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METHOD OF IMPROVING THE PROCESSING OF REFINED CHEMICAL PULP INTO VISCOSE

Paul Henry Schlosser and Kenneth Russell Gray, Shelton, Wash., assignors to Rayonier Incorporated, Shelton, Wash., a corporation of Delaware

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1

This invention relates to the production of cellulosic products, and has for its general object the provision of certain improvements in carrying out one or more of the processing steps in the production of such products. The invention is of special advantage in the preparation and processing of viscose derived from chemically prepared wood pulp, and in this connection aims to improve the steps of shredding and filtering. The invention provides a method of processing chemical wood pulp comprising the incorporation in the cellulosic material prior to shredding of an oxide of a tertiary amine in which there is attached to the nitrogen by a covalent bond a lipophilic surface-active inducing group. The invention also provides, as a new article of manufacture, a chemically prepared wood pulp product having incorporated therein an oxide of a tertiary amine containing a lipophilic surface-active inducing group. For simplicity of wording "an oxide of a tertiary amine" is hereinafter referred to as "a tertiary amine oxide." The agents of the invention are thus "cation-active tertiary amine oxides."

Chemically prepared wood pulps are extensively used in the industrial arts for the production of such cellulosic products as rayon and other synthetic fibers, nitrocellulose, cellulose acetate, cellulose ethers, Cellophane and similar cellulosic films or sheets, etc. Wood pulp is commonly prepared and marketed in sheet form, and comminution or shredding is customarily one of the initial steps in its subsequent processing. When wood pulp is used in bulk form, either wet or dry, shredding or comminution is frequently a step involved in the subsequent processing. Since such subsequent processing usually involves the conversion of the cellulose of the wood pulp to some cellulose derivative and the solution of the derivative in a suitable solvent, the purpose of shredding is to break down the sheet into a fluffy mass or crumb in which the individual fibers will be suitably exposed to the action of the derivative-forming reagent or reagents. Thus, for example, in the production of viscose, the wood pulp sheet is customarily steeped in a caustic soda solution to form alkali cellulose, and the alkali cellulose, after pressing and while still in sheet form, is shredded or comminuted to properly prepare the cellulose fibers for the xanthating reaction with carbon bisulfide. The resulting sodium cellulose xanthate is dissolved in dilute caustic soda to form the solution commonly known as viscose, which is opacified if desired, filtered, and spun into filaments.

2

Normal dissolving wood pulps in present use consist mainly of cellulose, but contain appreciable amounts of non-cellulosic impurities, such as hemi-celluloses, fats, resins, waxes, etc. One of the main objects in the manufacture of a highly refined dissolving pulp is to remove as much as possible of the non-cellulosic impurities, so that a whiter, purer pulp results, which is capable, in the manufacture of rayon, of producing a higher grade yarn.

We find, however, that not all of the non-cellulosic impurities which can be removed are undesirable, and in fact, certain of such impurities, normally present in small amounts, are highly beneficial in aiding the processing of the pulp into viscose, especially as regards the step of shredding the alkali cellulose. The beneficial impurities which aid the shredding operation are probably of the nature of fiber lubricants which permit the steeped and pressed pulp to be thoroughly shredded into a fluffy condition more readily and without mechanical damage to the alkali cellulose fibers, which would cause them to react incompletely with carbon bisulfide. In a pulp which has not been highly refined, most of these beneficial impurities constitute a portion of the materials removable by organic solvents, as for example, ether, benzene, alcohol, etc. These beneficial impurities, often loosely termed "resins," are mainly of the nature of waxes, fats and resins, the latter often being present in a relatively small amount.

In theory, the problem of making a good pulp could be solved by removing all the undesirable impurities while retaining those impurities which facilitate the shredding operation by lubricating the fibers or otherwise. In practice, such a clean-cut separation is difficult to accomplish directly. We have discovered that better results are obtainable by removing most or all of the impurities, including those which serve beneficially as lubricants to the alkali cellulose fibers during shredding, and then adding to the pulp or to the alkali cellulose prior to the completion of shredding a sufficient amount of a material of a class entirely different from the natural impurities originally present, and which greatly aids the steps of shredding and filtering.

