United States Patent

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[33] **Japan**
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[54] **WATER REPELLENT**
6 Claims, No Drawings

[52] **U.S. Cl.** **106/2,** 106/243, 117/135.5, 252/8.8, 260/404.5
[51] **Int. Cl.** **C09k 3/18**
[50] **Field of Search** 106/2, 243; 260/404.5; 252/8.8, 390; 117/135.5

[56] **References Cited**

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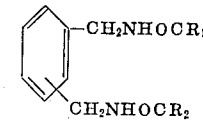
ABSTRACT: A water repellent containing as its active ingredient a compound represented by the general formula



wherein R_1 and R_2 are each selected from the group consisting of alkyl and alkenyl groups having from seven to 21 carbon atoms. One substituent is in either the meta or the paraposition with respect to each other. A process for imparting water repellent properties to a fiber is also disclosed which comprises applying the repellent material to the fiber, drying and heat treating the fiber.

WATER REPELLENT

BACKGROUND OF THE INVENTION

1. Field of Invention

The present invention relates to a water repellent and more particularly to a water repellent to be applied to textile fabrics. Further, this invention relates to a finishing process for water repelling by the use of said water repellent.

2. Discussion of Prior Art

Water repellents heretofore used for fiber products include silicone resins such as alkyl hydrosiloxane, alkyl siloxane, etc., fluorine-containing resins, pyridine derivatives, paraffins, and salts of aluminum, titanium, zirconium, and the like. These water repellents are absorbed into the fiber products and heat-treated so as to impart excellent water repellency to the finished products.

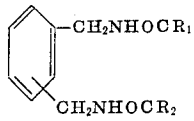
However, such conventional water repellents are limited in use because of their high cost, the excessively high temperatures required for heat-treatment, the need for a catalyst to effect water repellency and their lack of storage stability. Thus, these water repellents cannot be applied to every fiber product.

An object of this invention therefore is to provide a novel water repellent which is inexpensive and which has superior washing-durability.

Another object of this invention is to provide an efficient method for imparting waterH-repellency whereby the heat-treatment temperature required is comparatively lowered.

SUMMARY OF THE INVENTION

These objects have now been accomplished by the use of the following compound as a water repellent



wherein R_1 and R_2 are each alkyl or alkenyl groups having from seven to 21 carbon numbers, inclusive, and wherein one substituent is in either the meta or the paraposition to the other.

A process for treating a fiber with the water repellent comprising applying the water repellent material to the fiber, drying the treated fiber and heat treating the fiber. The heat treating temperature generally is from about 130° C. to about 200° C., with the preferred temperature range being from about 130° C. to about 150° C. Generally from about 3–4 minutes will be a sufficient heat treatment time at the above temperatures.

DETAILED DESCRIPTION OF THE INVENTION

The compound described in this invention can be obtained in accordance with conventional processes. For example, by the reaction of at least one aromatic diamine selected from meta-xylylene diamine and para-xylylene diamine, with a higher fatty acid chloride, a free higher fatty acid or an ester of a higher fatty acid. From the viewpoint of simplicity and economy of materials, it is advantageous to produce the water repellent compound of this invention from xylylene diamines and free fatty acids.

The fatty acids to be used include either saturated or unsaturated fatty acids having from eight to 22 carbon atoms. These fatty acids are usually obtained by the hydrolysis of mixtures of natural fats and oils and such mixtures may be directly used as the material for the synthesis. It is preferred, however, to use the fatty acid, when it contains a large proportion of unsaturated bonds, after it has been partially or completely hydrogenated, in order to obtain improved stability and water repellent properties for the above-mentioned compound.

The application of the compound of the present invention to fiber products may be effected by the use of a solution, but it is usually applied in the form of a suspension, in which the compound is dispersed in the amounts of from 0.1–3% by weight, preferably 0.5–2% by weight, in a suitable dispersion medium. The resultant suspension is then applied to the fiber products to be finished in a conventional manner, such as by dipping, brushing, spraying, and the like, to give water repellent fiber products. The proportion of the water repellent compound to the dispersion medium may be greater than 3% by weight, but further increases in the proportion will not generally impart significantly greater improvements in the water repelling properties. Similarly, the use of less than 0.1% by weight of the compound will generally give insufficient water repelling action.

