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(54) **A method and an apparatus for injecting a treating liquid into a woody material.**

(57) A method for injecting a treating liquid into a woody material (1) to be treated, which comprises subjecting the woody material (1) to a first pressure reducing treatment (a) in the presence or absence of the treating liquor in a receptacle (2) capable of being maintained at reduced or elevated pressure, and thereafter subjecting it to a second pressure reducing treatment via a wood piece contained in filter units (F) while the woody material is immersed in the treating liquor and pressure is applied thereto, and an apparatus for injecting the treating liquor into the woody material in accordance with the method described above.

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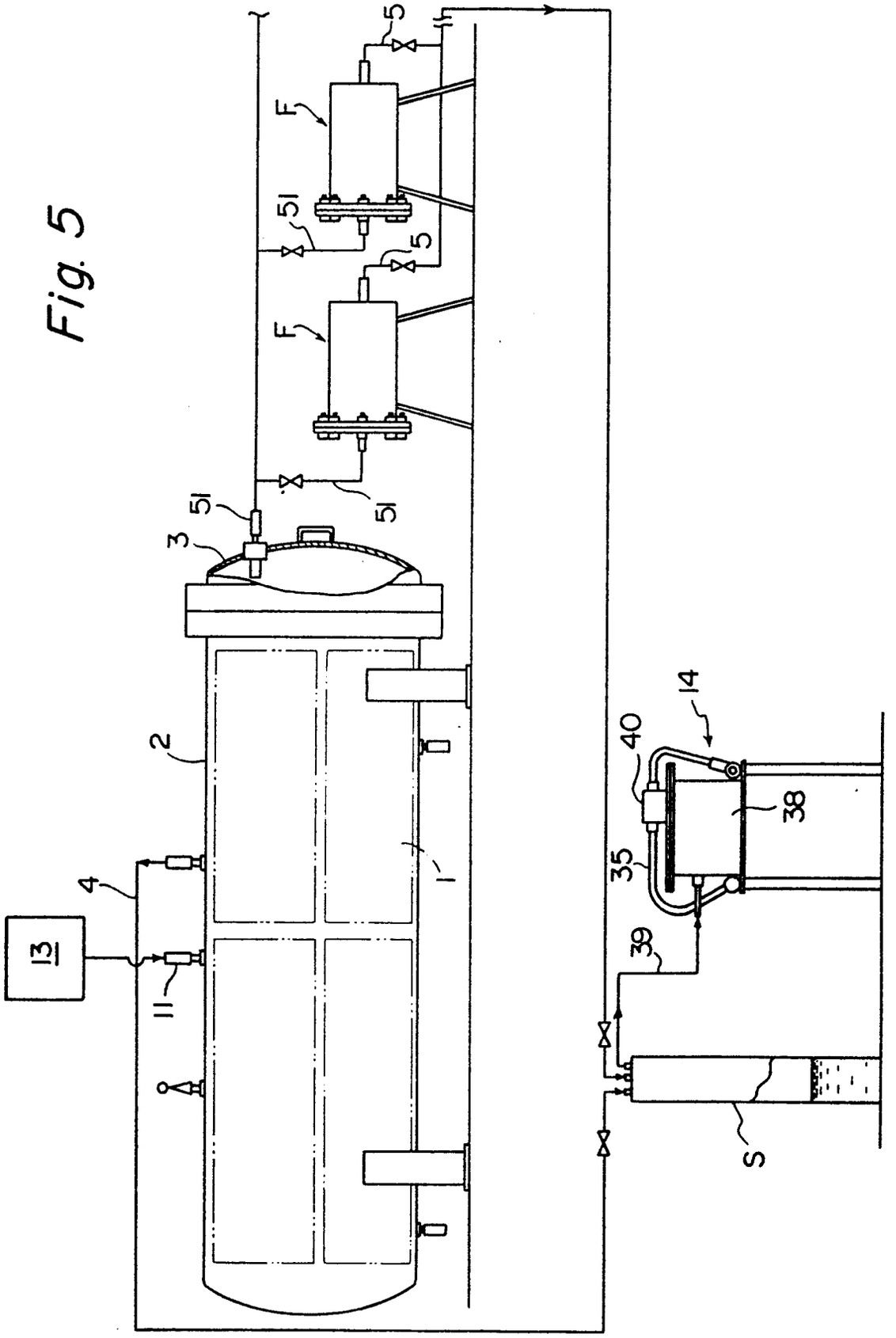
Fig. 5



# A METHOD AND AN APPARATUS FOR INJECTING A TREATING LIQUID INTO A WOODY MATERIAL

This invention relates to a method and an apparatus for injecting various treating liquors into woody materials for flame-retardation, dry cracking prevention, strengthening, antiseptic treatment, pest controlling treatment, dyeing, etc.

Heretofore, woody materials have been treated for flame retardation, dry cracking prevention, strengthening, antisepsis, pest control, dyeing, etc. by impregnating them. But none of such treatments have proved to be entirely satisfactory in effect, operability and economy, and particularly, no uniform and complete impregnation of the treating liquors in the woody materials has been achieved.

It is an object of this invention therefore to provide a method and an apparatus for impregnating a treating liquor uniformly and fully into a woody material within a short period of time.

The present inventor has now found that by using a specific method and a specific injecting device, a treating liquor can be uniformly and fully impregnated into a woody material.

According to this invention, there are provided

(1) a method for injecting a treating liquid into a woody material (I) to be treated, which comprises subjecting the woody material (I) to a first pressure reducing treatment (a) in the presence or absence of the treating liquor in a receptacle capable of being maintained at reduced or elevated pressure, and thereafter subjecting it to a second pressure reducing treatment via a wood piece (II) while the woody material is immersed in the treating liquor and pressure is applied thereto; and

(2) an apparatus for injecting a treating liquor into a woody material (I) to be treated, comprising a receptacle capable of being maintained at reduced or elevated pressure, a filter unit including a wood piece (II) as a filter, a pressure applying unit, and a pressure reducing unit, and optionally a gas-liquid separating unit.

