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2,876,140

SOIL RESISTANT TEXTILE MATERIAL AND METHOD OF MAKING THE SAME

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This invention relates to textile materials having improved soil resistant properties and process for making same.

Various methods have been employed or suggested for increasing resistance to soiling of woven fabrics, such as clothing, drapery and upholstery materials, and the like. These treatments generally take the form of filling the interstices of the fabric with resinous compounds or of applying a thick, smooth surface to the fabric. For the most part, these procedures suffer from various disadvantages such as causing matting of the fibers, adversely affecting the feel of the fabric and tenaciously retaining soil during laundering operations. The treatments have been of no value for use with pile or loop fabrics such as rugs where matting of the fibers cannot be tolerated.

The object of this invention is to provide a process for treating textile materials which markedly increases resistance to soiling without adversely affecting launderability or the physical characteristics of the fibers or fabric.

Another object is to provide a process for increasing soil resistance which is particularly advantageous for use with pile or loop fabrics such as rugs and carpeting.

Other objects and advantages of the invention will become obvious from the following detailed description.

My process is particularly suitable for use with textile materials comprising vegetable and/or synthetic fibers, such as cotton, viscose and acetate rayons, nylon and the like, which are particularly susceptible to soiling and which hold the soil tenaciously so that normal home cleaning treatments such as vacuuming are ineffective in removing the dirt. My treatment can also be efficaciously applied to wool although wool possesses considerable natural soil-resistant properties as compared with vegetable and synthetic fibers. It is especially advantageous for application to wool blends with vegetable and/or synthetic fibers. The treatment can be applied to the raw stock fiber, yarn or finished goods such as rugs, clothing, drapery and upholstery material.

Broadly speaking my process comprises impregnating the fiber, yarn or fabric with barium sulfate formed in situ, a cationic softening agent and carboxymethyl cellulose.

I have found that BaSO₄, when formed in situ so that it is deposited within the interstices of the fiber and fabric, imparts some soil resistance to the material. However, this advantage is off-set by the fact that the soil which does attach itself to the material after such treatment is held very tenaciously, is very resistant to vacuuming and can be removed only to a very limited extent by laundering. I have discovered that the intro-

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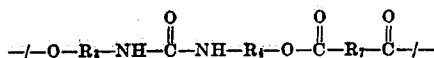
duction of a cationic softening agent and carboxymethyl cellulose along with the BaSO₄, greatly increases soil resistance and improves launderability to the point where it is substantially unimpaired. The treatment, furthermore, improves the luster of the material and does not adversely affect feel or texture. There is no matting or sticking of the fibers so that the treatment is particularly suitable for use with loop or pile fabrics such as rugs.

As aforementioned, the BaSO₄ should be formed in situ. This can be accomplished by separately impregnating the material with a solution of a soluble barium salt, such as the chloride or acetate, and a solution of a soluble sulfate such as sodium, potassium or magnesium sulfate to precipitate BaSO₄ in very finely divided form directly into the interstices and crannies of the fibers, yarn or fabric. The salt solutions can be applied in any desired sequence. Preferably the material is dried prior to impregnation with the second solution. For satisfactory performance, it is generally desirable to incorporate at least about 2% and, preferably, about 3% of BaSO₄ based on the weight of the material. There is no critical upper limit of BaSO₄ added, although at amounts above about 10%, the increase in soil-resistance may generally not be sufficiently appreciable to warrant the increased cost.

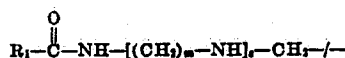
The impregnating solutions employed to deposit the BaSO₄ can be of any concentration up to the limit of solubility of the reacting salt so long as the amount of salt is adequate for the desired pick-up and the amount of solution is sufficient for thorough impregnation of the material. It is generally desirable to employ a somewhat higher concentration of salt in the second solution to ensure as complete reaction as possible.

Impregnation of the material can be carried out at any desired temperature as, for example, room temperature, although, somewhat elevated temperatures, e. g. about 100 to 200° F., facilitate more rapid penetration of the material.

