Title: NONFLAMMABLE MDF AND METHOD FOR PRODUCING THE SAME

Abstract: Disclosed is a nonflammable MDF obtained by forming an air-permeable inorganic coating film having a thickness of 0.2 to 2 mm on a surface of an MDF (medium-density fiber board) obtained by adding a fire retardant, as well as wood fiber and an adhesive, and forming by hot pressing, when produced; and a method for producing the same. The nonflammable MDF obtained by the invention is excellent in fire resistance and heat reflecting properties, as well as in nonflammability, high in mechanical strength, and has high weather resistance, so that it can be used as a fire protection structural material. Further, heat resistance, weather resistance and sound absorption performance are enhanced without impairing the original performances of the MDF such as bending strength, hardness, lightness in weight and processability, and moisture conditioning properties and soil resistance are also excellent.
Description

Nonflammable MDF and Method for Producing the Same

[Technical Field]

[0001]

The present invention relates to a nonflammable MDF (medium-density fiber board) and a method for producing the same.

More particularly, the present invention relates to a nonflammable MDF in which an air-permeable inorganic coating film having a thickness of 0.2 to 2 mm is formed on a surface of an MDF obtained by adding a fire retardant, as well as wood fiber and an adhesive, and forming by hot pressing, which is nonflammable, excellent in heat insulating properties, weather resistance, sound absorption, moisture conditioning properties, bending strength, hardness and processability, and usable as a light-weight nonflammable, fire protection structural, heat insulating and sound absorbing materials; and a method for producing the same.

[Background Art]

[0002]

As a means for making an MDF nonflammable, there has hitherto been known one in which various fire retardants, as well as wood fiber, adhesives and additives, are added in producing the MDF, one in which a surface of an MDF is coated with an inorganic coating such as an alkoxyym metal or alkali metal coating, one in which a fire retardant is combined with an inorganic coating, or the like.
However, under the present circumstances, all of these can achieve the approval of a fire retardant material, but are difficult to attain a semi-incombustible material, and do not reach an incombustible material at all. As a matter of course, a halogen-based one generating harmful gas in burning can not be used, and a nonflammable inorganic coating film has the problem that it is easily cracked by expansion and contraction in absorbing and releasing moisture to cause lack of stability.

[Disclosure of the Invention]

[Problems That The Invention Is to Solve]

The present invention has been made against the background of the prior art as described above, and an object of the present invention is to provide a nonflammable MDF enhanced in nonflammability, heat resistance, weather resistance and sound absorption of the MDF, and also excellent in moisture conditioning properties and soil resistance without impairing the original performances of the MDF such as bending strength, hardness, lightness in weight and processability; and a method for producing the same.

[Means for Solving the Problems]

The present invention relates to an nonflammable MDF characterized in that an air-permeable film having a thickness of 0.2 to 2 mm is formed using an inorganic coating agent on a surface of an MDF obtained by adding a fire retardant, as well as wood fiber and an adhesive, and forming by hot pressing.
The adhesive as used herein is preferably at least one thermosetting resin selected from the group consisting of a urea resin, a melamine resin, a phenol resin, an isocyanate resin, an epoxy resin and a polyester resin.

Further, the fire retardant is preferably at least one selected from the group consisting of a borate, a phosphate, a silicate, an ammonium salt, a metal hydroxide and other acids and bases.

Furthermore, the inorganic coating agent is preferably a composition composed of (a) 4 to 12 parts by weight (in terms of an SiO₂ basis) of a quaternary ammonium, (b) 45 to 75 parts by weight of an inorganic filler, (c) 2 to 12 parts by weight of a hardener and (d) 15 to 40 parts by weight of water and/or a hydrophilic organic solvent (with the proviso that (a)+(b)+(c)+(d)=100 parts by weight).

In addition, (b) the inorganic filler contained in the inorganic coating agent is preferably at least two selected from the group consisting of a metal oxide, a metal hydroxide, a metal nitride, a metal carbide, a metal carbonate and a silicate having an average particle size or an average length of 0.3 to 200 μm and insoluble in water.

Further, (c) the hardener contained in the inorganic coating agent is preferably at least one selected from the group consisting of a zinc compound, a magnesium compound, a calcium compound, a phosphate and a mineral acid.

Then, the present invention relates to a method for producing a nonflammable MDF comprising applying the above-mentioned inorganic coating agent onto an MDF subjected
to fire retarding treatment, and hardening the above-mentioned inorganic coating agent at ordinary temperature or by heating at low temperature to form an air-permeable coating film having a thickness of 0.2 to 2 mm.

[Advantage of the Invention]

[0006]

The nonflammable MDF of the present invention is excellent in fire resistance and heat reflecting properties, as well as in nonflammability, high in mechanical strength, and has high weather resistance, so that it can be used as a fire protection structural material. Further, in the heat insulating effect by the synergistic effect of the original heat insulating properties of the MDF and the heat insulating properties of the inorganic coating film, the nonflammable MDF having a thickness of 20 mm is comparable to a conventional heat insulating material having a thickness of 60 mm. Furthermore, when the nonflammable MDF is used as an interior material, the nonflammable MDF which forms the interior material having a high energy-saving effect can be provided, because it is excellent in heat radiation properties and dropwise condensation prevention (drying properties), as well as in nonflammability, in addition to heat insulating properties.

