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TETRASILICIC ESTERS AND THEIR USE FOR TEXTILE TREATMENT

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ABSTRACT OF THE DISCLOSURE

A new tetrasilicic ester of at least one unsaturated long-chain fatty alcohol or mixture of fatty alcohols which has an iodine number of at least 40, and a method of treating textiles in which the textiles are impregnated with the new tetrasilicic ester, preferably in the form of an aqueous emulsion, and if necessary then dried.

It is known that textile materials may be treated with tetrasilicic esters of saturated long-chain fatty alcohols. Such a treatment may serve for example to make the material being treated water-repellent or to improve the resistance to abrasion of crease-resist finished material. These prior art agents have a number of disadvantages. When they have been derived from linear alcohols, they are hard and rigid at room temperature and impart an unpleasant harsh handle to the textile material. When they have been derived from branched alcohols, although they are usually liquid at room temperature they have a very unpleasant odor which adheres firmly to the material treated therewith.

It is an object of this invention to provide an improved method of treating textiles with tetrasilicic esters which not only improves the abrasion resistance, tear resistance and sewing properties of the treated textiles but also imparts a pleasant soft handle thereto.

Another object of the invention is to provide an improved process for treating textiles with tetrasilicic esters which does not impart any unpleasant odor to the textiles.

It is moreover an object of the invention to provide aqueous emulsions of tetrasilicic esters which can be used as textile treating agents and are distinguished by particularly good stability in storage.

Finally it is an object of this invention to provide new tetrasilicic esters having valuable properties, which have a liquid to pasty consistency at room temperature and are free from unpleasant odor.

These and other objects are achieved by new tetrasilicic esters which are derived from at least one long-chain unsaturated fatty alcohol or a mixture having an iodine number of at least 40 of at least one long-chain unsaturated fatty alcohol and at least one long-chain saturated fatty alcohol.

The new tetrasilicic esters are eminently suitable as textile treating agents.

In the present specification, "long-chain" means containing at least twelve carbon atoms; tetrasilicic esters of alcohols of the abovementioned type which contain twelve to twenty carbon atoms have proved to be very good;

esters of alcohols containing fourteen to eighteen carbon atoms are preferred.

Examples of alcohols from which tetrasilicic esters in accordance with this invention are derived are n-hexadecenyl alcohol, oleyl alcohol, elaidyl alcohol, eicosenyl alcohol, linoleyl alcohol and linolenyl alcohol. Alcohols with single unsaturation are preferred. The tetrasilicic esters may be prepared from individual fatty alcohols or from mixtures of long-chain unsaturated fatty alcohols with one another or with long-chain saturated fatty alcohols, the content of unsaturated alcohol in the mixture in the latter case being at least so large that the mixture has at least the iodine number given above. For example alcohol mixtures having an iodine number of from 40 to 140 have proved to be very good; mixtures having an iodine number of 40 to 90 are preferred. Examples of saturated long-chain fatty alcohols which may be used in the alcohol mixtures are lauryl alcohol, n-tridecyl alcohol, myristyl alcohol, cetyl alcohol, n-heptadecyl alcohol, stearyl alcohol and arachidyl alcohol.

The tetrasilicic esters may be prepared by conventional methods, for example by reaction of silicon tetrachloride with the alcohols or alcohol mixtures at elevated temperature, if desired in indifferent solvents.

Tetrasilicic esters which contain only so small an amount of unreacted fatty alcohols that they have a hydroxyl number of not more than 5 are particularly advantageous for the purposes of the present invention.

The new tetrasilicic esters may be applied to the material being treated by various methods, for example in the form of their solutions in organic solvents, such as benzene, carbon tetrachloride, kerosine or other petroleum fractions. For reasons of economy and to minimize the risk of fire however it is preferred to use aqueous emulsions. Those esters which have a hydroxyl number of not more than 5 are particularly suitable for this type of application because their concentrated stock emulsions (unlike those of esters having higher hydroxyl numbers) do not tend to become thick and therefore unusable after prolonged storage. The concentrated stock emulsions may contain the tetrasilicic esters in amounts of up to 50% by weight or more. Stock emulsions containing about 40% by weight of tetrasilicic esters have proved to be very suitable.