White, highly purified or refined wood pulps are very advantageous for the production of high grade rayon yarns of superior strength and color, and for this reason, are highly esteemed by the trade. Such highly refined pulps are in general characterized by having an ether extract of about 0.15% or less where such values refer to the

amount of natural ether-extractable material left in the pulp after the purification processes. While our invention is particularly applicable to such highly refined pulps containing not more than about 0.15% of ether-extractable material, and increasingly so as the ether extract approaches zero, it may be applied with certain advantages to the processing of normal dissolving pulps containing substantially more than 0.15% of ether-extractable material, although such pulps do not generally yield the highest grade yarns and their processing into shredded alkali cellulose is not accompanied by any particular difficulty.

The surface-active portion of the beneficial impurities, naturally present as such in the refined pulps or formed during the processing, are predominately of the general class known as anion-active materials.

We have discovered that a class of cation-active compounds give important results in the processing of chemical pulp. In general, the compounds which we have discovered for use in our invention are much more effective than the beneficial portion of the impurities naturally present. Accordingly, the cation-active compounds can be used in very minute quantities. This is doubly advantageous, because such additions of materials are inexpensive and also because the very pure pulp treated with the compounds is substantially free from non-cellulosic materials due to the minuteness of the quantity of the compounds required. Based on these discoveries, our invention involves improving one or more of the processing steps in the production of cellulosic products from chemically prepared wood pulp by carrying out one or more of the processing steps in the presence of a compound of the class described. When applied to the preparation and processing of viscose derived from chemically prepared wood pulp, the invention particularly involves carrying out the shredding of the alkali cellulose, or at least the final stage of shredding, and the filtration of the viscose in the presence of said amine oxides.

The amine oxides used in our invention are of the cation-active class, which means that the group or groups responsible for surface activity are in the positive instead of the negative ion of the molecule. As a class, cation-active tertiary amine oxides are superior in a number of ways to the anion-active compounds for use in the viscose process prior to the completion of shredding, especially as regards treatment of the original pulp. A great many cation-active tertiary amine oxides within the concentrations concerned are soluble in all concentrations of caustic soda that would be encountered in any stage of the viscose process. This caustic soda solubility has certain advantages as regards the final viscose solution but would seem to preclude or limit their use to steps subsequent to steeping or to the use in the steeping caustic which would, however, be more expensive. We have made the important discovery that cation-active tertiary amine oxides possess the remarkable and important property of being adsorbed and held very firmly by cellulosic materials in the presence of steeping caustic soda solutions.

The closeness of the association and the firmness with which the amine oxides are held to the cellulose even in strong caustic soda solutions gives important results. Thus, even though the compound may be completely soluble in 18% caustic soda in the absence of cellulose, on the other hand when pulp is impregnated with the compound and then steeped in 18% caustic soda, as in the first step of the viscose process, the compound is substantially retained by the pulp due to the strong attraction for the cellulose.

Another great practical advantage in using cation-active tertiary amine oxides for treating the pulp itself results from the substantivity of these materials toward cellulose. Thus, the pulp may be treated with these compounds prior to sheet formation as, for example, in the form of a dilute suspension of pulp in water in a stock chest without the necessity of very completely recirculating the white water. This is in contrast to the practical limitations under which anion-active compounds would usually have to be used when being applied to the pulp. With anion-active compounds it is generally necessary to apply these after the sheet has been formed and the greater part of the water has been removed, or in the event of addition prior to sheet formation to provide for a system of almost complete recirculation of the white water to prevent excessive loss of the compounds. The cation-active amine oxides, on the other hand, will be substantially exhausted from a very dilute solution by a suspension of pulp fibers and the materials will be substantially retained by the fibers during the mechanical dehydration pursuant to sheet formation.