[Brief Description of the Drawings]

[0007]

[Fig. 1] Fig. 1 is a view for illustrating a state in which a test piece E is put between stainless steel plates, both ends of which are fixed with rod screws (Example 2).
[Fig. 2] Fig. 2 is a view for illustrating a method for heating the test piece of Fig. 1 by using a gas burner (Example 2).

[Description of Reference Numerals and Signs]

[0008]
a: Stainless Steel Pressure Plates
b: Rod Screws
c: Bolts
d: Test Piece
e: Gas Burners

1 to 4: Temperature Measuring Positions with Thermocouples

[Best Mode for Carrying Out the Invention]

[0009]

MDF: The MDF used in the present invention can be produced by known methods. For example, the MDF is formed by adding an adhesive and a fire retardant to wood fiber obtained by fibrillating wood chips, drying the resulting mixture, then, forming a mat with a dry sheet making apparatus (air filter), and hot pressing the mat.

The amount of the wood fiber used which constitutes the MDF herein is from 67 to 72% by weight, and preferably about 70% by weight, based on the whole, taking the water content as 6%.

[0010]
The above-mentioned adhesive is at least one of thermosetting resins such as a urea resin, a melamine resin, a phenol resin, an isocyanate resin, an epoxy resin and a
polyester resin. Although a melamine-urea mixed resin is frequently used, an alkali phenol or isocyanate resin which releases a smaller amount of formaldehyde has recently been increasing. However, some of the melamine-urea mixed resin is reduced in formaldehyde release with a fire retardant, and a catcher agent is used in some cases. The catcher agent as used herein is an adsorption decomposer, for example, silver zeolite or copper zeolite.

[0011]

The amount of the above-mentioned adhesive used is usually from 12 to 24% by weight, and preferably from 14 to 20% by weight, based on the whole. Less than 12% by weight results in weakened strength or decomposition, whereas exceeding 24% by weight unfavorably results in no air permeability, deteriorated heat insulating properties or a failure to obtain nonflammability.

[0012]

Further, the fire retardant is at least one selected from the group consisting of a borate, a phosphate, a silicate, an ammonium salt, and a metal hydroxide. The effect of these fire retardants is that the fire retardants themselves melt by heat to form coating films, thereby insulating materials from flames, for the borate, borax and the silicate, that nonflammable gases such as CO₂, NH₃, SO₂ and SO₃ are generated by decomposition by heat to weaken combustibility, for the ammonium salt, and that dehydration and carbonization are accelerated by heat to give no time for burning, for the phosphate and the other acids and bases.
The above-mentioned fire retardant is ordinarily used as a combination of two or more of them in an amount of 7 to 20% by weight, and preferably in an amount of 9 to 14% by weight, based on the whole. Less than 7% by weight results in insufficient nonflammability, whereas exceeding 20% by weight unfavorably results in poor adhesion.

Besides, other additives such as a wax, a preservative, a fungicide, an ant-preventing agent, a waterproof agent, a colorant and a deodorant can be added to the above-mentioned MDF.

The MDF used in the present invention is formed, for example, by fibrillating wood chips or another plant fiber, adding thereto an adhesive and a fire retardant, and other additives as needed, drying the resulting mixture, then, forming a mat, rapidly applying a pressure of 60 to 80 kg/cm² for several seconds at a first step, and pressing at 20 to 40 kg/cm² for 1 to 3 minutes at a second step. The hot plate temperature is from 200 to 260°C, but the forming process is not limited to the above-mentioned technique.

Properties of the MDF subjected to the above-mentioned fire retarding treatment are scarcely different from those of an ordinary MDF, a material thereof is homogeneous, a surface thereof is smooth, the density is from 0.6 to 0.8, the bending strength (N/mm²) is 30 or more, the thermal conductivity
(kcal/m sec) is from 0.04 to 0.08, and the releasing speed of formaldehyde (mg/m²h) is from 0.003 to 0.004.

[0017]

The MDF made nonflammable of the present invention is one obtained by forming an air-permeable inorganic coating film having a thickness of 0.2 to 2 mm on a surface of the MDF formed by hot pressing as described above, using an inorganic coating agent.

The inorganic coating agent as used herein mainly composed of, for example, (a) 4 to 12 parts by weight (in terms of an SiO₂ basis) of a quaternary ammonium, (b) 45 to 75 parts by weight of an inorganic filler, (c) 2 to 12 parts by weight of a hardener and (d) 15 to 40 parts by weight of water and/or a hydrophilic organic solvent (with the proviso that (a)+(b)+(c)+(d)=100 parts by weight).

[0018]

(a) Quaternary Ammonium Silicate:

(a) The quaternary ammonium silicate is one containing 15 to 45% by weight of a SiO₂ component, used as a main binder in the inorganic coating agent of the present invention, and represented by the following general formula:

\[(R₃N)₂·nSiO₂\] (wherein R is an alkyl group having 1 or more carbon atoms, and n is an integer of 1 or more.)

(a) This quaternary ammonium silicate has alkalinity, and reacts with (c) the hardener to strongly adhere to the MDF which is a substrate, as the following formulas (1) and (2), thereby imparting fire resistance, heat resistance, water resistance, weather resistance, hydrophilicity and air
permeability to the coating film formed.

