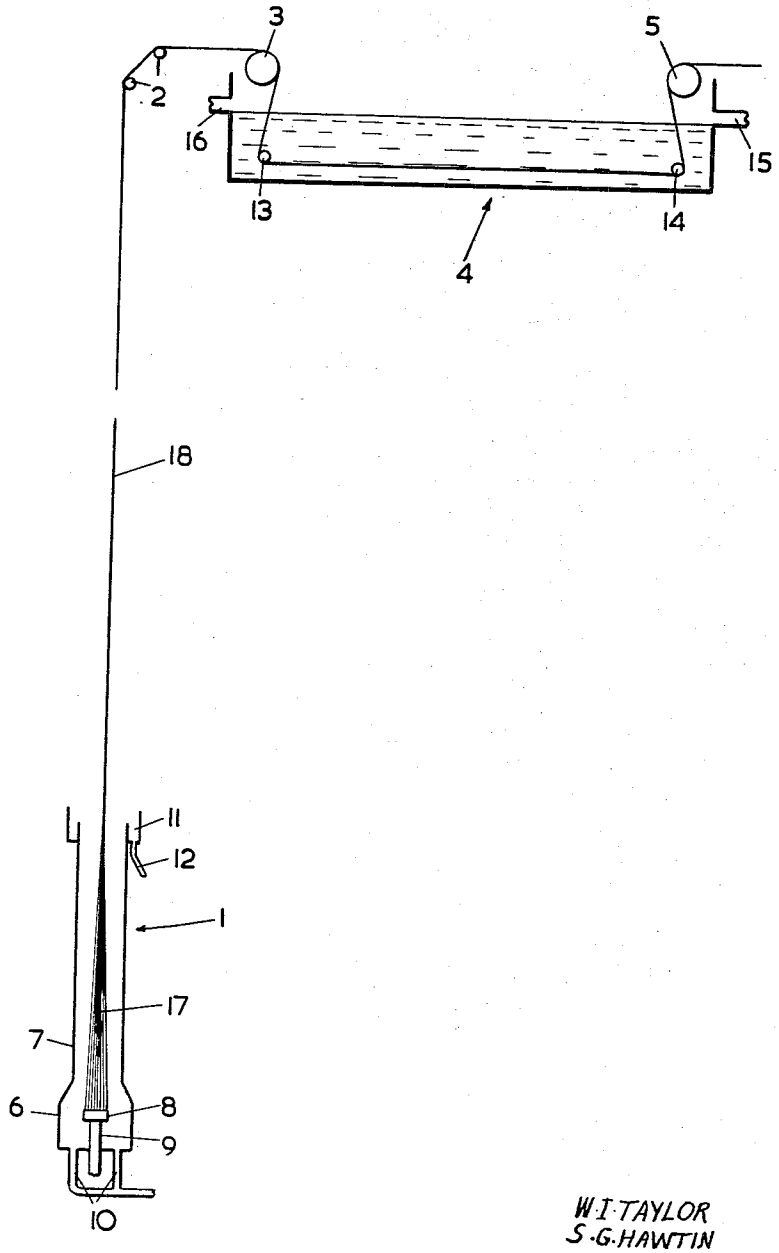
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BY A WET SPINNING PROCESS
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MANUFACTURE OF FILAMENTARY MATERIALS BY A WET SPINNING PROCESS

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This invention relates to the manufacture of filamentary materials having a basis of an acetone-soluble fibre-forming substance, especially an acetone-soluble cellulose acetate or other organic derivatives of cellulose, by a wet spinning process.

As is well known the great bulk of cellulose acetate yarn is, at present, made by dry spinning or evaporative processes, and yarn so obtained is very satisfactory for most purposes. For some applications, however, wet spun yarn is to be preferred, since it usually has a higher extensibility and more uniform and rounder cross-section than the dry spun products; moreover wet spun material has some advantage over dry spun for conversion into staple fibre.

In one method of making wet spun cellulose acetate yarn a spinning solution consisting of cellulose acetate dissolved in acetone is extruded into an aqueous solution of acetone as described for example in U. S. application Ser. No 217,816, filed March 27, 1951, of S. G. Hawtin and J. W. Grebby, and U. S. application Ser. No. 237,397, filed July 18, 1951, of the present applicants. This method gives very good results when used in the production of filaments of low or fairly low denier, and of yarns and staple fibre made from such filaments, but it suffers from the inherent disadvantage that the volatility of acetone necessitates special precautions to prevent excessive losses by evaporation; moreover it is not at all easy to employ it in the production of materials of high filament denier. Generally filaments obtained by spinning into aqueous acetone have only a moderate lustre.

It is an object of the present invention to provide a method of making filamentary material from cellulose acetate which shall be capable of giving material having not only a high extensibility and uniform cross-section, but also a high lustre and high filament denier. The present invention, by which these objects are achieved, involves the use not only of a particular class of substances as coagulating liquids, but also of certain specific spinning conditions. The new process may be applied also to other fibre-forming substances which are soluble in acetone.

