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YARN AND FILAMENT AND METHOD OF
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poration of DelawareNo Drawing. Application January 19, 1934.
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This invention relates to the treatment of products made of cellulose ethers and esters, such as filaments, threads, yarns, tapes, straws, ribbons, bristles and like textile materials, films, foils, slabs, sheets and other materials having flat, cast or rolled surfaces and fabrics woven, netted, knitted, braided, knotted or otherwise formed from the textile materials.

The principal object of the invention is to render yarns, films, foils and textile fabrics more amenable to textile operations as by rendering them free from the trouble occasioned by electrification and also to make them more pliable and supple, so that they may be woven, knitted or otherwise manipulated without danger of breaking.

Other objects of the invention will appear from the following detailed description.

I have found that if the materials and/or products made of or containing cellulose derivatives are treated with an aliphatic diamine salt of a hydroxy carboxylic acid or other organic or inorganic acid that their textile properties are greatly improved. These salts may be applied to or incorporated in artificial materials, depending on the method of forming the same. The treatment of yarns according to this invention produces yarns which will not take a charge of electricity upon friction with like yarns, yarns of other materials, glass rods, metal rods, etc., as the yarns become conductors of electricity and the electric charge is carried off as soon as it is formed. Once the yarns have been treated, this property of conducting electricity is not reduced upon ageing but is carried through the processing to the finished fabrics. The treating material does not oxidize or deteriorate upon standing nor does it decompose into injurious acids, thus permitting treatment of yarns etc. which are to be stored for long periods of time before final processing into finished articles.

The yarns, films and fabrics treated according to this invention have no corrosive action on metal and metal alloys with which they contact or upon which they may rest for days in the processing steps. The treating material contains no free acids such as chlorine and is neutral in action. The yarns and films treated with aliphatic diamine hydroxy salts of carboxylic acid or other acids are neither tacky nor gummy and do not become so upon standing and they therefore pass through all the guide means without difficulty or without formation of ridges or drops of the treating material about the guides. Yarns treated for anti-static properties accord-

ing to this invention that are formed into staple fibres by cutting etc., need not be subjected to treatment to remove the anti-static finish after cutting to prevent trouble by rusting or corrosion of card machines drafting rolls or other parts of spinning apparatus. In this way, danger of further packing and snarling up of the staples so that they would be difficult to open and card is eliminated. Also the property of not taking an electric charge is of advantage in the carding, drawing and other subsequent processes.

Yarns so treated are sufficiently lubricated as well as rendered immune from static electric charges that they may be warp knitted or otherwise fabricated, passing freely through the guides and without fluffing or spreading.

Fabrics treated according to this invention or made from yarns so treated are also conductors of electricity and do not retain a charge of static electricity. A great source of annoyance caused by static electricity is found in women's wearing apparel; for example when a fabric of artificial silk or a fabric containing derivatives of cellulose and/or regenerated cellulose is used as an outer garment over silk or wool undergarments, since the outer garment, if untreated, takes on a static charge which causes the outer garment to tend to adhere or cling to the wearer's body destroying the lines and design of the garment. Fabric treated according to this invention is free from static electricity and does not tend to adhere to the wearer's body but hangs free. The fabric so treated is not off odor nor tacky and retains its electrical conducting properties regardless of its age.

Another source of annoyance in fabrics which and non-conductors for electricity is that, after becoming charged, they attract dust particles, both organic and inorganic. Brushing to remove such particles merely increases the electric charge and the particles appear to cling to the fabric with even more tenacity. Fabrics treated according to this invention allow the static charge of electricity to leak out as fast as it is formed and the fabric therefor does not attract dust particles and, further, any dust particles thereon may be easily removed by brushing or shaking as there is no electrical force attracting them thereto and there is no moist or tacky substance tending to hold them to the fabric.

The treating material here described has a special applicability to films and foils as the material is not absorbed into the body of the films to any great extent but remains on or near the surface. Films therefore are not discolored or

made cloudy. Furthermore the static charge of electricity is a surface charge and conducting material not on the surface does not allow for the fast discharge of developed electric charges.

5 In accordance with my invention I treat or incorporate in yarns, films, foils and fabrics, a small amount of an aliphatic diamine salt of a hydroxy carboxylic acid or other acid with, or without lubricating materials, carriers and colors.

