Sept. 23, 1930. G. W. BLANCO ET AL 1,776,536

SHEET FOR THE MANUFACTURE OF VISCOSE

Filed Nov. 4, 1927

Fig. 1.

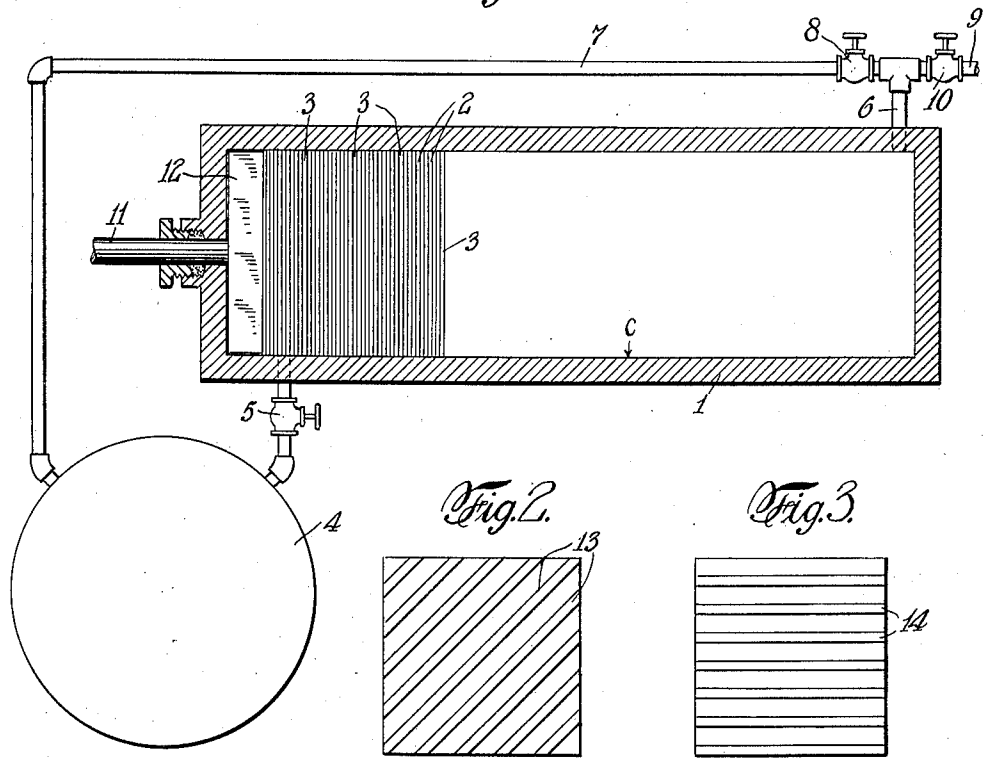


Fig. 2.

Fig. 3.

Fig. 4.

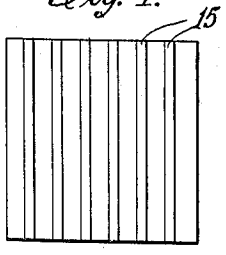


Fig. 5.

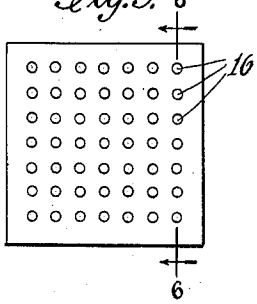
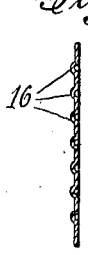


Fig. 6.



Galo W. Blanco
and Carleton Cummings INVENTORS

BY Geo. S. Butchard ATTORNEY

Patented Sept. 23, 1930

1,776,536

UNITED STATES PATENT OFFICE

GALO W. BLANCO AND CARLETON HENNINGSEN, OF BUFFALO, NEW YORK, ASSIGNORS, BY MESNE ASSIGNMENTS, TO DU PONT RAYON COMPANY, OF NEW YORK, N. Y., A CORPORATION OF DELAWARE

SHEET FOR THE MANUFACTURE OF VISCOSE

Application filed November 4, 1927. Serial No. 231,090.