Examples of the woody material to be treated by this invention include woody materials derived from coniferous trees typified by Japanese cedar, fir, Japanese cypress, hemlock-spruce, Japanese black pine tree, white fir, Ezo spruce, Japanese larch, Japaese red pine tree, sawara cypress, hatchet-leaved arborvitae, yew, spruce, radiata pine, western red cedar, white cedar, Douglas fir, Loblolly pine and western hemlock, and broad-leaved trees typified by Makaba birch, Siebold's beech, maple, elm, evergreen oak, zelkova, Nara oak, Japanese linden, Shiinoki, cherry, Japanese horse-chestnut, Katura, Sen, walnut, Nyatoh, Djohan, white mahogany, Mahogany, Calophyllum, Matoa, white ash, Moabi, Bubinga, Agathis, Teak, white lavan, Kapur, Kamerere, Yellow meranti, Ebony, Tracwood, silky oak, white oak, red lavan and Balsa. The woody material may be in the form of lumbers such as logs and beams and processed woody materials such as plywood, mosaic wood and particle boards.

The treating liquors used in this invention may, for example, include treating liquors for flame retardation, treating liquors for prevention of dry cracking, treating liquors for strengthening, antiseptic treating liquors, pest controlling treating liquors, dyeing liquors, etc.

The flame retarding treating liquors include, for example, solutions or dispersions of diammonium hydrogen phosphate, inorganic water-soluble salts such as boric acid salts, sulfamic acid salts, halogen compounds, compounds containing phosphorus and nitrogen, guanidine compounds and metal oxides such as antimony oxide.

Examples of the treating liquors for prevention of dry cracking are solutions or dispersions of polyethers such as polyethylene glycol or polypropylene glycol, saturated polyester resins, poly(meth)acrylic acid esters or copolymers thereof, urethane resins, polyvinyl alcohol, paraffins, vinyl acetate copolymers, polyamide resins, polyimino resins, fluorine resins, silicone resins, vinyl copolymer resins, SBR and NBR.

Examples of the treating liquors for strengthening include solutions or dispersions of vinyl monomers typified by styrene, (meth)acrylates, vinyl acetate, diallyl phthalate, divinylbenzene, (meth)acrylic acid, acrylonitrile and vinylidene chloride, mixtures of unsaturated polyesters with styrene monomer, reactive polyurethane resins, phenolic resins, urethane resins, alkyd resins, vinyl esters, urea resins, melamine resins and epoxy resins.

Examples of the antiseptic treating liquors include solutions or dispersions of water-soluble antiseptics such as copper sulfate, cupric chloride, cupric hydroxide, chromium compounds, arsenic compounds, boron compounds and fluorine compounds; oil-soluble antiseptics such as 8-quinolinol copper (oxine copper), metal naphthenates, pentachlorophenol, chloronaphthalenes, organotin compounds, captan, and other compounds (captan derivatives, carbanilides, propiolanilides, carboxanilides, urea compounds and chloronitrobenzenes); and contact-type antiseptics such as creosote oil/petroleum, creosote oil/pentachlorophenol, zinc chloride chromate, Walman salt, chloronaphthalines, creosote oil and copper/chromium/arsenic type compounds.

Examples of the treating liquors for pest control include solutions or dispersions of arsenic-containing pest controlling agents such as Wolman salt, Boliden salt K-33 and Celcure AP; and organic pest controlling agents such as organophosphorus compounds, carbamate compounds, organotin compounds, pyrethroid compounds, chlordane, Heptachlor, Dieldrine, Aldrine, Thiodane, gamma-BHC - (1,2,3,4,5,6-hexachlorocyclohexane), DDT [1,1,1-trichloro-2,2-bis(p-chlorophenyl)ethane], methoxychloro[1,1,1-trichloro-2,2-bis(p-methoxyphenyl)ethane], Toxaphene, sulfonamides, thiophene oil, and organic thiocyanates salts.

The dyeing liquors include, for example, solutions or dispersions of (1) water-soluble dyes, for example direct dyes such as Chrysophenine GX, Direct Brown M and Direct Fast Scarlet 4BS, acid dyes such as Suminol Fast Orange PO, Suminol Fast Brown R and Suminol Milling Scarlet FG, and basic dyes such as Safranine, Auramine and Bismarck Brown), (2) alcohol-soluble dyes and (3) oil-soluble dyes optionally together with dyeing adjuvants or surface-active agents.

Media that can be used in preparing these treating solutions include, for example, water, alcohols (e.g., methanol, denatured alcohol, ethanol, propanol, and butanol), glycols (e.g., ethylene glycol, diethylene glycol, glycerol and Cellosolves), aromatic hydrocarbons (e.g., toluene and xylene), aliphatic hydrocarbons (e.g., hexane, heptane, kerosene oil and terpene), alicyclic hydrocarbons (e.g., cyclohexane), ketones (e.g., methyl ethyl ketone, methyl isobutyl ketone and acetone), esters (e. g., ethyl acetate), halogen compounds (e.g., methylene chloride, trichloroethylene, perchloroethylene and 1,1,1-trichloroethane), acids (e.g., formic acid and acetic acid), dioxane, tetrahydrofuran, DMF and DMSO.

The treating liquors used in this invention have a viscosity of preferably not more than 1,000 cps. If it is above 1,000 cps, the treating liquors are difficult to impregnate into the woody material.

According to the method of this invention, the woody material (I) to be treated is subjected to a first pressure reducing treatment in the presence or absence of the treating liquor to remove most of the gas present in the woody material. Then, while the woody material is immersed in the treating liquor and pressure is applied thereto, the woody material (I) is subjected to a second pressure reducing treatment via the wood piece (II). By this treatment, the gas remaining in the woody material (I) moves to a pressure reducing sucking side through the treating liquor and preferentially discharged through vessels of the wood piece (II). Consequently, the treating liquor is uniformly and fully impregnated in the woody material (I) within a short period of time.

Preferably, the first pressure reducing treatment (a) and the second reducing treatment (b) are both carried out under an absolute pressure of not more than 200 mmHg. Under absolute pressures higher than it, the gas present in the woody material (I) is difficult to remove efficiently. The absolute pressure of not more than 200 mmHg in the second pressure reducing treatment (b) denotes a value measured on that side of the wood piece (II) which does not make contact with the treating liquor. The first pressure reducing treatment (a) can be efficiently carried out in a separate line from the second pressure reducing treatment (b) to be carried out via the wood piece (II).

