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3,033,699

ANTISTATIC COMPOSITION

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This invention relates to compositions and processes for improving synthetic fiber yarns, and is more particularly directed to compositions especially useful for decreasing the tendency of such yarns, specially nylon yarns, to develop static electricity, and at the same time increasing the soil resistance of the yarns, said compositions comprising an aqueous suspension of a magnesium montmorillonite clay and an alkali-stabilized colloidal silica sol in which the silica particles are substantially discrete and in the size range of about 3 to 150 millimicrons and the SiO_2 :alkali oxide mol ratio is in the range of about 60:1 to 500:1, and is further particularly directed to processes for producing such soil and static resistance in synthetic yarns, said processes comprising the steps of coating the yarn with a composition comprising a magnesium montmorillonite clay which has been dispersed in an aqueous medium by high shear mixing, the treating composition preferably also containing an alkali-stabilized colloidal silica sol of the type described, and drying the coated yarn.

Carpets in which the yarn is made up completely or in part of synthetic fibers, especially nylon fibers, have shown remarkable wear resistance and for this and other reasons have found wide acceptance in the carpet industry. At very low relative humidities, however, such carpets tend to develop static electricity when walked upon and this electrostatic is transmitted to the person of the user who thereupon experiences an electrical "shock" when he touches a grounded object. While these shocks are in no sense dangerous they are sometimes considered unpleasant and hence the existence of this phenomenon has tended to retard the fullest acceptance of synthetic fibers in carpet yarns.

By the nature of their use carpets are subjected to severe soiling conditions and it has long been recognized that any treatment which would retard the soiling of carpets would be highly advantageous. At about the time that such synthetic fibers as nylon became widely available for use in carpet yarns there also started a trend toward carpets of lighter colors such as pastel shades and even whites or off-whites. This made the problem of soil resistance particularly difficult.

To be most effective as an anti-static and anti-soil agent for synthetic fibers a treating agent should have at least some resistance to removal by ordinary cleaning methods. Obviously, no treatment which is readily removable by ordinary vacuum cleaning of the treated fibers would be of any practical value. Moreover, since rugs are often spot-laundered during use the ideal treatment will have resistance to removal by laundering.

Various treatments have already been suggested for increasing the soil resistance of carpets made from yarns of synthetic fibers. Probably the most effective of these has been the treatment described in Cogovan and Friderici Patent 2,622,307. Such treatments, however, were not concerned with the anti-static problem nor with the laundering problem.

Now according to the present invention it has been found that by coating synthetic carpet yarns such as nylon with an aqueous medium in which a magnesium montmorillonite clay has been dispersed under high shear, and drying the coating, the tendency of the yarns to develop static electricity during use in carpets is substantially reduced and the ability of the yarns to resist soiling is sub-

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stantially increased. When an alkali-stabilized colloidal silica sol of 3 to 150 millimicron-sized particles is also included in the treating compositions, the soil resistance of the treated yarns is further enhanced and the treating agents are surprisingly resistant to removal by laundering.

The yarns treated by the novel processes and compositions are made up of synthetic, man-made fibers as distinguished from such naturally occurring fibers as cotton, wool, flax and the like. Included in the former class are polyester fibers, polyacrylic fibers, polyolefin fibers, polyvinyl fibers and regenerated cellulose fibers including viscose fibers. The treatments are particularly effective when applied to the polyamide fibers known as nylon.

The treated yarns can be used for other purposes than in carpets, of course, such as in various kinds of textile fabrics. In automobile upholstery, suiting fabrics, and sheeting, for instance, and other uses where development of static in fabrics is objectionable or annoying, the treated yarns have particular applicability. However, the advantages of the treatment are most strikingly demonstrated in carpets and rugs since these are subjected to the most intense conditions of soiling and static generation. The yarns can be treated before or after incorporating them into the carpet or rug, and there is some advantage in the latter, since it does not require treatment of the portion of yarn which is not subjected to the severest soiling conditions—namely, the part woven into the backing.

The treatment can be applied to the yarn by any method which effects thorough distribution of the aqueous composition onto the fibers of the yarn. For instance, the treating compositions can be sprayed or brushed onto the yarn or the yarn can be immersed in a bath of the treating liquid.