In manufacturing viscose the common practice is to place sheets of wood pulp in a steeping press into which caustic solution is admitted, this forming the first step in the process. After the sheets are thoroughly steeped, they are compressed to squeeze out the excess caustic liquor. This liquor contains a certain amount of hemi-celluloses which are extracted from the pulp. By the term hemi-celluloses is generally meant that portion of the pulp which is most closely related in chemical constitution to the large class of compounds known as sugars or polysaccharides. No definite chemical formula can be assigned to the hemi-celluloses, as they are rather variable mixtures of compounds whose exact constitution is in many cases unknown. It has been customary to stop the operation of the press after a certain travel of the piston termed the cut-off, and to permit the liquor extracted up to this time to return to the source of supply for reuse. The press has been then put into operation again to extract the remaining liquor which is allowed to flow to waste. This operation has been followed because of the fact that the wasted liquor has been found to contain relatively high percentages (2% and over) of hemi-celluloses. If hemi-celluloses are used in the steeping solution in large percentages, a number of brown gelatinized areas will appear, not only on the surface of the sheets, but extending through the sheets as well. These areas increase in number in proportion to the increase of hemi-celluloses in the steeping liquor.

During succeeding steps in the process of making the viscose these gelatinized areas have been found to be very detrimental. During the shredding operation the gelatinized areas, because of their slimy and somewhat soapy-like nature, are extremely difficult to shred. Thereafter, when the alkali cellulose is treated with carbon bi-sulphide to form cellulose xanthate, the gelatinized areas fail to react normally to the bi-sulphide. As a result of the poor xanthation the subsequent solution in water is incomplete and thus the viscose tends to retain the slimy aggregate which soon plugs up to a very undesirable degree the cloths used for filtering and also the screens through which the viscose is passed before filtering. Charges which ordinarily are screened in a normal period would require three or four times as long to screen, when brown gelatinized areas were formed during steeping. It is, therefore, the custom to keep the percentage of hemi-celluloses in the caustic liquor below .5% or at the most 1%. This means that the cut-off in the steeping tank must be set at a point which results in the waste of about one-third to one-half of the caustic employed in the press. Many schemes have been suggested to reclaim and reuse the wasted liquor most of which are expensive and unsatisfactory.

Therefore, one of the objects of our invention is to provide a method and a means which shall avoid the necessity of adopting such expensive methods as referred to above and which shall at the same time permit the reuse of 100% of the caustic liquor which has formerly been wasted.

It has been found that, when all of the caustic liquor is returned to the make-up tank from the steeping press, the hemi-celluloses contained therein will gradually build up to about 3.5% and over. The percentage depends on the specific pulp used and may run very high. A high percentage of hemi-celluloses, however, has been accompanied with an increase in brown gelatinized areas referred to above, often as many as 75% of the sheets of pulp developing these areas. These areas cause the trouble in shredding xanthation and filtration referred to above.

It has been observed, however, that the finished rayon is not harmed by the presence of the hemi-celluloses in the caustic liquor. The trouble was caused almost entirely in the operating steps.

Another object of our invention, therefore, is to provide a novel and improved means which shall permit the use of a steeping caustic containing relatively high percentages of hemi-celluloses and which shall thus avoid the necessity of wasting such a large amount of steeping liquor.

In the accompanying drawing in which we

have shown certain means for practicing our invention:

Figure 1 is a diagrammatic plan view showing a steeping press, the tank being shown in section.

Figures 2 to 5 inclusive show various means by which our invention may be practiced.

Figure 6 is a section on the line 6—6 of Figure 5.

Referring now to the drawings in detail and more particularly to Figure 1, the numeral 1 designates the steeping tank of the press in which the raw material, in this case sheets of wood pulp, is deposited for treatment by a bath of caustic solution. The sheets of pulp are designated 2 and are arranged on edge between perforated iron plates 3, which hold the sheets upright. From five to twenty sheets of pulp are usually placed in a compartment formed between two plates and the number of compartments is determined by the size of the particular apparatus employed, and may range from ten to one hundred. For the purpose of illustration, however, and in order to better show the invention on a large scale, we have shown the space taken up by a compartment as being somewhat exaggerated. The object of the iron plates is to hold the pulp sheets upright and allow drainage of the liquor.