Preferably, the pressure applying treatment in the method of this invention is carried out under a pressure of 1 to 100 atmospheres, especially 8 to 50 atmospheres. Impregnation is better as the degree of pressurization is higher. Care should however be taken in this regard because some types of the woody material (I) undergo deformation under a pressure of more than 50 atmospheres during impregnation.

The wood piece (II) in this invention is provided for preferentially discharging the gas remaining in the woody material (I). The mechanism of this is not clear, but it is presumed that vessels in the wood piece serve as a filter for separating low-molecular-weight substances such as air or water from high-molecular-weight substances. The wood piece (II) further serves as a measure, or a monitor, of determining the penetrability of the treating liquor into the woody material (I). Preferably, the wood piece (II) is a piece of the same wood species as the woody material (I). Thus, if the woody material to be treated is wood from a coniferous tree, the wood piece (II) is preferably made of a coniferous tree. If the woody material to be treated is from a broad-leaved tree, the wood piece (II) is preferably made of a broad-leaved tree. Especially preferably, the wood piece (II) is made of the same wood as the woody material (I).

The apparatus of this invention for injecting the treating liquor into the woody material in accordance with the method described above is comprised of a receptacle capable of being maintained at reduced or elevated pressure, a filter unit including a wood piece (II) as a filter, a pressure applying unit, and a pressure reducing unit, and optionally a gas-liquid separating unit.

The apparatus of this invention will be described specifically with reference to the accompanying drawings.

Figure 1 is a side elevation, partly broken away, of one embodiment of the apparatus of this invention. In Figure 1, the reference numeral 1 represents a woody material (I) to be treated; 2, a receptacle capable of being maintained at reduced

or elevated pressure; 3, a closure; 4 and 5, suction tubes; 7, a pressing plate; 8, a screw seat; 9, a screw shaft; 10, a wood piece (II); 11, a pressurized injection tube; 12, a discharge tube; 13, a pressure applying unit; and 14, a pressure reducing unit. F represents a filter unit; X, the direction of pressure application; and Y, the direction of pressure reduction.

Figure 2 is an enlarged sectional view illustrating the intimate contact between the suction tube and the wood piece (II) in the filtering unit of the apparatus of Figure 1. In Figure 2, the reference numeral 5 represents the suction tube; 6, its forward end; 7, the pressing plate; 9, the screw shaft; and 10, the wood piece (II).

Figure 3 is a side elevation, partly broken away, of another embodiment of the apparatus of this invention. In Figure 3, the reference numeral 1 represents a woody material (I) to be treated; 2, a receptable capable of being maintained at reduced or elevated pressure; 3, a closure, 4 and 5, suction tubes; 51, a conduit; 11, a pressurized injection tube; 13, a pressure applying unit; 14, a pressure reducing unit; and 15, a valve. F represents a filter unit.

Figure 4 is an enlarged sectional view showing the details of the filter device of the apparatus shown in Figure 3. In Figure 3, the reference numeral 51 represents the conduit; 5, the suction tube; 6, the forward end of the suction tube 5; 7, a pressing plate; 8, a screw seat; 9, a screw shaft; 10, a wood piece (II); and 20, a shell capable of being maintained at reduced or elevated pressure.

Figure 5 is a side elevation, partly broken away, of still another embodiment of the apparatus of this invention. In Figure 5, the reference numeral 1 represents a woody material (I) to be treated; 2, a receptacle capable of being maintained at reduced or elevated pressure; 3, a closure; 4 and 5, suction tubes; 51, a conduit; 11, a pressurized injection tube; 13, a pressure application unit; 14, a pressure reducing unit; 35, a gas discharge tube; 38, a pressure reducing tank; 39, a suction tube; and 40, a pressure reduction nozzle block. F represents a filter device and S, a gas-liquid separating device.

Figure 6 is a sectional view of a pressure reduction nozzle block preferably used in the pressure reducing unit of the apparatus of Figure 5. In Figure 6, 21 represents a throat; 22, a forward end opening; 23, a nozzle; 24, a gas discharge hole; 25, a vacant chamber; 26, a suction passage; 27, a suction hole; 28, a cylindrical portion; 28', fructoconical portion; 29, an injection hole; d, the diameter of the nozzle; and D, the diameter of the forward end part of the throat.

Broadly, the apparatus of this invention includes two embodiments classified by the arrangement of the filter device. In a first embodiment, the filter unit including the wood piece (II) as a filter is included within the receptacle capable of being maintained at reduced or elevated pressure. In a second embodiment, the filter unit containing the wood piece (II) as a filter is included with a shell capable of being maintained at reduced or elevated pressure connected to the receptacle capable of being maintained at reduced or elevated pressure. The first embodiment is shown in Figure 1, and the second embodiment, in Figures 3 and 5. In the first embodiment, the wood piece (II) serves as a filter for separating low-molecular-weight substances from high-molecular-weight substances, and in the second embodiment, the wood piece (II) serves as a filter and also as a monitor.

For efficient suction of gas, the filter unit F, as shown in Figures 2 and 4, is preferably one consisting essentially of the suction tube 5 having a fructoconical end 6, the wood piece (II) 10, and the pressing plate 7 and assembled by holding the wood piece (II) between the end 6 and the pressing plate 7. The pressing plate 7 is adapted to move toward and away from the wood piece (II) 10 by known means utilizing the screw seat 8 and the screw shaft 9, for example. The pressing should be performed such that the front end 6 of the suction tube 5 fully bites into the surface of the wood piece (II) 10. Usually a vacuum pump is used as the pressure reducing unit. Furthermore, a pressure reducing unit of the structure capable of jetting out compressed air into the atmosphere at high speed may be used. When a highly viscous treating liquor is used, the pressure reducing unit should desirably be one suitable for it. One example of such a pressure reducing unit is the pressure reducing nozzle block shown in Figure 6. In the pressure reducing nozzle block, the nozzle 23 comprising the cylindrical portion 28 facing the front end 22 of the throat 21 and the fan-shaped frustoconical portion 28' following it is connected to the jet hole 29. The ratio of the diameter D of the throat to the diameter d of the nozzle is 1.5, and the distance from the tip of the nozzle to the front end of the throat is 0.1 to 1.0 mm, preferably about 0.5 mm. The throat 21 is connected to the gas discharge hole 24 in the fan-shaped frustoconical portion at its rear end. The vacant chamber 25 enclosing the front end opening of the nozzle and the front end opening of the throat is adapted to communicate with the suction hole 27 via the suction passage 26. Preferably, the cylindrical portion 28 has a length of about 0.3 mm, and the frustoconial portion 28' has a length of about 2.0 mm.

