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MANUFACTURE OF SHEETS OR WEBS
OF FIBROUS TEXTILE MATERIALS
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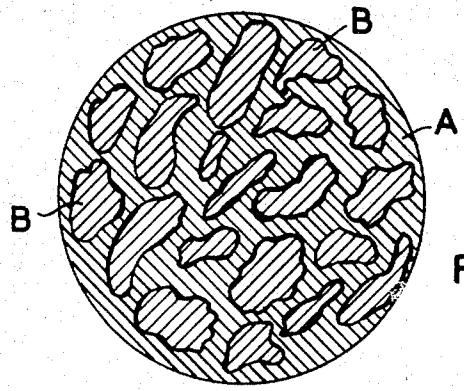


FIG.1.

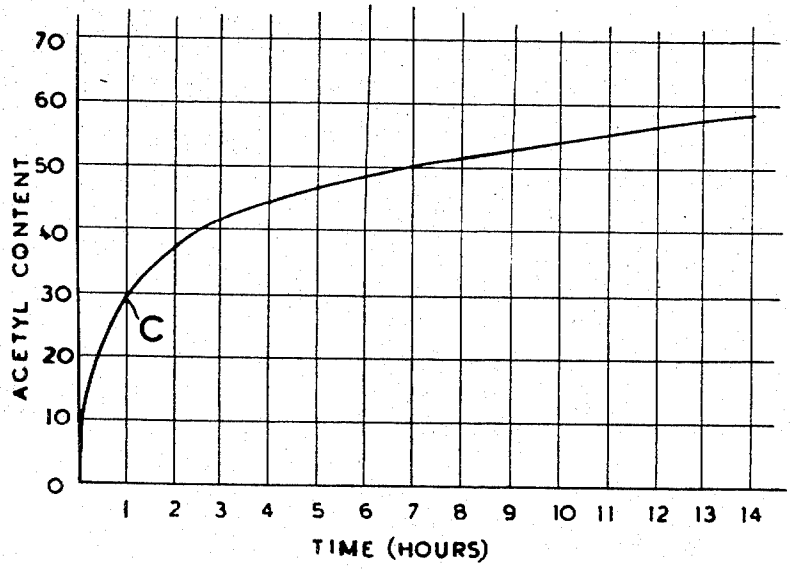


FIG.2.

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MANUFACTURE OF SHEETS OR WEBS OF FIBROUS TEXTILE MATERIALS

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2 Claims. (Cl. 154—101)

1

This invention relates to the manufacture of sheets or webs of fibrous textile materia's, such as for example felts, fleeces, and non-woven fabrics for use as towels or other cleaning tissues, hereinafter referred to generally as "sheets."

In the manufacture of fibrous textile materials in the form of felts, sheets and the like, it is known to blend non-adhesive fibres and potentially-adhesive fibres, that is fibres which can be rendered adhesive by suitable treatment such as by heating, pressing or treating with a solvent, and to treat the blend subsequently in order to render adhesive the potentially-adhesive fibres and to bind the fibres together. For example, in void British application No. 10,246/1938 as laid open to public inspection, sheets or other shaped bodies of fibrous materials and felt substitutes are made by carding natural or artificial fibres in admixture with threads of a thermoplastic artificial material and subjecting the card fleece or web to pressure, if desired with the application of heat.

The object of the present invention is to improve the manufacture of sheets of fibrous textile materials by the use of particular potentially-adhesive fibres.

In accordance with the present invention, a process for the manufacture of sheets of fibrous textile materia's comprises forming a layer of fibres at least part of which are incompletely-esterified cellulose fibres which have been prepared by esterifying cellulose fibres with an organic acid whilst maintaining their fibrous structure, and rendering the incompletely-esterified fibres adhesive so that the fibres in the layer are bonded together. The fibres are preferably incompletely-acetylated and they may be rendered adhesive by heating, preferably with the simultaneous or subsequent application of pressure, or by treatment with a solvent or plasticiser; methylene chloride is suitable for example as a solvent for cellulose acetate.

It is already known, see for example United States specification No. 1,861,320 that cellulose in fibrous form may be esterfied whilst maintaining its fibrous structure by immersing the fibres in an esterifying solution. We have investigated, particularly with the help of X-ray analysis, the course of the fibrous esterification process which will now be described with reference to the accompanying diagrammatic drawing in which:

Figure 1 represents a considerably enlarged cross-section of a cellulose fibre, and

2

Figure 2 is a graph showing one example of the course of fibrous acetylation using ramie fibres.

