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PROCESS OF WAX SIZING PAPERMAKING FIBERS USING A CATIONIC SURFACE ACTIVE AGENT

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The present invention relates to the sizing of paper. More particularly the present invention relates to the sizing of paper fibers by adding wax thereto.

The sizing of paper with wax is well known in the art and in general involves the addition of a suitable wax emulsion to the paper stock in a beater with the subsequent addition of alum or the like to break the emulsion and fix the wax on the fibers.

It has now been discovered in accordance with the present invention that wax may be deposited directly upon paper fibers by the addition to the fibers and wax of a cationic surface-active fixing agent. This fixes the wax directly on the fibers and gives a much higher take up of wax than is possible by utilizing a wax emulsion together with alum or the like.

It is one of the objects of the present invention, therefore, to provide a novel process for sizing paper fibers with wax by depositing the wax directly on the fibers from a molten condition.

A second object of the present invention is to deposit a high proportion of wax on the paper fibers by utilizing a cationic surface active agent for fixing the wax on the fibers.

Other objects and advantages of the present invention will become apparent from the present description and claims.

In practicing the present process a suitable quantity of pulp such as sulfite pulp either in bleached or unbleached condition is charged into a beater or other paper making apparatus capable of disintegrating the same. Thereafter the beater is heated to a temperature sufficient to melt the wax which is subsequently added. In general where ordinary wax having a melting point of approximately 120° F. is used, the beater is brought to a temperature above this point, such as approximately 130–160° F., by the use of steam or by other heating means. Thereafter a cationic surface active agent of the type hereinafter to be described is added in the proportion of at least 3% by weight of the wax. The agent apparently fixes the wax on the fibers for no wax is apparent in the apparatus or during the remainder of the process. Almost all of the wax introduced becomes fixed onto the pulp fibers, for when paper formed from this wax-sized pulp is extracted with a suitable solvent to determine its wax content the total quantity of wax in the paper is found to be nearly 100% of the weight of wax which was added to the pulp. The finished sheets thus prepared are smooth and show no evidence of wax spots and appear in every way to be suitably sized.

The cationic surface active agents used in accordance with the present invention include certain amino-amides, glyoxalidines cyclized from those amino-amides, and the solubilized derivatives of either. The amino-amides may best be described by the general formula:

$$R^1-\overset{\displaystyle O}{\overset{\|}{C}}-(NH-CH_2-CH_2)_n-N\begin{matrix}\diagup H\\ \diagdown R^2\end{matrix}$$

where R^1 represents an aliphatic chain containing from 7 to 23 carbon atoms; R^2 denotes a substituent selected from the group consisting of hydrogen and alkylol groups containing 1 to 3 carbons, and n is a number selected from the group consisting of 2 and 3 when R^2 stands for hydrogen and n is a number selected from the group consisting of 1, 2 and 3 when R^2 represents an alkylol radical. Glyoxalidines may be formed from amino-amides of the above formula by internal condensation wherein the carboxyl carbon atom of the amido group is linked to the nitrogen atom of the nearest amino group in the molecule. This glyoxalidine ring formation occurs when the oxygen and hydrogen atoms of the amido group combine with a hydrogen atom of the nearest amino group thereby liberating a molecule of water. Further, compounds corresponding to soluble acid salts or alkylated derivatives of the above type of compound may be used. These may be produced by treating a glyoxalidine or amide compound of the general formula above described with an aliphatic monocarboxylic acid of from 1 to 3 carbon atoms such as acetic, formic, glycolic, etc. or with a suitable ester of a mineral acid, as, for example, diethyl sulfate, triethyl phosphate, ethyl iodide, etc. The resultant solubilized compounds may then be illustrated by the general formula:

$$\left[R^1-\overset{\displaystyle O}{\overset{\|}{C}}-(NH-CH_2-CH_2)_n-N\begin{matrix}\diagup H\\ -R^2\\ \diagdown R^3\end{matrix}\right]Z$$

where R^1, R^2 and n have the values assigned previously, R^3 denotes a substituent selected from the group consisting respectively of hydrogen, when R^3Z is a 1 to 3 carbon aliphatic monocarboxylic acid, and an aliphatic alkyl group of from 1 to 3 carbon atoms, when R^3Z is an ester of a mineral acid with a 1 to 3 carbon aliphatic alcohol, and Z is selected from the group consisting of the respective residues of said acid and said ester; and where the formula also encompasses the same derivatives of glyoxalidines produced from the amino-amides prior to addition of the R^3 and Z radicals by linking the carboxyl carbon atom with the nitrogen atom of the nearest amino group accompanied by liberation of a molecular proportion of water.