Examples of emulsifiers which the emulsions may contain are anionic surfactants, particularly alkylsulfonates such as sodium octadecanesulfonate, or alkylarylsulfonates, such as sodium dibutylnaphthalenesulfonate and diethanolamine hexadecylbenzenesulfonate, or nonionic surfactants, particularly oxyalkylation products, such as the adducts of 2 moles of ethylene oxide to 1 mole of coconut fatty alcohol, of 6 moles of ethylene oxide to 1 mole of octadecyl alcohol, of 6 moles of ethylene oxide to 1 mole of stearic acid and of 40 moles of ethylene oxide to 1 mole of castor oil. Emulsions which contain, as emulsifiers, adducts of at least 20 moles, preferably 20 to 50 moles, of ethylene oxide to 1 mole of fatty alcohols containing twelve to twenty carbon atoms, such as sperm oil alcohol, octadecyl alcohol and oleyl alcohol or preferably alkali metal salts of sulfuric acid hemiesters of these adducts have proved to be particularly suitable. Emulsions of tetrasilicic esters which remain stable for several weeks can be prepared by means of these preferred emulsifiers. The emulsifiers are used in conventional amounts, i.e. in general in amounts of 2 to 6% on the silicic ester.

The stability of the silicic acid emulsions may be further increased by other additives. Thus it has been found that emulsions which contain water-miscible, low molecular weight organic solvents, such as acetone, methyl alcohol, ethyl alcohol, propyl alcohol, isopropyl alcohol, tert.-butyl alcohol or mixtures of the same as additives to increase stability are particularly suitable. The solvents not only improve the stability of the emulsions in storage so that they remain unchanged for months, but they also prevent foam formation in use and increase frost stability to such an extent that such emulsions may be frozen and thawed again without any change in structure. The organic solvents may be present in the emulsions for example in amounts of 5 to 20%, preferably 10 to 15%, with reference to the total weight of the emulsion; below 5% the effect declines markedly, whereas amounts of more than 20% in general do not add any other advantage. Another greatly recommended possibility for improving the stability of the emulsions consists in adding urea. Amounts of urea of 2 to 5%, with reference to the weight of silicic ester, are adequate. Urea may also be present in the emulsions together with the abovementioned solvents.

To prevent hydrolytic cleavage of the silicic ester it is advisable to be sure that the emulsions do not contain any free acid and if necessary to neutralize them with alkaline substances, such as alkali metal hydroxides or ammonia.

The solutions and emulsions of the new silicic esters may be used in the same way as prior art solutions and emulsions of silicic esters. For example the textile material to be treated may be impregnated with them alone by dipping or spraying, then squeezed in the usual way and dried at room or elevated temperature up to 150° C. There is also the possibility for example of applying the silicic ester emulsions together with agents which improve crease resistance to the material being treated and finishing off the material in the conventional manner.

It has also proved to be suitable to use the solutions or emulsions of the silicic esters in a concentration of 10 to 50 grams of ester per liter. With the conventional squeezing conditions used in practice, the amount of pure silicic ester applied is then about 1 to 5% with reference to the weight of the material being treated.

In these amounts they impart to the textile a pleasant soft handle and a considerable improvement in abrasion resistance, tear resistance and sewing properties, without the material acquiring an unpleasant odor.

Any textiles of natural or synthetic fibrous materials of all kinds may be treated with the new silicic esters, for example those of cotton, rayon staple, rayon, jute, linen, wool, cellulose acetate, linear polyamides and polyesters and acrylonitrile polymers.

The invention is illustrated by the following Examples; parts and percentages specified in the examples are by weight. Parts by weight bear the same relation to parts by volume as the kilogram to the liter.