When the yarn has already been incorporated as the pile of a carpet, it is particularly preferred to distribute the treating liquid over the surface of an applicator roll and bring just the pile of the carpet into contact with the roll.

The treated yarn can be dried in any convenient manner, as by passing the dried yarn through a circulating air oven.

The clay dispersion used in the treating process contains a magnesium montmorillonite clay which has been dispersed in an aqueous medium under high shear. Such clays are commercially available. One clay of this type appears in electron micrographs to have an ultimate particle which is about 1 micron long, 100 to 200 millimicrons wide and 1 to 10 millimicrons thick. When such clays are dispersed in water with ordinary mixing little viscosity increase is obtained even after prolonged contact with the water, and sedimentation occurs on standing. When high shear mixing is employed, however, such as is obtainable with a Waring blender, sols are obtained which are viscous and non-settling. For example, a thixotropic, non-flowing, almost paste-like dispersion is obtained by shear mixing 3 to 4% of the above-mentioned clay in water. The intensity of shear in the mixing process is therefore that which is sufficient to produce a substantial viscosity increase.

The aqueous medium in which the clay is dispersed under shear will, of course, contain water and can additionally contain suspending agents, wetting agents, and other adjuvants such as are commonly employed in aqueous compositions which are to be applied to textile fibers. The aqueous medium can also contain water-miscible, organic solvents although ordinarily such organic solvents are not necessary. The aqueous medium can contain acidic or basic constituents for adjusting the pH to optimum value for any particular treating situation.

In the novel compositions of this invention there is present, in addition to the aqueous clay dispersion, an alkali-stabilized colloidal silica sol in which the silica

particles have an average dimension in the range of about 3 to 150 millimicrons. It will be understood that the clays can be dispersed in the silica sol when the continuous phase of the sol is an aqueous medium.

Alkali-stabilized silica sols can be prepared by a variety of methods with which the art is already familiar. For instance, the sols can be prepared as described in the White Patent 2,375,738. These are not preferred because the particles are not discrete but are aggregated into clusters. They nevertheless will give some benefit in unusual circumstances and where whitening is not objectionable. The sols of the Bird Patent 2,244,325 and Voorhees Patent 2,457,971 can also be used. Generally these sols as prepared by ion exchange have a particle size below 5 millimicrons and hence are close to the lower limit of the desired size range, but the particles may be grown by heating them. Also the sols have a tendency to coalesce more than is desired.

Silica sols more effective than those just mentioned can be prepared according to processes shown in Bechtold and Snyder Patent 2,574,902. The sols of this patent have uniform, discrete, spherical particles of about 15 to 150 millimicrons in diameter. Sols produced as shown which have particles up to about 20 millimicrons are especially suitable for use according to processes of the present invention. Similarly suitable are the sols of the Rule Patents 2,577,484 and 2,577,485. These sols are composed of discrete silica particles in the diameter range of about 10 to 150 millimicrons; of these one may use to special advantage sols in which the particle size ranges from 10 to 20 millimicrons. The most preferred sols for use according to the present invention are silica sols in which the particle size range is from 5 to 20 millimicrons and which are otherwise like the sols of the Bechtold and Snyder and Rule patents. Such sols can be prepared by processes described in United States Patent 2,750,345, issued June 12, 1956, to Guy B. Alexander.

The silica sols just mentioned are alkali-stabilized, but the amount of alkali used is so small that the sols are not in any sense solutions of an alkali silicate. The stabilizing alkali can be an alkali metal hydroxide, such as sodium hydroxide, potassium hydroxide, lithium hydroxide, or it can be a nitrogen base such as ammonium hydroxide, or a quaternary ammonium base such as tetramethylammonium hydroxide. The mol ratio can be calculated as $\text{SiO}_2:\text{Na}_2\text{O}$ on the basis of stoichiometry even though the stabilizing base is actually some other material. The mol ratio will be in the range of 60:1 to 500:1.