Preferably, a plurality of the pressure reducing nozzle blocks are provided on the pressure reducing tank 38 as shown at 40 in Figure 5. At this time, the jet hole 29 is connected to a discharge tube of a compressor, the suction hole 27 is allowed to communicate with the pressure reducing tank 38, and the pressure reducing tank 38 is connected to the suction tube 39 leading from the gas-liquid separating unit S. On the other hand, the suction tube 4 from the receptacle 2 and the suction tube 5 from the filter unit F are connected to the gas-liquid separating unit S respectively so that they can be brought into and out of communication with the separating unit S. Accordingly, the receptacle 2 and the filter unit F are subjected to pressure reducing treatment by the pressure reducing nozzle blocks 40 via the gas-liquid separating unit S.

When such nozzle blocks are used, a highly viscous treating liquor does not adhere to and stagnate at the suction portion and therefore, no decrease in pressure reducing effect is caused.

As stated above, the apparatus of this invention consists essentially of the receptacle for injection of a treating liquor into the woody material (I) to be treated, the filter unit including the wood piece (II) as a filter, the pressure applying unit and the pressure reducing unit. Preferably, it further comprises the gas-liquid separating unit in order to prevent troubles in the pressure reducing unit, such as blockage.

The preferred apparatus of this invention including such an optional feature is illustrated in Figure 5. This apparatus is comprised of the receptacle 2 for receiving the woody material (I) to be treated and a treating liquor and adapted to be maintained at reduced or elevated pressure, a plurality of filter units F each comprising a shell 20 capable of being maintained at reduced or elevated pressure and the wood piece (II) 10 included therein, the pressure applying device 13, the pressure reducing unit 14 including the pressure reduction nozzle block 40, and additionally the gas-liquid separating unit S whose suction side is connected to the suction side of the pressure reducing unit 14. An apparatus of this structure can be continuously operated until the end of the treatment while the penetration of the treating liquor into the woody material is monitored. The use of this apparatus furthermore can avoid troubles of adhesion and stagnation of a viscous treating liquor at the pressure reducing unit.

The woody material into which the treating liquor has been injected by the method and apparatus of this invention is dried in air or in a heating oven or an oven under irradiation of radioactive rays, and then used in various applications as a material for civil engineering, building, furniture making or industries.

The following non-limitative examples illustrate the present invention more specifically.

EXAMPLE 1

The method of this invention was performed using the apparatus shown in Figure 5.

The closure 3 of the receptacle 2 was opened, and a woody material 1 (a cedar board having a size of 30 cm x 2 cm x 200 cm) was placed into it. The closure 3 was then closed.

Each of the filter units F was opened, and as shown in Figure 4, a wood piece 10 ( a cedar beam having a size of 5 cm x 4.5 cm x 41 cm) was held between the front end 6 of the suction tube 5 and the pressing plate 7, and the screw shaft 9 was tightened to cause the front end 6 of the suction tube 5 to bite into the wood piece 10. Then, each of the filter devices F was closed.

The conduit 51 was opened, and a treating liquor prepared by adding 500 parts of water-soluble trimethylated methylolmelamine resin having a non-volatile content of 70%, 250 parts of an aqueous solution of 3-(dimethylphosphono)-propionamide having a non-volatile content of 70%, 10 parts of ammonium chloride as a curing catalyst and 3 parts of a sodium salt of ethylene oxide sulfate succinate to 237 parts of water was injected into the receptacle 2 from the injection tube 11 attached to its upper surface to fill the receptacle to such a degree that some space was left in the upper part of the receptacle 2. Thus, the woody material 1 was immersed in the treating liquor.

The injection tube 11 and the conduit 51 were then closed, and the pressure reducing unit 14 was operated via the suction tube 4 and the gas-liquid separating unit S to reduce the pressure of the inside of the the receptacle 2 to 60 mmHg, and the pressure was maintained at this value for 40 minutes. The suction tube 4 was then closed, and the conduit 51 was opened. The pressure reducing device 14 was operated via the filter units F, the suction tube 5 and the gas-liquid separating unit S to reduce the pressure of the suction tube 5 to 60 mmHg.

While this state was maintained, the injection tube 11 was opened and compressed air under a pressure of 10 kg/cm$^2$ was sent to the receptacle 2 from the pressure applying unit 13. As the injection of the treating liquor under pressure proceeded,

the inflow of the treating liquor sucked from the receptacle through the filter units F was observed in the gas-liquid separating unit S. At this time, the suction system for one of the filter units F was shut off, and the wood piece 10 was taken out of the filter unit F. When its section was examined, it was ascertained that penetration of the treating liquor into the woody material was completed. Hence, the injecting operation was stopped.

The pressure inside the receptacle 2 was returned to atmospheric pressure, and the treating liquor was discharged. The closure was opened and the treated cedar board was taken out.

The rate of impregnation was 190%. The treated wood was dried at 25°C for 24 hours and then with hot air at 50°C for 24 hours. The P content was examined using test pieces sampled from the surface and inside of its end portion, and the surface and the inside of its center. The results are shown in Table 1. The treated wood was found to be acceptable by flame retardancy Class 3 in JIS 1302.

COMPARATIVE EXAMPLE 1

Example 1 was repeated except that the pressure reducing treatment via the filter units F was not performed. Specifically, the woody material 1 to be treated was put into the receptacle 2. The receptacle 2 was closed, and the pressure of the inside of the receptacle was reduced through the suction tube 4 to 60 mmHg, and maintained at this value for 60 minutes. While the reduced pressure was maintained, the treating liquor was injected from the injection tube 11 to fill the receptacle to such an extent as to leave some space in the upper part of the inside of the receptacle 2. Thus, the woody material 1 as immersed. Then, the pressure reduction line was closed, and compressed air under a pressure of 10 kg/cm² was sent from the pressure applying unit 13 for 60 minutes to inject the treating liquor into the woody material.