Tertiary amine oxides are compounds having the formula $R_1R_2R_3NO$ where R_1, R_2 and R_3 may be either similar or dissimilar and where the $R_1R_2R_3N$ portion of the molecule represents a tertiary amine. (It is to be noted that tertiary amine oxides are often referred to in chemical literature simply as "amine oxides.") Since nitrogen never seems to show a covalency of five, the constitution of the tertiary amine oxides is best represented not by the older formula $R_1R_2R_3N{=}O$, but by the newer formulae which represent the oxygen as being attached to the nitrogen by a semi-polar bond, i. e., by

$$R_1R_2R_3N^+{-}O^-$$

also written as $R_1R_2R_3N{\rightarrow}O$. In the hydrated form the compounds contain in addition at least one molecule of water and are believed to have the formula

$$\left(\begin{array}{c} R_2 \\ | \\ R_1{-}N{-}OH \\ | \\ R_3 \end{array}\right)^+OH^-$$

Tertiary amine oxides are frequently prepared in the hydrated condition by oxidizing tertiary amines with an oxidizing agent such as hydrogen peroxide or Caro's acid.

For the purposes of the invention it is immaterial whether the agents used be originally in the anhydrous or the hydrated condition. They will be applied in many cases in the form of aqueous solutions, where they will be wholly or largely in the hydrated condition. Accordingly, the term "tertiary amine oxide," in addition to referring to the anhydrous oxides, is also generically intended to include any hydrated products which will form or exist in the presence of water or moisture.

If at least one of the "R" groups (i. e., R_1, R_2 or R_3) attached to the nitrogen by a covalent linkage is a lipophilic group of sufficient magnitude, then the tertiary amine oxide will be cation-active. By a "lipophilic group" is meant a group having a substantial hydrocarbon character and having a definite affinity for fats and oils. Among the groups having the strongest lipophilic char-

acter are aliphatic hydrocarbon radicals such as are derived from fats, waxes, mineral oils, etc. The aliphatic hydrocarbon radicals which may be used to impart surface activity include straight chain, branched chain, saturated and unsaturated radicals. Other groups such as cyclic hydrocarbon groups or groups containing a small proportion of other elements than carbon and hydrogen are, however, not excluded.

In regard to the use of aliphatic hydrocarbon radicals to induce surface activity, suitable lipophilic groups will include those aliphatic hydrocarbon radicals having at least 7 carbon atoms. There is a practical upper limit for these or for any other type of lipophilic, surface-active inducing radical in that the compounds must be at least dispersible (and preferably soluble) in water or solutions of alkali metal hydroxide.

The formulae given for the anhydrous and hydrated forms of the amine oxides show attached to the nitrogen by covalent bonds, groups R_1, R_2 and R_3. It is to be particularly pointed out, however, that the cation-active amine oxides of the invention are not restricted to open chain compounds. Where R_1 is a lipophilic surface-active inducing radical, the radicals R_2 and R_3 may belong to the same system as, for example, in the employment of a cyclic nucleus such as the piperidine, or the pyrrolidine nucleus. In such cases the alkylene hydrocarbon radical which is attached to two of the valences of the nitrogen forming a ring counts as both groups R_2 and R_3.

When the lipophilic group attached to the nitrogen is not a hydrocarbon group, there is also the provision that the cation must be substantially stable in solutions of alkali metal hydroxide at the temperatures normally encountered in the viscose process up to and including the step of shredding.

The most preferred class of cation-active amine oxides are those wherein one of the groups (group R_1) attached to the nitrogen by a covalent bond is a normal, primary aliphatic hydrocarbon chain with from 7–20 carbon atoms and the other groups (groups R_2 and R_3) attached to said nitrogen by the remaining two carbon-nitrogen bonds are very small alkyl groups, the best being methyl groups.