Excellent washing-durability for the water repelling finish can be attained by a procedure which comprises dipping the fiber products in said suspension, drying by means of hot air or the like, and heat-treating at 130°–150° C. for 3–4 minutes. Although appreciable water repellency is obtainable by this treatment at temperatures below 130° C., it is preferred to conduct the heat-treatment at a temperature of about 130° C. to impart greater water repellency and washing durability.

The heat-treatment may be also carried out at temperatures above 150° C., to as high as 200° C., so long as the fiber products to be finished can endure the higher temperatures.

Similarly, the period of the heat-treatment may be shorter than 3 minutes to obtain the desired effect, but it is generally preferred to perform the treatment for 3–4 minutes. Heat-treatment may also be carried out for a period over 4 minutes, but this is generally unnecessary, since satisfactory results are obtainable within a period of 3–4 minutes.

The water repellent compounds to be used in this invention are only slightly soluble in almost all solvents at normal temperatures, so that purification is difficult. However, the water-repellent compounds of this invention can be used in their raw reaction form without any particular purification to impart general water repellent properties.

When purified products are needed, however, purification can be effected by a procedure which comprises dissolving the compound in a suitable solvent such as alcohol at high temperatures; discoloring, if necessary, with a suitable discolorant; and then cooling the solution so as to crystallize out pure material.

Suitable dispersion media which may be used in the practice of this invention include organic solvents having a boiling point below 150° C. For example, a lower alcohol with less than five carbon atoms, an acetic acid ester, benzene, toluene, cyclohexane and similar hydrocarbons, acetone, methyl ethyl ketone and similar ketones, dioxane and the like. In addition, aqueous media containing a hydrophilic organic solvent or a surface active agent may be used.

For convenience in transportation and storage, it is preferred that the liquid suspension of the present invention be first prepared as a highly concentrated liquid, for example, from about 30–50% by weight. Diluted to the desired concentration may be accomplished immediately before use.

The water repellent of the present invention may be used as a mixture, in any desired proportion, of meta-substituted and/or parasubstituted compounds.

The fiber products to be treated with the water repellent of this invention include a variety of synthetic and natural fibers. Superior effects are obtained when it is applied to synthetic fibers such as polyesters, Nylon, polyacrylonitrile, polyvinyl alcohol and the like.

Excellent water repellency can also be imparted when the water repellent of this invention is applied to cotton, wool, rayon and acetate fibers.

The water repellent compound of this invention can also give improved softness, as well as water repellency.

The invention is illustrated in further detail by the following examples in which "part" expresses parts by weight.

EXAMPLE 1

A reactor fitted with a stirrer was charged with 284 parts of purified stearic acid and 68 parts of meta-xylylene diamine was added thereto. After the addition was completed, the reaction temperature was raised to 160° C., and the reaction

was carried out for 3 hours while removing water formed by condensation. The reaction was continued for another hour after raising the temperature to 200° C.

The compound so obtained, namely, meta-xylylene diamine disteramide, was then suspended in 99 parts by weight of isopropyl alcohol using a homogenizer.

Cut pieces of cloth, 20 cm. square, made of various fiber products, were immersed for several minutes in the above suspension, then squeezed with a mangle, dried by the use of a hot-air dryer at 100° C. and finally individual groups were heat-treated at 130° C. and 150° C. for 4 minutes each. The cloths so treated were then used as test samples. The water repellency test was conducted according to standard spray testing techniques. The results are shown below.

	Heat Treatment Temp. °C.			
Fiber	Hot-air Drying	130° C.	150° C.	Not treated
Polyester	90° C.	100	100	0
Nylon	90° C.	100	100	0
Polyacrylonitrile	90° C.	100		0
Cotton	80°–90° C.	100	100	0

EXAMPLE 2

The same procedure used in example 1 was carried out with the exception that para-xylylene diamine was substituted for meta-xylylene diamine. The para-xylylene diamine distearamide so obtained was suspended in benzene at concentrations of 1% and 2% by the use of a homogenizer to form two suspensions.

