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POLYAMIDE FIBERS CONTAINING KAOLINITE AND PROCESS OF PREPARATION

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4 Claims. (Cl. 260—37)

ABSTRACT OF THE DISCLOSURE

Polycarbonamide fibers possessing improved friction properties are prepared through incorporation of kaolinite platelets specified dimension and certain heat stable deflocculant.

The following is a continuation in part of application No. 273,852 filed Apr. 18, 1963, now abandoned.

This invention relates to improved textile products such as yarns, filaments and fibers, and fabrics prepared therefrom. More particularly, it relates to an improved method of preparing polyamide textile fibers possessing novel luster and friction properties.

Melt-spun polyamide filaments are characterized by a smooth surface which gives high yarn running friction, high static friction, and high surface luster. Attempts have been made to reduce friction and luster by special sizing treatments or other surface coatings, and by adding finely divided inert materials to the polymer before spinning. Neither approach has been completely successful. The addition of many finely divided inert materials to molten polymer, for example, leads to the plugging of sand packs in the melt-spinning operation with a consequent reduction in efficiency of operation. On a commercial scale the reduction in efficiency has been prohibitive. Reduced luster has been achieved by adding pigments such as titanium dioxide, but TiO_2 also increases fiber opacity, which in many instances is undesirable. Furthermore, a conventional TiO_2 addition does not provide the desired reduction in friction.

It is an object of this invention to provide a procedure for preparing synthetic textile fibers having reduced surface luster. A further object is the provision of a method for preparing melt-spun polyamide fibers exhibiting reduced running and static friction. A further object is a procedure for incorporating an inert material in a polyamide fiber without significant loss of strength in the fiber. A still further object is the provision of a procedure for incorporating a finely divided inert material in a polyamide fiber without encountering excessive pack plugging during melt-spinning.

These and other objectives are achieved in a proces for preparing polyamide textile fibers which comprises adding to a mixture of polycarbonamide-forming reagents, or low molecular weight polymer, an aqueous slurry of (1) thin, hexagonal platelets of

$$Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$$

having the kaolinite crystal structure, with an average particle size of 0.2–2.0 microns and being substantially free of all particles larger than about 5 microns, combined with (2) a heat stable deflocculant at a concentration of from 0.05–3% by weight based on weight of kaolinite. The mixture is polymerized to give a synthetic linear polycarbonamide having an intrinsic viscosity of at least 0.4 which is subsequently melt-spun and drawn to give a textile fiber.

By this procedure, fibers containing as much as 17% by weight of kaolinite may be readily prepared. For most

textile purposes, it is preferred that the fibers contain from 0.5–5.0% by weight of kaolinite.

The above polymerization produces a synthetic linear polycarbonamide fibers having randomly dispersed therein hexagonal platelets of kaolinite so oriented that the long dimension of the platelet is substantially parallel to the fiber axis. Microscopic inspection reveals that each platelet is surrounded by an elongated void where the polymer has separated from the particle and that the filament exhibits a roughened surface. The filament surface has numerous small bumps, 1–3 microns in size, which are elongated in the direction of the fiber axis. The filaments show a subdued surface luster combined with markedly reduced dynamic and static frictional properties. Fiber strength is substantially equal to that of similar fibers containing no kaolinite.

The fibers produced by the process of this invention show reduced and more uniform running tensions, which leads to more uniform fabric structure. Unexpectedly, lower static friction provides improved fabric liveliness and recovery properties. In the knitting of hoisery, improved hosiery length control is achieved.

By adding varying quantities of conventional TiO_2 delustrant in the above process, it is possible to achieve a wide variety of combinations of surface luster and opacity. By proper choice of the kaolinite/TiO_2 ratio the polyamide yarn manufacturer is now able to prepare yarns with subtle luster effects hitherto unobtainable in a commercial process.

