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METHOD OF PRODUCING SHAPED RESINOUS ARTIFICIAL MATERIALS

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This invention relates to the production of artificial materials made of a fusible fibre or film forming resinous substance 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 fusible fibre or film forming resinous substances.

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

According to my invention the fusible fibre or film forming resinous substance is reduced to plastic or liquid form either by heating alone or by dissolving it 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.

Any suitable resin may be employed as the fusible film or fibre material. Examples of these are polyvinyl esters and ethers, e. g. polyvinyl acetate, polyvinyl chloride, polyvinyl-chlor-acetate and copolymers of vinyl acetate and vinyl chloride, polymerized esters of unsaturated acids, e. g. those of the acrylic and methacrylic acid series, fibre- or film-forming polyamides, e. g. the products obtained by condensation of diamines with dicarboxylic or disulphonic acids or by self-condensation of amino-carboxylic or amino-sulphonic acids, and other fibre- or film-forming polymers obtainable by condensation of bi-functional reagents.

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 fibre or film forming material and which is cooled to the low temperature required for lowering the temperature of the shaped material to the requisite degree. An example of such liquids is brine and other aqueous solutions of salts. 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 film or fibre forming resinous materials that melt easily, no plastic inducing substances need be added but they may be used if their effect is desired. With other materials, 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. Examples of volatile solvents for polyvinyl esters are acetone and ethyl alcohol. Examples of solvents for polyamides are hot glacial acetic acid, formic acid and phenol. These solvents may be employed in amounts of 10 to 250% of the weight of the resinous material. The plasticizers may be used in amounts of 5 or 10 up to 40% of the weight of the resinous material. The temperature at which the composition containing the resinous material may be maintained prior to shaping may be of the order of 100 to 230° C. or more.

When used, the plasticizers are preferably distributed throughout the resinous material in as uniform a manner as possible. This may be effected by treating the resinous material with a solution or dispersion of the plasticizer in a non-solvent for the resinous material, allowing the resinous material to absorb the plasticizer from the solution or dispersion and removing the solvent by evaporation. Another method by which

the plasticizers may be uniformly distributed comprises treating the resinous material with a solution or dispersion of the plasticizer in a volatile solvent for the resinous material or by adding the agents to a solution of the resinous material 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 fusible fibre or film forming resinous substance 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, especially those having a basis of a fibre- or film-forming polymer obtainable by condensation of bifunctional reagents, particularly fibre- or film-forming polyamides, are stretched while immersed in or wetted with water or other non-solvent containing hydroxyl radicals, 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 fibre- or film-forming resinous substance, 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 fusible fibre or film forming polyvinyl ester resin while in a soft and hot condition and immediately thereafter, while the shaped composition is still in a soft and hot condition, positively causing a drop of 50 to 200° C. in the temperature of said shaped composition in a period of 0.05 to 0.25 second.

2. Process for the production of artificial filaments, threads, bristles or straws comprising extruding a composition comprising a fusible fibre or film forming polyvinyl ester resin while in a soft and hot condition through an orifice under such conditions as to at least partially solidify the shaped composition and to maintain the same in a soft and hot condition, and immediately thereafter, while the shaped composition is still in a soft and hot condition, positively causing a drop of 50 to 200° C. in the temperature of said shaped composition 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 fusible fibre or film forming polyvinyl ester resin while in a soft and hot condition through an orifice under such conditions as to at least partially solidify and to maintain the same in a plastic condition, and immediately thereafter, while the shaped composition is still in a plastic condition, positively cooling the same to a temperature below 0° C. in a period of 0.05 to 0.25 second.

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