transverse strength and a higher degree of hardness. The object of my invention is to produce a pulp board whose fibres are coated with aluminum hydrate to such an extent that the board is highly fire-resistant and will smolder as little as possible, if at all, after having been exposed to a flame.

It is a further object of my invention to apply aluminum hydrate to fibres in such a manner that when such fibres are molded or otherwise formed into the preliminary shape of the finished board there will be as little interference as possible with the drainage of the water from the pulp and no undue warping of the wet board.

Other objects of my invention will be apparent from the following description thereof.

In order to produce my fire-resistant pulp board in the best manner known to me, I deposit upon the pulp fibres, before they are shaped into the form of a board, aluminum hydrate and calcium sulfate obtained preferably as precipitates from the double decomposition of aluminum sulfate and calcium hydrate. In the preferred form of my invention I deposit upon the fibres also a suitable quantity of a colloidal clay known as bentonite. The aluminum hydrate when obtained as a precipitate from the double decomposition referred to is in a fine state of subdivision, and the calcium sulfate is

in very fine crystalline form. The bentonite, which is inferior to aluminum hydrate as a fireproofing agent, appears to assist the aluminum hydrate in coating the fibres more effectively and thereby aids it to check smoldering of the board after the latter has been exposed to a flame. The calcium sulfate, while not so valuable per se as the aluminum hydrate from the standpoint of fireproofing, increases the fire-resistant qualities of the board because present in large quantities, and appears to perform a very valuable function in breaking up the continuity of the gelatinous deposit of aluminum hydrate, or aluminum hydrate and bentonite, on the fibres, and for this reason or for the reason that it acts as a spacing means to keep the fibres slightly separated, aids in permitting the water to drain from the pulp. Some of the calcium sulfate is, however, lost, being washed out in solution with the drainage water, but the loss of calcium sulfate in subsequent operations is diminished and eventually ceases, if the drainage water holding calcium sulfate in solution is re-used in subsequent operations.

I prefer to cause the reaction between the aluminum sulfate and the calcium hydrate to take place in the beater in which, and while, the pulp is being beaten, first adding the aluminum sulfate and, when this has been thoroughly dissolved in the beater, adding a suspension of calcium hydrate, but I may cause such reaction, and obtain the precipitates resulting therefrom, in a separate vessel, but then the precipitate should be used as soon as possible to prevent agglomeration thereof. The bentonite will have to be suspended in water, but as this is a somewhat difficult operation due to the finely divided nature of this material, I prefer to mix the bentonite with the calcium hydrate before making up the suspension of the former, thus eliminating one mixing operation and also reducing the viscosity of the bentonite suspension by the presence of the calcium hydrate.

The proportion of aluminum hydrate is preferably such as will result in as complete a fireproofing of the finished board as possible.



sible without interfering with the proper felting of the fibres. The proportion of calcium sulfate used is, for obvious reasons, conveniently such as would result from the double decomposition, when producing thereby the requisite amount of aluminum hydrate, but in any event should be just about sufficient to permit suitable drainage of the water from the pulp without being used in such quantities as will interfere with giving the maximum fire-resistant qualities to the finished board, or with a proper felting of the fibres. As hereinafter explained, the proportion of calcium sulfate may under some circumstances be reduced with advantage to the finished product. The proportion of bentonite to be used in such as will, together with the aluminum hydrate used, coat the fibres sufficiently to check smoldering as far as reasonably possible. The exact proportions of these various substances will depend largely upon the nature of the fibres to be used and the degree to which the resulting board is to be compacted but can, in any event, readily be ascertained by simple experiment, enough being used to effect the desired object, any more than this being mere surplusage or interfering with ease of manufacture of the board.

I prefer to render the board waterproof as well as fire-resistant and therefore to add to the beaten mixture of the ingredients hereinabove referred to the usual soap (sizing) and then to precipitate it onto the fibres.

