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# UNITED STATES PATENT OFFICE

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### ARTIFICIAL FILAMENTS AND METHOD OF MAKING SAME

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This invention relates to the preparation of artificial filaments, yarns, bristles, straw and the like containing organic derivatives of cellulose and an organic pigment material that imparts to the artificial material reduced lustre and other

desirable properties. An object of the invention is to prepare arti-

ficial filaments or yarns of subdued lustre especially adapted to be dyed a dark shade. Another 10 object of the invention is to prepare artificial

- filaments, yarns and the like of reduced lustre by incorporating therein agar-agar in a finely divided form. Other objects of the invention will appear from the following detailed description.
- Artificial filaments or yarns made of organic 15 derivatives of cellulose often have a lustre which is at times greater than desired for certain particular purposes. I have found that if finely divided agar-agar, which is not soluble in the  $_{20}$  solvents employed in making such filaments or
- yarns, but which is capable of being relatively permanently disseminated in such a solution, is incorporated therein, desired opacity and lack of lustre is imparted thereto and on dyeing less 25 dye is required than when dyeing other pigmented

yarns to the same depth of color. In accordance with my invention I prepare textile materials such as artificial filaments or yarns by the extrusion of a spinning solution 30 containing an organic derivative of cellulose in a

suitable solvent, which solution contains finely divided agar-agar in suspension, through orifices of appropriate size and shape into a medium causing solidification. The finely divided organic

35 material is disseminated in the filaments or yarns thus formed, and impart thereto a desirable subdued lustre, opacity and ability to take a dark dye appearing to have greater depth.

- By this invention, fine filaments of say from 1 40 to 25 denier or more may be formed, and a plurality of such filaments may be associated together by twisting to produce yarn. The yarns thus formed may be twisted to from 1 to 100 turns per inch. This invention also includes the yarns
- 45 formed from fine filaments that have been cut or broken to short lengths and the yarns formed therefrom by the woolen, worsted, cotton or French methods of forming yarns or threads from fibers. Heavier filaments to be used as artificial 50 bristles, horsehair, straw and the like of say 100

to 1000 denier or more may also be made. While cellulose acetate is preferred as the or-

ganic derivative of cellulose for making the filaments or yarns, any suitable organic ester or 55 ether of cellulose may be used. Examples of such organic esters of cellulose are cellulose formate, cellulose butyrate and cellulose propionate while examples of the ethers are methyl cellulose, ethyl cellulose and benzyl cellulose. In preparing the spinning solution, a suitable solvent or solvent 5 mixture for the organic derivative of cellulose may be used. Examples of such solvents or solvent mixtures are acetone, chloroform, mixtures of dichlorethylene and ethyl or methyl alcohol. The choice of the solvent or solvent mix- 10 ture will depend upon the solubility characteristics of the particular cellulose derivative employed. Generally the spinning solution contains from 15 to 35% of the organic derivative 15 of cellulose.

In order to impart subdued lustre, opacity and other desirable characteristics to the filaments or yarns that are to be formed there is incorporated in the spinning solution an appropriate amount of finely divided agar-agar that is not soluble in the solvent of the spinning solution nor hydrated by moisture or water contained in the spinning mixture. This material may be added to the spinning solution in amounts of from 0.5 to 5% or more on the weight of the cellulose derivative present.

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The agar-agar before being incorporated in the spinning solution is preferably ground, in a ball mill or similar device, in the presence of a dilute solution of a cellulose derivative say a 6 to 10% 30 solution of cellulose acetate in acetone if the spinning solution is to be cellulose acetate dissolved in acetone. The mixture is preferably milled until the agar-agar particle size is reduced to below 3 microns and 75% of them are below 35 1 to 1.5 microns. However the particle size may be as large as 4 microns.

Any suitable means may be employed for incorporating the finely divided agar-agar in the spinning solution. If the agar-agar is milled in a 40 dilute solution of the cellulose derivative corresponding or compatible with the spinning solution being formed, the necessary ingredients are added while mixing to raise the solution to the desired spinning concentration. If the agar-agar is ground while dry, the powder may be added directly to a pre-mixed spinning solution. If desired the spinning solution containing the finely divided material may be filtered before being spun. Because of the fine subdivision and the 50 low specific gravity of the agar-agar material employed, it remains in suspension in the spinning solution and passes without separation through filters and the spinning apparatus.

The spinning solution may contain besides the 55

agar-agar other effect materials such as metallic pigments, filling materials, soluble or insoluble dyes or lakes, fire retardants, plasticizers, sizes and lubricants. The lubricants may be the gly-5 cols or glycerols their substitution products and derivatives and/or oils such as sulphonated oils or oil acids, olive oil, teaseed oil, cotton seed oil or the animal and mineral oils.

The spinning solution containing the cellu-10 lose derivative and finely divided agar with or without effect materials may be extruded through suitable orifices into a drying evaporative atmosphere, as in dry spinning, or into a precipitating bath, as in wet spinning to form the desired

- 15 denier filaments. Due to the fineness of the agar-agar particles and other properties thereof such for example as their property of forming stable spinning solutions, filaments of exceedingly low denier may be formed. These fila-
- 20 ments may be grouped together by twisting to form yarns. The filaments thus formed, provided no sizing materials have been added thereto, are soft and pliable. They are also opaque due to the dispersion of light by the agar-agar
- 25 particles which have a different refractive index from that of the surrounding medium.