The above method of modifying polycarbonamides with kaolinite is also useful in preparing improved nylon molding powders. The nucleating effect of kaolinite raises the polymer freezing temperature by as much as 10° C. in comparison with unmodified polymer. As a result, normal injection molding cycle times may be cut in half, or less, without detactable losses in physical properties such as toughness, tensile strength or extensibility. For best molding performance, concentrations of 0.05–0.15% by weight kaolinite in polymer are preferred.

Hexagonal platelets of aluminum silicate having the kaolinite crystal structure are described by C. E. Marshall in The Colloid Chemistry of the Silicate Minerals, Academic Press, Inc., New York, New York (1949) pp. 49 and 72. For the purposes of this invention it is necessary to use a highly purified kaolinite which is substantially free of all metal oxides other than those of aluminum and silicon. It is further necessary to use a kaolinite in which the platelets have average maximum dimensions of from 0.2–2.0 microns and which is substantially free of platelets larger than 5 microns. The presence of even small quantities of particles larger than about 5 microns results in the plugging of sand packs in the melt-spinning operation. On the other hand, particles smaller than about 0.2 microns do not give the filament surface roughening effect which is necessary for reduced running tensions. In order to achieve the particle size range desired, highly refined commercial grades of kaolinite are further processed in accordance with the procedure described below.

The aqueous slurry of kaolinite is preferably prepared by mixing a sufficient quantity of a commercially available, highly refined kaolinite having a nominal particle size in the desired range with water to give a 20–60% slurry, along with a heat stable deflocculant as defined hereinafter, at a concentration of from 0.05–3% by weight based on weight of kaolinite. Preferably, the acidity of the slurry is adjusted to give a pH of 7 to 9. The slurry is stirred for at least 1 hour, preferably 4 hours, and then diluted to 10–25% solids and allowed to settle for a period of at least 6 hours, preferably about 20 hours. After settling, the slurry is decanted from the settled material, again stirred and sufficient water added to adjust the concentration to the desired level for intro-

duction into the polymerization system. Usually a slurry concentration of 10–25% is preferred when the polymer is to contain 0.5–2% kaolinite. After adjusting the concentration, the slurry is passed through a filter having a mean porosity rating of 5 microns (Cuno Filter, Cuno Engineering Corp., Meriden, Conn.) and thus made ready for injection into the polymerization system.

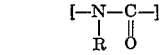
By "heat stable deflocculant" is meant a material which promotes dispersion of kaolinite in water by breaking agglomerates into single platelets and which continues to function in molten polyamides, i.e., is not chemically reactive. Furthermore, it does not lead to discoloration, illustrative of the reagents suitable in the process of this invention are tetrasodium pyrophosphate, sodium silicate, sodium hexametaphosphate, colloidal silica, lithium hydroxide and low molecular weight sulfonated naphthalene-formaldehyde condensates. The presence of a suitable deflocculant in the correct amount is essential to provide deflocculation of the kaolinite particles in the aqueous slurry and to prevent agglomeration of particles during the polymerization process.

The size or maximum dimension of the kaolinite particles is best determined by means of the electron microscope (ASTM E20–51T).

The prepared kaolinite slurry is incorporated in the polymer by addition to the aqueous solution of nylon salt, either at the beginning of the polymerization or at some later point during the polymerization procedure. Preferably, the slurry is added after polymerization has started but before an appreciable viscosity change has been realized according to the general procedure of Hoff in U.S. Patent No. 2,278,878. In continuous polymerization procedures, it is preferred that the kaolinite slurry be added according to the method of Heckert, as described in U.S. Patent No. 2,689,839.

Following the addition of the slurry to the polymerization mixture, the process is continued in the conventional manner to give a fiber-forming polymer. The polymer may be forwarded in the molten state through conduits to a spinning machine and there melt-spun into filaments which are subsequently drawn to give strong textile fibers. Alternatively, the polymer may be extruded as a ribbon, quenched, cut to flake and subsequently remelted for spinning into textile fibers on conventional melt-spinning equipment.