My preferred method (which results in the least costly board) therefore includes the following steps:

1. Preparation of a pulp suspension from any suitable fibre, such as wood fibre, chemically digested fibre, or mixtures thereof, as can be obtained, for instance, by beating up old newspapers; bagasse or straw may be used.

2. Preparation of aluminum hydrate in the requisite finely divided form, and calcium sulfate, by adding aluminum sulfate to the beater, and when this is dissolved, adding a suspension of calcium hydrate in which latter the desired quantity of bentonite is also suspended. It is advisable that the suspension of calcium hydrate be added gradually to the solution of aluminum sulfate and that enough be added to leave the mixture slightly alkaline at the end.

3. The pulp suspension and the precipitates, together with the bentonite, are mixed in the beater. If the pulp is to be subsequently sized care should be taken to keep or render the mixture neutral or slightly alkaline so as to avoid precipitation of the soap.

4. A suitable rosin or other soap in weight equal to 4%–5% of the total weight of fibre, bentonite, aluminum hydrate and calcium sulfate, is added to the beater charge as a sizing ingredient, preferably after the fireproof-

ing agents have been thoroughly mixed with the fibres.

5. The soap is then precipitated by adding a suitable quantity of a suitable precipitant, such as aluminum sulfate.

6. The pulp is then shaped in a mold or otherwise, and dried, or dried and compacted.

The resulting board is sufficiently fire-resistant to comply with the majority, if not all, of the building codes of American cities; incidentally, the product, if waterproofed as above described, is water-resistant so as to permit its use in construction work exposed to the elements; it has rigidity and good mechanical strength with freedom from brittleness and a low heat transmission as compared with boards composed of mineral products, such as cement, asbestos or plaster.

The following examples will illustrate the proportions which may be used in the manufacture of my board:

#### Example 1

	Pounds	
Fibre (paper pulp)-----	1000	
Sulfate of alumium-----	1000	90
and		
Slaked lime-----	360	
forming		
Aluminum hydrate-----	250	
and		95
Calcium sulfate-----	664	
Bentonite-----	100	
Water-----	49000	

This gives a board of the composition 100

52.2% fibre  
13.1% aluminum hydrate  
29.5% calcium sulfate  
5.2% bentonite

In the above example, about 100 lbs. of calcium sulfate were lost by having gone into solution in the drainage water. 105

#### Example 2

Same as Example 1 using 110

	Pounds	
Bentonite-----	300	

thus giving a board of the composition

47.4% fibre  
11.8% aluminum hydrate  
26.6% calcium sulfate  
14.2% bentonite 115

My invention may with substantially equally good results be employed in making a pulp board by the laying up process, that is, by forming paper-like layers of the board, for instance, on a paper making machine, and uniting the layers thereof in the usual manner, for instance, with the aid of sodium silicate. 120

The value of the bentonite arises from its extremely fine state of subdivision, so fine that the material swells when wetted. When 130



in my claims I refer to bentonite I intend to include as an equivalent any clay of such a degree of fineness, for instance ground china clay (kaolin) as will enable it to check smoldering to a substantially valuable degree.

The above examples give one illustration of a change in proportion made desirable by a change in the fibres used. In Example 1, the fibres used were obtained by grinding old newspapers and were fairly well hydrated and therefore tended to cling close together and thus prevent drainage. In Example 2, the fibres used were obtained from ground wood screenings and therefore constituted a free draining stock and were therefore capable of being made into a fire-resistant board with the use of a much higher proportion of bentonite.

Instead of producing aluminum hydrate by the double decomposition process with aluminum sulfate and calcium hydrate at the place of use, I may purchase aluminum hydrate of the required state of subdivision and calcium sulfate crystals and mix them, but these materials if purchased separately are very much more costly than if produced in the manner described, at or near the place of use. The aluminum hydrate should preferably be of a degree of fineness substantially equal to that obtained from the double decomposition hereinabove described. It is to substantially this degree of fineness that I refer when I speak in my claims of "aluminum hydrate of the degree of fineness described". If the particles of aluminum hydrate are of a much less degree of fineness a commercially valuable degree of fireproofing is not obtained, and if they are of a much higher degree of fineness the drainage of water from the wet pulp is so slow as to make the process commercially impracticable. In place of the calcium element or the sulphur element, or both, I may substitute in whole or in part an element or elements which when used with the aluminum element and the hydroxide radical give me aluminum hydrate in the fine state of subdivision described, and a crystal insoluble or difficulty soluble in water which can perform the function of the calcium sulfate crystals.