10 The yarns, films and fabrics to be treated may be made from regenerated cellulose, wool, cotton, silk, flax, or other fibres. This invention is, however, most applicable to yarns, films, foils, fabrics and other artificial materials formed at least in part of organic derivatives of cellulose which yarns, films, foils and fabrics are normally either non-conductors or weak conductors of electricity depending upon the effect materials used with the organic derivatives of cellulose. The most common of the organic derivatives of cellulose are the esters and ethers of cellulose. Examples of organic esters of cellulose are cellulose acetate, cellulose formate, cellulose propionate and cellulose butyrate, while examples of organic ethers of cellulose are ethyl cellulose, methyl cellulose, and benzyl cellulose.

The yarns and films or foils of derivatives of cellulose may contain incorporated as a part thereof or as coating thereon, effect materials such as pigments, filling materials, dyes or lakes, fire retardants, plasticizers, sizes and lubricants. Examples of fire retardants are beta chlornaphthalene, triphenyl phosphate and tricresyl phosphate. Examples of plasticizers are triacetin, diethyl phthalate, dibutyl tartrate, ethyl toluene sulfonamid, etc. Examples of lubricants are mineral oil, olive oil, teaseed oil and neat's-foot oil. Examples of filling materials are powdered metal, oxides of metal or fibres of other materials. Examples of sizes are wax, gelatins, starches, gums, salts of resin acids and glycerides. These effect materials may be incorporated as a part of the filaments or films by adding them to the spinning solution from which the filaments or films are formed or by softening or swelling the formed filaments or films with a swelling agent, such as thiocyanates of ammonia, sodium, potassium and calcium and ethyl ether of ethylene glycol, and adding the effect materials to the filaments and films while they are in the swollen condition.

Under the term yarns are included threads, tapes, straws, ribbons, bristles and like textile materials and assemblies or bundles of a number of continuous filaments which may be in parallel relationship or which may be twisted together, short lengths of staple fibres or yarns spun from such staple fibres.

While the treating material is preferably applied to the yarns, filaments and the like after their formation by suitable devices, the treating material may be incorporated in the solution of the derivatives of cellulose from which such artificial products are formed.

65 The fabrics to be treated may be formed of a single type of yarn, say one formed by twisting together fine filaments of an organic derivative of cellulose, or the fabric may be formed of a plurality of types of yarn, as a warp of silk and weft of a cellulose acetate. The fabrics may be coated after treatment according to this invention with coating materials such as rubber, latex, resins, lacquers, etc. for making rain coats etc. The anti-static finish does not interfere with the chemical make-up of such coatings and fur-

thermore it is not an impervious coating which would allow any future coating applied to the fabric to peel off.

The yarn, filaments or fabric are rendered completely effective in anti-static properties by the application of as little as $\frac{1}{2}\%$ by weight of the aliphatic diamine salt of hydroxy carboxylic acids. When it is desired to treat yarn and carry the effect into the fabric a slightly larger per cent say 2 to 5% will be found sufficient. Even as small as 0.1% of the aliphatic diamine hydroxy carboxylic acid may be used effectively if it is to be applied to the yarn, film or filaments along with a lubricant such as olive oil, teaseed oil, blown olive oil, sulphonated castor oil, light mineral oils, rape seed oil and animal oils or glycol and glycerol or their oleates and stearates.

It is often found valuable for the purpose of lubrication and identification when treating yarns with a mixture of an aliphatic diamine salt of a hydroxy carboxylic acid and diethylene glycol or similar lubricant to incorporate in the mixture a small per cent of a concentrated dye stuff such as the diamino-derivative of triphenylmethane which has been treated to remove the inorganic sodium salts contained therein. This dye stuff greatly enhances the lubricative properties of the mixture.

In treating formed fabrics not treated in the yarn with the anti-static material it is preferable to apply about 0.5% of same, after dyeing and before tentering. This may be done by passing the fabric over rollers which dip into a trough containing a solution or suspension of the anti-static material or by passing the fabric through a bath of about 1% of anti-static material either in a solution or emulsion and then through the tenter machine.

The anti-static material may be dissolved up to 50% concentration in aliphatic polyhydric alcohols and their substitution products. Emulsions of the anti-static material may be made with water and oil or modified oils.

