

[54] **DISPERSING CELLULOSE XANTHATE FIBERS IN AN ACIDIC AQUEOUS MEDIUM CONTAINING A CATIONIZED ALKYL BETAINE SURFACTANT**

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[30] Foreign Application Priority Data

June 18, 1973 Japan..... 48/68505

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[51] Int. Cl.²..... D21H 3/12

[58] Field of Search..... 162/157 C, 158, 183; 8/137, 137.5; 264/194; 252/357, 546

[56] References Cited

UNITED STATES PATENTS

3,718,537 2/1973 Kawai et al..... 162/157 C

3,320,117 5/1967 Aoki et al..... 162/157 C
2,702,279 2/1955 Funderburk et al..... 252/546
3,832,281 8/1974 Kawai et al. (I)..... 162/157 C

FOREIGN PATENTS OR APPLICATIONS

19,562 1972 Japan

OTHER PUBLICATIONS

Schwartz et al., Surface Active Agents & Detergents, Vol. II, 1958, p. 138. (GP 170)

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[57] ABSTRACT

Non-woven fabric or paper-like material can be produced from fibers comprising unregenerated cellulose xanthate or hydroxymethyl cellulose xanthate by treating the fibers with a cationic surfactant, obtained by acidifying a betaine surfactant under acidic conditions, and subjecting the treated fibers to a wet-method paper-making process.