As a matter of fact under certain circumstances I prefer to substitute for some of the calcium an element which will not cause the formation of crystals, so as to reduce the proportion of crystals to the aluminum hydrate. As long as a sufficient proportion of crystals is present to permit reasonably free drainage, substantially the full value of the presence of the crystals is obtained. The presence of crystals in excess of such proportion interferes to a certain extent with the felting of the fibres and thus results in a board of less hardness than could be produced if no such excess of crystals were present. As already indicated the proportion of crystals necessary to permit reasonably free drainage depends upon

the nature of the fibre. For instance, ground wood screenings, untreated, form a free-flowing stock which does not call for the presence of so high a proportion of crystals as does old newspaper stock which under ordinary circumstances permits only a very slow drainage. The following example will illustrate a modification of my process which has for its object the lowering of the proportion of crystals:

### Example 3

	Pounds	
Fibre	1000	
Sulfate of aluminum	1000	
Slaked lime	180	80
Magnesium oxide	98	
forming		
Aluminum hydrate	250	
Calcium sulfate	332	
Magnesium sulfate	294	85
Bentonite	500	
Water	49000	

This gives a board of the composition

50.3% fibre	90
12.5% aluminum hydrate	
12.0% calcium sulfate	
25.2% bentonite.	

In the above example the fibre used is about two-thirds newspaper stock and one-third ground wood screenings, constituting therefore, because of the presence of the screenings, a stock which is freer flowing than newspaper stock alone. The proportion of slaked lime used is one-half of that used in Example 1 and there is substituted for the remainder of the slaked lime of Example 1, 98 lbs. of magnesium oxide. The resulting magnesium sulfate does not form crystals but goes into solution in the drainage water and is removed therewith simultaneously with about 100 lbs. of the calcium sulfate as in Example 1. As a result the amount of calcium sulfate crystals in the board (232 lbs.) is far less than one-half of the amount of calcium sulfate crystals (564 lbs.) of Example 1. This permits a more complete felting of the fibres and therefore a harder board having a surface less liable to be injured by abrasion than the surface of the product of Example 1. This permits also the addition of a much higher proportion of bentonite than in Example 1, thus increasing the insurance against smoldering. The presence of the bentonite even in this high proportion does not interfere, at least to any substantial extent, with the free felting of the fibres, nor does it interfere with the drainage of the stock. The addition of this high proportion of bentonite in Example 1 would increase unduly the proportion of mineral matter to fibre.

In Example 3, the magnesium oxide may be replaced by caustic soda, sodium carbonate or ammonium hydrate, the resulting sodium or ammonium sulfate passing out with the



drainage water just as does the magnesium sulfate in such example.

The board produced in accordance with the preferred method, heretofore outlined in detail, is porous even after compaction because of the presence of the high proportion of crystals and therefore its surface, especially when the board is new, is readily injured. After the board has been exposed to the atmosphere for two or three weeks, the surface is hardened so that it may not be readily rubbed off. As already explained, a harder surface is obtained by diminishing the proportion of crystals in the manner indicated in Example 3. If a board having a harder surface is desired and particularly one which will permit the smoothing of such surface, for instance, by sanding, a metallic soap of a drying oil (f. i. linseed, China oil or perilla) is added to the furnish as the sizing ingredient (step 4) and the board, after it has been dried, is baked for a period of time, and at a temperature, sufficiently high to oxidize the oil. Of course, any suitable fillers or coating compositions (f. i. ethyl silicate) may be used which will improve the board without interfering with its fire-resistant qualities.