The term "synthetic linear polycarbonamide" is intended to include any linear polymer having recurring units of the formula



as integral parts of the main polymer chain, wherein R is hydrogen or a monovalent hydrocarbon radical, the average number of carbon atoms separating the amide groups being at least 2, said polycarbonamide having an intrinsic viscosity of at least about 0.4, as defined in U.S. Patent No. 2,130,948. Particular polycarbonamides included among those which are useful in this invention are as follows: polyhexamethylene adipamide, polyhexamethylene sebacamide, polymerized 6-aminocaproic acid, polytetramethylene sebacamide, polytetramethylene adipamide, polymetaxylylene adipamide, the polyamide from bis(4-aminocyclohexyl) methane and azelaic acid, sebacic acid or decamethylene-1,10-dicarboxylic acid, and the polyamide from 2-methylhexamethylene diamine and terephthalic acid. The invention is also applicable to various copolymers, ether block or random, such as the coploymer of polyhexamethylene adipamide and polyhexamethylene isophthalamide, and the copolymer of polyhexamethylene adipamide and polyhexamethylene-t-butyl isophthalamide. Other suitable polycarbonamides are disclosed in U.S. Patents Nos. 2,071,251 and 2,071,253.

The following examples are cited to illustrate the invention. They are not intended to limit it in any way.

Example I

Two hundred parts of a commercially available kaolinite powder, purified by an ultraflotation process (U.S. Patent No. 2,990,958) to substantially eliminate metal oxides other than aluminum and silicon oxide and classified by centrifugation to provide an average maximum dimension of 0.55 micron, are mixed with 300 parts water and 1.2 parts tetrasodium pyrophosphate decahydrate in a high-shear mixing mill. After milling for 1 hour, the mixture is diluted with 300 parts water, transferred to a tank and stirred for 24 hours. The slurry is allowed to settle for 20 hours, then decanted from the settled material and diluted with water to a concentration of 20% solids. The diluted slurry is then passed through a standard commercial filter having an average pore size of 5 microns and continuously stirred until used.

Examination of the particles in the final slurry by means of an electron microscope indicates substantially no particle dimensions above about 5 microns. The average maximum dimension is about 0.5 micron.

A stainless steel autoclave, modified as in U.S. Patent No. 2,278,878, is charged with 467 parts of an aqueous solution containing 47% by weight hexamethylene diammonium adipate and 2.1 parts of an aqueous solution contaning 25% by weight acetic acid, purged of air, filled with nitrogen and heated until its temperature reaches approximately 215° C. at 250 p.s.i. At this stage, bleeding off of water vapor is begun and an appropriate amount of the previously prepared kaolinite slurry is added to give the concentrations indicated in Table I. The polymerization cycle is continued as described in Example I of U.S. Patent No. 2,163,636, giving a molten polymer having a relative viscosity of 42 (relative viscosity is defined in U.S. Patent No. 2,385,890). The polymer is extruded in the form of a ribbon upon a casting wheel, quenched, and cut into chips suitable for remelting and melt-spinning as taught in Example I of U.S. Patent No. 2,289,774. Even at the higher concentrations of kaolinite, the polymer is noted to have the same degree of transparency shown by polymer containing no kaolinite, i.e., there is no delustering effect of the type achieved with TiO_2.

The polyhexamethylene adipamide chips obtained above are melt-spun into a 34 filament yarn, using the apparatus of Greenewalt (U.S. Patent No. 2,217,743), operating at a temperature of 290° C. The extruded filaments are quenched in cross-flow air, wound on a package, and subsequently drawn 3.5 times their extruded length, using the apparatus disclosed in U.S. Patent No. 2,289,232, to produce a yarn having a break elongation of 30%. Single filament friction measurements, as described hereinafter, are carried out on each of the several yarns. The data recorded in Table I show a marked reduction in friction obtained by the addition of kaolinite particles to the polymer.

