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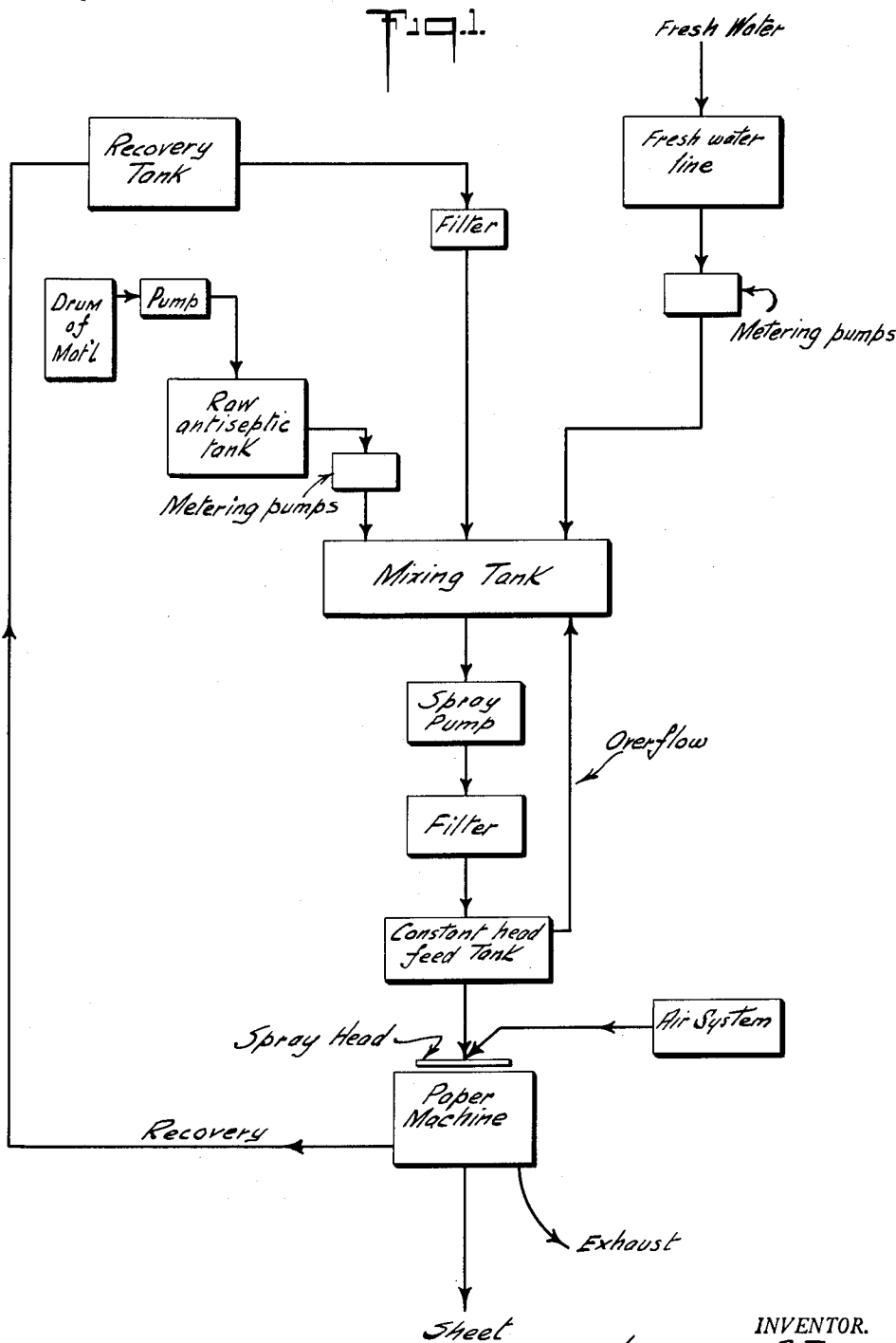
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METHOD OF INCORPORATING QUATERNARY AMMONIUM COMPOUNDS IN PAPER

Filed July 11, 1952

2 Sheets-Sheet 1



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2 Sheets-Sheet 2

Fig. 2.