According to the invention, we extrude an acetone solution of an acetone-soluble fibre-forming substance into a coagulating bath which consists essentially of a fatty oil at a temperature between 38° and 80° C. Advantageously the oil is kept at a temperature between 45° and 70° C., and especially between 48° and 56° C. It is necessary to ensure that the acetone content of the oil never rises above a limiting value which varies with different oils and which is equal to the highest acetone content at which a previously unstretched thread of the fibre-forming substance is stretched without breaking when subjected to a tension of 1 gram per denier for two minutes while immersed in a binary mixture of the oil and acetone at the spinning temperature. (The term "previously unstretched" is not intended to exclude a thread in the production of which some stretch has occurred during spinning and before the thread is fully set.)

Although this limiting value differs with different oils, its determination in any particular case is a matter of a simple test.

This limitation of the acetone content of the oil may in practice easily be effected by passing the oil through the spinning zone at a suitable rate such that it leaves the zone before its acetone content exceeds the limit. At spinning temperatures considerably above the boiling point of acetone, vaporisation of the acetone may of course prevent the acetone content reaching the limit however slowly the oil flows through the spinning zone, or even in the absence of flow.

The invention is of particular value when applied to the production of filamentary material having a basis of an acetone-soluble cellulose ester or ether, in particular cellulose acetate; it may however also be applied to the production of filamentary material from other acetone-soluble fibre-forming substances, for example polyvinyl acetate and acetone-soluble copolymers of vinyl acetate with other vinyl compounds, for instance vinyl chloride. The invention will be more particularly described by reference to the production of multi-filament yarns of cellulose acetate.

As examples of the fatty oils which have proved satisfactory for use in the coagulating bath may be mentioned coconut oil, palm oil, sperm oil and arachis oil, and of these we prefer to use the arachis oil. The temperature of the oil is preferably kept within the specified limits during the whole of the time the extruded filaments are immersed in it.

As already indicated, the acetone content of the oil is not allowed to exceed a limiting maximum which is specific for each oil. For example, when sperm oil is used at 50° C., the limiting maximum is an acetone content of about 8% by weight of the mixture of oil and acetone, and with an arachis oil at the same temperature it is about 14%. These figures represent approximately the limiting permissible acetone content after the oil has taken up acetone from the spinning solution. Subject to the maximum not being exceeded at any time, the oil may contain a certain amount of acetone before it enters the spinning zone.

The spinning solution may be filtered in the usual way before extrusion, and, if desired, may be heated, e. g. to about the same temperature as the oil or to a higher temperature, although we have found the use of unheated spinning solutions to be quite satisfactory. The spinning machine may be arranged so that the filaments formed travel through the oil in a horizontal or vertical direction or at an angle between the two. We prefer to extrude the spinning solution and to draw the filaments through the oil vertically upwards, and then to give the filaments a moderately long vertical air run, e. g. about 4–15 feet or more, so as to allow oil carried up with the filaments to drain back to the coagulating bath. The removal of adherent oil from the filaments may be assisted by causing the filaments to run in contact with guides or other members having a scraping action. It is also advantageous to wash the filaments, before they are wound up or otherwise collected, with a solvent for the oil which is without substantial effect on the substance of which the filaments are made; for this purpose a light hydrocarbon mixture such as petroleum ether or an aliphatic ether such, for instance, as di-isobutyl ether may generally be used. For example the filaments may be passed through a bath of the solvent for the oil.

It is advantageous that fresh oil be introduced continuously near the point of extrusion and that the oil should flow in the direction of travel of the filaments. This, however, is by no means essential, and if desired the oil may be caused to flow in the opposite direction or it may be introduced at more than one point into

the spinning zone and may flow in different directions through different parts of the spinning zone. Our preferred method is to spin upwardly through an upwardly flowing stream of the oil.

It is advisable when starting to spin to employ, in place of the fatty oil, a light hydrocarbon mixture, e. g. petroleum ether. A very short run through this hydrocarbon mixture is sufficient; for example when spinning upwards a depth of ½ to 1" above the jet face is all that is necessary. The light hydrocarbon mixture may be at about room temperature or at a lower or higher temperature. As soon as filament formation is proceeding satisfactorily, the hydrocarbon mixture is displaced by the heated fatty oil.

One unexpected result of the use of a fatty oil coagulating bath is that the filaments obtained can be allowed to come into contact with each other, without risk of their sticking together, while they still contain a proportion of acetone so high that if one of the known setting or coagulating methods were used adhesion would be practically unavoidable. Accordingly, quite a short run through the coagulating bath is sufficient, thus keeping small the volume of oil that needs to be used, and that has to be purified before reuse. If the amount of acetone remaining in the filaments warrants it, means may be provided for recovering acetone which evaporates from the filaments after they have left the coagulating bath; moreover acetone may be recovered from a solvent used to remove residual oil as described above, and/or the filaments may be washed with a solvent for acetone such as water after they have been freed from oil.