TABLE I

Kaolinite concentration	Coefficient of friction (f_{rd})
0.0	0.73
0.5	0.50
1.0	0.51
2.0	0.35
4.0	0.31
10.0	0.35
17.0	0.33

The coefficient of friction (f_{rd}) is measured by draping a single filament over a rotating cylindrical polished chrome mandrel (about 180° contact) with one end of the filament attached to a strain gauge and the other end attached to a free-hanging weight of about 0.3 g. After placing a drop of mineral oil on the mandrel and then wiping it off, the mandrel is rotated with a surface speed of 77 yards/minute in a direction which exerts tension on the strain gauge. The coefficient of friction (f) is calculated from the equation $T_2/T_1=e^{fa}$ where T_2 is the out-

put tension, T_1 is the input tension, and a is the angle of wrap in radians.

The yarns produced above show a subtle decrease in surface luster, which becomes more pronounced as the concentration of kaolinite increases. The effect achieved is quite different from that obtained with conventional amounts of pigments such as TiO_2 and does not suffer from the chalkiness exhibited by nylon yarns containing higher concentrations of TiO_2.

Example II

The general procedure of Example I is followed to produce a yarn containing 2% by weight of kaolinite. Melt-spinning is carried out at 290° C. using a sand pack of the type disclosed in U.S. Patent No. 2,266,368. Over a period of 24 hours, the average pressure rise in the sand pack is approximately 15 p.s.i.g./hour, which is considered acceptable for commercial operations.

For comparison, the above procedure is repeated in all details with the exception that no tetrasodium pyrophosphate is used in the kaolinite slurry. The rate of pressure rise in the sand pack is found to be approximately 280 p.s.i.g./hour. After 20 hours, spinning is terminated because of excessive pack pressure.

Example III

For comparison purposes, a highly purified commercial grade of kaolinite having an average particle size of 7 microns is used to prepare a nylon yarn containing 2% by weight of kaolinite, according to the general procedure of Example I except that the settling time of the slurry is fifteen minutes and the filtration step, using the five-micron filter, is eliminated. During melt-spinning, a rapid pack pressure increase is experienced and spinning is terminated after four hours because of excessive pack pressure.

Example IV

The general procedure of Example I is repeated, except that 0.4 part of sodium silicate is used in place of the tetrasodium pyrophosphate. Yarns containing 1.0, 2.0, and 4.0% by weight of kaolinite are prepared, with the effects on coefficient of friction and surface luster being substantially equivalent to those reported in Example I.

Example V

The general procedure of Example I is repeated, using a purified, classified kaolinite from a different commercial source, but having the same nominal specifications as that used in Example I. The slurry is prepared with 1.6 parts of sodium hexametaphosphate, instead of tetrasodium pyrophosphate. Yarns are produced having kaolinite concentrations of 0.5, 2.0 and 10.0% by weight. The effect on surface luster and coefficient of friction is substantially the same as reported in Example I.

Example VI

A 70 denier (7.8 tex.), 34 filament yarn composed of intermingled filaments of the monopolymer polyhexamethylene adipamide and the copolymer of polyhexamethylene adipamide containing 8 mol percent polyhexamethylene isophthalamide is prepared according to the procedure described in Example IV of Belgian Patent No. 615,857. The procedure is modified by adding to each polymer, during polymerization, a sufficient quantity of 20% aqueous slurry of kaolinite, prepared according to the procedure of Example I, to give a final concentration of 5 weight percent of kaolinite in the yarn produced. In addition, 2½% by weight of Igepal 880, an ethylene oxide condensation product of nonylphenol, is incorporated into the copolymer as an antistat. The 11% hot relaxation treatment given after drawing produces a yarn having no loops due to differences in instantaneous retraction upon removal of the yarn from its package, with consequent ease of handling during mill processing. When the yarn is woven into a fabric and subjected to a boiling water treatment, a differential shrinkage of 50% is observed between different filament species, which produces a warmer, dryer hand and higher bulk than observed with conventional nylon fabrics of the same general structure. Furthermore, the fabric shows improved liveliness in comparison with similar fabrics prepared from a control yarn made in the same way, but with no kaolinite added. The liveliness is attributed to the lower static friction shown by fibers containing dispersed kaolinite.