A commercial silica sol made according to the Bechtold and Snyder patent above mentioned is particularly suitable for use in the compositions of this invention and is preferred. This sol has an $\text{SiO}_2:\text{Na}_2\text{O}$ ratio of about 95:1 and contains silica in the form of substantially discrete particles in the size range of about from 12 to 17. It ordinarily has a pH of about 9.5, but this can be lowered, sometimes to advantage, as by acidifying with acetic or hydrochloric acids.

The proportions of clay and silica in the novel treating compositions of this invention, expressed on the solids weight basis can vary from 99:1 to 1:19. Mixtures in which the ratio of clay to silica is about 4:1 have especially optimum properties and hence are particularly preferred.

The invention will be better understood by reference to the following illustrative examples.

EXAMPLE 1

A dispersion containing 1% solids by weight of a magnesium montmorillonite clay and having a pH of 9.5 was made up by shear mixing the requisite amount of dry clay with water. Samples (6" x 6") of white type 66-501 nylon carpet were dip-treated in this clay dispersion, wrung out to 65% wet retention, oven dried at 110° C. and equilibrated sixteen hours at 75° F. and 50% relative humidity. The treated samples were then soiled

with a bulked Sanders and Lambert synthetic soil and then vacuum cleaned. The reflectance of the samples was measured, before and after soiling and after vacuuming, by means of a Photovolt reflectometer model 610 using a Tristimulus green filter. For comparison, a control was run using water instead of the clay dispersion.

The results of this treatment are shown in Table 1.

Table 1

Carpet sample treatment (1% solids dispersions)	Reflectance			Percent anti-soil after vacuuming ¹
	Before soiling	After soiling	After vacuuming	
Control-water	66.9	25.9	42.5	39.4
Clay, pH 9.5	67.0	33.9	52.2	

$$^1 \text{Antisoil ratings were made as follows: percent anti-soil} \\ \frac{\Delta R_c - \Delta R_t}{\Delta R_c} (100) = \frac{(66.9 - 42.5) - (67.0 - 52.2)}{66.9 - 42.5} (100) = \frac{24.4 - 14.8}{24.4} (100) = 39.4$$

where ΔR_c is the reflectance decrease, measured on the control carpet sample and ΔR_t is the reflectance decrease measured on the treated carpet sample. It was noted that the treated samples had definite resistance to the development of static.

EXAMPLE 2

This example demonstrates the superior results which are obtained using the magnesium montmorillonite clay-colloidal silica compositions of the present invention for treating nylon carpeting. The same type of carpeting as used in Example 1 was employed but the samples were 16 x 16 inches in this example. The treating baths were prepared by shear mixing the clay with water at 4% solids, diluting to 1% solids with normal rate agitation, and then blending with 1% SiO_2 sol which had been adjusted to pH 5 with acetic acid. The final blend pH was about 8.

Two sets of carpet samples were treated in 1% aqueous dispersions of the blends listed in Table 2. After treatment and drying, the 16 x 16 inch samples were evaluated for static characteristics. Additional 6 x 12 inch samples were prepared and used for soil testing. The soil test samples were cut in half; one half of each sample was soil tested as treated, and the other half was laundered in 0.1% aqueous solution of a sodium alkyl sulfate wetting agent, and then rinsed thoroughly and tumble-dried prior to soil testing. All treated and dried samples were allowed to equilibrate for sixteen hours at 75° F. and 50% relative humidity prior to soil testing.

Results are given in Table 2.

Table 2
TREATED AND SOILED

Treating agents ¹		Reflectance			Percent anti-soil ²	Static value ³
Percent clay	Percent silica sol	Before soiling	After soiling	After vacuuming		
100	0	70	38.6	55.9	14.1	-12
80	20	68.3	42.4	56.9	30.0	-5
50	50	66.9	42.3	55.1	27.6	+8
20	80	68.9	43.5	58.9	38.6	+13
10	90	67.0	45.7	58.5	47.3	+13
0	100	67.5	44.1	59.2	49.1	+17
Water	-----	67.1	24.9	50.8	-----	+37

TREATED, LAUNDERED AND SOILED

100	0	63.6	28.2	51.2	16.8	-29
80	20	63.2	31.0	51.5	21.5	-29
50	50	62.6	32.6	51.3	24.2	-18
20	80	63.8	30.8	51.6	18.1	+4
10	90	63.3	30.2	50.5	14.1	+27
0	100	63.8	26.9	49.3	2.7	+39
Water	-----	63.6	26.2	48.7	-----	+48

¹ 1% total solids; components expressed on solids basis.

