United States Patent Office

3,227,608

Patented Jan. 4, 1966

3,227,608

MANUFACTURE OF SYNTHETIC PAPER SHEET

Winfried Willicks, Hofheim, Taunus, Rolf Grossteinbeck, Oberhochstadt, Taunus, and Gert Tepel, Frankfurt am Main, Germany, assignors to Battelle-Institut e. V., Frankfurt am Main, Germany

No Drawing. Filed Aug. 21, 1964, Ser. No. 391,281
Claims priority, application Germany, Aug. 27, 1963, B 73,276
3 Claims. (Cl. 162—146)

The invention relates to a development of the method disclosed and claimed in application Serial No. 234,673, filed November 1, 1962, for a "Method of Making Paper and Non-Woven Fabric From Synthetic Fibers."

The invention relates to improvements in the preparation of paper and non-woven fabrics from synthetic fibers by the wet process.

According to the method of said earlier invention, a binder is incorporated in paper and non-woven fabrics made on papermaking machinery by the wet process in such a way that the binder joins the fibers only at their crossing points homogeneously throughout the entire web. This is accomplished by admixing to the fibers, prior to the formation of a mat or web, a lubricant which is insoluble in water and in the binder, and a binder which at normal temperature, for instance at 20° C., is solid and as little adhesive as possible but is fluid and becomes adhesive at elevated temperatures of about 80 to 120° C. In the liquid state, lubricant and binder must be incompatible under the operating conditions, that is they must be insoluble in each other. The fiber mixture is then formed to a sheet which is subjected to a heat treatment in the drying part of the paper machine. Due to the admixture of lubricant and binding agent to the fibers prior to the sheet formation, the additives are distributed very evenly in the sheet as it is being formed.

The lubricants employed are compounds producing a coating film on the fibers which presents a gliding surface on which the molten binder can glide in the form of droplets or globules to collect at the intercrossing points of the fibers. After solidification or curing of the binder, the lubricant may be washed out by a suitable solvent so as to leave the fibers of the fabric essentially with their original surface. The lubricants must be insoluble in water and are either liquid at room temperature or must become fluid at temperatures below 120° C., preferably at temperatures not higher than 80° C. In Serial No. 234,673, rosin and rosin soaps and alkyleneglycol esters of aliphatic monocarboxylic esters have been proposed as lubricants. We prefer to use natural and synthetic waxes, such as carnauba wax or montan wax, and other fatty acid esters of higher monohydric aliphatic and phytosterol alcohols, and polyalkylenediols of waxy consistency such as polyethylene glycol of a molecular weight of 950–1050 having a melting point of 34–40° C.

The preferred binder is a latent adhesive constituted by a polyurethane forming mixture of polyesters and polyisocyanates whose isocyanate groups are blocked by an alcohol or phenol and become reactive only at a temperature of about 100° C. Suitable blocking agents are, for instance, phenol, butanol, aliphatic amines, etc. (see Angewandte Chemie, vol. 59A, pp. 265–266 (1947)). The polyesters and polyisocyanates useful for the polyurethane formation are well known and described in many publications, for instance, in the book "Polyurethanes" by Bernard A. Dombrow, publ. by Reinhold Publishing Corp., New York, and in the various publications on "Desmodur" (=polyisocyanates) and "Desmophen" (=polyester) by Farbenfabriken Bayer AG., see also the articles on "Desmodur" and "Desmophen" in Römpp, Chemie-Lexikon.

An illustrative list of polyurethane forming polyisocyanates is given in British Patent No. 892,137, pages 11 and 12. Particularly suitable are, for instance, 2,4-tolylene diisocyanate, hexamethylene diisocyanate, triphenylmethane triisocyanate naphthylene 1,5-diisocyanate, and others.

Suitable polyesters are those of aliphatic polyols such as ethylene glycol, diethylene glycol, hexamethylene glycol, glycerol, and many others with one or more polycarboxylic acids such as adipic acid, sebacic acid, isosebacic acid, phthalic acids and others.

In order to ensure homogeneous distribution of the binder and to prevent or reduce the risk that binder is carried away by the white water during formation of the fiber sheet, the binder and lubricant have been added in form of filaments; this has been done by converting the binder to a filament and then coating the filamentary binder with the lubricant. This method has the drawback to require several process steps to produce the coated filaments.

In said earlier application, it has also been proposed to mix lubricant and binder to a homogeneous mixture and to add said mixture in form of filaments to the slurry of the synthetic fibers.

Said binder-lubricant filaments, however, are quite brittle and lack the flexibility desired for the purpose. We have now found that very flexible binder-lubricant filaments can be obtained by adding to the binder-lubricant mixture an alkyd resin and extruding the homogeneous binder-lubricant-alkyd resin mixture to filaments. The thus obtained filaments are cut to staple fibers of the desired length; said staple fibers are added to the paper stock, which is then processed to a sheet on a conventional papermaking machine. The temperature of the heated rolls of the dryer part of the machine is adjusted to a temperature suitable for curing the liquefied binder which has collected at the intercrossings of the textile fibers. After washing out the lubricant film formed on the synthetic textile fibers, the desired surface properties of said fibers are maintained. The initial wet strength of the web is also essentially maintained.

By adjusting the amount of synthetic textile fibers and binder-lubricant filaments, the number of bonded intercrossings, and thereby the elasticity of the final sheet, can be varied. Generally, the lubricant-binder fibers will be employed in an amount of 30 to 50 percent by weight of the weight of the dry textile fibers.

In the lubricant-binder filaments, the lubricant may be present in amounts of 10 to 100 percent by weight, calculated on the binder, and the amount of alkyd resin may be in the range of 5 to 25 percent, calculated on the lubricant-binder mixture.