Board hardened by the use of a metallic soap of a drying oil may be dried to bone-dryness before baking and in fact should be so dried. On the other hand, it is of very great importance that a board not containing such a soap should not be dried to bone-dryness, but should preferably have left within it as high as 2% by weight of moisture, as otherwise the fire-resistant qualities of the board are diminished to a very large extent. I am unable at this time to explain this phenomenon but believe that the removal of the final fraction of moisture results in chemical changes which destroy in part the protection afforded by the aluminum hydrate and the bentonite.

The proportion of crystals which under given circumstances may permit the freest drainage of the water from the pulp after the board has been shaped may, at least in some cases, be higher than desirable to permit the desired extent of felting, i. e., to produce the hardest possible board. Therefore if a relatively soft and highly porous board is the desired product, it will be profitable to use a comparatively large proportion of crystals so as to permit speed of operation resulting in reduced cost of production. If a comparatively hard board is the desired product, and therefore a smaller proportion of crystals must be used, speed of production must be sacrificed. The exact proportion of crystals may therefore have to be the result of a compromise in which some hardness of product is sacrificed in favor of speed of production, or vice versa.

I may substitute for some of the fibre, preferably about 20 percent, ground or granulated

cork. Such cork may be incorporated either in its natural state or impregnated with suitable fireproofing agents such as sulfate of ammonium, sodium molybdate, or boric acid, preferably to such an extent that the weight of the salt is about 20 percent of the weight of the fireproof cork. It may sometimes be found desirable to subject the cork to a mild alkaline treatment before fireproofing it so as to permit a higher degree of impregnation.

To increase the fireproofing qualities of the cork, the impregnated cork may be coated with a suitable coating, for instance, calcium carbonate, to prevent escape of the water soluble salts.

The aluminum hydrate referred to by me is  $\text{Al}(\text{OH})_3$ .

I claim:

1. A highly fire-resistant pulp board whose individual fibres are coated with aluminum hydrate and bentonite.

2. The method of making fire-resistant pulp board which comprises mixing fibres, water in an amount sufficient to float them, aluminum hydrate of the degree of fineness described in an amount substantially sufficient to coat them and calcium sulfate crystals in an amount sufficient to permit substantially free drainage of the water, shaping the resulting mixture into the form of a board, permitting the majority of the water to drain away and then drying the mixture.

3. In the process of claim 2, the step of producing the aluminum hydrate and the calcium sulfate crystals by double decomposition of aluminum sulfate and calcium hydrate.

4. A highly fire-resistant pulp board whose individual fibres are coated with aluminum hydrate, bentonite and an oxidized drying oil.

5. The method of making fire-resistant pulp board which comprises mixing fibres, water in an amount sufficient to float them, aluminum hydrate of the degree of fineness described in an amount substantially sufficient to coat them and crystals substantially insoluble in water in an amount sufficient to permit substantially free drainage of the water, draining the majority of the water away and then drying the mixture.

6. In the process of claim 5, adding bentonite in suspension to the mixture.

7. In the process of claim 5, the step of producing aluminum hydrate and the crystals by double decomposition of an aluminum salt and of a hydrate which will react to form aluminum hydrate and a crystal substantially insoluble in water.

8. In the process of claim 5, the step of producing some of the aluminum hydrate and all of the crystals which are substantially insoluble in water by double decomposition of an aluminum salt and a suitable hydrate and the remainder of the aluminum hydrate



by double decomposition of an aluminum salt and of a hydrate which will react to form aluminum hydrate and a substance soluble in water.

5 9. In the process of claim 5, the step of producing some of the aluminum hydrate and all of the crystals by double decomposition of aluminum sulfate and calcium hydrate and the remainder of the aluminum hydrate  
10 by double decomposition of aluminum sulfate and magnesium hydrate.

10. In the method of making fire-resistant pulp board claimed in claim 5, the step of  
15 adding to the mixture a metallic soap of a drying oil.

HUBERT L. BECHER.

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