Cellulose fibres are known to include at least two types of cellulose namely an amorphous cellulose and a highly crystalline cellulose. As shown in Figure 1, the amorphous cellulose A forms a continuous matrix or network which surrounds and merges into the highly crystalline cellulose B; in the drawing a line separation of the two varieties of cellulose A and B is shown for clarity but actually the two varieties are separated by zones of graded intercrystalline cellulose. When the fibre is esterified by the fibrous esterification process, the esterifying solution first penetrates the amorphous cellulose A and effects its esterification to the triester stage so that after a time (usually about 1–3 hours) the matrix of amorphous cellulose is converted into cellulose triester. At this stage of the esterification process, the highly crystalline cellulose B is substantially unchanged and X-ray analysis shows that substantially no crystalline cellulose triester has been formed. The amorphous triester formed swells and so opens up the structure of the fibre with the result that the esterifying solution now penetrates through the outer layers of highly crystalline cellulose B and as it penetrates, a highly-crystalline cellulose triester is formed and this triester can be detected by X-ray analysis. Until the esterification is complete therefore, the fibre will contain a number of highly crystalline cellulose nuclei surrounded by cellulose triester.

Figure 2 is a graph showing the rate of fibrous acetylation of ramie fibres. The acetylation was effected as follows, parts and percentages being by weight:

25 parts of ramie fibres, containing 3 per cent of water were pretreated for 24 hours with 12 parts of glacial acetic acid and the pretreated fibres were then immersed in an acetylation solution consisting of a mixture of 520 parts of toluene, 330 parts of acetic anhydride (97 per cent pure) and 1.8 parts of concentrated sulphuric acid. The mixture was maintained at 45° centigrade in a thermostat. Samples of the fibre were withdrawn at intervals and the acetyl contents calculated as acetic acid, were determined and X-ray analyses of the fibres were carried out. Up to and at point C on the graph (corresponding to a 29 per cent acetyl content after 1 hour's acetylation), the acetylation was rapid and no crystalline cellulose triacetate was detected by X-ray analysis. The graph from

the origin to point C therefore corresponds to the acetylation of the amorphous cellulose. Beyond the point C, X-ray analysis showed the presence of increasing quantities of highly crystalline cellulose triacetate, thus indicating that acetylation of the highly crystalline cellulose was being effected.

If the cellulose of the fibre is in a hydrated form, a similar two-phase product is obtained but in this case the triester has a different crystal lattice from that obtained on esterification of native cellulose, and the unreacted phase is hydrated cellulose.

The present invention takes advantage of the presence of the triester present in the fibre by causing it to act as the adhesive material for bonding the fibres together.

The incompletely-esterified cellulose fibres may be prepared from natural cellulose such as cotton and the bast fibres such as hemp, ramie, jute and flax, or from regenerated cellulose such as viscose rayon fibres in either continuous or discontinuous lengths.

The degree of esterification of cellulose fibres may be varied over a wide range and we have obtained good results by using a cellulose acetylated to give only an 8 per cent acetyl value, this value being defined herein as the percentage of acetic acid present based on the final weight of the fibre. In general for making non-woven tissues suitable for cleansing purposes, it is preferred to use incompletely-acetylated cellulose fibres containing from about 8 to about 40 per cent acetyl content. Should the incompletely-acetylated cellulose fibres be used in admixture with non-adhesive fibres, it is in general desirable to use fibres having a higher acetyl value than that of fibres used without such admixtures.

The layer of fibres may be built up from the incompletely-esterified cellulose fibres alone or from mixtures of these fibres and other natural or synthetic fibres such as cotton, wool, casein fibres, viscose rayon and cellulose acetate rayon. When using fibres of cellulose acetate rayon, a particularly good bond may be obtained by using incompletely-acetylated cellulose fibres as the potentially adhesive fibre in the fibre layer.

The layer of fibres may be prepared by carding the fibres from a carded web or several carded webs may be overlaid and laminated together. Continuous filaments may also be formed into several laps of single filament thickness and the layers assembled on top of each other with the filaments in adjacent laps crossing at approximately 90°. The layer may also be formed from a slurry of fibres in a manner similar to the process used in the paper industry.

The fibrous sheet made according to the invention may be treated if desired with a saponifying agent in order to remove some or all of the ester groupings, the physical form of the sheet remaining unchanged.

The present invention is illustrated by the following examples, parts and percentages being by weight unless otherwise stated:

Example 1

Ramie fibres were mixed with half their weight of glacial acetic acid and allowed to stand for 16 hours at ordinary temperature (15° to 20° centigrade). The fibres were then immersed for 1 hour at 45° centigrade in an acetylating bath containing 700 parts of sulphur-free toluene, 650 parts of acetic anhydride and 2 parts of sulphuric acid per 100 parts of ramie fibre. The ramie fibres on removal from the bath were washed with alcohol and water and were dried at 70° centigrade. The acetyl value of the fibres was approximately 8.5.

The incompletely-acetylated fibres were formed into three laps consisting of substantially parallel fibres, and the laps were placed on top of each other with the threads in adjacent layers at right angles. The assembly was then treated by spraying with methylene chloride to render the fibres adhesive; when the methylene chloride had evaporated, the fibres had united to form a non-woven fabric sheet. A more compact fabric sheet was obtained by lightly pressing the assembly at room temperatures.

