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TEXTILE PRODUCT AND METHOD OF
MAKING THE SAME

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This invention relates to a method of prepar-
ing artificial filaments, yarns and other textile
materials.

An object of our invention is to prepare arti-
ficial filaments having their appearance or other
properties modified by the incorporation therein
of finely divided solids. A further object of our
invention is to prepare such solids in a state of
minute subdivision. Other objects of this inven-
tion will appear from the following detailed de-
scription.

It has been previously proposed to incorporate
white inorganic pigment-like material in spinning
solutions in order to produce artificial yarns of
subdued lustre and increased opacity. In com-
mercial processes involving the use of such pig-
ments, it was necessary to subject the pigments
to prolonged and elaborate grinding processes in
order to attain the degree of subdivision neces-
sary to prevent clogging of the fine orifices of
the spinning jets and to avoid reducing the
strength of the resulting filaments to an undue
extent. However, even after such prolonged
grinding, the size of the pigment particles is not
reduced to as great a degree as is often desired.

Furthermore, it has been previously proposed
to incorporate colored pigments in spinning solu-
tions in order to produce colored yarn. However,
despite the great care taken in grinding such col-
ored pigments, the tinctorial value of the same is
relatively low so that relatively large amounts
thereof must be employed, and even the use of
such large amounts does not produce the desired
depth of shade.

We have found that if pigments or other solid
materials are prepared by subjecting them to
squeezing, rubbing or kneading action while they
are dispersed in a plastic mass, which mass pref-
erably comprises a derivative of cellulose, they
are reduced in a relatively short time to such a
fine state of subdivision as is not attainable by
prior methods. Not only is the time of treat-
ment reduced, but the particles of solids may be
used in smaller quantities to obtain effects such
as reduction of lustre or depth of colorations
heretofore unattainable.

In accordance with our invention then we pre-
pare artificial filaments containing cellulosic ma-
terial by incorporating in a spinning solution
from which they are formed a finely divided pig-
ment or other insoluble solid which has been pre-
pared by subjecting the same to a squeezing, rub-
bing or kneading action while dispersed in a plas-
tic mass which preferably comprises the same cel-

lulosic compound contained in the spinning so-
lution.

The filaments made in accordance with this
invention may be of fine denier which are associ-
ated together by twisting to form yarn, or the
filaments may be heavier, such as bristles, arti-
ficial horsehair and straw. Such filaments may be
made by the viscose, cuprammonium, Chardonnet
or other process, but this invention is of particu-
lar importance in connection with filaments made
of organic derivatives of cellulose such as organic
esters of cellulose and cellulose ethers. Examples
of organic esters of cellulose are cellulose acetate,
cellulose formate, cellulose propionate and cellu-
lose butyrate, while examples of cellulose ethers
are ethyl cellulose, methyl cellulose and benzyl
cellulose. The filaments containing the organic
derivative of cellulose may be prepared by dissolv-
ing the organic derivative of cellulose in a vola-
tile solvent such as acetone and extruding such
solutions through fine orifices into an evaporative
atmosphere, as in dry spinning, or into a precip-
itating bath, as in wet spinning.

As stated, we add a finely divided pigment or
solid modifying agent to the spinning solution or
dope. This pigment may be white to obtain sub-
dued lustre and increased opacity. Examples of
white inorganic pigments are tin oxide, tin phos-
phate, antimony oxide, titanium dioxide, barium
sulfate, lead sulfate, calcium sulfate, zinc oxide,
zinc carbonate, aluminum oxide, silicon dioxide,
barium borate, calcium borate or silicates such
as china clay or other clays, talc or mica. The
pigment may be of organic nature such as di-
acetyl benzidine, diacetyl toluidine, dibenzoyl
benzidine, naphthyl urea, anthraquinone, an-
thracene, suitable synthetic or natural resins, cel-
lulose in the form of fibres or powder, degraded
cellulose, starches etc.

This invention is of particular interest for the
preparation of colored filaments, in which case
a colored inorganic or organic pigment may be
employed to obtain the desired color or shade.
For a yellow color, ochre, sienna, chrome yellow,
tin bronze, etc. may be employed. For a red
color, Venetian red, red lead, vermillion, etc., may
be employed. For a blue color, ultramarine,
Prussian blue, Millori blue, etc., may be used. For
green, Guignet's green, verdigris, chrome green
may be employed. For brown, raw umber, burnt
umber or Vandyke brown may be used. For
black, lamp black, carbon black, graphite or
other black pigment may be used, which may
be toned with iron gallate or other toners to ob-
tain jet blacks. To obtain any other colors,

the pigments may be mixed as is well understood in the paint art. To obtain metallic effects, finely divided or colloidal metals may be employed.

The amount of pigment added will vary with a degree of subdued lustre and/or the depth of color desired and the nature of the pigment employed and will generally be from 0.1 to 10% of the weight of the cellulosic material present in the finished yarn.