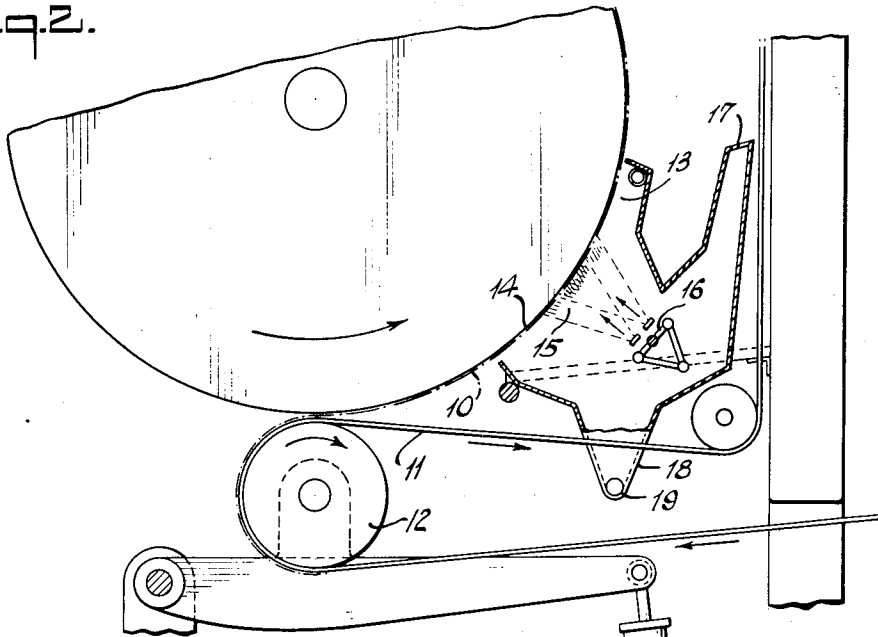


Fig. 3.

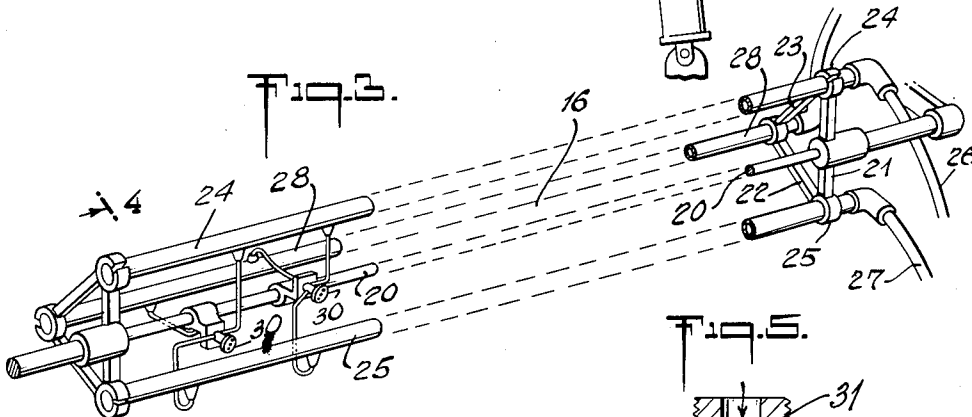


Fig. 4.

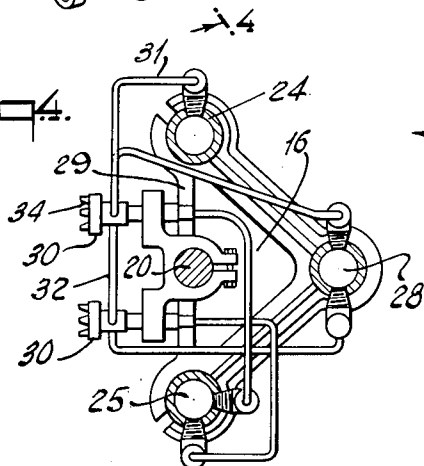
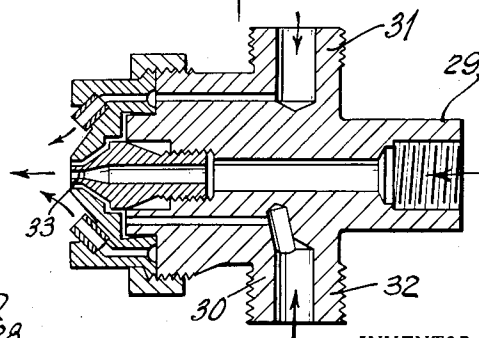


Fig. 5.