Example VII

Nylon flake of the 6—6 variety, containing 4 weight percent kaolinite, is prepared according to the general procedure of Example I. The flake is remelted in a screw extruder along with 5 weight percent of polyethylene oxide having an average molecular weight of 20,000 and melt-spun and drawn to give a 68 filament yarn having a denier of 1000 (110 tex.). The filament cross-section is similar to that shown in FIG. 2 of U.S. Patent No. 2,939,201. The yarn is subjected to 70 p.s.i.g. steam in a jet, according to the procedure described in French Patent No. 1,215,344, to give a bulky carpet yarn having permanent antistatic and anti-soiling properties. It is noted that the yarn exhibits a considerably reduced surface luster in comparison with an equivalent yarn prepared without the addition of kaolinite.

Example VIII

An aqueous solution of hexamethylene diammonium adipate and caprolactam in the mol ratio 85/15 is charged to an evaporator and heated at atmospheric pressure to remove excess water. The charge is then transferred to an autoclave, heated to a temperature of about 210° C. and brought to a pressure of 250 p.s.i. At this point, a 20% aqueous slurry of kaolinite prepared with tetrasodium pyrophosphate as in Example I is added in sufficient quantity to give a concentration of 2% kaolinite in the final polymer. Heating is continued at 250 p.s.i. until the temperature reaches 230° C., at which time 0.003% by weight manganese hypophosphite is added. Temperature is further increased to 274° C. and then the pressure gradually reduced to atmospheric with the temperature rising finally to 279° C. The polymer is extruded from the bottom of the autoclave in the form of a ribbon which is quenched on a water-cooled casting wheel and cut into ¼-inch flake in the conventional manner. The polymer flake contains 85% of 6—6 units and 15% of 6 units and has a relative viscosity of 42.

The polymer is melt-spun and drawn by known procedures to give a 15 denier (1.7 tex.) monofil hosiery yarn. The yarn is subsequently hot relaxed by passing it over a hot plate at a temperature of 175° C. with sufficient exposure time to reduce boil-off shrinkage to about 13%. Yarn tenacity is about 6 grams per denier and break elongation about 40%. Hydrodynamic friction coefficient measurements on the yarn indicate a reduction of more than 30% in friction in comparison with similar yarns prepared without the addition of kaolinite. It is further noted that the test yarn containing kaolinite shows reduced packaged yarn growth with a consequent reduction in yarn sloughing from the package. The test yarn also shows a reduced surface luster when compared with the control yarn.

Example IX

A fiber-forming polyamide is prepared from bis(paraminocyclohexyl)methane and azelaic acid by known procedures. During the early part of the polymerization, a sufficient quantity of kaolinite slurry of Example I is added to give a concentration of 3% kaolinite in the final polymer. The polymer is melt-spun and drawn to a 70 denier (7.8 tex.) 34 filament yarn in which the individual filaments have a trilobal cross-section. Woven fabrics prepared from these yarns show desirable differences in liveliness and handle, as well as reduced luster, when compared with fabrics prepared from similar yarns containing no kaolinite. Single filament friction measurements indicate

that the kaolinite has produced a reduction of 40% in hydrodynamic friction and also a substantial reduction in static friction.

Example IXa

Repeating the procedure of Example IX, sufficient of the kaolinite slurry is added to a polymerizing mixture of bis(4-aminocyclohexyl)methane and dodecanedioic acid to give a concentration of 2% in the final polyamide. The dispersing agent for the kaolinite is sodium silicate. The kaolinite particles are mostly below 2 microns in size, with less than 10% of the number in the two to three micron range.

The polymer mixture is melt spun to a yarn having 10 filaments of round cross section and then drawn to a final denier of about 18. Tests show that single filament friction is reduced from 0.95 (without kaolinite) to 0.54 with additive.