[0019]  
\[ \text{-Si-OH} + R_n\text{-NH}_3\cdot n \rightarrow \text{-Si-O-NH}_4\cdot n\cdot R_n \]  

(1)  

[0020]  
\[ \text{-Si-O-NH}_4\cdot n\cdot R_n + \text{Mg(OH)} \rightarrow \text{-Si-O-Mg-O-Si-} \]  

(2)  

Specific examples of (a) the quaternary ammonium silicates include liquid silicates such as dimethyl-ethanolammonium silicate, monomethyltribropanolammonium silicate, dimethyldiopropanolammonium silicate and monoethyltribropanolammonium silicate. (a) These quaternary ammonium silicates are easily obtained by (1) a method of adding a quaternary ammonium hydroxide to an active silica solution obtained by bringing diluted sodium silicate into contact with a hydrogen type cation-exchange resin, and carrying out condensation to a specified concentration, (2) a method of reacting a quaternary ammonium hydroxide and silica hydrosol with each other, or the like. The quaternary ammonium hydroxide used herein is usually obtained by a method of adding an alkylene oxide to ammonia or an amine, a method of deionizing a quaternary amine salt with an anion-exchange resin, or the like. However, a product containing a tertiary, secondary or primary amine in small amounts is also usable, and a quaternary ammonium silicate obtained using it is also usable as the coating agent used in the present invention.

[0022]  

It is necessary that SiO₂ is contained in (a) the
quaternary ammonium silicate within the range of 15 to 45% by weight, preferably within the range of 20 to 30% by weight. Less than 15% by weight results in the excessive necessary amount of component (a), or an increase in water to delay drying, whereas exceeding 45% by weight unfavorably results in conversely a decrease in adhesion force or development of cracks.

[0023]

Specific examples of (a) the above-mentioned quaternary ammonium silicates include NS-25 (trade name, aqueous solution, SiO₂ concentration=about 25% by weight) manufactured by KABUSHIKI KAISHA NIPPANKENKYUSHO and Qas 25 (trade name, aqueous solution, SiO₂ concentration=about 25% by weight) manufactured by Nissan Chemical Industries Ltd., both having a pH of 11 to 12. (a) These quaternary ammonium silicates can be used either alone or as a combination of two or more of them.

[0024]

The content of component (a) in the inorganic coating agent used in the present invention is from 4 to 12 parts by weight, and preferably from 5 to 8 parts by weight, in terms of a SiO₂ basis, based on 100 parts by weight of the total of components (a) to (d). Less than 4 parts by weight results in lack of bonding force or hardness, whereas exceeding 12 parts by weight unfavorably results in powdering or development of cracks. When used as an aqueous solution, it is from 20 to 30 parts by weight (from 4 to 12 parts by weight, in terms of a SiO₂ basis, based on 100 parts by weight of the total of components (a) to (d)).
The composition of the coating film of the inorganic coating agent of the present invention is the same as that of the inorganic coating agent used in the method for producing the inorganic coating agent of the present invention, from which solvent (d) is removed. Accordingly, taking the total of components (a) to (c) of the present invention as 100 parts by weight, the content of each of components (a) to (c) naturally amounts to a value similar to the content of each of the above-mentioned component (a) and the following components (b) and (c), taking the total of components (a) to (d) in the inorganic coating agent as 100 parts by weight.

[0025]

(b) **Inorganic Filler:**

(b) The inorganic filler is used in the inorganic coating agent for the purpose of preparing a coating film having good fire resistance, heat insulating properties, high heat resistance, heat reflecting properties, air permeability and the like, and further imparts coloration, decorating properties, high weather resistance and the like to the coating film.

[0026]

(b) The inorganic fillers is at least two selected from the group consisting of a metal oxide, a metal hydroxide, a metal nitride, a metal carbide, a metal carbonate, and a silicate having an average particle size or an average length of 0.3 to 200 µm and insoluble in water. Specifically, they are at least two selected from the group consisting of zinc oxide, aluminum oxide, silicon dioxide, titanium oxide,
zirconium oxide, chromium oxide, lanthanum oxide, iron oxide, copper oxide, aluminum hydroxide, zirconium hydroxide, manganese dioxide, aluminum silicate, calcium silicate, magnesium silicate, zirconium silicate, aluminosilica gel, zinc carbonate, calcium carbonate, nickel carbonate, magnesium carbonate, silicon nitride, silicon carbide, potassium titanate, diatomaceous earth, talc, kaolin, perlite, mica and various synthetic oxides.

[0027]

The shape of (b) the above-mentioned inorganic filler is preferably granular, acicular, fibrous or scaly, and they may be mixed. Further, the average particle size of component (b) is from 0.3 to 200 μm, and preferably from 1 to 100 μm. Less than 0.3 μm results in a failure to obtain a necessary film thickness, a failure to exhibit the functions of component (b) or increased production cost. On the other hand, exceeding 200 μm unfavorably results in loss of smoothness of the coating film and decrease in adhesion force. The above-mentioned average particle size is interpreted as the average value of the maximum length of the unit shape, when the shape of component (b) is acicular, fibrous or scaly. The content of component (b) in the inorganic coating agent used in the present invention is from 45 to 75 parts by weight, and preferably from 55 to 60 parts by weight, based on 100 parts by weight of the total of components (a) to (d). Less than 45 parts by weight results in the difficulty of expressing the desired performance, or lack of moisture retention or coloration. On the other hand, exceeding 75 parts by weight unfavorably
results in too thick a film, a decrease in adhesion force, or an increase in viscosity of the inorganic coating agent.