The rate of impregnation was 70%. The treated woody material was dried under the same conditions as in Example 1, and the same samples as in Example 1 taken from the treated woody material had the P contents shown in Table 1.

Table 1

| | End portion | | Central portion | |
|---|---|---|---|---|
| | Surface | Inside | Surface | Inside |
| Example 1 | 5.8% | 5.7% | 5.7% | 5.6% |
| Comparative Example 1 | 5.7% | 3.3% | 5.8% | 0.1% |

EXAMPLE 2

The method of this invention was performed using the apparatus shown in Figure 1.

A woody material 1 to be treated (a false arbovitae board having a size of 80 cm x 1 cm x 150 cm) was placed in the receptacle 2 after opening the closure 3. The suction tube 4 provided on the receptacle 2 and the suction tube 5 provided on the closure 3 were connected to the pressure reducing unit 14 (vacuum pump).

In the meantime, as shown in Figure 2, a wood piece 10 (made of false arbovitae; size 5 cm x 5 cm x 40 cm) was held between the front end 6 of the fructoconical portion of the suction tube 5 and the pressing plate 7, and the screw shaft 9 was tightened to cause the end portion 6 to bite into the wood piece 10. As a result, the filter unit F was built.

The closure 3 was closed, and the vacuum pump was operated to evacuate the inside of the receptacle 2 through the suction tubes 4 and 5. Thus, the inside of the receptacle 2 was maintained at 80 mmHg for 60 minutes. The suction tube 4 was then closed, and while the pressure reduction was continued only through the suction tube 5, a treating agent prepared by adding 600 parts of water-soluble urethane resin (Hydran HW-100, a tradename for a product of Dainippon Ink and Chemicals, Inc.) having an involatile content of 25% and 3 parts of Bismarck Brown (basic dye) to 393 parts of water was injected into the receptacle through the injection tube while applying a pres-

sure of 20 kg/cm² by the pressure appying unit 13, and this state was maintained for 60 minutes. As the injection proceeded water and air in the woody material were pushed out by the treating liquor until the treating liquor oozed out from the suction tube 5. At this point, the injection of the treating liquor into the woody material was completed.

Pressure application was stopped, and the pressure was returned to atmospheric pressure. The treating liquor was recovered from the discharge tube 12 at the bottom of the receptacle 2. The closure 3 was opened, and the treated woody material was taken out.

The rate of impregnation was 60%. When the treated woody material was cut, the cut surface was seen to be colored entirely. This indicated uniform impregnation. The treated woody material was dried under vacuum at 35°C for 36 hours, and left to stand at room temperature for 12 months. No cracking on drying occurred.

COMPARATIVE EXAMPLE 2

Example 2 was repeated except that the pressure reducing treatment through the filter unit F was not carried out. Specifically, the same woody material and treating liquor as in Example 2 were used, and the treatment was carried out by an ordinary pressure reduction and pressure elevation injecting method (60 minutes at 60 mmHg and 60 minutes at 20 kg/cm²).

The rate of impregnation was 20%. When the treated woody material was cut, the central part of the inside of the woody material was not colored at all, and no uniform impregnation was effected. The treated woody material was dried in vacuum at 35°C for 36 hours, and then left to stand at room temperature. Two weeks later, dry cracking occurred.

EXAMPLE 3

A Seabold's beach beam having a size of 4 cm x 10 cm x 200 cm was used as the material 1 to be treated. A solution composed of 800 parts of an unsaturated polyester resin for wood strengthening having a non-volatile content of 67% (a product of Dainippon Ink and Chemicals Inc.), 168 parts of styrene and 32 parts of benzoyl peroxide was used as the treating liquor. Makaba birch having a size of 4 cm x 5 cm x 40 cm was used as the wood piece (II) 10. The pressure reducing treatment was carried out at 120 mmHg for 30 minutes and the pressure applying treatment was carried out at 25 kg/cm² for 30 minutes. Otherwise, the injection of the treating liquor into the wood was carried out by the same method and apparatus as used in Example 1.

The rate of impregnation was 121%. The treated woody material was enclosed with an aluminum foil and heated at 80°C for 48 hours to cure the resin therein. Test pieces were sampled from the surface and inside of the end portion of the treated wood and the surface and inside of its central portion as in Example 1, and their tensile strengths (kg/cm²) were measured. The results are shown in Table 2.

COMPARATIVE EXAMPLE 3

The same injecting treatment as in Example 3 was carried out except that the pressure reducing treatment via the filter unit F was not carried out. Specifically, an ordinary pressure reducing and pressure applying treatments were carried out (30 minutes at 120 mmHg and 30 minutes at 25 kg/cm²).

The rate of impregnation was 46%. Test pieces were sampled from the treated woody material an Example 3, and their tensile strengths (kg/cm²) were measured. The results are shown in Table 2.

Table 2

| | End portion | | Central portion | |
|---|---|---|---|---|
| | Surface | Inside | Surface | Inside |
| Example 3 | 1,810 | 1,820 | 1,800 | 1,790 |
| Comparative Example 3 | 1,750 | 1,380 | 1,760 | 1,340 |

EXAMPLE 4

A Nara oak beam having a size of 10 cm x 20 cm x 150 cm was used as the woody material 1 to be treated. A solution of 34 parts of "Timbor" (a boron-containing antiseptic and pest controlling agent made by Borax Consolidated Limited, Britain) in 983 parts of water was used as the treating liquor. A Nara oak piece having a size of 5 cm x 5 cm x 40 cm was used as the wood piece II. The pressure reducing treatment was carried out at 70 mmHg for 90 minutes, and the pressure applying treatment, at a liquid pressure of 15 kg/cm² for 90 minutes. Otherwise the injecting treatment was carried out by using the same method and apparatus as in Example 2.