I have found that softening agents of the higher fatty acid amide type which are cationic at the time of addition or become cationic during processing, namely as a result of the acidification subsequently to be discussed, are particularly suitable for my purpose. A number of such textile assistants are known to the textile art. Especially satisfactory in my process are the reaction products of polybasic organic acids with dialkylol substituted carbamido compounds carrying side chains containing polyamino acid radicals and their salts. Such condensation products are disclosed in Morgan et al. patent U. S. 2,410,788 and are available commercially as, for example, Ahcovel R. They are characterized by one hydroxyl and one carboxyl end group and repeating units of



wherein R₁ is a hydrocarbon residue resulting from a polycarboxylic acid, R₂ and R₃ are simple alkylene groups of from 2 to 3 carbons. Attached to one of the carbons in R₂ and R₃, is an acid amide side chain of the general formula:



wherein R is hydrocarbon, e is an integer of from 0

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to 4 and m is an integer of from 2 to 3. To increase water solubility or dispersibility these compounds can be treated with an acid to form an acid salt or they can be alkylated. Formation of the acid salt also converts compounds of this type which are not already cationic, such as Ahcovel R, into the cationic state.

Other fatty acid amide cationic softeners which I have also found satisfactory include ester-amides, which are condensation products of a dihydroxy-alkyl amine and a higher aliphatic acid. Such products are available commercially as, for example, Finish 100-X.

For satisfactory results the cationic softening agent should be incorporated in an amount comprising at least about 0.1%, preferably about 0.25%, based on the weight of the material. Optimum range is about 0.25 to 1.0%. As much more can be introduced as desired but, generally speaking, at amounts above about 2 to 3% there is little advantage to be gained to warrant the increased cost.

The carboxymethyl cellulose, which I prefer to employ in the form of its alkali metal salt such as the sodium salt, is desirably added in an amount comprising at least about 0.1% of the material, optimum range being about 0.25 to 2.0%. There is no critical upper limit but, in most cases, there is little economic advantage with additions above about 3 to 5%.

The cationic softening agent and the carboxymethyl cellulose can be introduced into the material, e. g., fibers, yarn or fabric, by exhaustion from an aqueous solution after impregnation of the material with the BaSO_4 or can be added along with the second reaction solution employed for forming the BaSO_4 in situ. I prefer to use the latter procedure because of its convenience in eliminating an additional processing step.

Concentration of the softening agent and carboxymethyl cellulose in the aqueous treating bath is not critical. It is essential only to have sufficient present in the aqueous solution to obtain the desired degree of exhaustion on to the material. The treating bath may be maintained at ordinary or somewhat elevated temperatures. At temperatures of about 100 to 200° F., the material is more easily and rapidly penetrated by the solution so that the use of such elevated temperatures is a practical means for expediting processing.

Exhaustion of a fatty acid amide softening agent and the carboxymethyl cellulose from aqueous solution is best accomplished in an acid medium produced by the addition of an organic acid, such as acetic acid, in sufficient amount to lower the pH to about 6.5 or less. Where the carboxymethyl cellulose is added to the treating bath in the form of its sodium salt, it is exhausted onto the fibers as the parent compound with the sodium remaining in solution as a salt of the acidifying organic acid.

I have found that more complete exhaustion of the softening agent and carboxymethyl cellulose from the treating bath and more uniform distribution on the material are obtained when the pH is first reduced gradually by means of an acid salt, such as magnesium sulfate, aluminum sulfate or the like, in sufficient amount to bring the pH down to about 7.0 or less, preferably about 6.5, and then further reduced by addition of sufficient organic acid, such as acetic acid, to bring it to the desired level of 6.5 or less, preferably about 6.0 to 6.5. Although I prefer to use acetic acid because of its relatively low cost, other organic acids, such as propionic acid, succinic acid and the like can also be used.