[0028]

(c) Hardener:

(c) The hardener reacts with (a) the above-mentioned quaternary ammonium silicate in a short period of time to form the strong coating film excellent in water resistance, and has the effect of further improving fire resistance, heat resistance, weather resistance and air permeability of the resulting coating film.

[0029]

Component (c) is at least one selected from the group consisting of a zinc compound, a magnesium compound, a calcium compound, a phosphate and a mineral acid. Specifically, it is magnesium oxide, calcium oxide, zinc hydroxide, magnesium hydroxide, calcium hydroxide, phosphoric acid, aluminum orthophosphate, boric acid or the like. The above-mentioned components (c) may be used either alone or as a combination of two or more of them.

[0030]

The average particle size of component (c) is from 0.1 to 20 μm, and preferably from 0.5 to 10 μm. Less than 0.1 μm results in too rapid reaction with component (a) or an increase in viscosity, whereas exceeding 20 μm unfavorably results in loss of stability of the inorganic coating agent or loss of smoothness of the coating film. The content of component (c) in the inorganic coating agent used in the present invention is from 2 to 12 parts by weight, and preferably from 4 to 8
parts by weight, based on 100 parts by weight of the total of components (a) to (d). Less than 2 parts by weight results in too slow or insufficient reaction with component (a). On the other hand, exceeding 12 parts by weight unfavorably results in too rapid reaction with component (a) or development of cracks in the coating film.

[0031]

(d) Water and/or Hydrophilic Organic Solvent:

(d) The water and/or the hydrophobic organic solvent has the effects of adjusting viscosity of the inorganic coating agent, controlling the drying time and working time, and controlling dispersion of (b) the filler. As the water of component (d), there can be used service water, distilled water or ion-exchanged water. Further, the water also involves water contained in the silicate of the above-mentioned component (a), water contained in an emulsion type or water-soluble type synthetic resin used as an additive, and the like.

[0032]

The hydrophilic organic solvent of component (d) is an organic solvent compatible with water, and used as an agent for adjusting the solid concentration and viscosity of the inorganic coating agent, a drying speed adjusting agent or an antifreezing agent. (d) The above-mentioned hydrophilic organic solvents include an alcohol, a glycol and the like.

The alcohols include aliphatic alcohols having 1 to 8 carbon atoms, for example, methanol, ethanol, n-propanol, i-propanol, n-butanol, sec-butanol, t-butanol, methyl-
carbitol and the like. The glycols include, for example, ethylene glycol, propylene glycol, diethylene glycol and the like. The above-mentioned solvents may be used either alone or as a combination of two or more of them. As the preferred hydrophilic organic solvents, i-propanol, methylcarbitol and ethylene glycol are used either alone or as a mixed solvent of two or more of them.

[0033]

The content of component (d) in the inorganic coating agent used in the present invention is from 15 to 40 parts by weight, and preferably from 20 to 30 parts by weight, based on 100 parts by weight of the total of components (a) to (d). When the content is less than 15 parts by weight, the viscosity of the inorganic coating agent excessively increases, resulting in a decrease in storage stability, or deterioration of the dispersibility of components (a) to (c) in some cases. On the other hand, when it exceeds 40 parts by weight, other components relatively decrease, resulting in a decrease in adhesion force of the resulting coating film, or the coating film too thin to prepare a desired one in some cases, although the storage stability is improved.

[0034]

In the inorganic coating agent used in the present invention, components (a) to (d) are mixed, and dispersing agents such as various surfactants, hardening adjusting agents, antibacterial agents, synthetic resin emulsions, synthetic rubber emulsion, organic dyes and pigments, and other additives can be further contained as needed.
Further, the use of the synthetic resin and/or synthetic rubber emulsions as the above-mentioned additives can impart flexibility to the coating film, enhance adhesion force, and control water permeability of the coating film. Specifically, the above-mentioned emulsions include emulsions of an acrylic resin, a vinyl acetate resin, a silicone resin, an acrylonitrile-butadiene rubber, polybutadiene, a styrene-butadiene rubber and the like. The above-mentioned synthetic resins and/or synthetic rubbers may be used not only as emulsions, but also as aqueous solutions.

These resin and rubber components are well mixed with (d) the water and/or hydrophilic organic solvent, and form a water-insoluble transparent or translucent film when dried.

In the inorganic coating agent of the present invention, they can be used as an auxiliary agent for adhering (b) the filler, and further for improving impact resistance.

In the inorganic coating agent used in the present invention, components (a) to (c) are mixed, and component (d) and other additive components are blended as needed, thereby being able to adjust the total solid concentration to 60 to 85 % by weight, preferably to 70 to 75 % by weight. When the solid concentration is less than 60% by weight, it is difficult to obtain the necessary film thickness. On the other hand, when it exceeds 85% by weight, the viscosity unfavorably excessively increases to gel.

