

UNITED STATES PATENT OFFICE

2,022,411

MANUFACTURE OF ARTIFICIAL FILAMENTS,
THREADS, FILMS, FABRICS, AND THE
LIKE

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No Drawing. Application February 8, 1933, Serial
No. 655,772. In Great Britain February 26,
1932

6 Claims. (Cl. 106—40)

This invention relates to improvements in the manufacture of materials, for example filaments, threads, ribbons, films and the like, containing organic derivatives of cellulose, and to woven, knitted or netted fabrics and other products obtained therefrom, and in particular to processes whereby their resistance to heat may be increased.

I have discovered that by incorporating boron compounds, e. g. boric acid or borates, in materials made of or containing organic derivatives of cellulose, and particularly cellulose acetate, their resistance to heat may be improved and their safe ironing point may be raised. The products of the present invention may be obtained either by incorporating boron compounds in the spinning solution from which the materials are produced, or by treating the formed materials with such compounds.

The boron compounds may either be incorporated in the spinning solution or materials in the form in which they are to be present in the finished products, as, e. g., boric acid and alkali metal borates, or the compounds incorporated may afterwards be converted into their final form by treatment with suitable reagents. Examples of this latter type are many metallic borates and borate esters, e. g. alkyl borates such as the methyl and ethyl borates and glycerine borate, which may be decomposed by treatment with water.

As is stated above, the boron compounds may be incorporated in the filaments, films or other products either by treating the formed products, or by adding a suitable boron compound to the spinning solution. The latter method is preferably employed and in this case the boron compounds, whether they be such that they are present unchanged in the final products or such as are treated in order to convert them into other boron compounds, may be incorporated in the spinning solution by dissolving them directly in a portion or the whole of either the spinning solution itself or the solvent employed therein. Boric acid is particularly suitable for use in this connection, since it is appreciably soluble in acetone, and still more soluble in aqueous acetone. Alternatively the boron compound may be dissolved in any other suitable solvent and then added to the spinning solution or to the solvent thereof. In this case, if the solvent used for the boron

compound is not a solvent for the cellulose derivative, the amount of solvent employed for the boron compound should of course be adjusted so that, on addition to the spinning solution, precipitation of the cellulose derivative or other undesirable effect is avoided, while the proportion may or may not be such that precipitation of the boron compound occurs in the spinning solution.

Alternatively boron compounds insoluble in the spinning solution or solvent thereof may be incorporated therein in a finely divided form. This fine state of sub-division may be obtained by grinding the boron compound either alone or with part or all of the base of the spinning solution, and either dry or in the presence of liquids, e. g. water, or part of the spinning solution or of the solvent used in the spinning solution. A suitable means for carrying out the grinding is a ball mill or colloid mill. In order to improve the state of dispersion grinding may also take place in the presence of dispersing agents and/or protective colloids, for example Turkey red oil, preferably used in relatively small proportions, and/or in the presence of oils, such as oleic acid, linseed or olive oil and the like. Alternatively such substances may be added to the boron compound after grinding and before incorporating it into the spinning solution, or they may be incorporated directly with such solutions. Other liquids which may be employed are diethylene glycol, heavy white mineral oil and castor oil.

The ground boron compound either in a dry state or together with liquids with which it may have been ground as mentioned above, may then be incorporated directly in the spinning solution. Alternatively it may be worked with a small portion of the spinning solution or of the solvent used in the spinning solution and the dispersion thus obtained then incorporated with the main bulk of the spinning solution. Again the dispersing agent or protective colloid or a suitable oil such as those above mentioned, may be added to the ground boron compound and the mass worked up and then incorporated in the spinning solution or solvent from which such solution is to be prepared. Or again, the ground boron compound may be worked up with the cellulose derivative with or without the addition of oils or other suitable substances and the mass obtained

incorporated in a suitable solvent to form the spinning solution.

By whatever means the incorporation of the boron compound in the spinning solution is effected, the solution is then preferably thoroughly mixed and subjected to the usual filtration before spinning. If desired, however, the addition of the boron compound to the spinning solution may take place at any stage of the filtration process, or even after completion of the filtration.

When the invention is applied to the treatment of formed products the filaments, films or other materials may be treated with solutions or dispersions of the boron compounds, and in this case, particularly when dispersions of the compounds are employed, it is advantageous that a swelling agent should be applied to the materials, either prior to the treatment with the boron compound, or simultaneously therewith by incorporating a swelling agent in the bath containing such compound. Among suitable swelling agents for use in this process are thiocyanates, alcohols, aqueous solutions of formic or acetic acid. Other compounds may be employed, for example those described in U. S. Patent No. 1,821,464 and U. S. Patent application S. No. 374,288 filed June 27, 1929. A suitable method of carrying out this process comprises treating the materials with an alcoholic solution of a boron ester, in which case the alcohol itself may act as a swelling agent.