4 Claims, No Drawing Figures

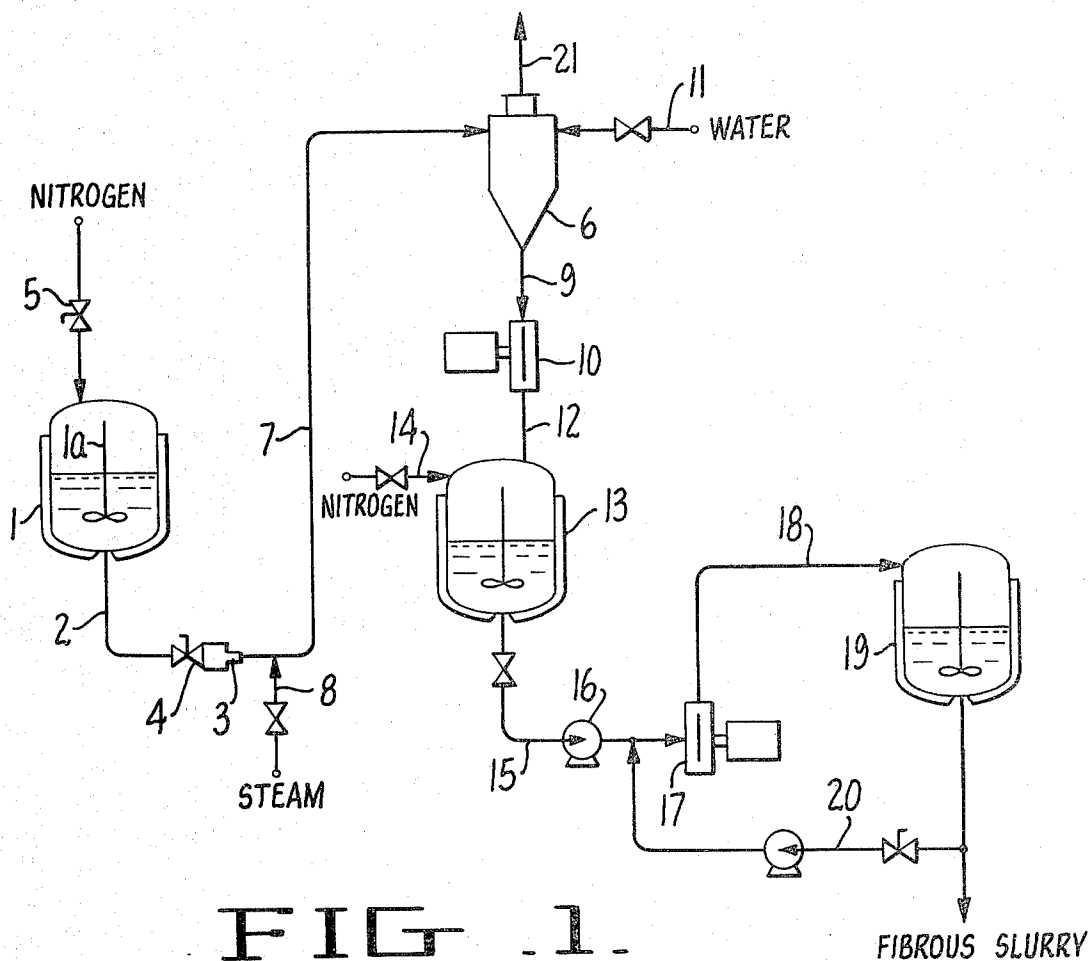


FIG. 1.

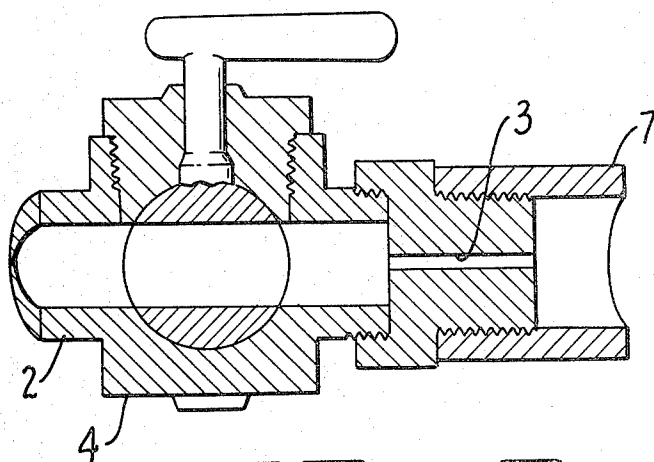


FIG. 2.

DISPERSING CELLULOSE XANTHATE FIBERS IN AN ACIDIC AQUEOUS MEDIUM CONTAINING A CATIONIZED ALKYL BETAINES SURFACTANT

BACKGROUND OF THE INVENTION

FIELD OF THE INVENTION

The present invention relates to a method for producing non-woven fabrics or paper-like materials having excellent properties by a wet method from fibers of unregenerated cellulose xanthate or hydroxymethyl cellulose xanthate.

DESCRIPTION OF THE PRIOR ART

Unregenerated cellulose xanthate and hydroxymethyl cellulose xanthate are methylolation products of cellulose xanthate. Their fibers are strongly acidic and stable, but are characterized by quite high chemical reactivity. Fibers comprising unregenerated cellulose xanthate and a method for producing non-woven fabrics or paper-like materials from the fibers are disclosed in U.S. Patent 3,320,117 and fibers comprising hydroxymethyl cellulose and a method for producing non-woven fabrics or paper-like materials from the fibers are disclosed in U.S. Patent 3,718,537 and U.S. Patent application Serial No. 267,994, filed June 30, 1972 (corresponding to Italian Patent 962,732).

These types of fibers have not heretofore been entirely successfully used in a wet process preparation of non-woven fabrics or paper-like materials, and it is now believed that the prior failures are attributable to an inability to properly disperse or distribute the fibers into the water. The use of dispersing accelerators has been considered, but heretofore, the high reactivity of the fibers have hindered effective use of such agents. It has now been discovered that a suitable such agent must have the properties of being:

1. acidic
2. highly water soluble

3. essentially chemically nonreactive with the fibers and cause no denaturation of the fibers
4. highly stable with respect to polyethylene oxide and polyacrylamide or the like, which are often used as dispersion stabilizing agents.