Tertiary amine oxides are weak bases forming salts with acids which are generally either largely or completely converted to the free base in the presence of an excess of a strong alkali. Since there is caustic soda present in all stages of the processing of the pulp into viscose, it is obviously an equivalent matter in the practice of the invention if the cation-active tertiary amine oxides are added in the form of salts with acids, rather than as the free bases. If the amine oxides of the invention are added as salts, these will become immediately converted to the free bases during the steeping operation, which is the initial step in the viscose process. The salt of any acid may be used where its anion would normally be innocuous as regards its presence in small amounts in the viscose process. In some cases there are actual practical advantages for addition of the agents in the form of salts rather than as the free base, as for example in certain cases where difficultly soluble amine oxides may be converted to more soluble salts.

Compounds which may be used in our invention include octyl dimethyl amine oxide, (2-ethyl hexyl) dimethyl amine oxide, lauryl dimethyl amine oxide, oleyl dimethyl amine oxide, cetyl dimethyl amine oxide, lauryl diethyl amine oxide,

cetyl diethyl amine oxide, lauryl piperidine oxide, lauryl methyl cyclohexyl amine oxide, and octadecyl dimethyl amine oxide.

An example of a compound which we have found to be particularly effective in improving the steps of filtering and shredding is lauryl dimethyl amine oxide (also known as dodecyl dimethyl amine oxide). This compound is particularly convenient to use since it is quite soluble in water, making it very simple to prepare treating solutions of any of the concentrations necessary to give the required amount of treatment within the range specified in the invention (0.01% to 0.20% based on the bone dry pulp), or to prepare even more concentrated stock solutions for later dilution to more dilute treating solutions. Other agents of greater molecular complexity, e. g., cetyl dimethyl amine oxide and oleyl dimethyl amine oxide, are also very effective in the viscose process, but their use is less simple as regards the matter of application from aqueous solutions, since the compounds (especially the cetyl derivative) are more difficultly water-soluble or tend to form viscous gelatinous aqueous solutions, or emulsions except in dilute solution. The difficulties connected with the lower solubility of these compounds or their tendency to form gelatinous aqueous solutions may be overcome in a number of different ways. Firstly, when treatments are being made from very dilute solutions, or from hot solutions, most or all of the agents will be soluble. When the desired treatment concentration based on bone dry pulp and method of application are such as to require more concentrated solutions, those agents which are difficultly soluble in water may be dissolved in isopropyl alcohol or a mixture of isopropyl alcohol and water. In some cases it may be advantageous to dissolve the agent in a small amount of isopropyl alcohol and then dilute this solution with water. Other water soluble volatile organic solvents may, of course, be used. If the pulp is to be treated before use in the viscose process, any volatile solvent in which the agents are soluble can be used whether water miscible or not, since it will be removed before use in the viscose process. Furthermore, the agents will be completely dissolved or dispersed in the viscose since their concentration at this stage will be very low.

In some but not all cases the acetates of the cation-active tertiary amine oxides are more water-soluble than the free bases. Thus in some cases where the agent is difficultly water-soluble and where making a treating solution or dispersion using water alone would be difficult or would give a viscous gelatinous solution, if the acetate is more water-soluble we prefer to add sufficient acetic acid to convert the base wholly or partly into the acetate. This is obviously considerably more economical than using isopropyl alcohol or a similar organic solvent. An example of an agent with which acetic acid may be advantageously used in making up a treating solution is oleyl dimethyl amine oxide. In preparing dilute solutions of this agent no particular difficulty is involved. In preparing more concentrated treating solutions of this agent, however, to avoid the formation of a gelatinous viscose solution, we prefer to add also acetic acid in amounts up to the theoretical equivalent.