Example 2

75 parts of viscose staple fibre (3 denier, 2½ inches staple length) were pretreated at 25° centigrade for 24 hours with 75 parts of glacial acetic acid containing 1 per cent by volume of concentrated sulphuric acid (98 per cent w./w.). The pretreated fibres were then immersed for 3 hours at 45° centigrade in an acetylating bath containing 750 parts of sulphur-free toluene and 375 parts of acetic anhydride (substantially 100 per cent). The fibres were then removed from the bath, washed in alcohol and water till free from acid and were dried at 60° centigrade. The acetyl value of the fibres was 30 per cent.

The incompletely acetylated fibre was then carded to form a web which was sprayed with methylene chloride, pressed lightly to compact the web and then allowed to stand till the methylene chloride had evaporated. The product was a non-woven tissue particularly suitable for wiping and cleansing purposes. A sample of the tissue was immersed in water for one month; it was found on drying the tissue that the fabric had maintained its strength and form.

Example 3

1 part of cotton linters was pretreated with ½ part of glacial acetic acid at 25° centigrade for 18 hours. The pretreated linters were then immersed at 45° centigrade for 1½ hours in an acetylating bath containing 10 parts of sulphur-free toluene, 5 parts of acetic anhydride and 0.002 part of sulphuric acid (98 per cent w./w.). The linters were then washed first with alcohol, then with water and were finally dried at 60° centigrade. The acetyl content of the fibres was 15 per cent.

The incompletely acetylated fibres were carded and treated with methylene chloride as described in Example 2 to form a non-woven tissue.

A similar type of product may be obtained by forming the web from a mixture of equal parts of the incompletely acetylated fibre and untreated viscose staple fibre.

Example 4

1 part of ramie fibre was pretreated with ½ part of glacial acetic acid at 25° centigrade for 8 hours. The pretreated fibre was then immersed at 45° centigrade for 1½ hours in an acetylating bath containing 10 parts of sulphur-free toluene, 5 parts of acetic anhydride and 0.002 part of sulphuric acid (98 per cent w./w.). The fibre was then washed with water and the toluene was removed by steam distillation. The product was then dried. Its acetyl content was 30 per cent.

The fibres obtained were then carded with ramie fibres, using a mixture of 3 parts of the acetylated fibre with 1 part of natural fibre, to

form a web which was treated as described in Example 2 to form a non-woven tissue.

Example 5

1 part of viscose staple fibre (3 denier, 2½ inches staple length) was pretreated at 25° centigrade for 18 hours with ½ part of glacial acetic acid. The pretreated fibres were then immersed at 45° centigrade for 24 hours in a bath containing 10 parts of carbon tetrachloride, 5 parts of acetic anhydride and 0.002 part of sulphuric acid (98 per cent w./w.). The fibres were then washed with hot water and carbon tetrachloride was removed by steam distillation. The product was then dried. Its acetyl content was 12 per cent.

The fibres were formed into a web and treated with methylene chloride as described in Example 2 to provide a non-woven tissue.

Example 6

1 part of viscose staple fibre (3 denier, 2½ inches staple length) was pretreated at 25° centigrade for 18 hours with ½ part of acetic acid containing 1 per cent by volume of sulphuric acid (98 per cent w./w.). The pretreated fibers were then immersed for 2 hours in an acetylating bath containing 10 parts of carbon tetrachloride and 5 parts of acetic anhydride. The product was separated and carbon tetrachloride was removed by steam distillation. The product was then washed with water and dried. The acetyl content of the product was 23 per cent.

The fibers were formed into a web and treated with methylene chloride as described in Example 2 to provide a non-woven tissue.

What we claim is:

1. A process for the manufacture of non-woven fabrics which comprises the steps of acetylating unspun cellulose fibers while maintaining their fiber structure until the acetyl content of the fibers is from about 8 to about 40 per cent, calculated as the percentage of acetic acid on the final weight of the fibers, forming a fibrous web of unspun fibers at least the major proportion of which consists of the unspun, incompletely-acetylated cellulose fibers so obtained, and rendering adhesive the cellulose triacetate phase of the unspun incompletely-acetylated cellulose fibers whereby the fibers in the web are bonded together at their points of contact to form a unified fibrous structure.

2. A process for the manufacture of non-woven bonded fabrics which comprises the steps of acetylating unspun cellulose fibers while maintaining their fiber structure until the acetyl content of the fibers is from about 8 to about 40 per cent, calculated as the percentage of acetic acid on the final weight of the fibers, forming a fibrous web of unspun fibers at least the major proportion of which consists of the unspun, incompletely-acetylated cellulose fibers so obtained, and rendering adhesive the cellulose triacetate phase of the unspun incompletely-acetylated cellulose fibers by treatment with a solvent for the cellulose triacetate followed by removal of the solvent; whereby the fibers in the web are bonded together at their points of contact to form a unified fibrous construction.

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REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
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2,387,354	Reed	Oct. 23, 1945

Certificate of Correction

Patent No. 2,535,919 December 26, 1950

FRANK HAPPEY ET AL.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows:

Column 3, line 49, for the word "from" read *to form*;

and that the said Letters Patent should be read as corrected above, so that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 13th day of February, A. D. 1951.

[SEAL]

THOMAS F. MURPHY,
Assistant Commissioner of Patents.