The caustic solution is supplied from the make-up tank 4 and the flow of the press 1 is controlled by the valve 5. An outlet 6 is provided from the other end of the tank and this outlet is provided with a connection 7 leading back to the make-up tank 4 and controlled by a valve 8, and also with a connection 9 through which the solution may be wasted, this connection being controlled by the valve 10. Slidably mounted in one end of the tank 1 is a piston rod 11 having a head 12 contacting with the contents of the press. The rod may be operated by any suitable means to compress the pulp.

In operation a solution of caustic soda of a normal strength of about 15% to 30% is maintained in the make-up tank, and this solution is run into the press through the valve 5 and to a height which will completely submerge all of the sheets 2. The sheets are left in the solution for a period which may vary from about 30 minutes to several hours in order to become thoroughly soaked therewith. Then the press is emptied of the excess solution or liquor by opening the valve 8 and permitting the liquor to run back to the tank 4, the valve 10 of course being closed. The piston is then moved until the head reaches some such point as that indicated by the arrow C, the length of travel of the piston head being termed the cut-off. The liquor which is pressed out of the caustic by this actuation of the piston is low in hemi-celluloses, and it has been the practice to permit this liquor to return to the make-up tank. The remaining liquor is relatively high in hemi-celluloses and therefore it has been the practice to waste it. Therefore, the next step in the operation has been to close the valve 8 and open the valve 10, after which the piston is again actuated to press out the remaining liquor from the pulp. The cut-off has been determined by the amount of hemi-cellulose content which it has been possible to use in the steeping liquor without producing the undesirable brown areas to such an extent as to make filtration difficult. As pointed out above this content has been kept at about .5%, or at most 1%, and, when the cut-off has been set so as to maintain this percentage of hemi-celluloses, the resulting waste of liquor has been large.

We have found that the brown gelatinized areas referred to above form on those portions of the pulp sheets which do not have free access to the caustic solution. The pulp absorbs the caustic soda out of the solution with great rapidity. The result is that the pulp to which the caustic solution has free access absorbs the caustic from the solution before the solution reaches that part of the pulp to which access is more difficult. Thus, in a relatively short time the concentration of the solution is substantially reduced. At the same time the pulp in these localized areas does not react normally to the caustic soda, since the concentration of the solution with which it is in contact has been materially reduced before it reaches these areas. This condition results in the formation of the brown areas. It has been found that after brown areas have once been produced, subsequent steeping in caustic of normal strength even for long periods of time will not serve to eliminate them. In other words, the pulp is permanently affected in these sections and cannot be subsequently converted into an alkali-cellulose suitable for viscose manufacture.

It has also been discovered that the number of gelatinized areas increases in proportion to the hemi-celluloses in the steeping liquor. It is believed that the increased viscosity of the steeping liquor causes this. The viscosity of the steeping liquor increases rapidly as the hemi-celluloses contained therein increase, thereby cutting down the rate at which diffusion of the caustic to the interior portions of the sheets will take place. Thus, it becomes increasingly difficult for solutions of higher viscosity (that is those containing large percentages of hemi-celluloses) to penetrate completely between and into the sheets as ordinarily placed in the steeping press.

When the sheets of pulp are wetted with the steeping solution they swell very rapidly. This swelling tends to press the sheets more closely together and tends to make the access

of the solution between and into the sheets more difficult.

We have discovered that, if the pulp sheets are so placed in the press as to permit free access of the solution to all the sheets whereby the sheets are rapidly and thoroughly wetted before swelling, the brown gelatinized areas are not formed. Experiments disclosed the fact that a solution containing a high content of hemi-celluloses then could be used without the brown areas occurring. Therefore, by adopting means to insure that the caustic solution reaches all parts of the sheets before they swell, the objectionable gelatinized areas are avoided and all the caustic solution may be returned to the make-up tank for reuse. The saving in this respect alone is large as indicated above. It was found that the increased percentages of hemi-celluloses were not only unobjectionable so far as the finished rayon is concerned but that, if the brown gelatinized areas were avoided, the soaking and subsequent operations were not adversely affected by the presence of the hemi-celluloses.

