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METHOD OF PRODUCING ARTIFICIAL
SHAPED ARTICLES

Camille Dreyfus, New York, N. Y.

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This invention relates to the production of artificial materials made of a thermoplastic derivative of cellulose of improved properties.

An object of my invention is to produce artificial materials of increased strength and other improved properties by suddenly chilling such materials immediately after their production from molten masses or solutions containing thermoplastic derivatives of cellulose.

Other objects of this invention will appear hereinafter from the following detailed description.

According to my invention the thermoplastic derivative of cellulose is reduced to plastic or liquid form either by heating alone or by dissolving in volatile solvents or plasticizers with or without heating, is then shaped and then immediately or as soon as possible thereafter, is suddenly chilled to temper the same and increase its tenacity or other strength characteristics.

The artificial materials made by this invention may be of any suitable form. Thus they may be filaments of 0.5 to 50 or more denier, which may be formed into yarns or threads either while in continuous lengths or while in the form of staple fibres. They may be in the form of heavier materials such as artificial bristles or horsehair, straw of varying widths of say $\frac{1}{8}$ to 3 inches, wider ribbons, or they may be in the form of foils or films. These filaments, bristles, straw or ribbons may be formed by extruding the liquid or plastic material through orifices of proper shape and size, while the foils or films may be formed by casting on the smooth surface of plates, bands or film wheels. Articles of heavier form such as may be formed by extruding the material while in plastic or liquid form through an orifice into a mold may also be produced by this invention.

While I prefer to use cellulose acetate as the thermoplastic derivative of cellulose, and the invention will be specifically described in connection therewith, other thermoplastic derivatives of cellulose may be used, such as cellulose acetopropionate, cellulose aceto-butyrate and cellulose aceto-stearate. However, the process of the invention is applicable to a large range of other thermoplastic derivatives of cellulose, such as, for example, other thermoplastic cellulose esters, e. g. cellulose propionate, cellulose butyrate, cellulose stearate and cellulose propio-stearate, cellulose ethers such as ethyl cellulose and benzyl cellulose, and ether-esters of cellulose such as ethyl cellulose acetate and oxy-ethyl cellulose acetate.

Immediately after the material is shaped, it is subjected to a considerable temperature drop, e. g. of the order of 50 to 200° C., and preferably 100 to 200° C. when the material is in molten

condition prior to shaping, the greater the temperature drop, the stronger the product formed, particularly when it is subjected to a stretching or drawing operation while being formed. In this manner the material may be cooled to a temperature of 0° C. or below. The time elapsing between when the material is shaped and when it is cooled is preferably very short and is of the order of 0.05 to 0.1 or 0.25 second.

The cooling of the shaped materials may be effected in any suitable manner. For instance, they may be subjected to a spray of or be immersed in a bath of a liquid that has no appreciable solvent action on the thermoplastic derivative of cellulose and which is cooled to the low temperature required for lowering the temperature of the shaped material to the requisite degree. Examples of such liquids, in the case of cellulose acetate, are brine and aqueous solutions of diacetone alcohol, diethyl tartrate or ethyl lactate of say 25 to 65% concentration. Alternatively the cooling may be effected by means of a blast or current of air or other gas of the requisite low temperature, or by contacting the materials with cold solid surfaces such as of internally cooled rods, tubes, rollers, plates, etc.

In case of thermoplastic derivatives of cellulose that melt easily, no plastic inducing substances need be added but they may be used if their effect is desired. With other thermoplastic derivatives of cellulose, such substances such as volatile solvents or plasticizers in amounts to render the material more easily reducible to liquid or plastic form are preferably employed. An example of a volatile solvent for cellulose acetate is acetone, which may be used in amounts of from 15 to 300% of the weight of the cellulose acetate. Examples of plasticizers for cellulose acetate and like materials are triacetin, triphenyl acetin, tripropionin, dimethyl phthalate, diethyl phthalate, dimethoxy ethyl phthalate, dibutyl tartrate and the sulphonamides, such as p-toluene sulphonamide. These plasticizers may be used in amounts of 5 or 10 up to 40% of the weight of the cellulose acetate. With such amounts of plasticizer the cellulose acetate material may be reduced to a sufficiently plastic state for shaping when subjected to temperatures of 150° to 230° C. or more.

When used, the plasticizers are preferably distributed throughout the cellulose acetate in as uniform a manner as possible. This may be effected by treating the cellulose acetate with a solution or dispersion of the plasticizer in water, benzene, ether or other non-solvent for cellulose acetate, allowing the cellulose acetate to absorb the plasticizer from the solution or dispersion and removing the solvent by evaporation as described in U. S. Patent No. 1,999,405. Another

method by which the agents may be uniformly distributed comprises treating the cellulose acetate with a solution or dispersion of the agents in a volatile solvent for the cellulose acetate or by adding the agents to a solution of the cellulose acetate in a volatile solvent. In the former case the materials are worked, e. g. by malaxation, until the substance is dissolved. When a uniform solution and/or dispersion of the components has been obtained, the volatile solvent may be evaporated, either wholly or in part, so as to leave the required composition.