EXAMPLE 1

400 parts of a tetrasilicic ester having a hydroxyl number of 3 (prepared from silicon tetrachloride and a fatty alcohol mixture (mainly C_{16} to C_{18}) having an iodine number of 40) is heated to 50° C. The silicic ester is allowed to flow into a solution (at 50° C.) of 464 parts of water, 110 parts of acetone, 10 parts of urea and 16 parts of the sodium salt of the sulfuric acid hemiester of an adduct of 20 moles of ethylene oxide to 1 mole of octadecyl alcohol while being emulsified with an impeller. The emulsion is homogenised three times at 150 atmospheres gauge in a homogenising machine and made neutral with a small amount of ammonia. The 40% emulsion, which remains stable and mobile for months, is diluted with water to a content of 25 g. of ester per liter. Rayon staple cloth is impregnated with this emulsion on a padding machine, squeezed to a 100% liquor pickup and dried at 110° C. on a tenter. The cloth has a very soft handle and is free from any unpleasant odor. Equally good results are achieved by using as the emulsifier an adduct of 25 moles of ethylene oxide to 1 mole of octadecyl alcohol.

EXAMPLE 2

400 parts of a tetrasilicic ester having a hydroxyl number of 2 which is liquid at room temperature (prepared from silicon tetrachloride and a fatty alcohol mixture (C_{16}–C_{18}) having an iodine number of 80) is allowed to flow while being emulsified with an impeller at room temperature into a solution of 444 parts of water, 130 parts of isopropanol, 10 parts of urea and 16 parts of the sodium salt of the sulfuric acid hemiester of an adduct of 25 moles of ethylene oxide to 1 mole of sperm oil alcohol. The pre-emulsion is then homogenised three times at 200 atmospheres gauge in a homogenising machine and made neutral with a small amount of ammonia. It may be kept unchanged for several months.

75 parts of this emulsion, 50 parts of N,N'-dimethylol-4,5 - dihydroxyimidazolidone - 2, 40 parts of N,N'-dimethylolbutanediol-1,4-diurethane, 50 parts of N,N'-dimethoxymethylurea and 5 parts of monoammonium phosphate are dissolved in water to form 1000 parts of finishing liquor. Cotton cloth is impregnated with this liquor, squeezed to 100% liquor pickup, dried to 8% residual moisture content and heated for three minutes at 145° C.

The cloth has a permanent crease resist finish; it has a very soft handle, high abrasion resistance, high tear resistance and good sewing properties. It is also free from unpleasant odor.

EXAMPLE 3

50 parts of the tetrasilicic ester specified in Example 2 and 3 parts of sodium octadecanesulfonate are dispersed in 948 parts of water with an impeller. To destroy the foam formed, 0.25 part of triisobutyl phosphate is stirred into the emulsion. The emulsion obtained is used to impregnate a cotton cloth which is squeezed to 100% liquor pickup and dried on a tenter at 110° C. The cloth has a very soft handle and is free from unpleasant odor.

EXAMPLE 4

25 parts of the tetrasilicic ester specified in Example 1 is dissolved in 1000 parts by volume of trichloroethylene. Cotton cloth is impregnated with this solution, squeezed and dried. The fabric has a fine silky handle without having acquired an unpleasant odor.

EXAMPLE 5

400 parts of the tetrasilicic ester of sperm oil alcohol is allowed to flow, while being emulsified with an impeller, into a solution of 454 parts of water, 120 parts of ethanol, 10 parts of urea and 16 parts of an adduct of 30 moles of ethylene oxide to 1 mole of oleyl alcohol. The emulsion formed is homogenised gauge three times at 200 atmospheres in a homogenising machine. It is stable for several months.

The sperm oil alcohol used for the preparation of the ester is a mixture of monounsaturated fatty alcohols having twelve, fourteen, sixteen, eighteen and twenty carbon atoms and saturated fatty alcohols having twelve, fourteen, sixteen and eighteen carbon atoms in such a ratio that the iodine number is between 80 and 90.

A very similar emulsion is obtained by using the same amount of the tetrasilicic ester of oleyl alcohol instead of the sperm oil alcohol ester.

Both emulsions may be used as described in Example 1 with the results given therein.

We claim:

1. A tetrasilicic ester of a mixture of saturated and unsaturated fatty alcohols having twelve to twenty carbon

atoms, said mixture having an iodine number of 40 to 90.

2. A tetrasilicic ester as claimed in claim 1, wherein said alcohols are sperm oil alcohols comprising a mixture of monounsaturated fatty alcohols having twelve, fourteen, sixteen, eighteen and twenty carbon atoms and saturated fatty alcohols having twelve, fourteen, sixteen and eighteen carbon atoms in such a ratio that the iodine number is between 80 and 90.

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