We have found that when a multi-filament yarn is being spun, the cone formed by the individual filaments usually has its apex at or very near the surface of the oil, so that at once or almost at once after leaving the oil the filaments come together; when other coagulating liquids are used the apex of the cone is usually well above the surface of the liquid, with the result that a column of liquid is drawn up with the filaments unless a special device is employed to force the apex down as described in U. S. application Ser. No. 217,816 of S. G. Hawtin and J. W. Grebby.

Besides being of use in the manufacture of multi-filament yarns, the new process may be applied to the manufacture of other filamentary material, for example single filaments (monofils) and tow for conversion into staple fibre. As has already been said, the process is well adapted to the production of heavy denier filaments, e. g. filaments above 20 denier, for example 25–35 denier, and it can also be used for the production of yet heavier structures such as artificial bristle or straw.

The term "fatty oils" is employed in this specification to denote not only the naturally occurring animal or vegetable oils (though as these are conveniently obtainable and relatively cheap their use will generally be preferred), but also analogous compositions and single compounds, e. g. hydrogenated natural animal and vegetable oils, single glycerides of one or more long chain fatty acids containing 12 or more carbon atoms in the molecule, and mixtures of such glycerides. It does not include mineral oils or esters of long chain monohydric alcohols such as the waxes. The use of natural oils or of single compounds or artificial mixtures having drying properties is, of course, best avoided.

Apparatus which may be employed in carrying out the process of the invention is illustrated diagrammatically in the accompanying drawing. The apparatus comprises a spinning cell **1**, a pair of thread guides **2**, a godet **3**, a bath **4** and godet **5**.

The spinning cell **1** comprises an extrusion chamber **6**, surmounted by a vertical tube **7** about 12 inches long and 2 inches in diameter. The extrusion chamber, which is somewhat broader than the vertical tube, contains a spinning jet **8** carried on a feed pipe **9** for spinning solution. Feed pipes **10** for coagulant enter the chamber at its base. A gutter **11** provided with a run-off **12** surrounds the upper end of the vertical tube **7**.

The thread guides **2** are positioned about 6 feet above the upper end of the tube **6**, and are designed to wipe off as much as practicable of the oil which still adheres to the filaments formed in the spinning device **1**. The godet **3** draws the filaments from the guides **2** and feeds them into the bath **4** under guides **13** and **14**. Washing liquid enters the bath by an inlet **15**, and leaves by an outlet **16**, flowing against the direction of travel of the filaments. The washed filaments are drawn from the bath by the godet **5**. The filaments pass up the tube **7** in the form of a truncated cone **17**, and come together to form a bundle or yarn **18** shortly after emerging from the tube **7** and thus form the coagulant.

The following example illustrates the invention.

Example

A form of apparatus such as that described above and illustrated in the drawing was employed, the liquid being fed continuously into the extrusion chamber **6** and collected continuously by the gutter **11** from the top of the tube **7**, and withdrawn by means of the run-off **12**.

The spinning solution was a 26% solution of cellulose acetate in anhydrous acetone and was supplied to the jet at a temperature of about 20° C. At the beginning of the operation the device contained petroleum ether at about 20° C. to a depth of ½–1 inch above the jet face, but as soon as filament formation had begun this was displaced by arachis oil entering the extrusion chamber at a temperature of 52° C. The rate at which the oil was fed into the device was so adjusted that when it overflowed at the top of the tube it contained 10–12% of acetone. The filaments formed were drawn from the tube vertically upwards through the atmosphere for 6 feet, after which they passed over the thread guides **2** to wipe off as much of the adherent oil as practicable. They then washed in the bath **4** which was continuously replenished with petroleum ether, and then after a further short air run they were wound up.

The filaments remained separate in the tube **7**, but came together to form an untwisted yarn almost immediately after leaving it. The resulting yarn showed no mutual adhesion of the filaments, and had a filament denier of 21, a tenacity of 1.02 gm./denier and an extensibility of 42%. The tenacity could be greatly increased by stretching the yarn, e. g. by 8–12 times its length in wet steam under pressure in accordance with known processes.

Having described our invention, what we desire to secure by Letters Patent is:

1. Process for the manufacture of filamentary material from acetone-soluble cellulose acetate by a wet spinning process using as the spinning solution an acetone solution of the cellulose acetate, which comprises initiating spinning by extruding the spinning solution upwardly into petroleum ether extending for ½ to 1 inch above the level of the orifices through which the solution is extruded, displacing the petroleum ether by a fatty oil selected from the group consisting of coconut oil, palm oil, sperm oil and arachis oil, at a temperature of 45° to 70° C. and maintaining the height of the fatty oil bath 12 inches above the level of said orifices, and maintaining the acetone content of the oil at a value which is at most equal to the highest concentration of acetone in a binary mixture of the oil and acetone at which a previously unstretched thread of the cellulose acetate, immersed in the mixture at the spinning temperature, can be stretched without breaking by applying a tension of 1 gram per denier for 2 minutes.

2. A process as in claim 1 where the fatty oil is arachis oil.

3. A process as in claim 1 where the fatty oil is sperm oil.

4. A process as in claim 1 where the fatty oil is coconut oil.

5. A process as in claim 1 where the fatty oil is palm oil.

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