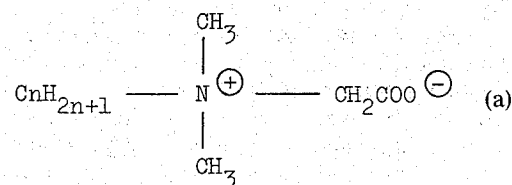
Accordingly, a need continues to exist for an improved wet process technique of forming non-woven fabrics or paper-like materials from unregenerated cellulose xanthate fibers.

SUMMARY OF THE INVENTION

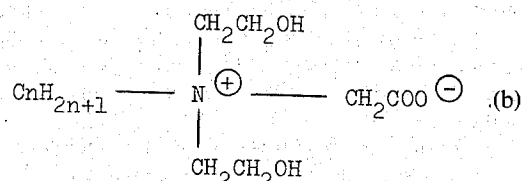
The objects of this invention are provided by the use of betaine surface active agents to impart good self-dispersibility to the unregenerated cellulose xanthate fibers. The betaine agent is used in an acidified condition.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

According to the present invention, fibers which can be used in preparing non-woven fabrics or paper-like materials, comprising unregenerated cellulose xanthate, or hydroxymethyl cellulose xanthate, are provided. The fibers are treated with a betaine surface active agent, and particularly those having the chemical structure of alkyldimethylbetaine or alkyl-dihydroxyethylbetaine. These betaines have the structure

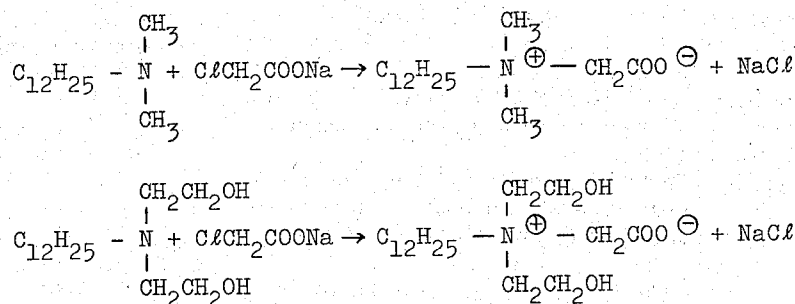


or



wherein n is an integer of 12-18. Especially suitable compounds of the formula (a) or (b) are lauryl dimethylbetaine and lauryldihydroxyethylbetaine, which contain the lauryl group, and stearyldimethylbetaine and stearyldihydroxyethylbetaine, which contain the stearyl group.

These betaine type surface active agents may be synthesized, for example, by the following reaction.



These betaine surfactants have relatively good water-solubility and stability in acidic, neutral and alkaline media and are especially stable in acidic and alkaline media.

In order to increase the affinity of these surfactants for fibers in the acidic media used in the present invention, the surfactant is advantageously cationized by acidification with acetic acid, lactic acid, or the like, whereby the adsorbability of the surface active

agents on the fibers will be increased and an extremely good dispersion effect will be obtained.

As mentioned above, the starting fibers used in the present invention are incompletely regenerated viscose fibers as used, for example, in U.S. Patent No. 3,320,117 of those comprising a derivative of cellulose xanthate, such as hydroxymethyl cellulose xanthate as used, for example, in U.S. Patent No. 3,718,537 and U.S. Serial No. 267,994. These fibers are usually cut into lengths of about 4–40 mm while wet and are used for the conventional paper-making as disclosed in said patents.

A method for using the surfactant of the present invention will now be explained. The starting fibers may be dipped into a liquid containing the above surfactant under acidic conditions or said liquid may be sprayed or applied onto the fibers under acidic conditions at the spinning step or at any of the stretching and the subsequent steps. By subjecting the fibers to these treatments, spontaneous dispersibility is imparted to the fibers and the thus treated fibers can then be dispersed into an acidic aqueous solution and used in a wet process technique for the formation of a paper-like product.

The starting fibers may also be directly dispersed into an acidic aqueous solution which contains the surfactant, and the dispersion may be subjected to paper-making.

It becomes possible, by using said surfactant, to homogeneously disperse the starting fibers and to produce homogeneous and excellent quality non-woven fabric or paper-like material on an industrial scale.