If it is desired to produce weighted filaments, the phosphate, silicate, oxide or other compound of tin, zinc, lead or other weighting metal may be incorporated in sufficiently large amounts, say from 10 to 40% or more of the weight of the cellulosic material present in the finished filaments.

Other solid modifying agents that are insoluble or incompatible with both the solvent and the derivative of cellulose present in the spinning solution may likewise be incorporated in accordance with our invention.

As stated, prior to incorporation in the spinning solution, the pigment or like material is reduced to a very fine state of subdivision by subjecting it to a manipulation involving kneading, squeezing or rubbing operations while dispersed in a plastic mass. This action causes the breaking up or disintegration of the agglomerates or aggregates in which the pigment particles may occur. These operations are conveniently carried out by processes ordinarily employed for making articles from cellulose derivative plastics. Thus a heavy solution or plastic mass may be formed by mixing the same derivative of cellulose as is employed for making the filaments with a relatively small amount of volatile liquid that is a solvent or becomes a solvent at elevated temperatures for the derivative of cellulose, whereupon the pigment or other modifying material is added and the mass is kneaded at elevated temperatures. During this kneading, part of the solvent evaporates to leave the heavy doughy mass which is then worked between heated malaxating rolls, the sheets formed by such rolls being repeatedly passed between the heated rolls which are spaced rather closely. This rolling operation is repeated until substantially all of the volatile liquid is evaporated. The sheets are finally broken into pieces which are then used for incorporation in the spinning solution.

This kneading, rubbing or squeezing operation reduces the pigments to extremely fine particle size which is usually less than one micron in diameter. As an example of the minuteness of subdivision attainable by this method, it is pointed out that if carbon black is treated, substantially all, or at least a major portion of the particles are too small to measure microscopically having a diameter less than 0.2 micron, there being present about 10% of aggregates having diameters of between 0.2 to 0.5 micron.

Because of the minute size of the particles, filaments containing them have substantially the same tenacity and elongation as filaments free of any pigments, while the incorporation of pigments ground by prior methods appreciably reduces the tenacity and elongation. The filaments prepared in accordance with this invention have a greater depth of color and/or subdued lustre than filaments containing appreciably larger amounts of pigments previously employed. By way of example, it is pointed out that whereas it is possible to obtain only a grey-black by

incorporating any amount of carbon black by the previous method, a jet black may be obtained by the incorporation in a spinning solution of but a very small quantity of carbon black prepared in accordance with this invention.

In order further to illustrate our invention but without being limited thereto, the following specific example is given.

Example

The following ingredients:

	Part by weight
Cellulose acetate	1
Carbon black	1
Acetone	1.5

are kneaded together in a heated kneading machine until a stiff doughy mass is formed. This mass is then worked between relatively closely spaced heated malaxating rolls until the product is substantially free of the acetone which is a volatile solvent for the cellulose acetate. The resulting sheet is broken into chips or pieces.

A spinning solution is formed by dissolving one part by weight of cellulose acetate in three parts by weight of acetone and to this is added an amount of above described pieces or chips so that the solution contains 0.03 part of carbon black. The ingredients are thoroughly mixed and are then filtered, and the filtered spinning solution is extruded through the fine orifices of a spinning jet into a heated evaporative atmosphere. The resulting filaments are twisted and wound onto a package.

The yarn so formed is jet black, and may be used for weaving or knitting fabrics that are as black as any cellulose acetate fabric that has been dyed in the piece.

It is to be understood that the foregoing detailed description is given merely by way of illustration and many variations may be made therein without departing from the spirit of our invention.

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

1. In a process for the manufacture of artificial filaments, yarns and the like from solutions of derivatives of cellulose by wet or dry spinning processes, the steps of working a plastic mass comprising a derivative of cellulose and a pigment-like material between rolls until the plastic mass is brought to a substantially dry state and the particle size of the pigment-like material is reduced, and dispersing pieces of the resulting product in the spinning solution containing the same cellulose derivative.

2. In a process for the manufacture of artificial filaments, yarns and the like from solutions of cellulose acetate by wet or dry spinning processes, the steps of working a plastic mass comprising cellulose acetate, a softening agent therefor, and a pigment-like material between rolls until the plastic mass is brought to a substantially dry state and the particle size of the pigment-like material is reduced to below 0.5 microns, and dispersing the pieces of the resulting product in the spinning solution.

3. In a process for the manufacture of artificial filaments, yarns and the like from solutions of cellulose acetate by wet or dry spinning processes, the steps of working a plastic mass comprising cellulose acetate, a softening agent therefor, and carbon black between rolls until the plastic mass is brought to a substantially dry state and the particle size of carbon black is reduced to below 75

0.5 microns, and dispersing the pieces of the resulting product in the spinning solution.

5 4. Artificial filaments comprising a cellulose derivative and finely divided pigment-like material produced from a spinning solution of a cellulose derivative in which has been dispersed pieces formed by working a plastic mass comprising the

same cellulose derivative and the pigment-like material between rolls until the plastic mass is brought to a substantially dry state and the particle size of the pigment-like material is reduced.

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