A further advantage of the treated yarns, and particularly continuous filament yarns, is that a separate application of lubricants may be rendered unnecessary in many textile operations, for example in the operation of winding or twisting and winding artificial yarns continuously with their production. The anti-static and lubricant may be applied simultaneously if desired or necessary, however, as stated above, a lubricant may also be applied to the yarn either in the same liquid as the one in which the anti-static material is applied or separately, the lubricant acting externally of the yarn to facilitate flexing, and enables it to withstand hard treatment, such as encountered in fine gauge knitting.

The anti-static material may be applied from solutions or suspensions in liquids which are hygroscopic for example ethylene glycol, diethylene glycol, glycerol etc. In order however, to prevent change in volume, viscosity and concentration of such liquids during use, it is preferred to add a quantity of water substantially equal to the maximum amount which the liquids tend to absorb in the atmosphere. Thus ethylene glycol may receive added water to the extent of 13% its original weight, glycerol 33% and diethylene glycol up to 30%. The hygroscopic material may be dissolved in water before mixtures of them are formed. Although the anti-static materials forming a part of this invention are somewhat hygroscopic in themselves they do not rely entirely upon this property as they permit the lead-

ing away of electric charges even in dry atmospheres.

The anti-static material with or without lubricants may be applied to running yarns at the spinning cabinet or any future winding operation by means of wicks or pads dipping in a solution or suspension of the anti-static material. Or it may be supplied by means of a drip tube or furnishing rollers supplied with a solution or suspension of the anti-static material. Whatever the means employed to apply the liquid, it is preferable to arrange that the liquid can be applied in a uniform manner and to a predetermined amount. Thus the concentration of solution and suspensions may be varied or the height of that part of the wick or rollers above the bath may be varied. A particular suitable wick device is the one described in U. S. application No. 538,484 filed May 19, 1931. Less care need be exercised in regards to uniformity of application in applying the anti-static material herein described than other anti-static materials as the one here described has a slight wetting action of its own which permits of a certain amount of spread on the yarns after application.

It is found that aliphatic diamine salts of hydroxy carboxylic acids or other acids with or without lubricating materials give an anti-static property to yarns, fabrics and filaments which are not tacky, do not give a harsh hand to fabrics, do not deteriorate on long standing, to give off colors and/or odors and do not give on long standing injurious acids or other reagents which would react with either the cellulosic material or the elements with which they come in contact and do not give rise to difficulties in future processing such as dyeing, coating, etc.

Examples of aliphatic diamine derivatives of hydroxy carboxylic acid are ethylene diamine lactate, ethylene diamine citrate, ethylene diamine tartrate. Either the mono- or di-acid salts of the diamine may be used. Thus the monolactates or dilactates of ethylene diamine may be employed. Besides the ethylene diamine the higher homologues and their isomers of that genus may be used as the base such as trimethylenediamine, tetramethylenediamine, pentamethylenediamine etc. Also the tartrates, citrates and lactates of the substituted aliphatic diamines may be employed. Any of the above aliphatic diamine salts may be used alone or as mixtures in place of, or in connection with ethylenediamine tartrate, citrate and lactate. Similar carboxylic acid radicles may be used, such as, for example: mesotartaric, saccharic, mucic acid and their isomers, gallic acid and salicylic acid. The borate salts of the aliphatic diamines may also be used.

To produce an anti-static material which allows for the readily conducting away of electric charges imposed by friction or other means, such as not rusting or exerting a corrosive action on the metals with which it comes in contact, it is necessary to employ a neutral electrolyte. The electrolyte therefore should be composed of an acidic and a basic material of substantially equal strengths. Upon hydrolysis, the acidic and basic materials must be such that neither leave the field of reaction as by precipitation, evaporation or reaction with adjacent materials, thus leaving an excess of the other present, nor should the electrolyte be sensitive to light and heat of normal strength and duration. All these features necessary to a good anti-static material are found in the aliphatic diamine salts of hydroxy car-

boxylic acids. However, the aliphatic diamine derivatives of other acids may be employed to form neutral derivatives or salts which are applicable to yarns. According to this invention, examples of such other acids are tannic acid, where a little stiffness or size is desired on the yarn, acetic anisic acid, benzoic acid, succinic acid, maleic acid and fumaric acid.

The following examples are given as description and not as limitations.