The treatment range of the invention is 0.01–0.20% based on bone dry pulp. The required concentrations for the solutions used in applying the treat are, however, not specified since these will not only depend somewhat on the amount

of treat per bone dry pulp desired, but will be greatly dependent on the method of application chosen. Thus with the more difficultly soluble agents in making up the treating solutions the question as to whether it will be practically necessary to use isopropyl alcohol, acetic acid or other aids to solution will depend greatly on the method chosen for applying the treat. Where the pulp is to be treated when still in slurry form prior to sheet formation, generally only very dilute solutions will be required. Where the pulp sheet is to be treated on the sheet-forming machine after removal of part of the water by means of a rotating roll partially immersed in a treating solution, in general more concentrated but still relatively dilute treating solutions will be required. Where the treating solution is to be sprayed on the partially dried sheet, usually it will be more practical and more economical to use somewhat more concentrated solutions than when the treatment is applied by a rotating roll. Where the material is to be sprayed in the shredder, it is advantageous to use as concentrated solutions as practically possible in order not to unduly wet the fibers so that they would stick together and shred up imperfectly.

It will be noted that in our preferred group of compounds the lipophilic surface-active inducing radical is a normal primary aliphatic hydrocarbon radical, either saturated or unsaturated, within the range of 7–20 carbon atoms. Commercially, the main source of such long chain normal primary aliphatic hydrocarbon radicals is the vegetable and animal fats and oils. Such fats or oils may be utilized in preparing the compounds of our invention, for example, by converting the fatty acids to the primary amines which may then be converted to tertiary amine oxides, by way of the tertiary amines, according to well-known practices. Other methods of preparing the tertiary amine oxides are, of course, possible. For example, the long chain fatty acids may be converted to fatty alcohols and then to long chain alkyl halides which may then be reacted with lower molecular weight compounds to give tertiary amines according to well-known practices. In preparing the compounds of the invention it is not necessary to use pure alkyl compounds (alkyl amines, alkyl halides, etc.). Indeed higher molecular weight alkyl compounds prepared as indicated from fats and oils are generally, if not always, offered in commercial quantities in the form of mixtures of different hydrocarbon chain lengths. Thus, for example, in place of using pure n-lauryl amine in the preparation of lauryl dimethyl amine oxide, technical lauryl amine that is such as may be prepared from coconut oil may be used. Such technical lauryl amine will be essentially a mixture of C_8, C_{10}, C_{12}, C_{14} and C_{16} carbon length chains with the C_{12} (i. e., the lauryl) chain predominating. Tertiary amine oxides prepared from such a mixture of normal primary amines function in our invention, we find, in a substantially identical manner as tertiary amine oxides prepared from the individual normal primary amines. Actually, however, when using such a mixture in which lauryl amine predominates and containing both lower and higher chain lengths, the result is almost the same as if pure lauryl amine were used for the preparation of the alkyl dimethyl amine oxide.

While highly refined wood pulps are advantageous for the production of high grade rayon yarns of superior strength and color, the reaction

of the shredded alkali cellulose from such pulps with carbon bisulfide is frequently incomplete. This impairment in the xanthating activity of the shredded alkali cellulose is due to some damage to the fibers during the shredding operation or to incomplete comminution or to formation of compressed fiber bundles. In the case of the conventional shredder having revolving blades coacting with a stationary saddle bar, the tendency for such damage is especially great if the clearance between the revolving blades and the saddle bar is a little less than the correct value. In an extreme case, with a very highly refined pulp, shredding, even under optimum conditions, may produce a shredded pulp which xanthates less completely than if the alkali cellulose were not shredded at all. In other cases, it is possible to obtain reasonably satisfactory shredding of the alkali cellulose from highly refined pulps by adjusting the shredder clearance and by experimenting to find the optimum shredding time for the particular pulp and particular shredder. In this manner it is possible in some cases to obtain almost as complete a degree of xanthation of the shredded alkali cellulose from a highly refined pulp as would be obtained with the shredded alkali cellulose from a less pure pulp. But the necessity of constant supervision and adjustment to assure optimum shredding conditions makes the shredding of highly refined pulps too sensitive for satisfactory commercial practice. This sensitivity to damage during shredding of the alkali cellulose from highly refined pulps is overcome, in our invention, by carrying out the shredding operation in the presence of a tertiary cation-active amine oxide which may be added to the pulp prior to use in the viscose process or to the alkali cellulose prior to the completion of shredding. The improvement in shredding produces a shredded alkali cellulose which reacts substantially completely with carbon bisulfide, and the resulting viscose is comparatively free of unreacted fibers and filtration proceeds more rapidly and economically.