The rate of impregnation was 50%. The treated woody material was dried with hot air at 40°C for 24 hours and then at 60°C for 12 hours. After drying, the treated woody material was cut at its end portion and central portion, and a 0.05% alcohol solution of curcumin was sprayed onto the cut surfaces. On drying, the surfaces of the wood became yellow. A solution obtained by adding 4 g of salicyclic acid to 20 ml of hydrochloric acid and 80 ml of alcohol was sprayed. The entire surface became crimson. It was therefore ascertained that the treating agent was present throughout the woody material. This treated woody material showed excellent antiseptic and pest controlling effects.

COMPARATIVE EXAMPLE 4

The same injection treatment as in Example 4 was carried out except that the pressure reducing treatment through the filter unit F was not carried out. Specifically, an ordinary pressure reducing and pressure applying treatment was carried out (90 minutes at 70 mmHg and 90 minutes at 15 kg/cm²).

The rate of impregnation was 23%. After drying, the treated woody material was cut at its end and central portions, and the cut surfaces were examined by a color forming test as in Example 4. A part 10 to 20 cm from the periphery of the cut surface became crimson, but the central portion remained yellow. This showed that the impregnation of the treating liquor was non-uniform.

## Claims

1. A method of injecting a treatment liquid into a woody material to be treated, which comprises subjecting the woody material to a first pressure reducing treatment in a receptacle capable of being maintained at reduced or elevated pressure, and thereafter subjecting it to a second pressure reducing treatment in which reduced pressure is applied through a piece of wood, and in which the woody material is immersed in the treating liquid and pressure is applied to the liquid.

2. A method according to claim 1 wherein the woody material is subject to the first pressure reducing treatment in the presence of the treating liquid.

3. The method of claim 1 or 2 wherein the first pressure reducing treatment and the second pressure reducing treatment are carried out under an absolute pressure of not more than 200 mmHg.

4. The method of claim 1, 2 or 3 wherein the pressure applying treatment is carried out under a pressure of 1 to 100 atmospheres.

5. The method of claim 4 wherein the pressure applying treatment is carried out under a pressure of 8 to 50 atmospheres.

6. The method of claim 1, 2, 3, 4 or 5 wherein the piece of wood is prepared from the same wood as the woody material being treated.

7. Apparatus for injecting a treating liquor into a woody material to be treated, comprising a receptacle capable of being maintained at reduced or elevated pressure, a filter unit having a piece of wood as a filter, a pressure applying unit, and a pressure reducing unit.

8. Apparatus according to claim 7, including a gas-liquid separating unit.

9. The apparatus of claim 7 or 8 wherein the filter unit is within the receptacle.

10. The apparatus of claim 7 or 8 wherein the filter unit is outside the receptacle.

11. The apparatus of claim 9 or 13 wherein the filter unit comprises a suction tube, with its front end formed in a frustoconical shape, the piece of wood and a pressing plate and is assembled by interposing the piece of wood between the front end of the suction tube and the pressing plate.

12. The apparatus of any one of claims 7 to 11 wherein the pressure reducing unit comprises a pressure reducing nozzle block, and the nozzle block comprises a throat with a fan-shaped frustoconical rear end portion, a nozzle having a cylindrical portion facing the front end of the throat and a fan-shaped frustoconical portion following the cylindrical portion, a jetting hole to which the nozzle is connected, a gas discharge hole to which the rear end of the throat is connected, a vacuum chamber enveloping the front end opening of the

nozzle and the front end opening of the throat, and a suction hole communicating with the vacuum chamber, the ratio of the diameter of the throat to that of the nozzle being 1.5, and the distance from the front end of the nozzle to the front end of the throat being 0.1 to 1.0 mm.