Example I

The yarn of an acetone soluble cellulose acid is drawn from the spinning cabinet in which it is formed by the dry or evaporative method and is caused to pass across a wick which dips into a solution composed of 90% of olive oil and 10% ethylene diamine borate. The wick is submerged in the solution such that at the rate of speed at which the yarn is spun and the area of the wick in contact with the yarn, it will apply to the yarn a coating of 2½% of this mixture based upon the weight of the yarn.

Example II

To a solution of cellulose acetate in acetone is added 5% based on the weight of the cellulose acetate of ethylene diamine tartrate. This solution is then spun into filaments by either the wet or dry method of spinning.

Example III

The yarn of an acetone soluble cellulose acetate is drawn from the spinning cabinet and is caused to pass across a wick which dips into a solution composed of 100 parts diethylene glycol, 30 parts of water and 10 parts of ethylene diamine citrate.

Having described my invention what I desire to secure by Letters Patent is:

1. Method of improving the properties of yarns, fibres, fabrics and films comprising incorporating therein an organic material comprising a salt of an aliphatic diamine.
2. Method of improving the properties of yarns, fibres, fabrics and films containing organic derivatives of cellulose which comprises incorporating therein an organic material containing a salt of an aliphatic diamine.
3. Method of improving the properties of yarns, fibres, fabrics and films containing cellulose acetate which comprises incorporating therein an organic material containing a salt of an aliphatic diamine.
4. Method of improving the properties of yarns, fibres, fabrics or films containing organic derivatives of cellulose which comprises applying to the yarns, fibres, fabrics and films a neutral salt of an aliphatic diamine.
5. Method of improving the properties of yarns, fibres, fabrics or films containing cellulose acetate which comprises applying to the yarns, fibres, fabrics and films a neutral aliphatic diamine salt of an organic acid.
6. Method of treating yarns, fibres, fabrics or films containing organic derivatives of cellulose which comprises applying to the yarns, fibres, fabrics and films an aliphatic diamine salt of a hydroxy carboxylic acid.
7. Method of treating yarns, fibres, fabrics or films containing cellulose acetate which comprises applying to the yarns, fibres, fabrics and films an aliphatic diamine salt of a hydroxy carboxylic acid.
8. The method of treating yarns, fibres, fabrics

or films containing organic derivatives of cellulose which comprises applying thereto an aliphatic diamine salt and a lubricant.

5 9. The method of treating yarns, fibres, fabrics or films containing cellulose acetate which comprises applying thereto an aliphatic diamine salt of an organic acid and a lubricant.

10 10. The method of treating yarns, fibres, fabrics or films comprising applying thereto an aliphatic diamine salt of an organic acid in a medium containing an oil.

15 11. The method of treating yarns, fibres, fabrics or films containing cellulose acetate which comprises applying thereto an aliphatic diamine salt of an organic acid in a medium containing an oil.

20 12. The method of treating yarns, fibres, fabrics and films containing organic derivatives of cellulose which comprises applying thereto an aliphatic diamine salt in a medium comprising an alcoholic body.

25 13. The method of treating yarns, fibres, fabrics and films containing cellulose acetate which comprises applying thereto an aliphatic diamine salt of a hydroxy carboxylic acid in a medium comprising an alcoholic body.

14. The method of treating yarns, fibres, fabrics

and films comprising applying thereto an aliphatic diamine derivative of a hydroxy carboxylic acid in a medium containing a polyhydric alcohol and a diamino-derivative of triphenylmethane.

5 15. The method of forming staple fibres formed from substantially continuous filaments containing organic derivatives of cellulose comprising treating the filaments with an aliphatic diamine salt in an alcohol and cutting the filaments to staple length.

10 16. Yarns, fibres, fabric and films comprising an aliphatic diamine salt.

17. Yarns, fibres, fabric and films of organic derivatives of cellulose comprising an aliphatic diamine salt of a carboxylic acid.

15 18. Yarns, fibres, fabric and films of organic derivatives of cellulose coated with an aliphatic diamine salt of a hydroxy carboxylic acid.

19. Yarns, fibres, fabric and films of cellulose acetate coated with an aliphatic diamine salt of a hydroxy carboxylic acid.

20 20. Yarns, fibres, fabric and films of cellulose acetate coated with an aliphatic diamine salt of a hydroxy carboxylic acid and a glycol.

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