In the case of the production and treatment of filaments, yarns, bristles, straws and the like, the plasticizer may be removed from the formed materials by extraction with a solvent for the plasticizer, e. g. aqueous alcohol.

The compositions comprising the thermoplastic derivative of cellulose that is shaped may also contain other agents in order to modify their properties or to give various effects in the products. They may contain, for example, high boiling solvents, pigments, dyes and/or fire retardants.

When the compositions contain volatile solvents they may be subjected after extrusion or shaping for a short period of time to heated evaporative atmosphere in order to impart to them sufficient solidity prior to their being given the chilling treatment.

The shaped articles may be subjected to various treatments after they have been set, with a view to modifying their characteristics. For example, in the case of filaments, yarns, bristles, straws and the like, they may be subjected to a stretching operation, whether or not they have been stretched before setting. Before such a stretching operation applied after setting, the materials may be treated with a softening agent, e. g. a solution of a solvent or plasticizer or a solvent vapor, and/or they may be softened by heat. Advantageously, the materials are stretched while immersed in or wetted with water or other non-solvent containing hydroxyl radicles, whether in liquid or vapor form. Thus they may with advantage be stretched while immersed in hot water or while in a steam chamber. In this way, the tenacity of the products may be still further increased.

Alternatively or in addition, the articles may be subjected to the action of a shrinking agent, e. g. a solution of a solvent for the thermoplastic derivative of cellulose in the absence of stretching tension so as further to modify their properties.

It is to be understood that the foregoing detailed description is merely given by way of illustration and that many variations may be made therein without departing from the spirit of my invention.

Having described my invention, what I desire to secure by Letters Patent is:

1. Process for the production of artificial shaped articles comprising shaping a composition comprising a thermoplastic derivative of cellulose while in a soft and hot condition and immediately thereafter sharply cooling the same by positively causing a drop of 50 to 200° C. in temperature in a period of 0.05 to 0.25 second.

2. Process for the production of artificial shaped articles comprising shaping a composition

comprising cellulose acetate while in a soft and hot condition and immediately thereafter sharply cooling the same by positively causing a drop of 50 to 200° C. in temperature in a period of 0.05 to 0.25 second.

3. Process for the production of artificial filaments, threads, bristles or straws comprising extruding a composition comprising a thermoplastic derivative of cellulose while in a soft and hot condition through an orifice under such conditions as to at least partially solidify the same and immediately thereafter cooling the same by positively causing a drop of 50 to 200° C. in temperature in a period of 0.05 to 0.25 second.

4. Process for the production of artificial filaments, threads, bristles or straws comprising extruding a composition comprising cellulose acetate while in a soft and hot condition through an orifice under such conditions as to at least partially solidify the same and immediately thereafter cooling the same by positively causing a drop of 50 to 200° C. in temperature in a period of 0.05 to 0.25 second.

5. Process for the production of artificial filaments, threads, bristles or straws comprising extruding a composition comprising a thermoplastic derivative of cellulose and a plasticizer therefor while in a soft and hot condition through an orifice under such conditions as to at least partially solidify the same and immediately thereafter cooling the same by positively causing a drop of 50 to 200° C. in temperature in a period of 0.05 to 0.25 second.

6. Process for the production of artificial filaments, threads, bristles or straws comprising extruding a composition comprising cellulose acetate and a plasticizer therefor while in a soft and hot condition through an orifice under such conditions as to at least partially solidify the same and immediately thereafter cooling the same by positively causing a drop of 50 to 200° C. in temperature in a period of 0.05 to 0.25 second.

7. Process for the production of artificial filaments, threads, bristles or straws comprising extruding a composition comprising a thermoplastic derivative of cellulose while in a soft and hot condition through an orifice under such conditions as to at least partially solidify the same and immediately thereafter cooling the same by positively causing a drop of 50 to 200° C. in temperature in a period of 0.05 to 0.25 second to a temperature below 0° C.

8. Process for the production of artificial filaments, threads, bristles or straws comprising extruding a composition comprising cellulose acetate while in a soft and hot condition through an orifice under such conditions as to at least partially solidify the same and immediately thereafter suddenly and positively cooling the same to a temperature below 0° C. in a period of 0.05 to 0.25 second.

9. Process for the production of artificial filaments, threads, bristles or straws comprising extruding a composition comprising cellulose acetate while in a soft and hot condition through an orifice under such conditions as to at least partially solidify the same, immediately thereafter suddenly and positively cooling the same to a temperature below 0° C. in a period of 0.05 to 0.25 second and then stretching the same.

CAMILLE DREYFUS.