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1

2,756,647

METHOD OF INCORPORATING QUATERNARY AMMONIUM COMPOUNDS IN PAPER

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5 Claims. (Cl. 92—40)

This invention relates to a method of incorporating quaternary ammonium compounds into soft paper tissue.

Quaternary ammonium compounds of the type that have at least one long chain alkyl radical in the molecule exhibit important bactericidal and deodorizing activity and accordingly the incorporation of substantial quantities of such compounds into soft paper tissue has become important in industry. In order to take advantage of the beneficial properties of quaternary ammonium compounds it is important that these compounds be distributed substantially evenly throughout the soft paper tissue. On the other hand, quaternary ammonium compounds are not usually compatible with the chemicals used for best results in paper stock systems and can not be present in such systems except in very low, ineffective concentrations. Many past efforts to incorporate the quaternary ammonium compounds in the white water or with the fiber prior to formation of the paper tissue have shown that these compounds accumulate in the white water forming dark, gummy substances in the nature of pitch. This accumulation results in the formation of unsatisfactory paper products presenting pitch marks, dark specks and holes in the paper, and prove costly as they necessitate frequent shutdowns and cleaning of the equipment. The accumulations also interfere with the proper working of the water clarification system.

Another difficulty encountered in prior art efforts to incorporate quaternary ammonium compounds prior to formation of the complete sheet lies in the uneven distribution of the quaternary ammonium compounds throughout the tissue finally formed. Thus there is no direct control over the amount of quaternary ammonium compound in each tissue area. Such operation is obviously unsatisfactory where a controlled effect of the bacteriostatic agent is desired. Last but not least in importance, prior efforts, because of the difficulties mentioned above, resulted in excessive loss of the costly chemicals intended for tissue treatment.

Moreover, incorporation of the quaternary ammonium compound into the completed paper tissue results in substantial waste in view of the difficulties of additional wet handling of paper tissue and requires additional wetting and drying steps. Such additional treatment causes loss of strength in the final product, if indeed it is possible to avoid tearing of the tissue when the compounds wet it. Preservation of sufficient strength, however, is particularly important in tissues treated with quaternary ammonium compounds as any treatment with quaternary ammonium compounds results in some decrease in the tensile strength of the sheet.

Accordingly, it is an object of this invention to provide a method for making a bacteriostatic paper tissue. It is a further object of the invention to provide a method for making a paper tissue treated uniformly with quaternary ammonium compounds. Still further objects of the invention reside in a method of providing paper tissue that shows even distribution of substantial quantities of

2

quaternary ammonium compounds and that has the quaternary ammonium compound distributed all over its surface and into its interior. Yet a further object of the invention is the preparation of a quality paper tissue impregnated with quaternary ammonium compound free of pitch marks and dark spots and without the substantial accumulation of pitch substances in the white water. Another object of the invention is the economical preparation of paper tissue treated with quaternary ammonium compound. Still other objects of the invention will be obvious from the consideration of the following specification particularly when it is considered in conjunction with the drawing wherein similar numerals refer to similar parts throughout the several views shown and wherein:

Fig. 1 is a schematic view of the process;

Fig. 2 is a vertical sectional view showing a mechanism for incorporating quaternary ammonium compound into paper tissue in accordance with applicant's invention;

Fig. 3 is a fragmentary perspective view of a spray head for incorporating quaternary ammonium compounds into paper tissue in accordance with the invention;

Fig. 4 is a vertical section through lines 4—4 of Fig. 3;

Fig. 5 is a vertical sectional view through the center of one of the nozzles of a spray head.

In accordance with the invention use is made of the relatively high specific gravity and boiling point of quaternary ammonium compounds and also of their cationic characteristics which cause them to attach themselves under certain conditions to paper tissue and to remain with this tissue even after prolonged wetting and drying. The tissue 10 is first formed on the screen (not shown), then carried by the felt 11 resting on rolls such as roll 12 and dried partially to the point where from fifty to eighty-five per cent moisture and preferably sixty to seventy-five per cent moisture remains in the tissue, all in absence of treatment with quaternary ammonium compounds, and the quaternary ammonium compound is applied to this partially moist tissue at this particular stage by intensive spray in a restricted area 13 just before the final drying step on the drum drier 14.