Where the softening agent and the carboxymethyl cellulose are introduced in the bath containing the second reaction component for formation of the BaSO_4 in the material, it is advisable to agitate the material with the aqueous solution for several minutes to accomplish BaSO_4 formation in the material before addition of the acid salt to exhaust the softening agent and carboxymethyl cellulose. The gradual lowering of the pH by

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the acid salt to the desired level is generally accomplished within several minutes, e. g. about 5 minutes, and the organic acid can then be added in requisite amount to produce the desired acidity. The material can then be removed from the aqueous treating bath and dried.

The treatment of the material, e. g. fibers, yarn or fabric, can be carried out in any suitable device or machine, such as dyeing or laundry equipment, which provides sufficient agitation for thorough impregnation of the material with the treating solutions. Drying similarly can be performed with any suitable, standard equipment.

The soil-proofing treatment aforescribed can be carried out in whole or in part at any stage in the fabrication of the textile material. It can be applied to the raw stock fiber, to the yarn or to the finished fabric, e. g. woven, pile or loop fabric such as clothing, drapery or upholstery materials or rugs. The BaSO_4 or one component such as the barium or sulfate salt can be deposited at one stage, e. g. the fiber or yarn, and the rest of treatment completed on the finished fabric.

EXAMPLE I

A white cut pile rug made with viscose rayon yarn, comprising 8 denier, $1\frac{1}{16}$ inch fibers, on a cotton backing was treated in a laundry-type machine with a solution comprising 1 part barium chloride to 9 parts water at 110° F. for 15 minutes. The ratio by weight of barium chloride solution to material was 30:1. The material was extracted to a 100% wet pick-up and dried at a temperature of 200° F. The resulting barium chloride pick-up was 10% by weight.

The dried material was then treated with 30 parts by weight of an aqueous solution containing 15% by weight of the material of sodium sulfate, 2% by weight of the material of Ahcovel R, which comprises 30% by weight of the solid fatty amide polymer, and 2% by weight of the material of sodium carboxymethyl cellulose at 110° F. for 5 minutes. 2% magnesium sulfate by weight of the material was then added to the solution and treatment continued for 5 more minutes. 1% by weight of the material of 56% acetic acid was added to the bath and agitation of the material continued for 5 more minutes. The material was then extracted and dried. The final pH was about 6.2. Solids pick-up of the dried material was 9.2% barium sulfate, 0.5% Ahcovel R and 0.5% carboxymethyl cellulose.

EXAMPLE II

Raw stock viscose rayon fibers were treated at 120° F. for 20 minutes in a raw stock pressure dyeing machine with a 5% aqueous barium chloride solution. Weight of solution to fibers was 10:1. The treated fibers were extracted to an 80% wet pick-up and dried in a stock dryer. Barium chloride pick-up was 4% by weight of the fibers.

The fibers were then processed into yarn and made into cut pile rugs on a cotton backing. The rugs were placed in an agitation-type laundering machine and treated at 110° F. for 5 minutes with an aqueous solution containing 15% sodium sulfate by weight of the material and 2% sodium carboxymethyl cellulose by weight of the material. 2% magnesium sulfate by weight of the material was then added and treatment continued for 5 more minutes. 1% of 56% acetic acid by weight of the material was added and agitation continued 5 minutes longer. The material was then extracted and dried to give a solids pick-up of 3.78% barium sulfate, 0.5% Ahcovel R and 0.5% carboxymethyl cellulose. Final pH was about 6.2.

EXAMPLE III

A cut pile rug made of a nylon and rayon blend on a cotton backing was treated as in Example I except that the temperature of the treating solutions was maintained at 200° F. Solids pick-up was the same.

EXAMPLE IV

A cut pile rug made of a cotton and rayon blend on a cotton backing was treated with a 5% aqueous solution of barium chloride at a temperature of 160° F. for 15 minutes, extracted to 100% wet pick-up and dried at 200° F.

It was then placed in an agitation-type laundering machine and treated as in Example II except that treatment temperature was 160° F. Solids pick-up was 4.76% barium sulfate, 0.5% Ahcovel R and ½% carboxymethyl cellulose.