The most practical and economical manner of securing the desired presence of cation-active tertiary amine oxides of our invention during the shredding of the alkali cellulose and during the filtration of the viscose is to incorporate the amine oxides in the wood pulp. This may be advantageously effected by adding the compounds to the pulp on the sheet-forming machine subsequent to sheet formation but prior to complete drying, by spraying the pulp sheet with an aqueous solution of the compound or by means of a rotating roll partly immersed in such a solution. If desired, the compounds of the invention may be incorporated in the pulp prior to sheet formation, as, for example, by adding the agent to a suspension of pulp in water in a stock chest. In the latter case, the white water need not be re-circulated in order to prevent considerable loss of the agent when eliminating water in sheet formation in view of the substantivity of the products toward cellulose. In any case, there is produced a chemically prepared wood pulp product having a cation-active tertiary amine oxide incorporated therein. When the compound is so incorporated in the wood pulp, by the manufacturer thereof, the pulp comes to the rayon manufacturer in a form calculated to secure the full advantages of the invention in the preparation and processing of the viscose into high grade rayon yarns.

The amount of the cation-active tertiary amine

oxide used in the practice of the invention is relatively small, ranging from 0.01–0.20%, and preferably from 0.03–0.12% by weight based on the bone dry weight of the wood pulp used, when the agent is incorporated in or added to the pulp or to the alkali cellulose. So far as the objectives of the invention are concerned, there is little if any improvement by increasing the amount of the compound above 0.20%, and such higher amounts frequently give rise to certain disadvantages. These disadvantages include the causing of excessive softness in the sheet, resulting in mechanical difficulties in steeping, excessive ball formation in xanthation, difficulties in the dissolving operation due both to the excessive ball formation in the xanthating step and due to excessive foaming in the viscose solution. Also there will be considerable difficulty in obtaining a completely de-aerated viscose which is absolutely necessary for satisfactory spinning. Higher concentrations of the compounds also unduly lower the surface tension of the viscose, thus changing the coagulating conditions so that the viscose cannot be satisfactorily spun by standard methods, causing the filaments to break and the thread to stick to the godet wheels or thread guides.

While it is our preferred practice to incorporate the cation-active amine oxide in the wood pulp, preferably a highly refined pulp containing not more than about 0.15% of ether extractable material, the presence of the compound during the processing step of shredding may be secured in any other appropriate manner. Moreover, when the pulp is in sheet form, the compound need not be incorporated in every sheet, but may be incorporated in only alternate sheets. It may be added to only a portion of the pulp in whatever form it is marketed. Alternatively, the compound may be sprayed upon or otherwise suitably added to all or a portion of the alkali cellulose prior to shredding or prior to the completion of shredding. However, we believe it will generally be found more advantageous to incorporate the compound in the initial wood pulp product, both as a matter of convenience and economy in preparing and processing the viscose, and because a very uniform distribution of the compound throughout the viscose is easily attained.

