Sept. 7, 1926. 1,598,640

W. W. CARTER

METHOD OF IMPREGNATING CELLULOSE FIBERS

Original Filed Dec. 27, 1922

Fig. 1.

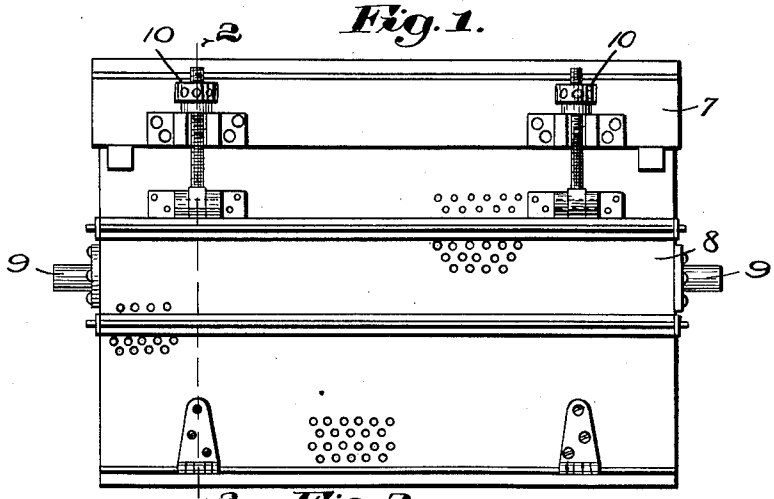


Fig. 2

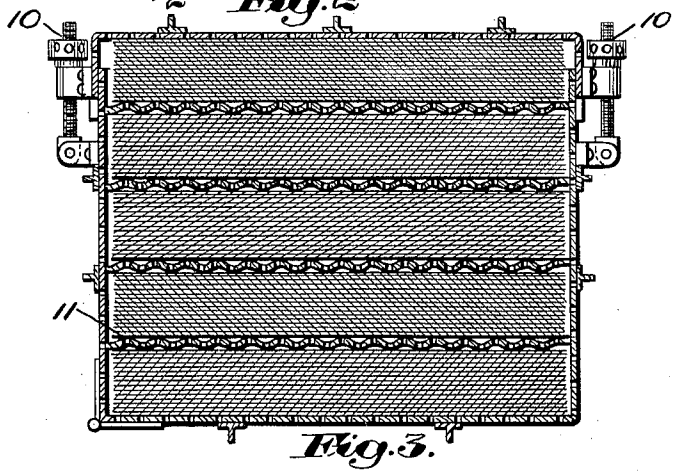
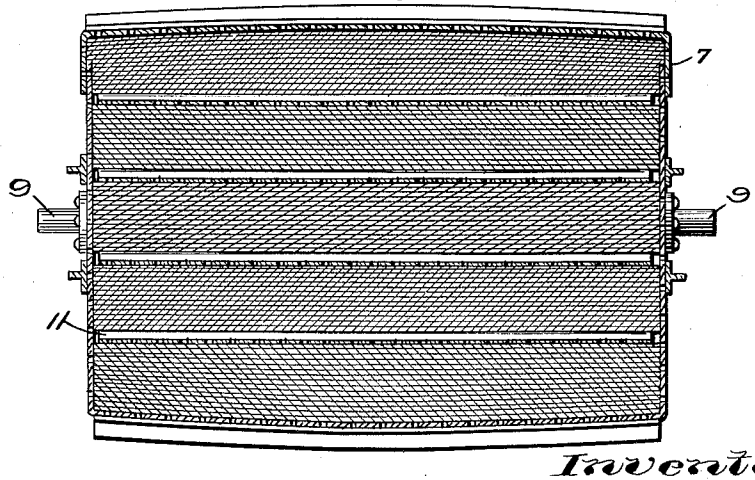


Fig. 3.



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Patented Sept. 7, 1926. 1,598,640

UNITED STATES PATENT OFFICE.

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METHOD OF IMPREGNATING CELLULOSE FIBERS.

Original application filed December 27, 1922, Serial No. 609,297. Divided and this application filed July 22, 1924. Serial No. 727,583.

This invention pertains to improvements in method of impregnating cellulose fibers. More particularly, though not exclusively, the invention relates to steps in the manufacture of waterproof paper rendered tough by a binder including fibers toughened by the thiocarbonate or xanthate reaction.

The application is a division of my prior application, Serial No. 609,297, filed December 27th, 1922.

In the drawings, which show a preferred form of one embodiment of an apparatus in which my preferred method may be carried out:

Figure 1 shows a side elevation of a crate adapted to contain the fibrous stock during chemical reaction and physical admixture of various elements.