Fig. 2

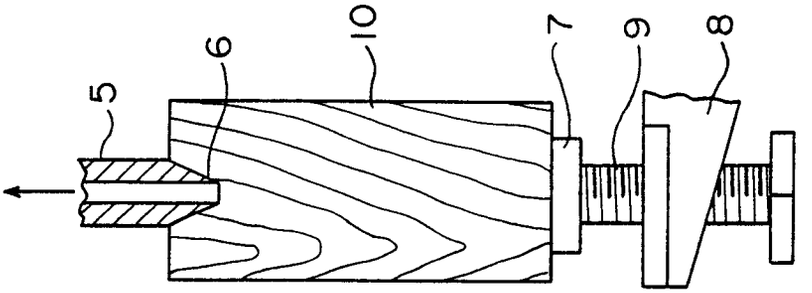



Fig. 1

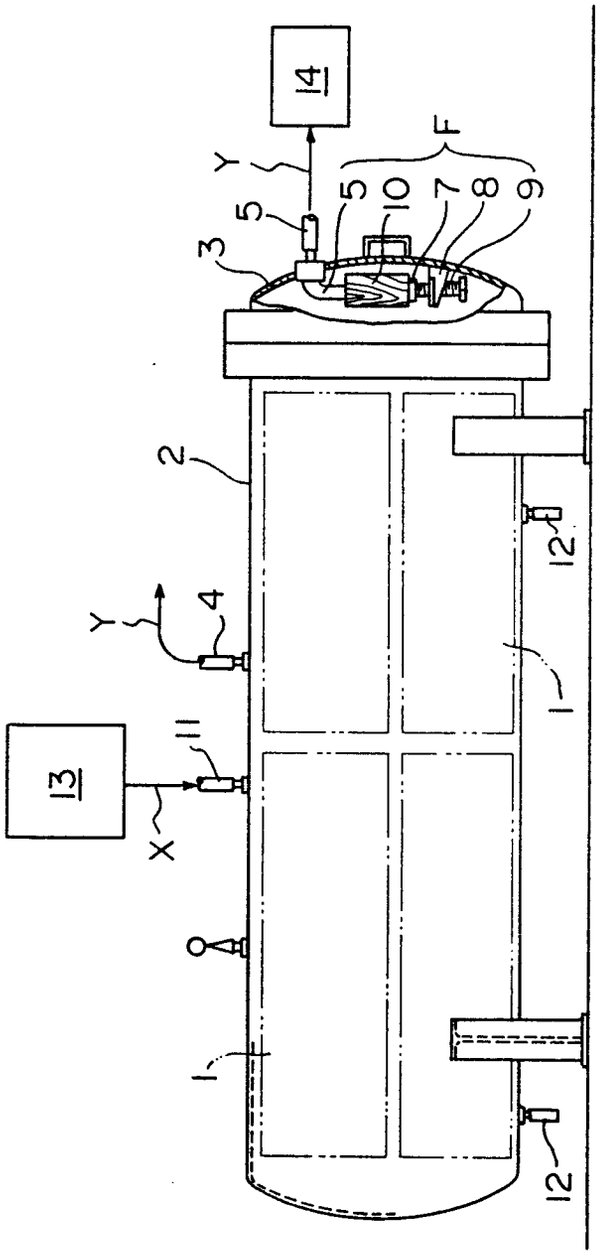
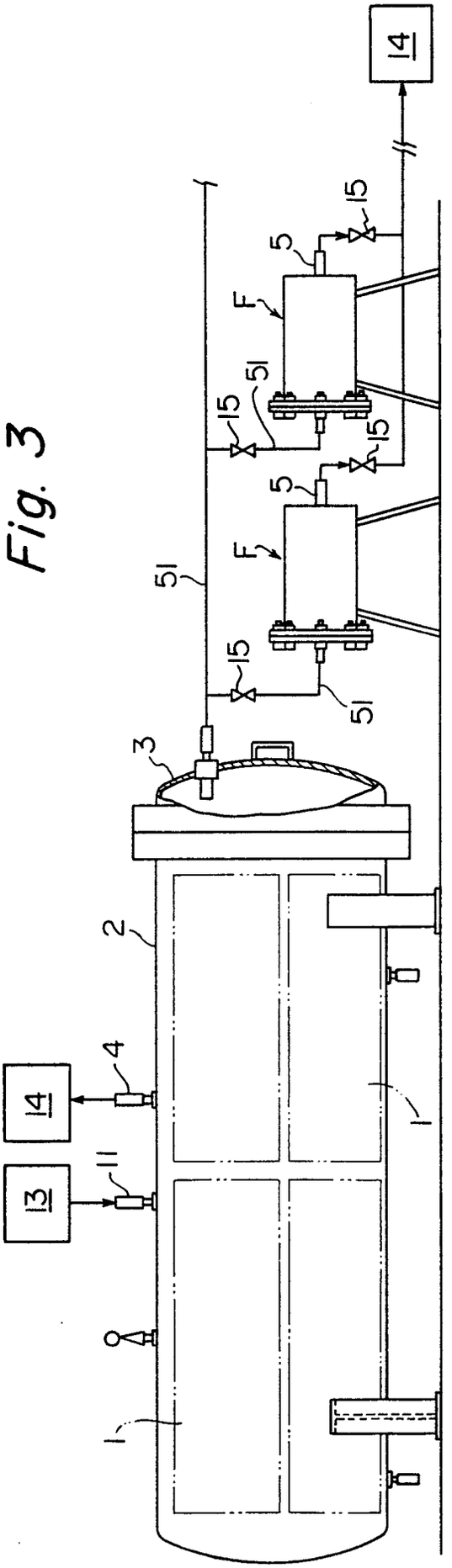


