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PAPER AND PROCESS FOR MAKING SAME OF SYNTHETIC FIBERS BONDED AT THEIR INTERCROSSING POINTS BY A THERMOPLASTIC POLYAMIDE RESIN

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ABSTRACT OF THE DISCLOSURE

In an unwoven web of synthetic fibers, the crossing points of the fibers, and only those points, are bonded by passing an aqueous slurry of said fibers and fibers of a thermoplastic adhesive together with a lubricant through a paper-making machine, thereby forming a temporary film of the lubricant on the fibers. The temporary film provides a gliding surface for the molten adhesive which collects at the crossing points.

The invention relates to novel non-woven fibrous structures made from synthetic fibers and bonded at the crossing points of the fibers, and to a method of producing such structures.

Various methods of making such products are known which may be divided into dry processes and wet processes. While the dry processes require special machines, the wet processes may be carried out in conventional paper-making machines.

In the known methods, a binder is generally applied to the fibers or to an already formed sheet or mat, whereby the fibers are bonded at their crossing points. Subsequently, the binder is cured or hardened by a heat treatment of the mat. The thus obtained web has the drawback that each fiber is coated with a thin cured film of the binder and that, therefore, not the original fiber but the binder film largely determines the properties of the web.

In order to avoid this drawback, a dry process has been developed where a conventional staple fiber is treated with a hydrophobic or water repellent agent and the dry felt of such fibers is sprayed with an aqueous solution or dispersion of a binder. The droplets or globules of such solution or dispersion do not spread on the hydrophobic fiber surface but flow thereon to the crossing points of superposed fibers and remain there suspended. On heating, the water is evaporated and the binder cured.

Such a method cannot be used in the wet process on papermaking machines because an aqueous solution or dispersion of the binder would be flowed away in the wet end of the machine with the waste water; when applied in the dry end, by spraying, the binder could not penetrate deeply enough into the rather strongly compacted sheet.

The present invention is a further development of the invention disclosed and claimed in Ser. No 234,673 now Patent No. 3,200,033, filed Nov. 1, 1962 by us together with Winfried Willicks and Herfried Mindermann.

In said application, we have described a method which makes it possible to incorporate a binder in paper and non-woven fabrics made on papermaking machinery in such a way that the binder joins the fibers only at their crossing points homogeneously throughout the entire web. Preferably the binder is added only in the amount required for bonding the crossing points of the fibers.

The process of Patent No. 3,200,033 consists in 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 synthetic fibers employed include polyamides such as nylon 66 (hexamethylene diamine-adipic acid condensation product), nylon 6 (polycaprolactam) and other nylon products (nylon 6/10; nylon 11); polyesters from dicarboxylic acids, such as terephthalic or isophthalic acid and diols or polyols (Dacron, Diolen, Terylene); vinyl polymers and copolymers on vinyl chloride or vinyl acetate basis (Vinyon); vinylidene chloride polymers and copolymers (Saran); polyacrylics (Dralon; Orlon; Acrylan; Creslan; Acrylast) and copolymers, e.g. of acrylonitrile with styrene; polyolefines such as polyethylene or polypropylene; polytetrafluoroethylene (Teflon); modified and regenerated cellulose fibers such as viscose, cuproammonium, cellulose acetate; also glass fibers and other synthetic fibers may be used, as well as mixtures of different fibers. Up to 50 percent of the synthetic fibers may be replaced by natural fibers.

For lack of a better term, we have called "lubricants" compounds used to produce on the fiber a coating film presenting a gliding surface for the binder droplets which collect finally at the crossing points of the fibers and form there a bond, without covering the remaining part of the fibers in the finished article. The lubricants must be insoluble in water; they may be liquid at room temperature and must have a melting point below 120° C., preferably not higher than 80° C. In the liquid state, they must be unmiscible with the liquid binder. Suitable lubricants are rosin and rosin soaps, or waxes such as paraffin wax. Preferred lubricants are compounds conventionally used as plasticizers for resins, particularly phthalates such as dimethylglycol phthalate, benzylbutyl phthalate, dibutyl phthalate, phthalic esters of C_6–C_9 alcohols such as dioctyl phthalate, and others. Also esters of aliphatic or aromatic dicarboxylic acids with aliphatic and/or aromatic monohydric or dihydric alcohols, such as benzyl octyl adipate, may be used. Suitable plasticizer-lubricants available in commerce are, for instance, sold under the trade-names Scadoplast RA 3L (polyester of adipic acid) and Scadoplast RS 20 (polyester of sebacic acid); polyesters of phthalic acid are marketed under the trade-names Scadoplast W L and plasticizer CEL (Farbenfabriken Bayer AG.)