The above-mentioned inorganic coating agent can be
obtained as a homogeneous dispersion with an ordinary stirrer, a butterfly mixer for high viscosity or another disperser, or by mesh filtering.

[0037]

The inorganic coating agent used in the present invention is aqueous and easy to be handled, and is hardened for a short period of time even at ordinary temperature after coating. It is therefore excellent in functionality. Accordingly, it is applied to the MDF, and dried by heating at 60 to 100°C, preferably 70 to 80°C for 5 to 20 minutes, preferably for 10 to 15 minutes, or dried at ordinary temperature for 3 to 6 hours, preferably for about 4 to 5 hours, thereby hardening it to be able to form an air-permeable coating film having a thickness of 0.2 to 2 mm, preferably 0.5 to 1.2 mm. For application of the coating agent to a surface of the MDF, coating means such as a brush coating, spray coating, roll coating and flow coating can be used. The coating film obtained by the inorganic coating agent used in the present invention is air-permeable, so that it can usually be finished by one application in coating without giving attention to pinholes as previously applied. However, it can also be applied twice or more.

[0038]

The amount of the inorganic coating agent applied to the surface of the MDF herein is from 300 to 2,000 g/m², and preferably from 600 to 1,800 g/m², in terms of a solid content basis, per one application. When the amount coated is less than 300 g/m² in terms of a solid content basis, the film thickness becomes 0.2 mm or less, resulting in difficulty to
exhibit the performance of the coating film of the present invention. On the other hand, exceeding 2,000 g/m² is unfavorable because the coating film becomes easy to crack. Usually, the total amount coated is from 300 to 3,000 g/m², and preferably from 1,000 to 2,400 g/m², in terms of a solid content basis. When the total amount coated is less than 300 g/m² in terms of a solid content basis, the film is too thin, resulting in difficulty to exhibit the performance of the coating film of the present invention. On the other hand, exceeding 3,000 g/m² unfavorably results in easy cracking.

Further, the dry thickness of the coating film of the present invention obtained by coating, drying and hardening is from 0.2 to 2 mm, and preferably from 0.4 to 1.2 mm. When the film thickness is less than 0.2 mm, the performance of the coating film of the present invention is difficult to be exhibited. On the other hand, exceeding 2 mm unfavorably results in easy cracking.

[0039]

The nonflammable MDF of the present invention becomes excellent in water resistance and strong, because (c) the hardener is used. Further, the inorganic coating agent forming the coating film is mainly composed of (a) the quaternary ammonium silicate and (b) the inorganic filler, so that the coating film protects a base material, has good adhesion to the base material, is excellent in nonflammability, heat resistance and soil resistance, and also has air permeability and hydrophilicity. The coating film follows expansion and contraction of the MDF because of its good air
permeability, has good freezing thawing resistance because of no occurrence of internal dew condensation, and is also excellent in weather resistance and natural pollution resistance. In addition, it reflects heat energy radiated to the nonflammable MDF, and further, it is difficult to conduct heat to an unheated surface because of its low thermal conductivity. Accordingly, it can be used as an excellent fire-resisting material or heat insulating material, and further as a sound absorbing material caused by its porosity.

[Examples]

[0040]

The present invention will be illustrated with reference to the following examples, but it is to be understood that the present invention is not limited to the following examples.

[0041]

Reference Example 1

<Preparation of Inorganic Coating Agent>

An inorganic coating agent was prepared according to compounding amounts shown in Table 1. Compounds (a) to (e) were placed in a stirring tank and mildly stirred, followed by stirring at about 5,000 rpm for 10 minutes by using a high-speed stirrer and filtration through a 60-mesh filter. Thus, an inorganic coating agent was prepared.

[0042]

Symbols in Table 1 indicate the following:

(a); Quaternary ammonium silicate (NS-25, manufactured by KABUSHIKI KAISHA NIPPANKENKYUSHO, SiO₂ concentration= about 25% by weight)
(b): Inorganic filler
   (b)-1: Silicon dioxide (average particle size=100 μm)
   (b)-2: Magnesium silicate (average length=15 μm, acicular)
   (b)-3: Potassium titanate (average size=0.2 to 0.5 μm, whisker)
   (b)-4: Titanium oxide (white) (average particle size=0.5 μm)
   (b)-5: Iron oxide (yellow) (average particle size=0.5 μm)

(c): Hardener
   (c)-1: Magnesium oxide (average particle size=2 μm)

(d): Solvent
   (d)-1: Ion-exchanged water
   (d)-2: Propylene glycol

(e): Other additive components
   (e)-1: Leveling agent (silicone-based)
   (e)-2: Synthetic rubber emulsion (solid concentration=48% by weight)
   (e)-3: Waterproof agent (silicone-based)
   (e)-4: Antibacterial agent-deodorant (silver zeolite)
[0043]

[Table 1]

<table>
<thead>
<tr>
<th>Compounding Formulation</th>
<th>Compounding Amount (g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>(a)</td>
<td>200</td>
</tr>
<tr>
<td>(b)-1</td>
<td>500</td>
</tr>
<tr>
<td>(b)-2</td>
<td>60</td>
</tr>
<tr>
<td>(b)-3</td>
<td>20</td>
</tr>
<tr>
<td>(b)-4</td>
<td>40</td>
</tr>
<tr>
<td>(b)-5</td>
<td>10</td>
</tr>
<tr>
<td>(c)</td>
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</tr>
<tr>
<td>(d)-1</td>
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</tr>
<tr>
<td>(d)-2</td>
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<tr>
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</tr>
<tr>
<td>(e)-3</td>
<td>30</td>
</tr>
<tr>
<td>(e)-4</td>
<td>5</td>
</tr>
</tbody>
</table>

[0044]

Example 1

<Preparation of Fire Retardant MDFs>

In order to evaluate nonflammability, fire retardant MDFs were prepared.