Soiling of the samples was accomplished by wrapping them around a screen mesh cylinder in a soiling drum, charging the drum with a weighed amount of the soiling composition and then revolving the drum for 30 minutes. The 30 minute exposure under these conditions is equivalent to about 8 months exposure in a large business office. After each 30 minute soiling cycle, the rugs were vacuum cleaned and light reflectance measured in terms of photovolt readings on a reflectometer.

The rugs were laundered in a tumbler type laundering machine using 2% olive soap flakes based on the weight of the fabric. They were washed for 15 minutes at 140° F., rinsed, extracted and dried.

Table I

Sample	Reflectance before soiling	Number soiling cycles	Reflectance after soiling	Reduction in reflectance, percent	Reflectance after laundering	Reduction in reflectance, percent
Untreated	92.5	2	49.5	46.5	92.7	0
BaSO ₄ + Ahcovel R + CMC	99.2	4	65.7	33.8	96.0	3.2
BaSO ₄ + Ahcovel R	99.0	4	64.6	34.8	81.1	18.1
BaSO ₄ + CMC	96.7	4	59.7	38.7	88.0	40.0
BaSO ₄	96.0	4	58.5	40.1	60.2	37.3

EXAMPLE V

Raw stock viscose rayon fibers were treated with a 10% solution of barium chloride in a raw stock pressure dyeing machine for 30 minutes at 140° F., extracted to 80% pick-up and dried, leaving 8% barium chloride deposited. The fibers were processed into yarn and sewn into cut pile rugs on a cotton backing. They were then treated with sodium sulfate, Ahcovel R and sodium carboxymethyl cellulose, magnesium sulfate and acetic acid in the same percentages and manner as described in Example II. Solids pick-up was 7.61% barium sulfate, 0.5% Ahcovel R and 0.5% carboxymethyl cellulose.

The materials treated according to my process exhibit excellent soil-resistant properties which are retained through numerous launderings and dry cleanings. Such soil as does accumulate after an extended period of time is readily and substantially completely removed by ordinary laundering or dry cleaning procedures. The treatment imparts excellent luster and improved texture and feel to the materials.

Table I summarizes characteristic results obtained with my treatment and demonstrates the great improvement in luster and soil resistance. The table also shows the marked superiority of the treatment comprising a combination of the barium sulfate, cationic fatty amide softening agent and carboxymethyl cellulose in terms of soil-resistance and launderability as compared with the use of barium sulfate alone or barium sulfate with either the softening agent or carboxymethyl cellulose.

After only two soiling cycles the untreated cut pile rug lost 46.5% of its reflectance whereas the rug subjected to the full treatment lost only 33.8% of its original improved reflectance after four soiling cycles. Where the rug treated with barium sulfate alone showed a reduction in reflectance of 37.3% after laundering, the fully treated rug showed a reduction of only 3.2% and the actual reflectance value after laundering was higher than that of the original unsoiled control.

The floor coverings treated in the series of tests given in Table I comprised white, cut pile rugs made of 8 denier, 1¹/₈ inch bright viscose rayon sewn on a white cotton backing. The proportions of treating agents introduced were as follows:

	Percent
BaSO ₄	10.0
Ahcovel R	0.3
Carboxymethyl cellulose	0.1

The various treating agents were introduced in a manner similar to those disclosed in the preceding examples.

Although this invention has been described with reference to illustrative embodiments thereof, it will be apparent to those skilled in the art that the principles of this invention may be embodied in other forms but within the scope of the appended claims.

I claim:

1. A method for imparting soil resistance to textile material which comprises separately impregnating the material with an aqueous solution of a water soluble barium compound and an aqueous solution of a water soluble sulfate, said solutions being applied in any desired sequence, whereby at least about 2% barium sulfate by weight of the material is deposited in the material, impregnating the material with a higher fatty acid amide softening agent in aqueous solution, said softening agent being selected from the group consisting of cationic compounds and potentially cationic compounds which form cationic acid salts upon acidification and sodium carboxymethyl cellulose in aqueous solution, exhausting the softening agent and carboxymethyl cellulose onto the material by adding an acid salt in sufficient amount to reduce the pH of the solution to about 6.5 to 7.0 and then adding an organic acid in sufficient amount to further reduce the pH to about 6.0 to 6.5, said softening agent being introduced into the material in an amount comprising at least about 0.1% of weight of the material and said carboxymethyl cellulose being introduced into the material in an amount comprising at least about 0.1% by weight of the material, and then drying the material.