As already stated boron compounds may be incorporated in the materials and subsequently decomposed by means of a suitable reagent. Thus for instance such decomposition may occur during the production of the spinning solution, in the spinning solution itself, during the actual formation of the materials or at any other stage of their manufacture or in the finished materials. For example a suitable solution of the boron compound to be decomposed may be mixed directly with an acetylation or other esterification mixture. Upon precipitation of the cellulose acetate or other cellulose ester the boron compound may be decomposed and the decomposed product obtained in a finely divided form intimately mixed with the cellulose acetate or other base. Furthermore, the decomposition of the boron compounds may take place in the spinning solution or other solution to be used for the manufacture of the artificial materials. Thus, for instance, ethyl borate may be dissolved in alcohol and added to a solution of cellulose acetate in aqueous acetone. The ethyl borate may thus be decomposed with the formation of boric acid in the acetone. Again, the decomposition may be effected during the actual formation of the materials by spinning the solutions containing the boron compound to be decomposed into suitable media. The invention also includes the process of decomposing the boron compounds in the formed materials by means of any suitable after-treatment.

Conversion of the boron compounds into the form in which they are to be present in the final products may be carried out in any suitable manner, for example by treating the solution or dispersion of the boron compound in the spinning solution or solvent or portion thereof with a reagent which reacts with the boron compound, or by treating the products after their formation with such reagents. Thus, for example, the esters of boric acid and certain metallic borates decompose more or less readily upon treatment with aqueous media. In some cases a simple treatment of the materials with a moist atmos-

phere or with steam, preferably moist, is sufficient to effect decomposition of the boron compounds. In other cases a somewhat more drastic treatment, for example treatment with dilute acids or dilute alkali may be used to effect decomposition. Acids or other reagents may thus take part in the formation within the materials of the boron compounds.

The incorporation of the boron compounds in the materials may be carried out at normal or increased temperatures and the proportion of compound incorporated in the artificial materials may vary within wide limits. In general, however, proportions of from 1 to 10% or 15% and particularly 5-8% on the weight of the cellulose derivative have been found suitable.

The following examples are given by way of illustration only, and are not to be considered as limiting the invention in any way.

Example 1

1 part by weight of an acetone-soluble cellulose acetate is dissolved in 3 parts by weight of 95% aqueous acetone, containing 0.06 part of boric acid. The solution is spun into an evaporative medium to form filaments, the filaments being collected together to form a thread and wound or twisted and wound. The fabric made from such threads is found to have an increased safe ironing point.

Example 2

Triethyl borate (obtained for example by the reaction between alcohol and boracetic anhydride) is dissolved in alcohol. Sufficient of this solution is mixed with an acetone solution of cellulose acetate to give a proportion of boron compound equal to 3% on the weight of the cellulose acetate. The solution is then spun into filaments in an evaporative medium, the filaments being collected together to form a thread and wound or twisted and wound. The threads obtained are then washed, treated with water or dilute acid and products containing boric acid distributed therein are obtained.

Though the invention has been described with particular reference to cellulose acetate it is also applicable to other organic derivatives of cellulose, for example esters, such as cellulose formate, propionate and butyrate, mixed esters, for example cellulose nitroacetate, cellulose ethers, such as ethyl, butyl and benzyl cellulose and mixed ethers and ether-esters, e. g. ethyl cellulose acetate, and oxyethyl cellulose acetate.

Moreover, while the invention is of particular value in increasing the heat resistance of artificial materials containing organic derivatives of cellulose, yet it is not limited to this purpose but may also be applied to obtain other improved properties, such as weighting or reduction or modification of lustre.

What I claim and desire to secure by Letters Patent is:—

1. Process for the production of filaments, threads, yarns, ribbons and the like from organic derivatives of cellulose, comprising incorporating therein an alkyl borate, and subsequently decomposing the said alkyl borate.

2. Process for the production of filaments, threads, yarns, ribbons and the like from organic derivatives of cellulose, comprising incorporating into the spinning solution an alkyl borate, and subsequently decomposing the said alkyl borate during the spinning process.

3. Process for the production of filaments,

threads, yarns, ribbons and the like from organic derivatives of cellulose, comprising incorporating into the spinning solution an alkyl borate, and decomposing the said alkyl borate by treating the formed products with an aqueous medium.

5 4. Process for the production of filaments, threads, yarns, ribbons and the like from an organic derivative of cellulose, comprising incorporating therein an alkyl borate, such that the amount of alkyl borate contained in the finished materials is from 5 to 8% by weight of the organic derivative of cellulose.

5. Process for the production of filaments, threads, yarns, ribbons and the like from organic derivatives of cellulose, comprising incorporating therein an organic compound of boron selected from the group consisting of ethyl and methyl borate.

6. Artificial filaments, threads, yarns, ribbons and the like composed of an organic derivative of cellulose and having an alkyl borate incorporated therein.

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