The preferred concentration of the aqueous solution of the surfactant used for dispersion is 10–100 ppm, although higher concentrations may also be employed, if desired.

The preferred amount of the surfactant to be used is 0.05 to 1.5% based on the weight of the fibers.

In the present invention, known dispersion stabilizing agents may, of course, be used in the dispersion step and the generally employed conditions for dispersion and paper-making may be used without any difficulty.

Paper-making processes from the fibers of the present invention are explained in detail in U.S. Patent 3,320,117, U.S. Patent 3,718,537 and U.S. Serial No. 267,994.

In U.S. Serial No. 267,994, now U.S. Patent 3,832,281, non-woven fabrics or paper-like materials are produced by

(a) dispersing spun and stretched viscose fibers comprising hydroxymethyl cellulose xanthate in an aqueous medium having a pH of lower than 6.0 at a temperature of lower than 30°C,

(b) forming the dispersed fibers into a web by a wet forming method,

(c) dehydrating the web to the extent that the water content of the web becomes lower than 700%, said fibers after dehydration being characterized by:

(1) a hydroxymethyl cellulose xanthate content in terms of γ -value of greater than 30,

(2) a decomposition degree of less than 75%, and

(3) a process swelling degree of lower than 250%,

(d) subjecting at least a portion of the surface of dehydrated web to a pressure of greater than 2 Kg/cm

at a temperature of 90° to 180°C, thereby fusing and decomposing the hydroxymethyl cellulose xanthate in the pressed portion of the fibers and simultaneously bonding the fibers in said portions to each other, and (e) subjecting the pressed web to regeneration treatment to decompose the remaining hydroxymethyl cellulose xanthate into cellulose.

Having generally described the invention, a more complete understanding can be obtained by reference to certain specific examples, which are included for purposes of illustration only and are not intended to be limiting unless otherwise specified.

EXAMPLE 1

A 40% aqueous solution of lactic acid-acidified stearyldimethylbetaine (Product A) was diluted with hot water containing 1% acetic acid at 80°C to prepare a 4% aqueous solution having a pH of 3.0.

A viscose containing 7.5% cellulose and 4% alkali and having a γ value of 55 was prepared in the usual manner. This viscose was extruded into a coagulation bath containing 25 g/l of sulfuric acid, 0.5 g/l of zinc sulfate and 40 g/l of sodium sulfate at 23°C. The resultant filament tow was stretched by 150% in a bath containing 2 g/l of sulfuric acid, and 300 ppm (0.3% based on the weight of fibers) of product A at 20°C and was cut into lengths of 15 mm.

The resultant cut filament tow were dispersed in an aqueous solution containing 20 ppm of product A and having a pH of 5.0 to obtain an extremely homogeneous dispersion. This dispersion was subjected to paper-making by employing a cylindrical paper machine to obtain a uniform sheet. The thus obtained sheet was allowed to pass through an embossing roll having projections and having a surface temperature of 150°C to effect partial fusion bonding. The thus treated sheet was then subjected to scouring treatments, such as hot acid treatment at 80°C, water washing, bleaching or the like, and dried.

For comparison, a filament tow which was not treated with product A was dispersed in a bath containing no product A and having a pH of 5.0 to cause dispersion of fibers. Many undispersed cut filament tow were present after dispersion. Dispersibility of this Example and the Comparative Example are compared in Table 1.

TABLE 1

	Number of undispersed fibers (/400 cm ²)			
	L	M	S	Total
The present Example.....	0	1	14	15
Comparative Example.....	3	8	20	31

Method of measurement of number of undispersed fibers:

A square 20 cm × 20 cm was drawn on the dried sheet as the area to be measured and this sample was placed on a measuring stand with a fluorescent lamp. Undispersed fibers within said area of 400 cm² were marked with red magic ink and the number of undispersed fibers was counted with a counter. This measurement was effected two times and the mean

value was taken as the number of undispersed fibers within said area. In Tables 1 and 2, "L", "M" and "S" have the following meanings.