Fig. 2 is a section on the line 2—2 of Fig. 1, showing the crate with sheets of paper stock enclosed therein and ready for immersion in the treating bath; and

Fig. 3 is a section of the crate and contents on the line 3—3 of Fig. 2 after treatment in the chemical bath and illustrating the limitation of the swelling of the contained fiber stock.

Waterproof papers are ordinarily made from paper stock and gum, such, for instance, as resin size. It is among the purposes of the present invention greatly to increase the strength of such waterproof papers. It is well known that paper stock treated by the xanthate or thiocarbonate methods is exceedingly strong, but such paper is not waterproof. It has been attempted to waterproof papers or similar products made partially or entirely from stock treated by thiocarbonate reaction, but great difficulty has been experienced because the gum sizes will not permeate the mass of stock which has been subjected to the xanthate reaction. Moreover, where stock already treated and aged by the xanthate reaction is not in and of itself in any way fortified against the absorption of water, a mere coating of size on the outside of fibers so treated is exceedingly susceptible to the action of water because, if there is the slightest break in the waterproof coating of the fiber, the water will penetrate, cause the fiber to swell, break the waterproof gum coating and lose all protection.

It has previously been practised to treat paper stock successively with alkaline gum emulsions and cellulose xanthate to provide a binder stock which is later mixed in the beater with the main body of paper stock and the gum emulsions. While the product provided by this method is to some extent waterproof and very tough, it is not entirely waterproof because the stock forming the binder and which has been subjected to the xanthate reaction is very absorbent, in fact, being more absorbent than the original unsized fiber.

I have found that very satisfactory results are obtained by providing a binder of fibers which are permeated with a waterproof size and also more or less xanthated. The size cannot be satisfactorily introduced after the xanthate reaction.

I have found that while xanthated fibers may be rendered exceedingly tough by completing the xanthate reaction, and while they may be rendered exceedingly absorbent by the xanthate reaction, the xanthated fiber when in such condition as to be adherent is ordinarily readily dissolved in water and therefore to an exceedingly large extent washed away and lost in the paper machine.

The solubility of the xanthated fiber is to a large extent dependent on the amount of absorption during treatment with sodium hydroxide as the initial step in the xanthate method. The more sodium hydroxide that is absorbed and combined during this operation, the greater is the absorptive property of the stock if later subjected to water. It has been heretofore attempted to limit the amount of sodium hydroxide in the fibers by pressing the fibers after they have been immersed in the sodium hydroxide or equivalent treating agent. It has also been attempted to limit this absorption by only supplying to the fiber the desired amount of the sodium hydroxide. The first of the above-described prior methods, while satisfactory for artificial silks, has proved unsatisfactory for paper-making processes because the entire structure of each fiber is effected by the sodium hydroxide and the whole fiber is therefore soluble in water when the xanthate reaction is completed. The second of the above-described mehods is unsatisfactory because it is a physical impossibility, regardless of the extent of kneading, measuration or mixing effected, to distribute evenly the fluid among the mass of fiber stock. Those portions of the fiber stock

which first receive the liquid become saturated, while the remainder are insufficiently treated even as to their surface.

I have found that where I limit the action of the chemical on the fibrous stock by confining the stock so as to limit the swelling thereof during the chemical reaction of the sodium hydroxide, that the surface of each of the fibers is to the desired extent acted upon by the sodium hydroxide and therefore conditioned to complete the xanthated reaction when immersed in the carbon disulphide. On the other hand, the interior of each fiber has not been enabled to swell to any considerable extent and for this reason has not permitted entrance therein of the treating fluid and is not in condition fully, or even partially in some instances to be affected by the reaction when immersed in the carbon disulphide. While some of the treating fluid reaches the centre of a fiber, the alkaline content of the fluid reaching the centre is so reduced, by combination with the exterior portions of the fiber through which it has passed, as to minimize any reaction; such fluid is, however, such as to maintain the gum emulsion. The paper stock may, if desired, be confined in a container adapted to limit swelling in one dimension only, as by exerting pressure on the top and bottom only, the crate or container being without sides. I prefer, however, to limit swelling in all directions. Where each fiber provides in its interior a non-xanthated or partially xanthated core, the chemical bond of the xanthated surface of the fiber for the core is so great as to overcome the tendency which would otherwise exist, for the xanthated portions of the fiber to be dissolved in the water.

