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**MANUFACTURE OF PAPER-LIKE MATERIALS  
COMPRISING SYNTHETIC FIBRES**

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The present invention relates to the manufacture of paper-like materials comprising synthetic fibres.

The term "paper-like materials" as used herein denotes materials in sheet form such as can be manufactured by means known in the paper making art. In the manufacture of such materials the fibrous starting material must be homogeneously distributed in water. Where some or all of the fibres used are synthetic, then, since synthetic fibres are generally difficult to wet, they may be given a coating of a dispersant in order to assist their distribution. A wide variety of compounds have been proposed as dispersants for synthetic fibres, for example cellulose ethers, partially hydrolysed polyvinyl acetate, polyethylene glycols, and also cationic and anionic compounds.

However, despite the use of dispersants a considerably difficulty involved in the manufacture of paper-like materials consisting of or containing synthetic fibres has been that in the preparation of the aqueous suspension synthetic fibres rapidly form coherent flocks; this has a disadvantageous effect on the formation of sheets. Furthermore, disturbing foaming may occur when unsuitable preparations are used.

An object of the present invention is to provide a dispersant for synthetic fibres which substantially reduces the difficulties heretofore involved in the preparation of paper-like materials comprising synthetic fibres.

A further object of the invention is to provide paper-like materials comprising synthetic fibres displaying a more even distribution of the fibres. Other objects will appear hereinafter.

It has now been found that improved dispersions of synthetic fibres having a reduced tendency to flocculate can be obtained by coating the fibres with a water-soluble N-ethoxylated polyamide. The present invention therefore provides a process for the production of paper-like material comprising synthetic fibres by forming the paper-like material from an aqueous suspension containing the synthetic fibres wherein the synthetic fibres have a coating of a water-soluble N-ethoxylated polyamide.

The invention further comprises the paper-like materials so-produced which consist partially or wholly of synthetic fibres.

The degree of ethoxylation of the polyamide used as dispersant suitably corresponds to 8 to 12 mols of ethylene oxide per gram atom of nitrogen. Particularly suitable N-ethoxylated polyamides are N-ethoxylated polycaproamide and N-ethoxylated copolyamides obtainable from  $\epsilon$ -amino-caprolactam and hexamethylene diamine adipate, e.g., using 50 to 70%, especially 60%, of  $\epsilon$ -amino-caprolactam and 50 to 30%, especially 40%, hexamethylene diamine adipate. These compounds dissolve in water readily, both insofar as the speed of solution and the maximum concentration of the solution are concerned.

To coat the synthetic fibres with the N-ethoxylated polyamide, they are preferably treated with an aqueous solution containing 10 to 25 grams of N-ethoxylated polyamide per litre of water at a moderately raised temperature, e.g., 30 to 50° C.

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If it is not desired to use the resulting aqueous dispersion directly the fibres can then be removed from the dispersion and dried at room temperature or with the aid of heat, and later redispersed.

The polymers from which the artificial fibres may be prepared include polyamides, for example polycaproamide, the polyamide of  $\omega$ -amino-undecanoic acid, polyhexamethylene adipamide, polyhexamethylene sebacamide, and condensation products of dicarboxylic acids with aliphatic, alicyclic or aromatic diamines generally; polyurethanes; polyesters, e.g., polyethylene terephthalate and copolymers of terephthalic and isophthalic acids and ethylene glycol; polyvinyl acetate and copolymers of vinyl acetate, e.g., with vinyl chloride, vinylidene cyanide or acrylonitrile; polyacrylonitrile and copolymers of acrylonitrile, e.g., with vinyl chloride; and poly-tetrafluoroethylene.

The synthetic fibres may be formed by melt, dry or wet spinning methods, as by extruding spinning compositions through spinnerets, or by precipitation from solutions.

The application of the N-ethoxylated polyamide to the synthetic fibres may be performed at any stage of their manufacture. The treatment of spun synthetic fibres with N-ethoxylated polyamides can be carried out during spinning, prior to the stretching operation, before or after cutting the filaments up into staple, or immediately prior to incorporation in the paper stock. If the synthetic fibres have previously been treated with other agents, it is in general possible to remove the latter (if desired) as by washing with a warm dilute aqueous soda solution.

In the manufacture of the paper-like materials it is of particular importance to be able to vary the proportion of synthetic fibres in the mixture with other fibres (i.e., natural or modified natural fibres) within wide limits, because this makes it possible to manufacture papers having particular properties. This is readily achieved in the present invention. For example, synthetic fibres coated with N-ethoxylated polyamide as described above may be introduced into a suspension of cellulose fibres prepared in known manner, and from this mixture a sheet of adequate strength is produced in the wet state on a paper making machine. If in this method the cellulose component used is in the form of so-called polynosic short staple fibres (see article in "Reyon Zellwolle und andere Chemiefasern," 9, page 431 [1959]) or other viscose short staple fibres, it is possible to incorporate up to 70% by weight of polyamide or polyester fibres in the aqueous dispersion, from which a sheet having the corresponding content of synthetic fibres and having special properties can be made. With ground cellulose and rag cellulose, for example, paper-like materials containing up to 90% by weight of synthetic fibres can be manufactured. A sheet produced in this manner is characterised by the even distribution of the fibres, as can be seen, for example, by looking through the finished paper. Papers made from fibre dispersions prepared with unsuitable dispersants display an uneven distribution of the fibres when inspected in transmitted light.

The invention is illustrated in the following examples, in which ratios and percentages given are by weight.

