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# UNITED STATES PATENT OFFICE

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## IDENTIFIABLE MATERIAL AND METHOD OF MAKING THE SAME

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This invention relates to the preparation of artificial yarns or other materials which yarns or other materials have therein a foreign substance that may be readily identified, especially when present in small quantities.

An object of our invention is to render artificial yarn, films, plastic masses or coating compositions readily identifiable by the addition to the solution of which they are formed, or to the yarn or other articles after their formation, of a compound of a metal that can be readily identified. Other objects of our invention will appear from the following detailed description.

Often it is desirable to have artificial yarns, films, plastic masses or coating compositions prepared so that they may be readily identified as to the nature of the materials employed in their manufacture, the method of preparing them and the source of manufacture. When only small amounts of the yarn or other articles are available, it is difficult to identify the same by ordinary methods of chemical or physical analysis. We have found, however, that if a small amount of readily detectable compounds are added to the yarns, they may be easily identified.

In one form of our invention, we prepare artificial yarns of any nature by adding either to the solution from which they are spun, or to the yarns after they are formed, a small amount of a compound that does not occur in the substances from which the yarn is prepared, but which may be readily detected either by physical tests or by chemical tests. Likewise, when films, plastic masses or coating compositions are to be treated in accordance with our invention, such foreign compound may be added to the solutions from which they are formed.

The yarn that forms the subject of this invention may be made of any suitable cellulosic material or its derivatives. Thus the yarn may be made of organic derivatives of cellulose which include organic esters of cellulose or organic ethers of cellulose. Examples of organic esters of cellulose are cellulose acetate, cellulose formate, cellulose propionate and cellulose butyrate; while examples of cellulose ethers are ethyl cellulose, methyl cellulose and benzyl cellulose. The yarns are prepared from these organic derivatives of cellulose by dissolving the same in a suitable volatile solvent, and extruding such solution through the orifices of spinerettes, either into a heated evaporative atmosphere as in dry spinning or into a precipitating bath as in wet spinning. This invention, however, is not restricted to yarn made of organic derivatives of cellulose, but also includes yarns of the reconstituted cellulose type such as are made by the viscose, cuprammonium or the Chardonet process.

The foreign substance added may be any suitable compound that is readily detectable. As an example of a compound that may be detected by physical means, strontium salts, particularly strontium chloride, may be mentioned. The yarn containing strontium salts may be tested either by the flame test, or in the spectroscope for the characteristic strontium spectrum. As an example of a compound that may be added to the yarn and which may be readily detected by chemical analysis, nickel salts, and particularly nickel chloride, is mentioned. The nickel compounds give a very delicate reaction with dimethyl glyoxime $(CH_3)_2C_2(NOH)_2$.

The proportions of the detectable material that may be added to the yarn may vary from 10 to 40 or more parts of the compound to one million parts of the yarn. Generally we prefer to add as small an amount of the compound as possible because of regard to the cost and other considerations. However, we prefer to add a sufficient amount of this compound so that it may be detected with samples of yarn as small as one gram. Thus we find that when we employ a water soluble salt such as nickel chloride ($NiCl_2.6H_2O$), 40 parts of this material per million parts of the yarn produce a product that is not substantially affected by subsequent washings, dyeings, scourings and the like.

The yarn, when prepared as above, may be readily identified. A convenient method of making the analysis of such yarn when it contains a strontium compound is to reduce the same to an ash and then to test the ash either

in a flame or by the aid of a spectroscope. For the testing of yarn containing a nickel salt, the yarn is ashed and the nickel contained therein is dissolved in the form of a salt in a small quantity of slightly alkaline water. This solution is then treated with an alcoholic solution of dimethyl glyoxime. The nickel combines with the dimethyl glyoxime to form a characteristic red crystalline precipitate. Of course it is understood that if volatile salts, such as nickel chloride, are employed, it is preferable to ash the same in the presence of sulphuric acid, so as to convert the volatile chloride to the less volatile sulphate.

While we have specifically cited compounds of nickel and strontium, it is to be understood that compounds of other metals may be employed. The reason that these metals have been specifically named is that they give pronounced physical or chemical tests and that they do not occur in the ingredients commonly employed for making artificial yarns. Furthermore, nickel is very resistant to the acids, alkalies, etc., employed in the manufacture and treatment of textile materials, and therefore yarns do not ordinarily become contaminated with nickel that may be used in apparatus employed for producing such yarn.

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

*Example*

A spinning charge is made by dissolving one part of an acetone soluble cellulose acetate having an acetyl value of 54 to 55% in 3 parts by weight of acetone. To this solution is added an aqueous solution of crystalline nickel chloride in such amounts that there are present 20 parts of nickel per million parts of cellulose acetate. The solution is then spun through the orifices of a spinerette into the metier of a dry spinning machine, wherein the acetone is evaporated by heated air. The filaments thus formed are drawn, twisted into yarn and wound into packages. The yarn thus made gives the characteristic nickel reaction with dimethyl glyoxime when treated as above set forth.

Although this invention has been described specifically in connection with the identification of yarns, it is equally applicable in the preparation of films, lacquers or plastic masses containing derivatives of cellulose such as cellulose nitrate or the organic derivatives mentioned above. These films, plastic masses and coating compositions will also contain low, medium, and/or high boiling solvent and plastifiers, as is well understood in the art.

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

The term "foreign substance" as used in the claims is to be interpreted as meaning substances which are not necessary for yarn, fabric or film formation. Substances which normally are not present in the yarn, fabric or film are thus included in this term.

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

1. Artificial silk containing 10 to 40 parts of a readily identifiable metal compound per one million parts of the silk.

2. Organic derivative of cellulose artificial silk containing 10 to 40 parts of a readily identifiable metal compound per one million parts of the silk.

3. Cellulose acetate artificial silk containing 10 to 40 parts of a readily identifiable metal compound per one million parts of the silk.

4. Cellulose acetate artificial silk containing 10 to 40 parts of strontium chloride per one million parts of silk.

5. Cellulose acetate artificial silk containing 10 to 40 parts of nickel chloride per one million parts of silk.

In testimony whereof, we have hereunto subscribed our names.

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