## UNITED STATES PATENT OFFICE

1,995,623

## MANUFACTURE OF IMPREGNATED ARTICLES

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No Drawing. Application January 20, 1932, Serial No. 587,790

8 Claims. (Cl. 91—70)

There are classes of articles or products which are impregnated through the use of impregnants contained in an aqueous vehicle, the water serving as the solvent or suspending medium for the impregnant and further for carrying the impregnant into and through the articles or product. In such cases, the impregnant is usually either dissolved in the aqueous medium or is held in colloidal suspension therein so as to be capable of entering into articles or products whose foundation or base is more or less porous. The present invention relates to this class of impregnation and the impregnated articles or products realized therefrom.

In accordance with the present invention, I carry out such impregnation in the presence of so-called wetting-out agents, which may be applied to the base being impregnated either before or together with the aqueous vehicle con-20 taining the impregnant. After such impregnation has been effected, I then destroy the wetting-out agent in the sense that the agent is either removed or chemically decomposed subsantially in entirety and hence no longer functions in the impregnated article or product. While not limited thereto, my invention is especially useful in the treatment of fibrous foundations when it is desired to impregnate them with aqueous impregnants imparting such qualities as water re-30 pellency thereto. The presence of wetting-out agents during impregnation phenomenally increases the rate at which water penetrates into a fibrous foundation, and thus promotes a uniform impregnation of the foundation with the impregnant contained in the aqueous medium. When the impregnated product is dried, however, it is not possessed of the desired water repellency because of the presence therein of the wetting-out agent, which accelerates the penetration of water into the product even though a waterproofing agent is associated with the product throughout. By destroying the wetting-out agent after it has performed its function, as my invention requires, it is possible to arrive at a product having the desired water-repellent quality.

There are various wetting-out agents which are commercially available for use in accordance with my invention, for instance the wetting-out agents sold under the trade names "Nekal A," "Nekal AEM," "Indrapid," "Nekal S," "Nekal BX," "Neomerpin," "Neomerpin N," all of which are either benzene, naphthalene, or anthracene derivatives having side chains. Thus, Nekal BX, which is a highly effective wetting-out agent, is the sodium salt of a naphthalene sulphonic acid with side

chains. Nekal A is the crude sodium salt of octohydro-anthracene metasulphonic acid, while Indrapid and Nekal S are similar but contain the free acid. Nekal AEM is a combination of Nekal A with methylhexalin. Neomerpin N is a high molecular weight sulphonic acid. Some of these wetting-out agents can be readily decomposed by heat or by the use of suitable acids or alkalies, but these treatments should not be practised when the heat, acid, or alkalies necessary for such 10 destruction also injures the impregnant. For instance, an acid treatment should not be practised when the impregnant is rosin soap or other aciddecomposible body. On the other hand, an acid treatment in which the acid is, for instance, dis- 15 solved in water or exists as a vapor or gas phase, might be relied upon when inert waterproofing ingredients like asphalt, paraffin, or the like have been used in the form of aqueous dispersions as the impregnating media. All of the wetting-out 20 agents can be removed, however, from the impregnated foundation by leaching out with water, wherefore, after the impregnated foundation has been dried to fix the waterproofing ingredients therein in a water-insoluble state, the dried, im- 25 pregnated foundation can be steeped in a water bath until the wetting-out agent has been substantially completely extracted, and then redried. The water bath may be heated, if desired, to promote the extraction of the wetting-out agent from 30 the impregnated foundation.

The principles of the present invention may be applied to advantage, for example, in producing an effectively sized paper, especially when the starting paper base is not especially porous and 35 it is to undergo impregnation with aqueous impregnants of a colloidal nature, like rubber latex, aqueous glue or rosin size solutions, and aqueous dispersions of asphalts, waxes, resins, or the like, which do not penetrate the base with facility. In 40 such case, I incorporate Nekal BX, for example, into the paper. This incorporation can take place before the paper is dipped into the bath of aqueous sizing medium. On the other hand, the Nekal BX can be added directly to the bath so 45 as to enter into the paper simultaneously with the size. In either event, the wetting-out agent, even when used in exceedingly small amount, vastly enhances the speed and uniformity of penetration of the size into and through the 50 paper. For example, as little as, say, from 0.05% to 0.25% of Nekal BX added to a sizing bath works an astonishing increase in penetrability of the size into and through the paper. The sized paper is then dried and the Nekal BX deposited 55

therein may be destroyed by suitable heat or acid treatment, or by extraction with water, the particular mode of destruction depending on the specific sizing agent used. When gelatin or glue 5 is used as a size, a suitable tanning or insolubilizing agent, e. g., alum, for such size may be added with the size or subsequent to the addition of the size for the purpose of inhibiting undesirable action upon size of water or aqueous chemical solu-10 tions subsequently employed for extracting the wetting-out agent. The resulting sized paper possesses a surface which can be satisfactorily written or printed upon with the usual kinds of printing or writing inks made up with water as the ink vehicle. Had the Nekal BX been permitted to remain in the paper, the printing or writing with such inks on the paper would have been attended by a highly objectionable spreading of the ink and the formation of feathered or