A typical process is illustrated in schematic Fig. 1. In accordance with this schematic illustration the material passes through a pump into the tank of raw antiseptic and is distributed in a mixing tank which combines it with the correct proportion of water by means of metering pumps. For a typical operation the percentage of quaternary ammonium compound in the water in the mixing tank may vary from five-tenths of one per cent to about ten per cent by weight. Most quaternary ammonium compounds mix fairly easily with water although special prolonged agitation may be necessary under certain circumstances.

From the mixing tank the aqueous solution passes through a spray pump into a filter for removal of any impurities. This filter may be of the full or continuous flow type involving for instance passage of the material through cotton twine material. Other filters, particularly cartridge type filters, are of equal utility provided they are used properly. After the filter the solution goes into a feed tank having a head. In case of overflow a small part of the solution returns from here to the mixing tank by way of an overflow tube. From the feed tank the material passes into the spray head where it is mixed with air in an atomizing process that will be described and illustrated more fully later on in this specification.

The spray head applies the atomized solution against the partially dried formed tissue. Material that is not absorbed by the paper tissue is caught within the catch pan or flows over the side of the hood and is recovered into a recovery tank from which it returns through a filter such as a filter press into the mixing tank.

In accordance with preferred embodiments of the inven-

3

tion the solution is applied in atomized spray form to the paper 10 through the upward spray 15 of the spray head 16 upwardly against a large drum drier 14. At this point spray head 16 and spray 15 are enclosed upwardly by the paper 10 backed by the drum drier 14 on one side and on the other side by a hood 17 blocking passage of the spray into the outside air. Downward escape of unused solution is prevented by a catch pan 18 and by a drain 19 leading to the recovery tank. Use is made at this point of the high specific gravity and boiling point of the spray material which discourages its escape into the outside air if reasonable precautions are taken to enclose the area. An airtight connection between parts is usual at this point, but is not absolutely necessary. In some embodiments of this invention a suction pump may be provided with the hood so as to discourage escape of the solution. The suction outlet pulls the vaporized solution away from the hot drier surface to speed its condensation in a cooler area.

The spray head is best illustrated in Figs. 3 and 4. It consists of a supporting rod 20 resting on channel iron 21 upon which in turn rest connecting channel irons 22 and 23 which support three headers. The two outside headers 24 and 25 supply air from controlled pressure lines 26 and 27. The air pressure used is usually about fifteen pounds per square inch and may vary conveniently from eight to thirty pounds. The third, central header 28 supplies solution from the feed tank and in turn supplies the leads 29 to the individual nozzles 30.

These nozzles 30 are each connected to two air lines 31 and 32 and one solution line 29. As the solution passes under pressure through the orifice 33 out of the nozzle 30 it contacts air blown against it from both sides under pressure and is thus atomized just at the point where it takes its upward journey toward the partially dried paper tissue.

Each spray head 16 has a substantial number of nozzles. Thirty to sixty nozzles may be used conventionally and two or more banks of nozzles may be used if desired as illustrated in Fig. 4. Contacting the paper in this manner the solution is well distributed throughout the paper and is easily absorbed because of the moist condition of the paper and because of its elevated temperature as it is then undergoing the drying process. Typical temperatures of the paper at this spot are 160–190° Fahrenheit and the speed of the paper going past the spray head may be approximately 1,200 feet per minute, with variations from a few hundred to several thousand feet per minute. These conditions may be varied for various machines, processes and special circumstances.

The hood 17 and the catch pan 18 collect a very high percentage of the unused solution and prevent any escape of the unused solution into the white water or any other part of the paper machine. As has been explained above the drain of the catch pan returns unused material through the recovery tank and the filter into the mixing tank from where it returns to the spray head through the system described above. This final step including the two filters eliminates fibrous impurities from the solution and acts to avoid frequent blocking of the fine orifices of the nozzles.