Example X

Polyhexamethylene adipamide containing 1% by weight kaolinite and having a relative viscosity of 40 m-cresol is prepared by adding, during polymerization, an aqueous slurry of kaolinite dispersed with colloidal silica at a concentration of 0.5% by weight based on weight of kaolinite. This polyamide is used to prepare side-by-side composite filaments along with polyethylene terephthalate which was prepared according to the general procedure described in U.S. Patent No. 2,465,319 and modified by adding a glycol slurry of kaolinite during polymerization in sufficient quantity to produce a concentration of 1% by weight kaolinite in the final polymer. The polyethylene terephthalate has a relative viscosity of 29 in a solution of trichlorophenol (7 parts) and phenol (10 parts). The polymers are melted separately and the melts led separately to a spinning unit of the type shown in FIG. 6 of U.S. Patent No. 2,931,091 and there joined and extruded in side-by-side arrangement through spinneret holes having a Y-shape. The two polymers are extruded in the ratio of 37% polyamide to 63% polyester by weight. The filaments produced have a trilobal cross-section (U.S. Patent No. 2,939,201) with a modification ratio of about 2.0. The polyamide part of the cross-section is ribbon shaped and the polyester portion bell shaped. The filaments are attenuated by winding them up at 500× the speed at which they leave the spinneret. The quenched filaments are drawn at 90° C. over a pin using a draw ratio of 3.7 and then wound into a package. The final drawn yarn consists of 26 filaments of 2.7 denier (0.3 tex.) each, the polyamide component being 1.0 denier (0.11 tex.) and the polyester component 1.7 denier (0.19 tex.). The yarn shows spontaneous crimping when immersed in boiling water. Fabrics woven from these yarns show reduced luster, dryer handle, and improved liveliness when compared with similar fabrics prepared from equivalent yarns containing no kaolinite.

Example XI

An aqueous slurry of kaolinite is prepared according to the general procedure of Example I with the exception that 3.3 parts of a 30% aqueous suspension of colloidal silica is added in place of tetrasodium pyrophosphate. The slurry is used to prepare four different batches of polyhexamethylene adipamide with different concentrations of kaolinite, as indicated in Table 2, following the general polymerization procedure of Example I. An aqueous dispersion of TiO_2 is also added to the polymer batches along with the kaolinite, in amounts indicated in the table. The polymerization of each batch is continued until a polymer relative viscosity of about 46 is reached, whereupon the polymer is extruded as a ribbon, quenched, and cut to flake.

The nylon flake prepared above is remelted in a screw extruder and melt-spun at 290° C. using a 34-hole spinneret. The extruded filaments are quenched in cross-flow air according to the method of Brand, U.S. Patent No. 3,067,459, and wetted with a spinning finish on a rotating finish roller as shown in U.S. 3,067,458. The undrawn yarn is then passed around a feed roll rotating with a surface speed of 834 y.p.m., then through a first draw zone and around a first draw roll rotating with a surface speed of 1870 y.p.b. The partially drawn yarn is then passed through a second draw zone to and around a pair of combination draw-anneal rolls heated to a temperature of 190° C. and rotating at a surface speed of 3000 y.p.m., after which it is passed to a surface driven bobbin windup where it is wound into a package without twisting. The yarn produced has a denier of 70 (7.8 tex.), a tenacity of 5.4 g.p.d., and an elongation of 15% under 300 grams load. The initial modulus of the yarn is 30 g.p.d.

The coefficient of hydrodynamic friction (f_h) for each yarn prepared is measured by passing the yarn at a speed of 200 y.p.m. over a cylindrical polished chrome surface with the yarn contacting the chrome surface through a wrap angle of 164°. Input tension is set as 10 grams and the output tension measured by means of a strain gauge. The coefficient of friction is calculated from the Melt equation shown in Example I. The results are summarized in Table 2 below. The sharp reduction in friction obtained by adding kaolinite is clearly evident from an examination of the data in the table.