2. The process of claim 1 in which the cationic softening agent is the reaction product of polybasic organic acid with dialkylol substituted carbamido compound carrying side chains containing a polyamino acid radical.

3. The process of claim 2 in which the textile material is a pile fabric containing fibers selected from the class consisting of vegetable and synthetic fibers.

4. The process of claim 1 in which the acid salt is magnesium sulfate and the organic acid is acetic acid.

5. The process of claim 4 in which the softening agent is introduced into the material in an amount comprising at least about 0.25% by weight of the material.

6. The process of claim 5 in which the barium sulfate is introduced in an amount comprising at least about 3% by weight of the material.

7. The process of claim 6 in which the textile material is a pile fabric containing fibers selected from the class consisting of vegetable and synthetic fibers.

8. A method for imparting soil resistance to textile ma-

terial which comprises separately impregnating the material with an aqueous solution of a water soluble barium salt and an aqueous solution of a water soluble sulfate, said solutions being applied in any desired sequence, the material being dried between application of said first and second solutions, whereby at least about 2% barium sulfate by weight of the material is deposited in the material, including in the second reacting solution a higher fatty acid amide softening agent selected from the group consisting of cationic compounds and potentially cationic compounds which form acid salts upon acidification and sodium carboxymethyl cellulose, adding to said second solution after precipitation of the barium sulfate an acid salt in amount sufficient to reduce the pH of the solution to about 6.5 to 7.0 and then adding to said second solution an organic acid in amount sufficient to further reduce the pH to about 6.0 to 6.5, the softening agent and carboxymethyl cellulose each being introduced into the material in an amount comprising at least about 0.1% by weight of the material and then drying the material.

9. The method of claim 8 in which the textile material contains fibers selected from the group consisting of vegetable and synthetic fibers and the softening agent is the reaction product of polybasic organic acid with dialkylol substituted carbamido compound carrying side chains containing a polyamino acid radical.

10. The method of claim 9 in which the softening agent is introduced into the material in an amount comprising at least about 0.25% by weight of the material and the barium sulfate in an amount comprising at least about 3% by weight of the material.

11. Textile material, characterized by improved soil resistant properties, impregnated with finely divided barium sulfate deposited in situ in amount at least about 2% by weight of the material, at least about 0.1% by weight of the material of a cationic softening agent and at least about 0.1% by weight of the material carboxymethyl cellulose.

12. The product of claim 11 in which the cationic softening agent is a higher fatty acid amide.

13. The product of claim 12 in which the textile material contains fibers selected from the class consisting of vegetable and synthetic fibers.

14. The product of claim 13 in which the textile material is a pile fabric.

15. The product of claim 11 in which the softening agent is the reaction product of polybasic organic acid with dialkylol substituted carbamido compound carrying side chains containing a polyamino acid radical and is present in an amount comprising at least about 0.25% by weight of the material.

16. The product of claim 15 in which the barium sulfate is present in an amount comprising at least about 3% by weight of the material.

17. A method for imparting soil resistance to textile material which comprises separately impregnating the material with an aqueous solution of a water soluble barium compound and an aqueous solution of a water soluble sulfate, said solutions being applied in any desired sequence, whereby at least about 2% barium sulfate by weight of the material is deposited in the material, impregnating the material with a higher fatty acid amide softening agent in aqueous solution, said softening agent being selected from the group consisting of cationic compounds and potentially cationic compounds which form cationic acid salts upon acidification and an alkali metal salt of carboxymethyl cellulose in aqueous solution, exhausting the softening agent and carboxymethyl cellulose onto the material by acidifying the solution, said softening agent and said carboxymethyl cellulose each being introduced into the material in an amount comprising at least about 0.1% by weight of the material and then drying the material.