In preferred embodiments of the invention the quaternary ammonium compounds used are alkyl dimethyl benzyl ammonium chloride, alkyl methyl pyrrolidinium chloride, dimethyl benzyl ammonium chloride, parateritary ethoxy ethyl dimethyl benzyl ammonium chloride, alkyl tolyl methyl trimethyl ammonium chlorides and other alkyl aryl methyl trialkyl ammonium chlorides wherein the carbon atoms in the alkyl group run from about nine to about fifteen and wherein the trialkyl group consists of methyl and/or ethyl; and alkyl aroxyl ethoxy ethyl dimethyl benzyl ammonium chlorides such as diisobutyl phenoxy ethoxy ethyl dimethyl benzyl ammonium chlorides, or, sometimes diisobutyl cresoxy ethoxy ethyl dimethyl benzyl ammonium chloride. The problem of

4

incorporating such compounds is particularly acute where more than one tenth of one per cent is to be incorporated into the tissue. Usable ranges of such compounds comprise upward of fifteen hundredths of one per cent of the quaternary ammonium compound and preferably not more than five tenths of one per cent by weight. More than one of the treating agents under discussion may be used at one time within the suggested quantity limitations. While certain of the quaternary ammonium compounds have been particularly listed above the invention is broader than quaternary ammonium compounds alone in many of its aspects and seems equally applicable to the incorporation of other heavy cationic agents into tissue.

Using quaternary ammonium compounds it has been found most advantageous to dilute the standard compounds to a concentration of from one to about three per cent by weight in aqueous solution or dispersion and apply it through the spray in this concentration. The pressure of the air at the nozzle may vary somewhat depending upon the particular treatment effect desired but air pressures of from three to twenty pounds per square inch are usual to spread and atomize the liquid from the spray nozzles. A typical bank of nozzles may be located about three feet away from the wet end of the drum drier. Experimental measures using the methods described in this application show that about thirty-five to seventy-five per cent of the spray is usually absorbed by the tissue with the remainder of the spray being condensed from the drier's upward flow and returned through the system.

Experiments show that use of the particular quaternary ammonium compounds described above results in elimination of bacterial growth and particularly in elimination of those types of bacteria usually present in the bacterial spectrum of the common cold. Among bacteria thus normally present and absent in view of the use of applicant's process are: *Staphylococcus*, *Streptococcus*, *pneumococcus*, *Micrococcus catarrhalis* and *influenza bacilli*, *diphtheria vacilli* and *meningococci*.

The invention has been described in its preferred forms. Many other embodiments will be obvious from the above description.

The claims are:

1. A process of uniformly incorporating into soft tissue a heavy cationic quaternary ammonium compound, comprising the steps of diluting the quaternary ammonium compound in water to a concentration of from about one to about three per cent, partially drying the tissue after its formation to the point where it comprises from about fifty to about eighty-five per cent moisture, and force spraying the dilute metered compound from the nozzle of an atomizer against said partially dried tissue.

2. A process of uniformly incorporating into soft tissue a heavy cationic quaternary ammonium compound, comprising the steps of diluting the quaternary ammonium compound in water to a concentration of from about one to about three per cent, partially drying the tissue after its formation to the point where it comprises from about fifty to about eighty-five per cent moisture, force spraying the dilute metered compound from the nozzle of an atomizer against said partially dried tissue said spray being within a relatively narrow and closed area, collecting the sprayed material not absorbed by said tissue and recirculating it through filters for renewed spraying.

3. A process of uniformly incorporating into soft tissue a heavy cationic quaternary ammonium compound, comprising the steps of diluting the quaternary ammonium compound, in water, to a concentration of from about one to about three per cent, partially drying the tissue after its formation to the point where it comprises from about fifty to about eighty-five per cent moisture, force spraying the dilute metered compound from the nozzle of an atomizer against said partially dried tissue said spray being within a relatively narrow and closed area, collecting the sprayed material not absorbed by the

5

tissue using a vacuum and recirculating it through filters for renewed spraying.

4. In the process of applying heavy cationic quaternary ammonium compounds to paper tissue, the step of spraying in atomized form onto the wet tissue prior to completion of the original drying steps a dilute solution of a heavy cationic quaternary ammonium compound and then drying said tissue so as to provide the dried tissue with from about fifteen-hundredths of one per cent to about five-tenths of one per cent of quaternary ammonium compound. 5 10

5. In the process of applying heavy cationic quaternary ammonium compounds to tissue during the paper-making process, the step of force spraying in atomized form a dilute metered aqueous solution of the cationic material 15

6

into the moist tissue at elevated temperature within a confined relatively narrow area.

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