TABLE 2

Sample	Kaolinite, percent	TiO_2, percent	Coefficient of Friction (f_h)
XI-a (Control)	0	0.5	0.85
XI-b	0.3	0.5	0.74
XI-c	0.7	0.45	0.68
XI-d	1.0	0.3	0.68
XI-e	2.0	0	0.65

Example XII

In a polymerization otherwise identical to that described in Example I, another crystalline form of aluminum silicate, montmorillonite, was used in plate of kaolinite. Particle size was substantially the same as described in Example I. The chips contained 2% by weight of montmorillonite. Spinning of filaments was interrupted after twenty minutes because of pack plugging.

It is apparent that the over-all polymerization may be changed and modified in many respects without departing from the spirit of the present invention which is, therefore, intended to be limited only by the scope of the appended claims.

What is claimed is:

1. In the polymerization of a synthetic linear polycarbonamide, the steps of: heating a polycarbonamide-forming salt solution under condensation temperature and pressure conditions and, before completion of the polymerization, adding thereto an aqueous slurry of kaolinite platelets having an average maximum dimension of 0.2–2.0 microns and an actual maximum dimension of about 5 microns to provide from about 0.3 to about 17% by weight of the said platelets based on the weight of the polycarbonamide to be produced, said slurry also containing a heat stable deflocculant selected from the group consisting of tetrasodium pyrophosphate, sodium silicate, sodium hexamethaphosphate, colloidal silica, lithium hydroxide and low molecular weight sulfonated naphthalene-formaldehyde condensates in a concentration of from 0.05–3% by weight based on weight of kaolinite.

2. In the polymerization of a synthetic linear polycarbonamide, the steps of: heating a nylon salt solution under condensation temperature and pressure conditions and, before completion of the polymerization, adding thereto a 10–25% aqueous slurry of kaolinite platelets having an average maximum dimension of 0.2–2.0 microns and an actual maximum dimension of about 5 microns to provide from about 0.3 to about 17% by weight of the said platelets based on the weight of the polycarbonamide to be produced, the said aqueous slurry containing also from 0.05–3% by weight based on weight of kaolinite of

a heat stable deflocculant, said salt solution being selected from the group consisting of hexamethylene diammonium adipate, caprolactam, hexamethylene diammonium adipate and caprolactam, hexamethylene diammonium adipate and hexamethylene diammonium isophthalate, the salt of bis-(paraminocyclohexyl)methane and azelaic acid, and the salt of bis-(paraminocyclohexyl)methane and dodecanedioic acid, said deflocculant being selected from the group consisting of tetrasodium pyrosphate, sodium silicate, sodium hexamethaphosphate, colloidal silica, lithium hydroxide and low molecular weight sulfonated naphthalene-formaldehyde condensates, completing polymerization of the said salt solution to produce a fiber-forming polycarbonamide and thereafter melt spinning filaments from the said polycarbonamide.

3. A textile fiber consisting essentially of a polycarbonamide containing dispersed therein (a) from about 0.3 to about 17% by weight of kaolinite based on the weight of the said fiber, the said kaolinite being in the form of platelets having an average diameter of 0.2–2.0 microns and a maximum diameter of 5 microns and (b) from about 0.05% to about 3% by weight based on the weight of the said kaolinite of a compound selected from the group consisting of tetrasodium pyrosphate, sodium silicate, sodium hexametaphosphate, colloidal silica, lithium hydroxide and low molecular weight sulfonated naphthalene-formaldehyde condensates.

4. A textile fiber consisting essentially of polyhexamethylene adipamide containing dispersed therein (a) from about 0.3 to about 17% by weight of kaolinite based on the weight of the said fiber, the said kaolinite being in the form of platelets having an average diameter of 0.2–2.0 microns and a maximum diameter of 5 microns and (b) from about 0.05% to about 3% by weight based on the weight of the said kaolinite of a compound selected from the group consisting of tetrasodium pyrophosphate, sodium silicate, sodium hexametaphosphate, colloidal silica, lithium hydroxide and low molecular weight sulfonated naphthalene-formaldehyde condensates.

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