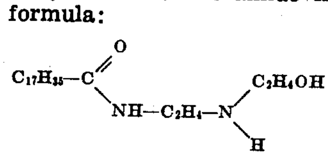
In preparing the cationic compound which fixes the wax on the fibers in accordance with the present invention, a suitable fat or fatty acid is reacted with a polyalkylene polyamine compound in order to form an amino-amide, or in the alternative, the fatty acid or fat is reacted with an alkylene alkylol polyamine or a polyalkylene alkylol polyamine compound. Thereafter, the amino-amide may be heated to a relatively high temperature to drive off water in order to form a cyclic compound. The amino-amide, or the cyclic compound equivalent to the amino-amide, may then be treated with an agent rendering the compound water soluble such as a suitable ester of a mineral acid, for example diethyl sulfate, triethyl phosphate, ethyl iodide, or an aliphatic monocarboxylic acid of from 1 to 3 carbon atoms such as acetic, formic, glycolic, etc., or the amino-amide may be added to the paper fibers and wax and thereafter the acid.

In general, temperatures in the neighborhood of 200° C. or from 180 to 210° C. are utilized for the amidification, and in the event the cyclic compound is desired, temperatures in the neighborhood of 300° C. are used. Various polyalkylene polyamine, alkylene alkylol polyamine or polyalkylene alkylol polyamine compounds having from 2 to 4 amino groups may be used as, for example, diethylene triamine, ethanol ethylene diamine, ethanol diethylene triamine, triethylene tetramine, etc. Almost any fatty acid or fatty acid glyceride having from 8 to 24 carbon atoms in the chain may be utilized as the fatty acid portion of the compound. Thus, oleic acid, ricinoleic acid, lauric acid, stearic acid, palmitic acid, etc. are examples of the fatty acids which may be utilized in preparing the amino-amide compound.

The following specific example serves to illustrate but is not intended to limit the present invention.

Example I

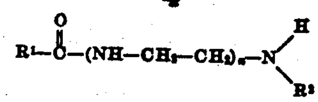
47.8 parts by weight of hydrogenated soybean oil and 25.2 parts of ethanol ethylene diamine were heated in a closed vessel at 150° C. for approximately 6 to 8 hours in order to condense the same. The resultant product is believed to consist largely of an amino-amide having the following formula:



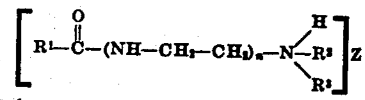
Approximately 1500 grams of unbleached sulfite pulp and 1000 grams of bleached sulfite pulp were disintegrated in a laboratory beater with 50 l. of water and heated to 160° F. 50 grams of scale wax were added to the beater followed by 5 grams of the amino-amide prepared as above set forth. Beating was then continued for 30 minutes. Hand sheets made from the stock had a good appearance and feel. Samples of these sheets were extracted with petroleum ether to determine the wax content and the wax content thereof was approximately 1.98% which was evidence of excellent take up of the wax.

Having described my invention, what I claim as new and desire to secure by Letters Patent is:

1. A process for wax-sizing paper fibers, the steps consisting of suspending the fibers in water, heating the suspension to a temperature sufficiently high to melt wax, adding wax to the aqueous suspension, heating the suspension until the wax has completely melted and mixed with the fibers, and fixing the wax on the fibers by adding a cationic surface active substance to the fiber and wax suspension, said cationic substance being selected from the group consisting of amino-amides having the general formula:



and salts of said amino-amides having the general formula:



where R^1 represents an aliphatic chain containing from 7 to 23 carbon atoms, R^2 denotes a substituent selected from the group consisting of hydrogen and alkylol groups containing 1 to 3 carbons, and n is a number selected from the group consisting of 2 and 3 when R^2 stands for hydrogen and n is a number selected from the group of 1, 2 and 3 when R^2 represents an alkylol radical, R^3 denotes a substituent selected from the group consisting respectively of hydrogen, when R^3Z is a 1 to 3 carbon aliphatic monocarboxylic acid, and an aliphatic alkyl group of from 1 to 3 carbon atoms, when R^3Z is an ester of a mineral acid with a 1 to 3 carbon aliphatic alcohol, and Z is selected from the group consisting of the respective residues of said acid and said ester.

2. A process according to claim 1 in which the quantity of said cationic surface active substance added is at least 3% of the weight of wax present in the suspension.

3. A process for wax-sizing paper fibers, the steps consisting of suspending the fiber in water and heating the water suspension to a temperature sufficiently high to melt wax, adding wax thereto, heating the wax and fiber until the wax has completely melted and mixed with the fiber and adding ethanol amino ethylene stearamide to fix the wax on the fibers.

4. A process according to claim 3 in which the quantity of ethanol amino ethylene stearamide added is at least 3% of the weight of wax present in the suspension.

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