*Example 1*

The dispersing material used is an ethoxylated copolyamide from 60% of  $\epsilon$ -aminocaprolactam and 40% of hexamethylene diamine adipate containing 2.4% of nitrogen, which corresponds to a degree of ethoxylation of 10.5 mols of ethylene oxide per gram atom of nitrogen. The compound is soluble in water to an extent of 25 to 30%, and a 2% aqueous solution has at 20° C.

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a viscosity of 1.6 centipoises. It softens at 135° C. and melts at 140° C.

Nylon short staple fibres 3 denier/6 mm. long were first freed from dressing by being washed with water containing soda and then centrifuged. They were then treated with an aqueous solution of the dispersing material. From part of the dispersion so obtained the fibres were removed and then dried for 2 hours at 65° C. In both cases the fibres were very easily wetted by water, as revealed by the speed at which the fibres settled down on being introduced into water. When the suspension was thoroughly stirred, very even suspensions substantially fast to flocculation were obtained.

These tests were repeated using polyester short staple fibres 1.5 denier/4 mm. long. Similar results were obtained.

#### Example 2

The dispersing material used was an N-ethoxylated polycapraamide which contained 2.68% of nitrogen, corresponding to a degree of ethoxylation of 9 mols of ethylene oxide per gram atom of nitrogen. A 2% aqueous solution of the product has a viscosity of 1.6 centipoises at 20° C. The product melts at 151° C. After this N-ethoxylated polycapraamide has been absorbed on the fibres referred to in Example 1 in the manner described above, they are easy to moisten and are homogeneously distributed in water, that is to say their behaviour is identical with that of the fibres pretreated with the N-ethoxylated copolyamide described in Example 1.

#### Example 3

The fibres used in this example were not melt spun through spinnerets but precipitated, being moist polyester fibrils (product of E. I. du Pont de Nemours and Co.) Type 201, as described for instance in French specification No. 1,214,126, corresponding to 100 grams of dry substance, are stirred for 40 minutes at 35° C. in 10 times their own weight of a 2% solution of N-ethoxylated caproamide, as described in Example 2, then suction-filtered and dried for 48 hours at room temperature over phosphorus pentoxide. In an identical manner 100 grams of dry substance of the above fibrils are treated with water only and then dried.

After having been thoroughly dried for 48 hours and then moistened with water the fibrils treated with N-ethoxylated caproamide could be dispersed homogeneously within a short time. The fibrils treated with water only, on the other hand, are difficult to moisten with water, and they require a much longer mechanical treatment in water to achieve their homogeneous distribution.

From the dispersions of each batch in water sheets weighing about 100 grams per square metre were made on a sheet making machine. The sheet made from the pretreated fibrils has a better water retention capacity than the sheet made from fibrils not pretreated with N-ethoxylated polyamide.

#### Example 4

So-called polynosic short staple fibres 0.4 denier/4 mm. long are ground in a Hollander to 42° S.R. (Schopper-Riegel degrees). 0.36 kg. (30%) of this ground material is suspended within 15 minutes in a stock blending chest in 60 litres of water with the aid of a vane-type stirrer. Another 40 litres of water and, portionwise, 0.48 kg. (40%) of viscose short staple fibres 1.5 denier/4 mm. long are added, and the whole is stirred for 10 minutes. There are then further added 100 litres of water and at the same time, while stirring vigorously,

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0.36 kg. (30%) of polyamide fibres 3 denier/6 mm. long which have been treated with 10 grams per litre of the N-ethoxylated copolyamide described in Example 1 at a goods-to-liquor ratio of 1:20 for 30 minutes at 40° C., then centrifuged and finally dried at 90–105° C. From this stable suspension a sheet is made on a Fourdrinier machine having a fourdrinier wire 40 cm. wide. After drying, this sheet material can be impregnated with resins on a padder, then dried, pressed and calendered between metal rolls and paper rolls at 150–180° C. The papers manufactured in this manner have excellent properties and in them the fibres are evenly distributed, as can be seen on inspection in transmitted light.

#### Example 5

660 grams of aspen sulphite cellulose and 180 grams of fibrillated rag cellulose are thoroughly beaten in 100 litres of water in a stuff blending chest, and 360 grams of polyhexamethylene adipamide fibres (2 denier/4 mm. long) are stirred portionwise into the fibre suspension. Before being mixed with the cellulose pulp the polyamide fibres are treated for 30 minutes at 45° C. with a solution containing per litre 10 grams of an ethoxylated polycapraamide (degree of ethoxylation: 9 moles of ethylene oxide per gram atom nitrogen), then centrifuged and dried in air. The mixture in the stock blending chest is made up to 200 litres, and the fibre suspension (stuff density of 0.2 to 0.3%) is processed on a Fourdrinier machine having a fourdrinier wire 40 cm. wide into a uniform, paper-like sheet. When viewed in transmitted light the paper has a much better appearance than similar paper made with polyamide fibres treated with conventional dispersants. On the Fourdrinier machine no disturbing foaming is observed nor does sticking at the wet end or in the fourdrinier section occur.

We claim:

1. In a process for the production of a paper-like material comprising synthetic fibers, in which process a uniform suspension of said fibers in water is formed into a sheet, the improvement which comprises a step of coating said fibers with a solution consisting of water and N-ethoxylated polyamide, which N-ethoxylated polyamide has a degree of ethoxylation corresponding to 8 to 12 mols of ethylene oxide per gram atom of nitrogen, prior to the formation of said sheet.

2. A process in accordance with claim 1 in which the fibers include fibers selected from the group consisting of natural polymeric fibers and modified natural polymeric fibers.

3. A process in accordance with claim 1 in which the N-ethoxylated polyamide is the N-ethoxylated copolyamide of  $\epsilon$ -aminocapro lactam and hexamethylene diamine adipate.

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