18. A method for imparting soil resistance to textile material which comprises separately impregnating the

material with an aqueous solution of a water soluble barium compound and an aqueous solution of a water soluble sulfate, said solutions being applied in any desired sequence, the material being dried between application of said first and second solutions, whereby at least about 3% barium sulfate by weight of the material is deposited in the material, impregnating the material with a higher fatty acid amide softening agent in aqueous solution, said softening agent being selected from the group consisting of cationic compounds and potentially cationic compounds which form cationic acid salts upon acidification and an alkali metal salt of carboxymethyl cellulose in aqueous solution, exhausting the softening agent and carboxymethyl cellulose onto the material by gradually acidifying the solution first by addition of an acid salt and then by addition of an organic acid, said softening agent being introduced into the material in an amount comprising at least about 0.25% by weight of the material and said carboxymethyl cellulose being introduced into the material in an amount comprising at least about 0.1% by weight of the material and then drying the material.

19. A method for imparting soil resistance to textile material which comprises separately impregnating the material with an aqueous solution of a water soluble barium compound and an aqueous solution of a water soluble sulfate, said solutions being applied in any desired sequence, whereby at least about 2% barium sulfate by weight of the material is deposited in the material, impregnating the material with a softening agent in aqueous solution, said softening agent being selected from the group consisting of cationic compounds and potentially cationic compounds which form cationic acid salts upon acidification, and an alkali metal salt of carboxymethyl cellulose in aqueous solution, exhausting the softening agent and carboxymethyl cellulose onto the material by acidifying to a maximum pH of about 6.5, said softening agent and said carboxymethyl cellulose each being introduced in an amount comprising at least about 0.1% by weight of the material, and then drying the material.

20. The process of claim 19 in which the softening agent is a higher fatty acid amide.

21. A method for imparting soil resistance to textile material which comprises separately impregnating the material with an aqueous solution of a water soluble barium compound and an aqueous solution of a water soluble sulfate, said solutions being supplied in any desired sequence, whereby at least about 2% barium sulfate by weight of the material is deposited in the material, impregnating the material with a softening agent in aqueous solution, said softening agent being selected from the group consisting of cationic compounds and potentially cationic compounds which form cationic acid salts upon acidification, and an alkali metal salt of carboxymethyl cellulose in aqueous solution, exhausting the softening agent and carboxymethyl cellulose onto the material by adding first an acid salt and then an organic acid in sufficient amount to reduce the pH to a maximum of about 6.5 and then drying the material.

22. The process of claim 21 in which the softening agent is a higher fatty acid amide and the carboxymethyl cellulose is sodium carboxymethyl cellulose.

References Cited in the file of this patent

UNITED STATES PATENTS

152,903	Gender et al.	July 14, 1874
2,410,788	Morgan et al.	Nov. 5, 1946
2,570,750	Bauer	Oct. 9, 1951
2,693,247	Kingsford	Nov. 2, 1954
2,701,218	Nickerson	Feb. 1, 1955

FOREIGN PATENTS

705,672	Great Britain	Mar. 17, 1954
---------	---------------------	---------------

(Other references on following page)

OTHER REFERENCES

Mollient: Silk Journal and Rayon World, May 1943, pp. 27, 28 and 30; 117-139.5.

Compton et al.: "Soiling and Soil Retention in Textile Fibers," Ind. and Eng. Chem., vol. 45, No. 3, pp. 597-602.

Feuell: "Desoiling Properties of Carboxymethyl Cellulose," Journal of Textile Institute, vol. 40, No. 8, August 1949, 8-137, pp. T-523-525.

Bayley et al.: "The Influence of Sodium Carboxymethylcellulose on the Suspending Power of Build Soap

Solutions," Textile Research Journal, July 1950, pp. 510-513; 8-137.

Untermohlen et al.: "Improvement of Cotton Cloth in Resistance to Soiling and in Ease of Washing," Textile Research Journal, July 1951, pp. 510-521.

Pollok: "Sodium Carboxymethylcellulose," Journal Textile Institute, vol. 343, No. 4, April 1952, p. A-256; 8-137.

Zimmerman et al.: Handbook of Material Trade Names, 1953, pp. 17 and 18.