When the invention is practiced for effecting the hereinbefore-mentioned improvements in shredding and filtering, certain further economies are effected in the subsequent steps of xanthating, dissolving and filtering. In viscose solutions there is usually a certain amount of undissolved fibers and gel-like material due to the incomplete reaction of the cellulose with the carbon bisulfide during xanthation. Prior to spinning, the viscose solutions are filtered several times to remove these gels and undissolved fibers. In the event that the viscose solutions contain excessive amounts of undissolved and partially dissolved fibers, filtration is an expensive operation. In such cases the filters become rapidly clogged and the filter media must be changed frequently in order that the viscose will pass through in a reasonable time. Frequent changing of the filter media is expensive, not only as regards consumption of filter cloth but also in view of the very considerable amount of labor involved and also since a certain amount of viscose is lost every time the filter is opened up. Furthermore, when the viscose solutions contain very large proportions of gel-like material, filtration is usually not altogether satisfactory in that some of the smaller gel-like particles tend to pass through the pores of the cloth with adverse effect upon the spinning operation. It has heretofore been the practice in the viscose industry, when processing pulps which tend to yield viscose solutions high in undissolved material and having poor filtration properties, to minimize such difficulties by carrying out the xanthation with amounts of carbon bisulfide considerably in excess of that normally required. Use of excess carbon bisulfide is expensive and in addition is technically undesirable in that it may adversely affect certain properties such as the ripening of the viscose and yarn characteristics. We have found that when processing highly purified pulps which would normally tend to give poorly filtering viscose solutions, the addition of minute amounts of the compounds of the invention so improves the shredding operation that the alkali cellulose subsequently reacts much more completely with carbon bisulfide and yields a viscose solution very free from undissolved and partially dissolved cellulose particles and having good filtration properties. This result can be accomplished not only without the use of excess carbon bisulfide, but in many cases satisfactory viscose solutions can be obtained using amounts of carbon bisulfide very substantially less than the amounts normally required.

We claim:

1. The method of improving the processing of refined chemical pulp containing not more than 0.15% ether extractable matter into viscose comprising adding to one of the viscose-forming materials at a stage prior to completion of shredding a cation-active tertiary amine oxide at least dispersible in, and whose cation is substantially stable in, solutions of alkali metal hydroxide, said amine oxide being added in a range from 0.01% to 0.20% by weight, such percentages being based on the weight of the bone dry pulp.

2. The method of improving the manufacture of shredded alkali cellulose from refined chemical pulp containing not more than 0.15% ether extractable matter comprising adding to one of the alkali cellulose-forming materials at a stage prior to the completion of shredding a cation-active tertiary amine oxide at least dispersible in, and whose cation is substantially stable in, solutions of alkali metal hydroxide, said amine oxide being added in a range from 0.01% to 0.20%, such percentages being based on the weight of the bone dry pulp.

3. The method of improving the processing of refined chemical pulp containing not more than 0.15% ether extractable matter into viscose comprising adding to such refined chemical pulp prior to use in the viscose process a cation-active tertiary amine oxide at least dispersible in, and whose cation is substantially stable in, solutions of alkali metal hydroxide, said amine oxide being added in a range from 0.01% to 0.20% by weight, such percentages being based on the bone dry weight of the pulp.

4. As a new article of manufacture, a refined chemical pulp product containing not more than 0.15% of natural ether extractable matter and having incorporated therein a cation-active tertiary amine oxide at least dispersible in, and whose cation is substantially stable in, solutions of alkali metal hydroxide, the amount of the incorporated amine oxide being from 0.01% to 0.20% by weight, such percentages being based on the bone dry weight of the pulp.

5. The method of improving the processing of

refined chemical pulp containing not more than 0.15% ether extractable matter into viscose comprising adding to one of the viscose-forming materials at a stage prior to completion of shredding a cation-active tertiary amine oxide at least dispersible in solutions of alkali metal hydroxide and wherein one of the groups attached to the nitrogen by a covalent bond is an aliphatic hydrocarbon chain of 7–20 carbon atoms, said amine oxide being added in a range from 0.01% to 0.20% by weight, such percentages being based on the weight of the bone dry pulp.

6. The method of improving the processing of refined chemical pulp containing not more than 0.15% ether extractable matter into viscose comprising adding to such refined chemical pulp prior to use in the viscose process a cation-active tertiary amine oxide at least dispersible in solutions of alkali metal hydroxide and wherein one of the groups attached to the nitrogen by a covalent bond is an aliphatic hydrocarbon chain of 7–20 carbon atoms, said amine oxide being added in a range from 0.01% to 0.20% by weight, such percentages being based on the bone dry weight of the pulp.