The lubricant and binder may be added to the fibers either separately or together. This can be done in various ways. For instance, the fibers may be first contacted with the liquid or liquefied lubricant so as to receive a thin coating of the same; subsequently, the binder is applied in a similar manner so as to produce a thin film on the lubricant coating. Another possibility consists in mixing the lubricant and binder to a homogeneous mixture which is added in form of a powder or of fibers to the slurry of the fibers to be bonded in the box of a paper machine. In another modification of the process, the binder is converted to a filament and coated with the lubricant, whereupon the coated binder filament is comminuted to staple fiber and added to the aqueous slurry of the fibers forming the mat.

The use of the binder in a filamentary foam has the

advantage to prevent or reduce the risk that binder is carried away by the white water during formation of the sheet. Therefore, it is not necessary to use an excess of the binder.

If not already added in liquid foam, the lubricant is molten in the drying part of the machine and covers the fibers with a thin film. When the temperature is further increased, the binder added in powdery or filamentary form melts also and flows on the coated fiber surface to the crossing points of the fibers. The droplets or globules formed at said crossing points form a kind of knot. The even distribution of the binder in the mat ensures a bonding of the crossing points not only at the surface of the sheet but also in the inside layers thereof.

Heretofore, the binder used was a polyurethane forming mixture of polyesters and polyisocyanates whose isocyanate groups were blocked by an alcohol or phenol and became reactive only at a temperature of about 100° C. Said latent adhesive agent, when exerting its bonding properties on heating, had then to be cured by further heating.

We have now found that the bonding operation can be considerably simplified and shortened when the previously used polyurethane forming mixtures, which have first to be reacted to become adhesive and then must be cured, are replaced by certain thermoplastic adhesives known under the trade-name "Versamides" manufactured by General Mills, Inc. Said adhesives are polyamide resins resulting from the reaction of polymeric fat acids and polyamines. Their preparation is described in various patents, for instance Nos. 2,450,940, 2,728,737, 2,767,089, 2,811,459, 2,908,584, 2,929,117.

The resins are applied in the same manner as set forth above for the binder. For instance, they may be spun to filaments which are then cut to staple fibers of the desired length and admixed in the machine chest to the fibrous slurry from which the web is formed. The synthetic fibers may be mixed previously with the lubricant, or the binder filaments may be coated with the lubricant. While the batt passes through a heating zone, the lubricant melts and coats the synthetic fibers. When the temperature is further raised, also the thermoplastic binder fiber melts and contracts to separate droplets. As the synthetic fiber and the droplets of the binder material is separated by the film of the lubricant, said droplet glides to the next crossing point. When the web is cooled, the droplets solidify and bond the fibers together at their intersections.

The polyamide resin must be resistant to oxygen at temperatures up to 150° C. and must not crack. It must further combine a suitable melting point, for instance about 100 to 120° C., with a suitable viscosity in the molten state at said temperature, for instance about 2500–3500 centipoises, which viscosity corresponds to a flow-out time of about 50 to 80 seconds at 110° C. in the DIN-cup of the German testing standards having a 4 mm. nozzle.

It is difficult to produce individual polyamides presenting both the desired melting point and the desired viscosity; we prefer to obtain such properties by mixing a polyamide of relatively high melting point and viscosity with a polyamide of lower melting point and viscosity in suitable proportions.

It is, of course, also possible to add to the fibrous slurry the thermoplastic binder also in powdery form homogenized with the lubricant.