In practicing the specific embodiment of my invention selected for illustrative purposes I take ordinary paper stock, which may be in sheet or board form, and place any desired number of sheets or boards in a crate. Referring to Fig. 1, the crate formed of perforated metal, is provided with a removable top 7 and a hinged front 8, the ends, bottom and back preferably fixed and the ends provided with trunnions to be engaged by suitable tongs on a crane or hoist. In loading, about four inches of the sheets or boards are first placed on the bottom, a separator 9 is then introduced, another four inches of stock above this and so on until the crate is filled, when the side 8 is swung up and the top 7 is screwed down by suitable clamping devices. The separators 11 are preferably corrugated and perform the function of permitting the liquid readily and quickly to flow to the inner portion of the crate. The fiber stock in sheet or board form is so loosely pressed that capilary action of the liquid causes entrance thereof to the center of the four inch layers of sheets or boards very quickly. When the pulp sheets and separators are all in position and the crate closed, I immerse the entire crate in a vat containing the desired chemicals, which may be a 15% solution of sodium hydroxide forming a carrier for an emulsion of a suitable size which may be resin or other gum commonly used as a paper size. Where the desired ultimate product is not ordinary paper or paper board, but one having electrical insulating properties, I may utilize an emulsion of artificial gums or resins suitable for dielectrics. For purposes of this application, however, all products of sheet, molded or other form containing cellulose fibers may be considered a paper.

The sizes when in the emulsion are very finely divided particles having little if any adhesive properties. They are, however, so small as to permeate within the structure of the individual fibers of the stock.

The crate of paper stock may be left in the bath of 15% sodium hydroxide solution until it has swelled sufficiently completely to fill and to exert some pressure on the crate. This expansion may preferably be such as to provide ninety cubic inches of swelled cellulose fiber for each pound of stock introduced into the crate. The crate is preferably left in the fluid with the sheets of paper stock in such position as to permit ready and complete escape of air, such position preferably with the sheets vertical. The crate may then be removed, its top and bottom released and its contents permitted to age until the reaction is complete. The reaction will be complete in about twelve hours under normal conditions, and may continue for a longer period so long as the stock does not dry out and so long as carbon dioxide of the air does not combine with the sodium and prevent proper reaction therewith of the carbon disulphide. When aging is complete, the reaction may be completed by immersing the stock in carbon disulphide for a sufficient period, usually three or four hours. The stock may be somewhat broken up or shredded prior to its treatment with carbon disulphide. After completion of the xanthate reaction, water is added to make a water paste which will just flow out of the containers into the beaters. To provide this, approximately twice its own weight of water is added to the stock. This paste is thoroughly mixed and goes to the beaters where it is mixed with the remaining untreated paper stock and with the ordinary sizing. The operation from this point on may be identical with that ordinarily used in manufacturing paper products. Where the emulsion of the gum does not break in the beater, but has to be broken by chemicals, it is important that the chemicals used to break the emulsion should be such as do not

affect the xanthate. Thus, the use of alum and sulphuric acid should be avoided and acetic acid or sulphurous acid or sodium hydrogen sulphite or hyposulphites may be used in their place or similar salts of that nature.

While I have shown and described apparatus which constitutes a preferred embodiment of my invention, and while I have described a preferred way of practicing my improved method, it will be understood that major changes in the apparatus and even changes in the method described may be made without departing from the scope of my invention, which is best defined in the following claims.

1. The method of limiting the action of chemicals on fibrous stock which comprises loosely confining the stock to permit some swelling thereof, while limiting the swelling of the stock thereby controlling the chemical action thereon.

2. The method of limiting the depth of the xanthate reaction on paper stock which comprises loosely confining the stock to permit some swelling thereof, while limiting such depth of reaction by limiting the ultimate swelling of the stock.

3. The method of limiting the action of chemcals on fibrous stock which comprises loosely confining the stock in a container adapted to admit the treating fluid, immersing the container in the treating fluid and permitting absorption until the stock in swelling exerts pressure on the container and then removing the container with the stock from the fluid.

4. The method of limiting the action of chemicals on fibrous stock which comprises loosely confining the stock in a container or holder adapted to admit the treating fluid, immersing the container in the treating fluid and permitting absorption until the stock in swelling exerts pressure on the container and then removing the container with the stock from the fluid, permitting drainage and removing the stock from the container for aging or completion of the desired organic reaction.

5. The method of limiting the action of a solution of a reactive chemical on fibrous stock which comprises confining the stock in a container which permits some swelling of the stock while it opposes, in at least one dimension, swelling of the stock beyond a desired point when absorbing the solution and thereby limits the amount of reactive chemical which acts upon the body of the stock.

6. The method of holding pulp sheets during chemical treatment thereof which comprises confining the sheets in a container which opposes swelling of the sheets beyond a desired point, while admitting the treating liquor.

7. The method of limiting the action of chemicals on fibrous stock which comprises loosely confining the stock in a container or holder adapted to admit the treating fluid, applying the treating fluid to the material in the container and permitting absorption until the stock in swelling exerts pressure on the container and then discontinuing treatment of the stock by the treating fluid.

In testimony whereof, I have signed my name to this specification.

WILLIAM W. CARTER.