² Anti-soiling rated as in Example 1.

³ 0 to 20, no spark discharge; 20 to 25 borderline; greater than 25 unsatisfactory.

The static value is determined from deflection on indicating galvanometer. Test consists of conditioning car-

pet specimen for at least 12 hours at 72° F. and 20% relative humidity, generating a static charge on the carpet surface by an observer wearing dry, leather-soled shoes shuffling one foot while standing erect on the carpet sample through a series of 10 brisk strokes, and then touching the sensory probe of the galvanometer to transfer any static charge which has developed. Tabulated values are a measure of relative static charge and are further qualified by the following observations: 0 to 20=no spark discharge perceptible to observer; 20 to 25=spark discharge is weak but perceptible; greater than 25=spark discharge and shock sensation is readily detectible by feel, sight, and sound.

It will be noted that the combination of clay dispersion and silica sol had a synergistic effect in that after laundering better soil resistance was obtained for blends containing 50% or more of clay than for either clay alone or silica sol alone.

We claim:

1. A composition for decreasing the tendency of synthetic fiber yarns to develop static electricity and at the same time increasing their soil resistance, in which composition the antistatic and the soil resistant components consist essentially of an aqueous suspension of a particulate magnesium montmorillonite clay and an alkali-stabilized colloidal silica sol in which the silica particles are substantially discrete and in the size range of about 3 to 150 millimicrons and the SiO₂:alkali oxide mol ratio is in the range of about 60:1 to 500:1, the weight ratio of clay to silica being from 99:1 to 1:19.

2. A composition according to claim 1 in which the proportion of clay to silica is about 4:1 by weight.

3. A composition for decreasing the tendency of synthetic fiber yarns to develop static electricity and at the same time increasing their soil resistance, in which composition the antistatic and the soil resistant components consist essentially of an aqueous suspension of a shear-dispersed magnesium montmorillonite clay and an alkali-stabilized colloidal silica sol in which the silica particles are substantially discrete and in the size range of about 5 to 20 millimicrons and the SiO₂:alkali oxide mol ratio is in the range of about 60:1 to 500:1, the proportion of

clay to silica being about 4:1 by weight, said composition having a pH in the range of about 5 to 9.

4. In a process for decreasing the tendency of synthetic fiber yarns to develop static electricity and at the same time increasing their soil resistance, the steps comprising coating the yarn with a composition in which the antistatic and the soil resistant components consist essentially of a magnesium montmorillonite clay dispersed in an aqueous medium under shear and an alkali-stabilized colloidal silica sol in which the silica particles are substantially discrete and in the size range of about 3 to 150 millimicrons and the SiO₂:alkali oxide mol ratio is in the range of about 60:1 to 500:1, the weight ratio of clay to silica being from 99:1 to 1:19, and drying the coated yarn.

5. A process according to claim 4 in which the proportion of clay to silica in said coating composition is about 4:1 by weight.

6. In a process for decreasing the tendency of nylon yarn to develop static electricity and at the same time increase its soil resistance, the steps comprising coating the yarn with a composition in which the antistatic and the soil resistant components consist essentially of a magnesium montmorillonite clay dispersed in an aqueous medium under shear and an alkali-stabilized colloidal silica sol in which the silica particles are substantially discrete and in the size range of about 3 to 150 millimicrons and the SiO₂:alkali oxide mol ratio is in the range of about 60:1 to 500:1, the weight ratio of clay to silica being from 99:1 to 1:19, and drying the coated yarn.

7. A process according to claim 6 in which the proportion of clay to silica in said coating composition is about 4:1 by weight.

References Cited in the file of this patent

UNITED STATES PATENTS

2,574,902	Bechtold et al. _____	Nov. 13, 1951
2,584,337	Famulerer et al. _____	Feb. 5, 1952
2,622,307	Cogovan et al. _____	Dec. 23, 1952
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