The lubricant-binder mixtures or filaments may contain the lubricant in amounts of 2 to 5 percent by weight, the balance being polyamide resin. About 25 to 50 percent, calculated on dry fiber weight, are added to the fibrous slurry.

The invention will be further illustrated by the following example showing a preferred mode of operation.

EXAMPLE

Nylon staple fibers of 6 mm. length and 1.5 den. were beaten in water and passed into the machine chest. Subsequently, staple fibers cut from binder-lubricant filaments were introduced into the chest in an amount of 30 percent by weight, calculated on dry weight basis of the nylon fibers.

The binder-lubricant fiber had been prepared by mixing 85 percent by weight of a polyaminoamide resin A with 15 percent of a polyaminoamide resin B. Resin A had been obtained by a reaction as described in Example 3 of Patent No. 2,728,737 and had a viscosity of 30–45 poise at 150° C. and a molecular weight of about 7400. Resin B had been obtained by a reaction as described in Example 1 of Patent No. 2,767,089 and had a viscosity of 10–15 poises at 150° C. The mixture was heated at 150° C. and the molten resin was spun through a spinneret to a filament. The solidified filament was passed through a bath of dimethylglycol phthalate and then between squeeze rolls to strip the excess of the dimethylglycol phthalate. The filament thus provided with a thin film of the lubricant was finally wound upon a reel and later cut to staple fibers of the desired length which were added to the fibrous slurry in the chest.

The fibrous mixture in the chest is then formed into a web on a conventional papermaking machine. During the manufacture of the paper, the web is heated in the dryer section of the machine for a short time at a temperature of about 150° C.; thereby, the melting point of the thermoplastic polyamide adhesive is exceeded and the adhesive melts, which produces a separation of the lubricant and adhesive.

The lubricant forms a thin coating on the nylon fibers while the polyamide resin breaks up to droplets which migrate on the lubricant film to the points of intersection of the fibers. Following the heat shock, the web is cooled, whereby the resin droplets solidify at said points of intersection and form a firm bond. Subsequently, the lubricant is washed off, and the web is wound up.

We claim:

1. A method of preparing an unwoven web material from at least 50 percent of synthetic fibers, comprising adding to an aqueous slurry of said fibers passing through a papermaking machine a water insoluble lubricant material selected from the group consisting of natural and synthetic resins, waxes, and plasticizers, said material forming in the liquid state a removable film on said fibers, and fibers of a thermoplastic polyamide composition, said lubricant material having a melting point not higher than 120° C. and said polyamide composition having a melting point higher than said lubricant material, said polyamide composition having the polyacyl group of polymeric fat acids, and the polyamino group of an aliphatic polyamine, subjecting said fibers to a temperature sufficient to melt said polyamide composition, said molten polyamide composition collecting on the film of the lubricant material formed on the fibers as droplets at the intercrossing points of the fibers and solidifying thereon cooling to form bonds at said intercrossing points.

2. The method as claimed in claim **1** wherein said lubricant material is a dialkylglycol phthalate.

3. The method as claimed in claim **1** wherein said polyamide fibers are coated with said lubricant material.

4. The method as claimed in claim **1** wherein said lubricant material and said polyamide fibers are applied in intimate mixture.

5. An unwoven fibrous product consisting essentially of intercrossing non-fibrillating synthetic fibers, the fibers being bonded at their intercrossing points by a thermoplastic polyamide resin, the polyamide having the polyacyl group of polymeric fat acids and the polyamino group of an aliphatic polyamine, the fibers being otherwise substantially free of polyamide coating.

6. The method as claimed in claim **1** wherein up to 50

percent of the synthetic fibers in said web material are replaced by natural fibers.

7. The method as claimed in claim 1 wherein lubricant and binder are added in a total amount of at least 25 percent, based on the dry weight of the fibers, the lubricant comprising about 2 to 5 percent of said total amount.

8. The method as claimed in claim 1 wherein said polyamide fibers consist of a mixture of a high melting polyamide having at 150° C. a viscosity of 30–45 poises and a low melting polyamide having at 150° C. a viscosity of 10–15 poises, said mixture having a susbtantially uniform

melting point of 100 to 120° C. and a viscosity of 25 to 35 poises.

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