Fig. 3



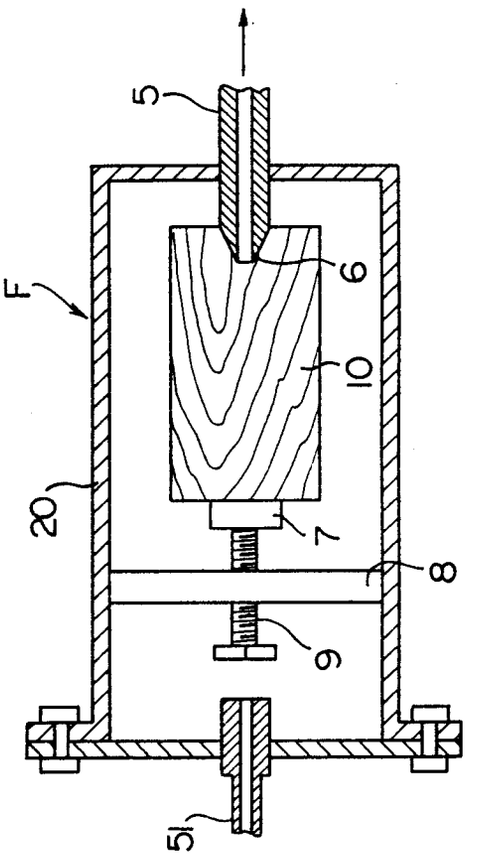




Fig. 4

Fig. 5

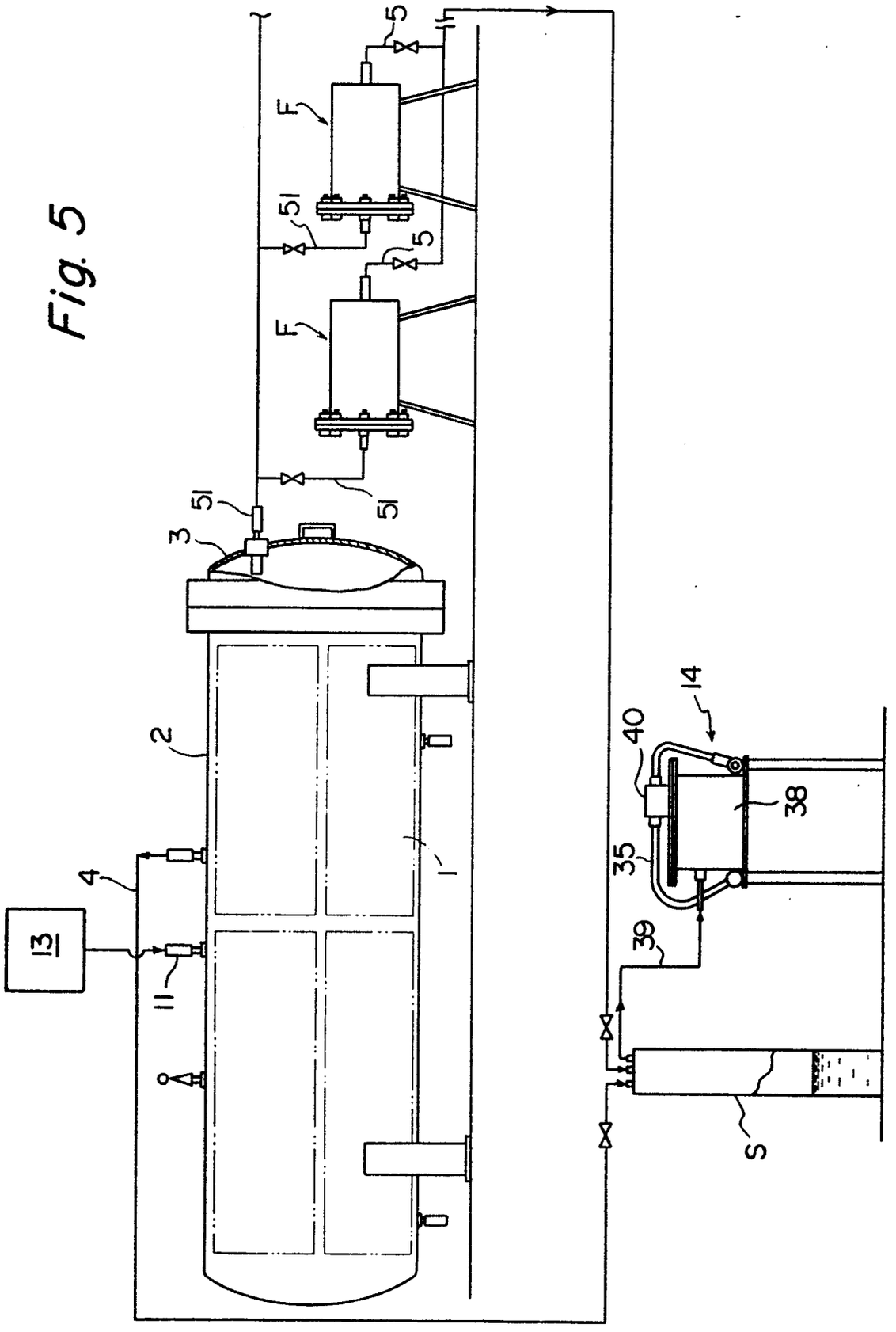
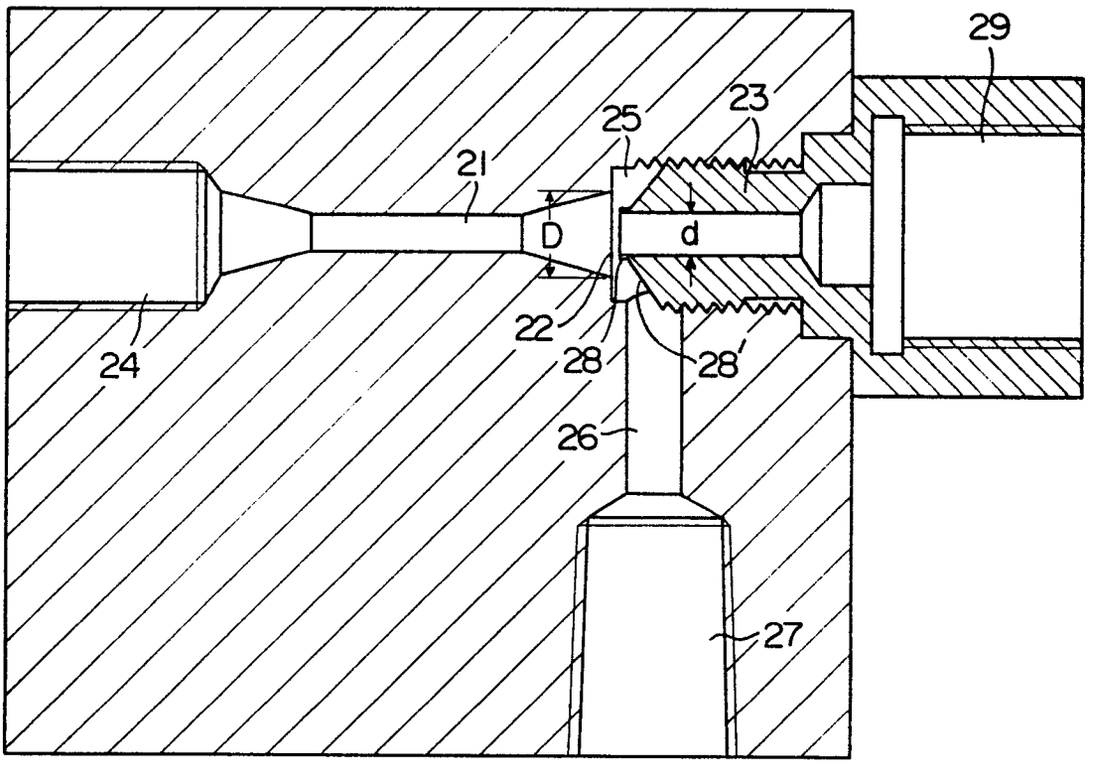


Fig. 6



European Patent Office

# EUROPEAN SEARCH REPORT

Application number

EP 86 30 5821

## DOCUMENTS CONSIDERED TO BE RELEVANT

| Category | Citation of document with indication, where appropriate, of relevant passages | Relevant to claim | CLASSIFICATION OF THE APPLICATION (Int. Cl.4) |
|---|---|---|---|
| A | GB-A-1 572 715 (BALFOUR BEATTY LTD.)<br>* Page 1, line 71 - page 2, line 5; page 2, line 88 - page 3, line 44; claims * | 1-6 | B 27 K 3/10<br>B 27 K 3/08 |
| A | DE-C- 862 663 (ATLASMASTE KG)<br>* Page 1, line 17 - page 2, line 20 * | 1-6 | |
| A | GB-A-1 169 958 (BOLIDEN AB)<br>* Page 3, line 78 - page 5, line 21; claims * | 1-6 | |
| A | GB-A-1 374 277 (BOLIDEN AB)<br>* Claims * | 1-6 | |

TECHNICAL FIELDS SEARCHED (Int. Cl.4)

B 27 K

The present search report has been drawn up for all claims

| Place of search | Date of completion of the search | Examiner |
|---|---|---|
| THE HAGUE | 06-11-1986 | FLETCHER A.S. |

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