Wood chips were fibrillated, and 19% by weight of a melamine-urea resin and 11% by weight of a silicate-based fire retardant, SIRIONO (manufactured by F-Stop, Belgium) were added thereto, followed by drying. Then, a mat was formed with a dry sheet making machine, and preliminarily compressed. The preliminarily compressed mat was hot pressed to prepare two kinds of fire retardant MDFs having thicknesses of 12 mm and 18 mm. Then, they were cut to 100×100 mm and 500×500mm to form test pieces.

The inorganic coating agent prepared was applied to a single side or both sides of each test piece in an amount of about 1,400 g per m² with an air spray, and dried at ordinary temperature for 3 hours, followed by standing in a room for
7 hours. The thickness of the coating film was 750 μm. The contents of the test pieces are shown in Table 2. An uncoated test piece was taken as F.

[0045]

<table>
<thead>
<tr>
<th>Table 2</th>
</tr>
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<tbody>
<tr>
<td>Name of Test Piece</td>
</tr>
<tr>
<td>A</td>
</tr>
<tr>
<td>B</td>
</tr>
<tr>
<td>C</td>
</tr>
<tr>
<td>D</td>
</tr>
<tr>
<td>E</td>
</tr>
<tr>
<td>F</td>
</tr>
</tbody>
</table>

[0046]

Examples 2-5, and Comparative Example 1

Using test pieces A to D and F, the burning test (according to ISO 5660) with a cone calorimeter was made. The results are shown in Table 3.
[Table 3]

<table>
<thead>
<tr>
<th></th>
<th>Example 2</th>
<th>Example 3</th>
<th>Example 4</th>
<th>Example 5</th>
<th>Comparative Example 1</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Test Time (min)</strong></td>
<td>20</td>
<td>20</td>
<td>20</td>
<td>20</td>
<td>20</td>
</tr>
<tr>
<td><strong>Radiant Intensity (kw/m²)</strong></td>
<td>50</td>
<td>50</td>
<td>50</td>
<td>50</td>
<td>50</td>
</tr>
<tr>
<td><strong>Ignition Time (sec)</strong></td>
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<td>No ignition</td>
<td>No ignition</td>
<td>No ignition</td>
<td>No ignition</td>
</tr>
<tr>
<td><strong>Maximum Heat Generation Rate (kw/m²)</strong></td>
<td>7.64</td>
<td>6.46</td>
<td>6.23</td>
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<td>31.91</td>
</tr>
<tr>
<td><strong>Maximum Heat Generation Time (sec)</strong></td>
<td>435</td>
<td>321</td>
<td>692</td>
<td>869</td>
<td>544</td>
</tr>
<tr>
<td><strong>Total Heat Generation Amount 1 Minute after Start of Test (MJ/m²)</strong></td>
<td>0.18</td>
<td>0.09</td>
<td>0.11</td>
<td>0.05</td>
<td>0.32</td>
</tr>
<tr>
<td><strong>5 Minutes after Start of Test</strong></td>
<td>2.91</td>
<td>1.57</td>
<td>1.18</td>
<td>1.11</td>
<td>11.06</td>
</tr>
<tr>
<td><strong>10 Minutes after Start of Test</strong></td>
<td>7.31</td>
<td>3.92</td>
<td>3.48</td>
<td>2.87</td>
<td>16.73</td>
</tr>
<tr>
<td><strong>20 Minutes after Start of Test</strong></td>
<td>9.68</td>
<td>7.85</td>
<td>7.17</td>
<td>6.19</td>
<td>38.12</td>
</tr>
<tr>
<td><strong>Total Heat Generation Amount at Termination of Test (MJ/m²)</strong></td>
<td>9.68</td>
<td>7.85</td>
<td>7.17</td>
<td>6.19</td>
<td>38.12</td>
</tr>
<tr>
<td><strong>Termination Time (min)</strong></td>
<td>20</td>
<td>20</td>
<td>20</td>
<td>20</td>
<td>20</td>
</tr>
<tr>
<td><strong>Heat Generation of 200 kw/m² or More Continuing for 10 Seconds or More</strong></td>
<td>No</td>
<td>No</td>
<td>No</td>
<td>No</td>
<td>No</td>
</tr>
<tr>
<td><strong>Occurrence of Deformation, Melting, Cracks and Others Harmful in Terms of Fire Prevention</strong></td>
<td>No</td>
<td>No</td>
<td>No</td>
<td>No</td>
<td>Yes</td>
</tr>
<tr>
<td><strong>Carbonization of Back Side</strong></td>
<td>Yes</td>
<td>No</td>
<td>No</td>
<td>No</td>
<td>Yes</td>
</tr>
</tbody>
</table>

From the results of Table 3, test piece A conformed to

5 a semi-nonflammable material (the total heat generation amount
(MJ/m²) after 10 minutes is 8 or less), and test pieces B, C
and D conformed to a nonflammable material (the total heat
generation amount (MJ/m²) after 20 minutes is 8 or less).
Uncoated test piece F was not ignited, but unsuitable for the
nonflammable material (the total heat generation amount
(MJ/m²) after 20 minutes is 8 or less).