A wide range of alkyd resins may be used, for instance glycerol or pentaerythritol esters of phthalic anhydride, short oil alkyds modified with linseed oil or other vegetable oils or with saturated lower fatty acids, medium oil alkyds based on dehydrated castor oil, styrene modified alkyds, and others.

Conventional driers such as cobalt or rare earth salts of naphthenic acid, 2-ethylhexoic acid, and other acids used for this purpose may be added to shorten the curing time of the binder after the web has been formed.

The following example is given to illustrate and not to limit the invention.

Example

Polyester fibers were beaten in water, and the fibrous mass was introduced into the headbox of a paper forming machine. At the same time, binder-lubricant filaments cut to staple length were added, and the fiber mixture was slurried with water to a dispersion containing a solids concentration of about 0.01. Said highly diluted fiber

dispersion was then passed onto the Fourdrinier wire where the desired web was formed and the major part of the water was separated from the fibers.

The lubricant-binder filaments had been prepared by extruding the following mixture, all percentages being given by weight:

	Percent
Desmodur AP stable	33
Desmophen 1200	18
Alkydal RD–18	10
Alkydal BG	5
Wax E	30
Soligen cobalt (Gebr. Borchers AG.)	4

Desmodur AP stable is toluylene diisocyanate whose isocyanate groups are blocked by phenol.

Desmophen 1200 is a polyester of adipic acid with a mixture of diols and triols, dissolved in acetone and methylglycol acetate.

Alkydal RD–18 is prepared from phthalic acid and trimethylolpropane and modified with 25% saturated low molecular fatty acid.

Alkydal BG is an unmodified phthalic acid resin.

Wax E is an ester wax marketed by Badische Anilin- & Sodafabrik. It is a montan wax (C, number 28) esterified with ceryl and myricyl alcohol and has a melting point of 79–82° C., a setting point of 73–75° C., an acid number of 17–25 and a saponification number of 158–178.

Soligen cobalt is cobalt naphthenate.

After passage through the wire section of the paper-forming machine, the binder-lubricant filaments were distributed throughout the web. In the drying section, the web passed over a plurality of heated rolls, the first of which had a surface temperature of 120° C. The web was passed through a heating zone of a total length of about 150 m. within about 15 minutes and was then rewound.

In the heating zone, the lubricant-binder filaments melted and formed individual droplets which run on a thin layer of the liquid wax, formed on the textile fibers, to the intercrossing points of the fibers, where they started being cured. Due to the presence of the drier catalyst, the cure was completed within 3 to 5 minutes at 150° C.

For the manufacture of the web, all synthetic fibers can be used which, due to their preparation from a melt, by extrusion or spinning, and by the subsequent treatments, have a smooth non-fibrillated surface; stretched, crimped, or hollow fibers may be used. Examples of such synthetic fibers are regenerated cellulose fibers from viscose or cuproammonium; polyamides as obtained by the condensation of hexamethylene diamine salt with adipic acid (nylon 66) or ε-caprolactam (nylon 6); polyesters from dicarboxylic acid, such as terephthalic or isophthalic acid, with diols or polyols (Diolene, Terylene, Dacron); polyvinyls, e.g. from vinyl chloride, vinyl acetate, styrene, vinylene chloride, and copolymers thereof; polyacrylics from acrylonitrile (e.g. Dralon); copolymers of vinyl compounds and/or styrene and acrylonitrile; polyolefins, for instance polyethylenes and polypropylenes. Also glass fibers may be used.

The synthetic fibers may be substituted up to 50 percent by natural fibers without requiring modification of the described procedure.

We claim:

1. A method of preparing an unwoven web material consisting to at least 50 percent by weight of synthetic textile fibers, the balance being natural textile fibers, on a conventional paper-forming machine comprising adding to said textile fibers, prior to their passage through the paper-forming machine, about 30 to 50 percent, calculated on the dry weight of said textile fibers, of fibers consisting essentially of a homogeneous mixture of

(a) a polyurethane forming mixture of a polyester having at least one free hydroxyl group and a polyisocyanate whose isocyanate groups are blocked by a member of the group consisting of alcohols and phenols but are reactive at a temperature of about 100° C.,

(b) a wax having a melting point below the melting point of said polyurethane, and

(c) an alkyd resin,

the proportion of said polyurethane forming mixture to said wax being about 1:0–1–1, and the amount of said alkyd resin being about 5 to 25 percent by weight of the total polyurethane-wax mixture.

2. A filamentary composition, suitable as addition in the manufacture of unwoven web material, consisting essentially of a homogeneous mixture of

(a) a polyurethane forming mixture of a polyester having at least one free hydroxyl group and a polyisocyanate whose isocyanate groups are blocked by a member of the group consisting of alcohols and phenols but are reactive at a temperature of about 100° C.,

(b) a wax having a melting point below the melting point of said polyurethane, and

(c) an alkyd resin,

the proportion of said polyurethane forming mixture to said wax being about 1:0.1–1, and the amount of said alkyd resin being about 5 to 25 percent by weight of the total polyurethane-wax mixture.

3. The filamentary composition of claim 2 containing, in addition, a drier in an amount efficient to cure said polyurethane-forming mixture on heating.

References Cited by the Examiner

UNITED STATES PATENTS

2,680,995	6/1954	Frost	162—185
2,962,762	12/1960	Hartmann	162—157
3,049,466	8/1962	Erlich	162—157
3,053,609	9/1962	Miller	162—157
3,096,231	7/1963	Griggs	162—157

FOREIGN PATENTS

572,962	10/1945	Great Britain.

DONALL H. SYLVESTER, *Primary Examiner.*