20 blurred imprints. There are instances when a paper or other foundation has already undergone impregnation with waterproofing ingredients like those already mentioned and so is difficultly, if at all, penetrable 25 even by aqueous solutions of compounds which may be desirable secondary impregnants. For example, a web of interfelted fiber may have been bituminized, as by dipping into molten asphalt, to produce a dense, impregnated sheet highly 30 resistant to penetration by water on account of the fibers being enveloped by a continuous bitumen phase amounting by weight to much more than even the fibers. Yet it might be desirable to incorporate into the bituminized sheet 35 agents from an aqueous vehicle, for instance, agents like the phosphates and tungstates, which are water-soluble and which impart fire resistance to the sheet. The property of fire resistance is, of course, desirable when the 40 bituminized sheet is to be used as a building material, e. g., on a roof. I have found that in this connection the use of Nekal BX or equivalent wetting-out agent in amount of, say, 0.1% to 0.2% in the aqueous vehicle containing a fire re-45 tardant or other secondary impregnant is most advantageous, as it makes possible the desired secondary impregnation. A bituminized sheet which has thus been impregnated with phosphates or tungstates as the fire retardants con-50 tain these retardants where they are most effective in their action, namely, on the bitumen phase, which is the phase first exposed to the fire. The bitumen phase contributes to the fire hazard properties even more than the fiber foundation. 55 especially when present in amount much more than the fiber, as is usually the case in bituminized webs for roofing or other building pur-Moreover, it is easy to diminish the combustibility of the fibrous base by making it partly or entirely of incombustible fiber, such as asbestos, or if made of combustible fiber such as cellulose, by treating such fiber with fire retardants of the type mentioned. The destruction of the wetting-out agent after the drying of the 65 bituminized sheet containing fire retardants as the secondary impregnants, as by heating, is of course most desirable, since otherwise the wetting-out agent would promote the imbibition of water by the sheet, and would thus depreciate 70 the value of the sheet for roofing or other purposes where water resistance is required. Moreover, the presence of a wetting-out agent in the sheet would conduce to a leaching out by water of water-soluble fire retardants incorporated 75 into the sheet.

In those cases where a foundation of cellulose fibers is impregnated in the presence of a wetting-out agent with rubber latex and/or aqueous dispersions or emulsions of asphalt, paraffin, or other bodies which are practically inert with respect to strong solutions of alkali, the wetting-out agent may be destroyed, after the impregnated foundation has been dried, by strong solutions of alkali, for instance, solutions of caustic soda of about 10% to 18% or higher concentration. The 10 treatment with such caustic soda solutions can be carried out at room temperature and be followed by the use of wash water to remove the alkali.

The foundation material being treated in accordance with the present invention need not necessarily be fibrous, although, so far as I am now aware, the greatest utility of my invention resides in the treatment of fibers of vegetable, mineral, or animal origin, in bulk condition or 20 in the form of fibers integrated either on textile machines as bats, felts, or woven fabrics, or on machines of the papermaking class as webs or papers of interfelted texture.

I claim:

1. A process which comprises impregnating a base in the presence of a wetting-out agent with an aqueous medium containing a dispersed waterproofing agent for said base, drying the impregnated base, and destroying the wetting-out 30 agent in said impregnated base.

2. A process which comprises impregnating a fibrous sheet in the presence of a wetting-out agent with an aqueous medium containing a water-repellent size for said sheet, drying the sized 35 sheet, and destroying the wetting-out agent in said sheet.

3. A process which comprises impregnating a fibrous sheet carrying a water-repellent impregnant in the presence of a wetting-out agent with 40 an aqueous medium containing a second impregnant for said sheet, drying the sheet containing both impregnants, and destroying the wetting-out agent in said sheet.

4. A process which comprises impregnating a 45 bituminized fibrous sheet in the presence of a wetting-out agent with an aqueous solution of a fire retardant, drying the impregnated sheet, and destroying the wetting-out agent in said sheet.

5. A process which comprises impregnating a 50 bituminized fibrous sheet in the presence of the wetting-out agent with an aqueous solution of a fire retardant and drying the impregnated sheet.

6. A process which comprises impregnating a 55 base in the presence of a wetting-out agent with an aqueous medium containing a dispersed, thermoplastic waterproofing agent for said base, drying the impregnated base, immersing the dried base in water to leach out the wetting-out agent 60 therefrom, and re-drying the base.

7. A process which comprises impregnating a bituminized fibrous sheet with an aqueous solution of a fire retardant in the presence of a wetting-out agent destructible by heat, drying the 65 impregnated sheet, and heating it to destroy said wetting-out agent.

8. A process which comprises impregnating a bituminized fibrous base made up at least in part of asbestos fiber with an aqueous solution of a 70 fire retardant in the presence of a wetting-out agent destructible by heat, arying the impregnated base, and heating it to destroy said wetting-out agent.