[0049]

Example 6

Using test piece E of Table 2 described above, a test
based on the fire-resisting performance test necessary for
fire-resisting construction performance (Building Standard
law, article 2, item 7) was made. The test method is shown
in Fig. 1 and Fig. 2. In Fig. 1, test piece E was put between
stainless steel plates having a size of 700×120 mm and a
thickness of 5 mm, both ends of which were fastened with rod
screws and fixed by application of pressure. In the state of
Fig. 1, test piece E was heated with two gas burners at 900
to 1,000°C for 1 hour, as shown in Fig. 2. The state of a heated
surface was observed, and the temperatures of four positions
on an unheated surface shown in Fig. 2 were measured with
thermocouples. After the termination of heating, the test
piece was allowed to stand for 3 hours, and the state of test
piece E was observed. The states of the heated surface and
the unheated surface are shown in Table 4.
[0050]

[Table 4]

<table>
<thead>
<tr>
<th>Position in Drawing</th>
<th>No continuous spurt of flame to the unheated surface (back side) exceeding 10 seconds is observed at all, and changes in color are scarcely observed</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>No continuous flaming exceeding 10 seconds is observed at all on the unheated surface.</td>
</tr>
<tr>
<td>2</td>
<td>Damages such as cracks through which flame passes do not occur.</td>
</tr>
<tr>
<td>3</td>
<td>No continuous spurt of flame to the unheated surface (back side) exceeding 10 seconds is observed at all, and changes in color are scarcely observed</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Changes in temperature of the unheated surface</th>
<th>Measured point</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
</tr>
</thead>
<tbody>
<tr>
<td>Temperature (°C) 10 minutes after the start of heating</td>
<td>45 65 68 53</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Temperature (°C) 30 minutes after the start of heating</td>
<td>71 79 84 74</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Temperature (°C) 60 minutes after the start of heating</td>
<td>78 87 92 81</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

[0051]

As shown in Table 4, the temperatures of the unheated surface are less than 100°C, which shows extremely high heat reflective heat insulating properties. The effect on the unheated surface is scarcely observed. Three hours after the termination of heating, the heated surface is cracked at two positions of the coating film to come into a state in which the surface slightly rises. However, no changes to pressure are observed, although about 2 to 3 mm of a surface layer of the base material (MDF) is carbonized.

[0052]

Example 7

15 Using test piece B, various tests were made by the following techniques. The results are shown in Table 5.
<Measurement of Heat Insulating Properties>
The thermal conductivity was measured, based on the plate comparison method of JIS A1412.

<Measurement of Density>
The density was measured, based on JIS A5906.7.2

<Sound Absorption>
The rate of sound absorption by the reverberation room method was measured, based on JIS A1409. The thickness of test piece B is 19.6 mm.

<Water Absorption>
The water absorption was measured, based on JIS A5907.5.5.

<Water Content>
The water content was measured, based on JIS A5906.7.3.

<Bending Strength>
The bending strength was measured, based on JIS A5906.7.4.

<Wet Bending Strength>
The wet bending strength was measured, based on the wet bending strength test A of JIS A5908.5.5.1.

<Accelerated Weather Resistance>
The color difference after the termination of the QUV test (repeat test of UV irradiation at 70°C for 8 hours and dew condensation at 50°C for 4 hours) for 500 hours was measured.

<Amount of Formaldehyde Released>
The amount of formaldehyde released was measured, based on the "fiber board" formaldehyde released amount test of JIS A5905.
[Table 5]

<table>
<thead>
<tr>
<th>Item</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Heat Insulating Properties (kcal/m.h.deg)</td>
<td>0.04</td>
</tr>
<tr>
<td>Density</td>
<td>0.78</td>
</tr>
<tr>
<td>Sound Absorption Rate of sound absorption</td>
<td></td>
</tr>
<tr>
<td>Center frequency c/s</td>
<td>125</td>
</tr>
<tr>
<td></td>
<td>250</td>
</tr>
<tr>
<td></td>
<td>500</td>
</tr>
<tr>
<td></td>
<td>1,000</td>
</tr>
<tr>
<td></td>
<td>2,000</td>
</tr>
<tr>
<td></td>
<td>4,000</td>
</tr>
<tr>
<td>Water Absorption (%)</td>
<td>0.17</td>
</tr>
<tr>
<td>Water Content (%)</td>
<td>0.46</td>
</tr>
<tr>
<td></td>
<td>0.68</td>
</tr>
<tr>
<td>Bending Strength (kgf/cm²)</td>
<td>0.88</td>
</tr>
<tr>
<td>Wet Bending Strength (kgf/cm²)</td>
<td>0.75</td>
</tr>
<tr>
<td></td>
<td>0.80</td>
</tr>
<tr>
<td>Accelerated Weather Resistance</td>
<td>22</td>
</tr>
<tr>
<td>Amount of Formaldehyde Released (mg/L)</td>
<td>12</td>
</tr>
<tr>
<td>Average value: 0.2, maximum value: 0.2</td>
<td>334</td>
</tr>
<tr>
<td></td>
<td>193</td>
</tr>
<tr>
<td></td>
<td>ΔE 1.8</td>
</tr>
</tbody>
</table>