The yarn may be processed to fabrics as by weaving, warp knitting, circular knitting, knotting or netting in the usual manner. The soft 30 agar-agar particles at the surface of the yarn aid the yarn in passing through guides and

needles, without any abraiding action, in the various processing steps. While the yarn is in hank form or after it has

- 35 been processed into a fabric it may be subjected to an aqueous bath. This yarn is a sub-boil delustering yarn showing first signs of enhanced delustre at between 60° and 70° C. and gives a quick and pronounced delustre at 80° C. The
- 40 delustering properties caused by the change in the physical properties of the finely divided agaragar and that effect transmitted to the filaments, lends to the yarn an enhanced dyeing property i. e. in dyeing dark shades the yarn appears to 45 have a greater depth than normal yarn dyed
- with the same dye. The yarn after the treatment in the aqueous bath is opaque, soft and has a full hand.

As an illustration and not as a limitation the 50 following example is given.

#### Example

Agar-agar is milled with a 6 to 10% solution of cellulose acetate in 95/5 acetone/water solvent 55 until the particles are reduced in size to below 3 to 4 micron 75% of which are below 2 microns. A 26% spinning solution of cellulose acetate in acetone is prepared to which is added sufficient of the cellulose acetate agar-agar mixture to 60 form a mixture containing about 3% agar-agar on the weight of the cellulose acetate present. The solution is filtered and spun into filaments having a denier of from 2 to 3 by a dry method of spinning. The filaments are twisted to a yarn 65 and circular knit into a fabric. The fabric is treated in an aqueous bath containing from 2 to 3 grams per litre soap as a wetting out agent. The strength of the fabric is approximately normal The fabric is opaque dewhen either wet or dry. 70 lustered and soft and full in hand. The finished fabric containing permanently a large percent of the agar-agar which aid in imparting a good color to the goods when dyed, requiring less dye than for untreated yarns or yarns treated with 75 metallic pigments.

It is to be understood that the foregoing detailed description is merely given by way of illustration and many alterations 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 filaments, yarns and the like of subdued lustre, which comprises forming a solution containing an organic 10 derivative of cellulose and fine particles of solid agar-agar, extruding said solution through suitable orifices into a setting medium and subjecting the formed materials to the action of a hot 15 aqueous medium.

2. Process for the production of filaments, yarns and the like of subdued lustre, which comprises forming a solution containing an organic ester of cellulose and fine particles of solid agaragar, extruding said solution through suitable 20 orifices into an evaporative atmosphere and subjecting the formed materials to the action of a hot aqueous medium.

3. Process for the production of filaments, yarns and the like of subdued lustre, which com- 25 prises forming a solution containing cellulose acetate and fine particles of solid agar-agar, extruding said solution through suitable orifices into an evaporative atmosphere and subjecting the formed materials to the action of a 2-3% 30 soap solution at 60-80° C.

4. Process for the production of filaments, yarns and the like of subdued lustre, which comprises forming a solution containing cellulose acetate and a proportion of 0.5-5.0% by weight 35 of the cellulose acetate of fine particles of solid agar-agar having a particle size less than three microns, extruding said solution through suitable orifices into an evaporative atmosphere and subjecting the formed materials to the action of 40 a hot aqueous medium.

5. A spinning solution for the production of artificial filaments, yarns and the like of subdued lustre, comprising an organic derivative of cellulose dissolved in an organic solvent and contain-45 ing fine particles of solid agar-agar distributed throughout the solution.

6. A spinning solution for the production of artificial filaments, yarns and the like of subdued lustre, comprising cellulose acetate dissolved in 50 an organic solvent and containing fine particles of solid agar-agar distributed throughout the solution.

7. A spinning solution for the production of artificial filaments, yarns and the like of subdued 55 lustre, comprising an organic derivative of cellulose dissolved in a volatile organic solvent and containing particles of solid agar-agar having a size of less than 3 microns distributed throughout the solution. 60

8. A spinning solution for the production of artificial filaments, yarns and the like of subdued lustre, comprising cellulose acetate dissolved in a volatile organic solvent and containing particles of solid agar-agar having a size of less than 3 65. microns distributed throughout the solution.

9. A spinning solution for the production of artificial filaments, yarns and the like of subdued lustre, comprising cellulose acetate dissolved in a volatile organic solvent and containing a propor-70 tion of 0.5-5.0% on the weight of the cellulose acetate of fine particles of solid agar-agar distributed throughout the solution.

10. Filaments, yarns and the like having a basis of organic derivatives of cellulose, and con-75

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taining finely divided solid agar-agar distributed throughout their mass, which filaments, yarns and the like are readily delustered by the action of a hot aqueous medium at a temperature below 5 the boiling point of water.

11. Filaments, yarns and the like having a basis of cellulose acetate, and containing finely divided solid agar-agar distributed throughout their mass which filaments yarns and the like

their mass, which filaments, yarns and the like 10 are readily delustered by the action of a hot aqueous medium at a temperature below the boiling point of water.

12. Filaments, yarns and the like having a basis of organic derivatives of cellulose and con-15 taining distributed throughout their mass finely divided solid agar-agar, the particle size of which agar-agar, before treatment with an aqueous bath, is less than 3 microns, which filaments, yarns and the like are readily delustered by the action of a hot aqueous medium at a temperature of about  $80^{\circ}$  C.

13. Filaments, yarns and the like having a basis of cellulose acetate and containing distributed throughout their mass finely divided solid agar-agar, the particle size of which agar-agar, before treatment with an aqueous bath, is 10 less than 3 microns, which filaments, yarns and the like are readily delustered by the action of a hot aqueous medium at a temperature of about  $80^{\circ}$  C.

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