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## MANUFACTURE AND PRODUCTION OF ARTIFICIAL FILAMENTS, THREADS, FILMS, AND THE LIKE

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This invention relates to the production of artificial filaments, threads, films and the like with a basis of alginic acid. Alginic acid, when prepared from seaweed, is generally obtained in the form of its calcium salt, which is insoluble in water, and in order to obtain from this calcium alginate a solution which can be spun into filaments and threads, or converted into films, it has been customary to treat the calcium alginate with hydrochloric acid, and then to remove the soluble calcium chloride from the insoluble alginic acid, and thereafter to dissolve the alginic acid in caustic soda solution, thus obtaining a solution of sodium alginate, which is viscous, and then to extrude this viscous solution, using the ordinary technique for the production of artificial threads, into a solution of a calcium salt, whereupon threads or filaments of insoluble calcium alginate are produced. It has also been proposed to dissolve alginic acid in a mixture of caustic soda and ammonia, and to spin the solution thus obtained into a solution of calcium chloride, or to form a film of the solution by drying it on a glass plate, and then to render the film insoluble by treatment with a solution of calcium chloride.

We have now found that filaments, threads and films, all of which we will hereinafter include under the term filaments, can be obtained by extruding into a suitable setting medium an aqueous solution of an alkali metal alginate containing in suspension a finely divided alkaline earth metal salt which is less soluble than the corresponding alkaline earth metal alginate. Such a suspension can be obtained for instance by treating an alkaline earth metal alginate, such for example as calcium alginate or barium alginate, with a solution of a salt of an alkali metal including ammonium and of an acid of which the alkaline earth metal salt is less soluble than the corresponding alkaline earth metal alginate.

As examples of suitable acids of which the alkaline earth metal salts are more insoluble than the corresponding alginates we mention oxalic acid, phosphoric acid and carbonic acid, while as examples of the alkali metal we mention sodium, potassium and ammonium.

The suspension of the alkaline earth metal salt in the aqueous solution of alkaline metal alginate may be extruded through a nozzle adapted to bring the solution into the desired shape into a setting bath containing a dilute acid, such as hydrochloric acid or acetic acid, the alkaline earth metal salts of which are soluble in water,

or give rise to water-soluble salts. Although there may theoretically be sufficient alkaline earth metal present to combine with the whole of the alginic acid, the filaments formed consist generally of a mixture of the alkaline earth metal alginate and alginic acid, while the alkali metal salt which was originally added to the alkaline earth metal alginate in order to obtain the spinning solution remains in solution in the spinning bath and is thereby eliminated from the filaments. We generally prefer to extrude the suspension into a setting bath containing both acid and also a soluble alkaline earth salt, such as calcium chloride. In order to ensure that the filaments are completely converted to the alkaline earth metal alginate we can subject them to further treatment with an alkaline earth metal compound. For example, filaments of calcium alginate and alginic acid may be treated with a solution of calcium chloride containing calcium hydroxide or finely divided calcium carbonate.

When ammonium carbonate is used according to the present invention to bring the alginic acid radical into solution the suspension obtained may be extruded into air and as the filaments begin to dry, the ammonia tends to evaporate leaving the free alginic acid to react with the alkaline earth metal carbonate in the suspension. Carbon dioxide is evolved and the filaments are converted to the insoluble alkaline earth metal alginate. Sometimes, the last traces of ammonia leave the filament very slowly, and in this case the completion of the conversion of the ammonium alginate to the alkaline earth metal alginate may be hastened by treatment with a dilute solution of a salt of the same alkaline earth metal, or by treatment with steam. When the suspension containing ammonium alginate and alkaline earth metal carbonate has been extruded into an acidified spinning bath the filaments formed may, if desired, be exposed to the air to complete the volatilisation of the ammonia, or may be further treated with a solution of an alkaline earth metal salt to ensure complete conversion of the alginic acid to alkaline earth metal alginate.

If, when carrying out the process of this invention an alkali metal carbonate is used for bringing the alginic acid radical into solution, and it be desired, during the extrusion into an acidified bath to avoid the formation of bubbles in the filaments, the temperature and acidity of the bath into which the suspension is extruded should be kept sufficiently low.

The process according to this invention presents advantages since it avoids the necessity of removing the alkaline earth metal from the alkaline earth metal alginate, which is employed as raw material, thus enabling the manufacture of filaments, threads, films and the like to dispense with a laborous process, before the extrusion process is carried out.

The following examples serve to illustrate the nature of this invention which however is not limited to these examples:

*Example 1*

8.4 kilograms of calcium alginate containing 20 per cent of moisture are dissolved in 90 kilograms of water containing 1.90 kilograms of sodium carbonate and 0.5 kilogram of 40 per cent formaldehyde as preservative. The suspension obtained is subsequently extruded into a solution containing 5 per cent of calcium chloride and 0.25 per cent of hydrochloric acid. The filaments thus produced are then treated with 0.1 per cent of hydrochloric acid at 20° centigrade for 30 minutes, then with 0.75 per cent of hydrochloric acid for 15 minutes and finally for 15 minutes with 5 per cent of calcium chloride saturated with calcium hydroxide. The filaments are then washed and dried.

*Example 2*

7 kilograms of calcium alginate, 2.05 kilograms of ammonium carbonate, 0.6 kilogram of 0.880 ammonia and 90.35 kilograms of water are mixed for four hours at the ordinary temperature. The solution is then extruded through a suitable jet into a bath containing 5 per cent of calcium chloride. The threads thus formed are treated for 10 minutes with a saturated solution of calcium hydroxide, washed and dried.

What we claim is:

1. A process for producing artificial filaments, threads, films and the like which consists in reacting an alkaline earth metal alginate with an aqueous solution of an alkali metal salt thereby producing a mixture comprising a solution of alkali metal alginate in water having therein finely divided particles of alkaline earth metal salt, and extruding said mixture into a setting medium.

2. A process for producing artificial filaments,

threads, films and the like which consists in reacting an alkaline earth metal alginate with an aqueous solution of an alkali metal salt thereby producing a mixture comprising a solution of alkali metal alginate in water having therein finely divided particles of alkaline earth metal salt, and extruding said mixture into a setting medium containing an acid the alkaline salts of which are soluble in water.

3. A process for producing artificial filaments, threads, films and the like as claimed in claim 2, in which the setting medium contains a water-soluble alkaline earth metal salt.

4. A process for producing artificial filaments, threads, films and the like, which consists in reacting an alkaline earth metal alginate with an aqueous solution of ammonium carbonate thereby producing a mixture comprising a solution of ammonium alginate in water having therein finely divided particles of alkaline earth metal carbonate, and extruding said mixture into a setting medium.

5. A process for producing artificial filaments, threads, films and the like, which consists in reacting calcium alginate with an aqueous solution of sodium carbonate thereby producing a mixture comprising a solution of sodium alginate in water having therein finely divided particles of calcium carbonate, and extruding said mixture into a setting medium.

6. A process for producing artificial filaments, threads, films and the like, which consists in reacting calcium alginate with an aqueous solution of ammonium carbonate thereby producing a mixture comprising a solution of ammonium alginate in water having therein finely divided particles of calcium carbonate, and extruding said mixture into a setting medium.

7. A process for producing artificial filaments, threads, films and the like, which consists in reacting calcium alginate with an aqueous solution of ammonium carbonate thereby producing a mixture comprising a solution of ammonium alginate in water having therein finely divided particles of calcium carbonate, and extruding said mixture into a setting medium containing an acid and about 5% calcium chloride, the alkaline salts of said acid being soluble in water.

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