7. The method of improving the processing of refined chemical pulp containing not more than 0.15% ether extractable matter into viscose comprising adding to one of the viscose forming materials at a stage prior to completion of shredding a cation-active tertiary amine oxide wherein one of the groups attached to the nitrogen by a covalent bond is a normal primary aliphatic hydrocarbon chain with from 7–20 carbon atoms and the other groups attached to said nitrogen by the remaining two carbon-nitrogen bonds are methyl groups, said amine oxide being added in a range from 0.01% to 0.20% by weight, such percentages being based on the weight of the bone dry pulp.

8. The method of improving the manufacture of shredded alkali cellulose from refined chemical pulp containing not more than 0.15% ether extractable matter comprising adding to one of the alkali cellulose forming materials at a stage prior to the completion of shredding a cation-active tertiary amine oxide wherein one of the groups attached to the nitrogen by a covalent bond is a normal primary aliphatic hydrocarbon chain with from 7–20 carbon atoms and two other groups attached to said nitrogen by the remaining two carbon-nitrogen bonds are methyl groups, said amine oxide being added in a range from 0.01% to 0.20%, such percentages being based on the weight of the bone dry pulp.

9. The method of improving the processing of refined chemical pulp containing not more than 0.15% ether extractable matter into viscose comprising adding to such refined chemical pulp prior to use in the viscose process a cation-active tertiary amine oxide wherein one of the groups attached to the nitrogen by a covalent bond is a normal primary aliphatic hydrocarbon chain with from 7–20 carbon atoms and the other groups attached to said nitrogen by the remaining two carbon-nitrogen bonds are methyl groups, said amine oxide being added in a range from 0.01% to 0.20% by weight, such percentages being based on the bone dry weight of the pulp.

10. As a new article of manufacture, a refined chemical pulp product containing not more than 0.15% of natural ether extractable matter and having incorporated therein a cation-active tertiary amine oxide wherein one of the groups attached to the nitrogen by a covalent bond is a normal primary aliphatic hydrocarbon chain with from 7–20 carbon atoms and the other groups attached to said nitrogen by the remaining two carbon-nitrogen bonds are methyl groups, the amount of the incorporated amine oxide being from 0.01% to 0.20% by weight on the bone dry pulp.

11. The method of improving the processing of refined chemical pulp containing not more than 0.15% ether extractable matter into viscose comprising adding to one of the viscose forming materials at a stage prior to completion of shredding lauryl dimethyl amine oxide in a range from 0.01% to 0.20% by weight, such percentages being based on the weight of the bone dry pulp.

12. The method of improving the processing of refined chemical pulp containing not more than 0.15% ether extractable matter into viscose comprising adding to such refined chemical pulp prior to use in the viscose process lauryl dimethyl amine oxide in a range from 0.01% to 0.20% by weight, such percentages being based on the bone dry weight of the pulp.

13. The method of improving the processing of refined chemical pulp containing not more than 0.15% ether extractable matter into viscose comprising adding to one of the viscose forming materials at a stage prior to completion of shredding oleyl dimethyl amine oxide in a range from 0.01% to 0.20% by weight, such percentages being based on the weight of the bone dry pulp.

14. The method of improving the processing of refined chemical pulp containing not more than 0.15% ether extractable matter into viscose comprising adding to such refined chemical pulp prior to use in the viscose process oleyl dimethyl amine oxide in a range from 0.01% to 0.20% by weight, such percentages being based on the bone dry weight of the pulp.

15. The method of improving the processing of refined chemical pulp containing not more than 0.15% ether extractable matter into viscose comprising adding to one of the viscose forming materials at a stage prior to completion of shredding cetyl dimethyl amine oxide in a range from 0.01% to 0.20% by weight, such percentages being based on the weight of the bone dry pulp.

16. The method of improving the processing of refined chemical pulp containing not more than 0.15% ether extractable matter into viscose comprising adding to such refined chemical pulp prior to use in the viscose process cetyl dimethyl amine oxide in a range from 0.01% to 0.20% by weight, such percentages being based on the bone dry weight of the pulp.

PAUL HENRY SCHLOSSER.
KENNETH RUSSELL GRAY.

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