"L": Undispersed fibers larger than 1.0 mm in width

"M": Undispersed fibers 0.5-1.0 mm in width

"S": Undispersed fibers less than 0.5 mm in width.

EXAMPLE 2

A 50% aqueous solution of lactic acid-acidified stearyl dihydroxyethyl betaine (Product B) containing 10% sodium chloride was diluted with hot water containing 1% acetic acid at 80°C to prepare a 5% aqueous solution having a pH of 3.0.

A viscose containing 7% cellulose and 4.2% alkali and having a γ value of 80 was prepared in accordance with the conventional method. This viscose was extruded into a coagulation bath containing 35 g/l of sulfuric acid, 8 g/l of formaldehyde and 80 g/l of sodium sulfate at 26°C. The thus obtained filaments were stretched by 150% in a second bath containing 10 g/l of sulfuric acid at 60°C to obtain a filament fiber tow. This tow was continuously cut to lengths of 20 mm and at the same time, the thus cut fibers were continuously introduced and dispersed in an aqueous solution containing 70 ppm (0.15% based on the weight of fibers) of product B and having a pH of 3.5 to obtain an extremely homogeneous dispersion. This dispersion was immediately subjected to papermaking by employing a cylindrical paper machine to obtain a sheet of homogeneous texture. The thus obtained sheet was passed through an embossing roll having a surface temperature of 130°C to cause partial fusion bonding and then was subjected to scouring treatments, such as hot acid treatment at 80°C, water washing, bleaching, or the like, and dried.

For comparison, the above procedure was repeated except that cut fibers were dispersed in an aqueous solution containing no product B. Table 2 shows the comparison of dispersibility in this Example and the comparative Example.

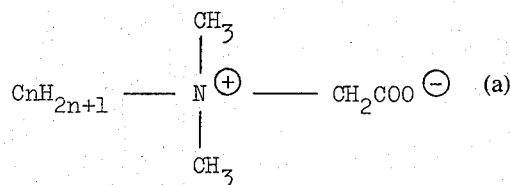
TABLE 2

	The number of undispersed fibers (/400 cm ²)			
	L	M	S	Total
The present Example.....	9	4	18	31
Comparative Example.....	10	21	46	77

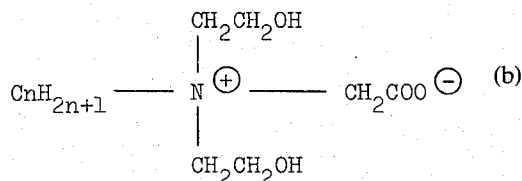
Having now fully described the invention, it will be apparent to one of ordinary skill in the art that many changes and modifications can be made thereto without departing from the spirit or scope of the invention as set forth herein.

What is claimed is:

1. In a method for producing a non-woven fabric or a paper-like material by cutting fibers comprising unregenerated cellulose xanthate or cellulose xanthate derivative, dispersing the cut fibers in an acidic aqueous medium and then forming the dispersed fibers into a sheet, the improvement which comprises: treating said fibers under acidic conditions with a surfactant having the following structure:



or



wherein n is an integer of 12-18, which surfactant is cationized by acidification, so as to impart good self-dispersibility to said fibers.

2. The method of Claim 1, wherein the treatment of the fibers with the cationized surfactant is carried out by dipping the fibers into a solution containing said cationized surfactant or by spraying or applying the solution to the fibers.

3. The method of Claim 1, wherein said surfactant is used in an amount of 0.05 to 1.5% based on the weight of fibers.

4. The method of Claim 1, wherein the concentration of the cationized surfactant in the aqueous solution is 10-100 ppm.

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,907,633 Dated September 23, 1975

Inventor(s) Migaku Suzuki et al.

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Cancel the sheet of drawing containing Figs. 1 and 2.

Signed and Sealed this

Sixteenth Day of November 1976

[SEAL]

Attest:

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