Another feature of very great importance that has been discovered is that the high content of hemi-celluloses materially decreases the time necessary for ripening of viscose. When operating with caustic having a high concentration of hemi-celluloses, the ripening time of viscose is decreased as much as 50%. It is believed that this is due to the presence of small quantities of hemi-celluloses which are carried down in the pulp from the steeping operation into the viscose. The time for ripening is usually several days, and, as by our invention the time has been reduced by approximately one-half, the resulting saving is large.

We have found that another way to reduce the time necessary to ripen the viscose is to introduce hemi-celluloses into the viscose before the ripening step takes place. By introducing hemi-celluloses in this manner also the time of ripening can be very materially reduced. If desired, the reduction in time may be caused entirely by the hemi-celluloses left over from the steeping operation or this may be supplemented as desired by the addition of extra quantities. Or, if desired, the steeping process may be carried on in the old manner and hemi-celluloses may be introduced independently of the steeping operation.

The means for insuring a complete and thorough wetting of the pulp may take a variety of forms. In Figures 2 to 6 inclusive we have shown the various means which have been tried with success and others will no doubt suggest themselves to those skilled in the art.

One means which has been found to be very successful is the provision of the sheets with corrugations. The corrugations may be arranged as desired but we have found that, when they are arranged diagonally to each edge of the sheet as indicated at 13 in Figure 2, the best results are obtained. If the corrugations are arranged horizontally as shown at 14 in Figure 3, excellent results are also obtained. If the corrugations are arranged vertically as shown at 15 in Figure 4, good results are also obtained, although not so good as when they are arranged as shown in Figures 2 and 3.

Another means is indicated in Figures 5 and 6 wherein is shown a plurality of dimples 16 formed by running the sheets through a suitable machine.

When any one of the embodiments illustrated is employed, it is not necessary that every sheet shall be constructed as shown. We have found that it is sufficient to have, for example every other sheet made in special form to insure free flow of caustic between the sheets. We have shown a number of means for achieving this object but obviously other forms also may be developed by those skilled in the art without departing from the scope of our invention. Obviously, also, it is possible to insure the thorough wetting by separately depositing sheets of pulp or other raw material in the caustic. This operation, however, is dangerous to the operators because of the likelihood of splashing. For that reason this method is undesirable.

In practicing our invention the sheets of raw material are placed in the steeping press in the compartments as is usual. The corrugations or other means used in place thereof will temporarily hold the sheets in position so that the greater portions thereof will be exposed to the action of the steeping liquor. The steeping liquor is then permitted to enter the press. The sheets swell rapidly as the liquor comes in contact therewith, but the means which we employ permit the liquor to come into contact with all portions of the sheets and thoroughly saturate them before they swell and come in contact with one another throughout their surfaces. After the material is thoroughly steeped, the piston then is actuated to press the material and to extract therefrom the excess liquor, all the liquor thus extracted being returned to the make-up tank for reuse.

While we have referred to the raw material as being made of wood pulp, it is obvious that our invention may be practiced with any other kind of raw material to which it is applicable. We are aware that various modifications and changes may be made in the practice of our invention as disclosed in this application and without departing from the scope of our invention. We therefore do not intend to limit ourselves except by the appended claims.

We claim:

1. A sheet composed of wood pulp from

which viscose suitable for rayon manufacture is to be made having projections on one of its surfaces and corresponding depressions on the other surface, for the purpose set forth.

2. A sheet composed of wood pulp from which viscose suitable for rayon manufacture is to be made having corrugations, for the purpose set forth.

3. A sheet composed of wood pulp from which viscose suitable for rayon manufacture is to be made having corrugations, said corrugations being diagonally arranged in respect to the edges of said sheet, for the purpose set forth.

4. A sheet composed of cellulosic material from which viscose suitable for rayon manufacture is to be made having projections on one of its surfaces and corresponding depressions on the other surface.

In testimony whereof, we have affixed our signatures to this specification.

GALO W. BLANCO.
CARLETON HENNINGSEN.