[Industrial Applicability]

[0054]

The nonflammable MDF of the present invention becomes a strong one which is nonflammable, excellent in fire resistance, heat resistance, heat insulation, heat reflecting properties, sound absorption, moisture conditioning, weather resistance, water resistance, processability and mechanical strength, light in weight, and has air permeability.

Accordingly, it can be extensively used for interior furnishings, exterior claddings and structural materials of architectural structures such as houses, mansions, office buildings, factories and warehouses, vehicles, ships and vessels, airplanes, electric products, acoustic equipments, chemical plants, power equipment, indoor and outdoor pipes, containers, freezers and the like, and exhibits effects of improvements in fire protection and earthquake resistance, weight saving, an increase in comfort, energy saving, a
reduction in cost and the like.
Claims

[Claim 1]
A nonflammable MDF characterized in that an air-permeable film having a thickness of 0.2 to 2 mm is formed using an inorganic coating agent on a surface of an MDF (medium-density fiber board) obtained by adding a fire retardant, as well as wood fiber and an adhesive, and forming by hot pressing.

[Claim 2]

The nonflammable MDF according to claim 1, wherein the adhesive is at least one thermosetting resin selected from the group consisting of a urea resin, a melamine resin, a phenol resin, an isocyanate resin, an epoxy resin and a polyester resin, and used in an amount of 12 to 24% by weight based on the whole.

[Claim 3]

The nonflammable MDF according to claim 1, wherein the fire retardant is at least one selected from the group consisting of a borate, a phosphate, a silicate, an ammonium salt and a metal hydroxide, and used in an amount of 7 to 20% by weight based on the whole.

[Claim 4]

The nonflammable MDF according to claim 1, wherein the inorganic coating agent mainly composed of (a) 4 to 12 parts by weight (in terms of an SiO$_2$ basis) of a quaternary ammonium silicate, (b) 45 to 75 parts by weight of an inorganic filler, (c) 2 to 12 parts by weight of a hardener and (d) 15 to 40 parts by weight of water and/or a hydrophilic organic solvent (with the proviso that (a)+(b)+(c)+(d)=100 parts by weight).
[Claim 5]

The nonflammable MDF according to claim 4, wherein (b)
the inorganic filler is at least two selected from the group
consisting of a metal oxide, a metal hydroxide, a metal nitride,
a metal carbide, a metal carbonate, and a silicate having an
average particle size or an average length of 0.3 to 200 μm
and insoluble in water.

[Claim 6]

The nonflammable MDF according to claim 4, wherein (c)
the hardener is at least one selected from the group consisting
of a zinc compound, a magnesium compound, a calcium compound,
a phosphate and a mineral acid.

[Claim 7]

A method for producing the nonflammable MDF according
to any one of claims 1 to 6, which comprises applying an
inorganic coating agent onto an MDF subjected to fire retarding
treatment, and hardening the coating agent at ordinary
temperature or by heating at low temperature to form an
air-permeable coating film having a thickness of 0.2 to 2 mm.
INTERNATIONALSEARCHREPORT

International application No
PCT/JP2004/011733

A. CLASSIFICATION OF SUBJECT MATTER
Int.Cl.7 B27K3/02, 3/20, B27N3/04

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)
Int.Cl.7 B27K3/00 - 3/52, B27N3/04

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
Published examined utility model applications of Japan 1992-1996
Published examined utility model applications of Japan 1971-1996
Published registered utility model specifications of Japan 1994-2000
Published registered utility model applications of Japan 1994-2000

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

<table>
<thead>
<tr>
<th>Category</th>
<th>Citation of document, with indication, where appropriate, of the relevant passages</th>
<th>Relevant to claim No.</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>JP 11-349950 A (Sun Techno Chemical Kabushiki Kaisha), 1999.12.21, Full text (Family: none)</td>
<td>1-7</td>
</tr>
<tr>
<td>A</td>
<td>JP 49-31084 B1 (Keisuke Shimizu), 1974.08.19, Full text; all drawings (Family: none)</td>
<td>1-7</td>
</tr>
<tr>
<td>A</td>
<td>JP 51-24370 B2 (Agency of Industrial Science &amp; Technology), 1976.07.23, Full text; all drawings (Family: none)</td>
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<tr>
<td>A</td>
<td>JP 52-3237 B2 (Okura Kogyo Kabushiki Kaisha), 1977.01.26, Full text (Family: none)</td>
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<tr>
<td>A</td>
<td>JP 6-198611 A (Fujimori Kogyo Kabushiki Kaisha), 1994.07.19, Full text (Family: none)</td>
<td>1-7</td>
</tr>
</tbody>
</table>

Further documents are listed in the continuation of Box C. See patent family annex.

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Date of mailing of the international search report 26.04.2005

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