Into the resultant suspensions, various cloth samples were immersed for 1 minute, dried by means of a hot-air dryer at 80° C. and the groups were heat-treated at 130° C., 140° C., and 150° C., respectively, for 4 minutes each. The results are shown below.

	Heat-treatment temperature—					
	130° C.		140° C.		150° C.	
	Suspension concentrate		Suspension concentrate		Suspension concentrate	
Fiber	1%	2%	1%	2%	1%	2%
Polyester	100	100	100	100	100	100
Nylon	100	100	100	100	100	100
Polyacrylonitrile	90	100	100	100	100	100
Cotton	100	100	100	100	100	100
Wool	90	100	90	100	100	100

EXAMPLE 3

Mixed xylylene diamine palmitic amide was synthesized by reacting palmitic acid (Neutralization value = 216, Iodine = 1) with a mixed amine comprising 95% by weight of meta-xylylene diamine and 5% by weight of para-xylylene diamine. This amide was suspended in 50% methanol-water at a concentration of 3% by weight.

Various cloth samples were immersed in this suspension, dried, and heat-treated at 140° C. for 3 minutes. The results are shown below.

	Heat Treatment Temperature		
Cloth	Naturally dried	140° C.	Not treated
Polyester	80	100	0
Nylon	80	100	0
Cotton	70	100	0
Wool	90	100	50

EXAMPLE 4

A fatty acid amide was prepared by the reaction of beef tallow fatty acid (a mixture of saturated and unsaturated fatty acids containing from 14–18 carbon atoms) with a mixed diamine comprising 50% by weight of meta-xylylene diamine and 50% by weight of para-xylylene diamide. The resultant diamine was suspended in benzene to prepare a 0.5% by weight suspension thereof.

Various cloth samples were immersed in the suspension, dried by means of a hot-air dryer and heat-treated at temperatures of 130° C., 140° C., and 150° C., respectively, for 3 minutes. The results are shown below.

	Heat Treatment Temperature		
Fiber	130° C.	140° C.	150° C.
Polyester	100	100	100
Nylon	100	100	100
Cotton	90	100	100

EXAMPLE 5

Stearamide was prepared from industrial grade stearic acid and meta-xylylene diamine, and suspended in 5% by weight of isopropyl alcohol-water to prepare aqueous suspensions of various concentrations.

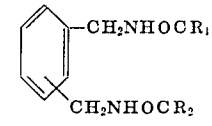
Polyester fiber cloth was immersed in the resultant suspension for 1 minute, dried by means of hot-air and then heat-treated at 150° C. for 3 minutes. The results are shown below.

	Concentrations			
Not treated	0.1%	0.5%	1%	3%
Not treated	70–80	100	100	100

While the invention has been described with regard to particular materials and means, it will be obvious to one skilled in the art that various changes can be made within the scope of the invention, and is limited only by the scope of the following claims.

What is claimed is:

1. A water repellent composition containing in an inert liquid medium an amide as the active ingredient, said amide being represented by the formula:



wherein R_1 and R_2 are each selected from the group consisting of an alkyl radical, said alkyl radical having from seven to 21 carbon atoms, and an alkenyl radical, having from seven to 21 carbon atoms and being selected from the group consisting of an alkenyl radical containing one internally-positioned double bond and an alkenyl radical containing two internally-positioned double bonds, said substituents containing R_1 and R_2 on said benzene ring being in a meta or pararelationship.

2. The water repellent of claim 1 wherein R_1 and R_2 are each a linear alkyl radical containing 17 carbon atoms.

3. The water repellent of claim 1 wherein R_1 and R_2 are each a linear alkyl radical containing 15 carbon atoms.

4. The water repellent of claim 1 wherein R_1 and R_2 each contain from 13 to 17 carbon atoms and wherein each is derived from beef tallow fatty acids.

5. The water repellent of claim 1 wherein R_1 and R_2 are in the meta position with respect to each other.

6. The water repellent of claim 1 wherein R_1 and R_2 are in the paraposition with respect to each other.

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