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(54) **Fabric cleaning compositions.**

(57) Fabric cleaning compositions having improved properties are provided and comprise a carrier material and at least 5% by weight of the composition of the solvent diglyme.

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FABRIC CLEANING COMPOSITIONS

This invention relates to fabric cleaning compositions, more particularly to dry compositions useful for cleaning carpets, curtains and similar furnishing fabrics.

Carpets and other furnishing fabrics have, for many years, been subjected to cleaning using various wet systems involving shampooing, steam-cleaning and straightforward washing where the fabric can be conveniently treated by such methods. For carpets a particular problem arises from wet methods in the time taken for the carpet to dry prior to being available for its normal use as a floor covering. For example, the pile of a carpet can be flattened by people walking on it, or placing furniture on it when it is wet. In addition, unless traces of surfactant or soap used in the wet cleaning methods are removed, there is a tendency for the fabric to pick up soil at an enhanced rate.

Many proposals have been made for the preparation of dry cleaning compositions for fabric cleaning, some of which overcome the re-soiling problem by having little or no surfactant present in the compositions. For example, United States Patent Specification No 3 630 919 discloses a dry cleaning composition containing a minor amount of a colloidal silica having a particle size in the range 0.01 to 20 microns, a major amount of finely divided carrier having a bulk density in the range 6 to 22 lbs/ft^3 and water. Suitable carrier materials disclosed include various diatomaceous earths, sawdust, talc, cork, corn cob, Fuller's earth and the like. United States Patent Specification No 4 035 148 discloses a further surfactant-free composition consisting essentially of a water-soluble phosphate and a water-insoluble alumina having a particle size less than 3 microns. This composition is said to impart a soil repellent finish to carpets and the like. The preferred particle size for the alumina is from 0.005 to 0.1 microns. British Patent Specification No 2 037 311 discloses a pulverant, non-toxic cleaning agent comprising 70 to 30 parts by weight of the solvent mixture consisting of water and certain organic solvents, and a pulverant carrier of which the particle size is between about 5 and 1000 microns consisting of starch, flour or woody material. A very wide range of organic solvents is listed in this British patent specification including terpenic hydrocarbons which are liquid at 20°C and have a boiling point under atmospheric pressure between 150 and 250°C, aliphatic hydrocarbons which are liquid at 20°C and have boiling points under atmospheric pressure of between 90 and 300°C and oxygenated solvents selected from mono- and di-alkyl ethers of ethylene glycol and acetates of ethylene glycol mono-alkyl ethers.

It has now been found that from the wide range of solvents either suggested or claimed in the many prior art references, one solvent has outstanding properties. This particular solvent gives outstanding cleaning properties to compositions incorporating it and does not cause whitening problems of the type encountered in certain known cleaning compositions.

Accordingly the present invention provides a fabric cleaning composition comprising a carrier material and as cleaning liquid at least 5% by weight of the composition of diethylene glycol dimethyl ether. Diethylene glycol dimethyl ether is also known as diglyme.

The liquid component in the fabric cleaning composition may also comprise water up to 94% by weight of liquid and, optionally, a small quantity of a surfactant may be included. Preferably the surfactant is in the range 1 to 5% by weight of the total composition. Compositions containing a surfactant which produces a dry residue which can be readily removed are preferred.

The carrier material may be selected from a wide range of solid material including solids such as sodium sulphate, calcite and many other cheap carrier materials. Preferably, the carrier material is a porous material able to absorb a significant amount of the preferred solvent and to reabsorb the liquid component from the fabric.

A preferred carrier material is a silica comprising at least 5% by weight of the composition of the preferred solvent. Preferably the silica is a silica hydrogel. Suitable silica hydrogels for use in the compositions provided by this invention can be obtained from Joseph Crosfield & Sons Limited, Warrington, England, and are

usually sold in a form containing about 68% by weight water.

Another preferred carrier material is a particulate porous polymeric material. Suitable polymeric materials are disclosed in, for example, British Patent Specification No 1 411 230 and No 1 576 228 and in European Patent Applications Nos 006 138 and 0 105 634.

Other useful carrier materials include cellulosic composition and urea/formaldehyde resin powders such as those disclosed in British Patent Specification No 2 112 013 and Japanese Patent Specification 1981-12242.

Certain of the inorganic carriers can usefully be used in combination with the more expensive inorganic or polymeric carriers or as extenders in more complex mixtures.

Various additives may be incorporated into the fabric cleaning compositions provided by this invention such as perfumes, antistats, bacteriostats, fillers such as inorganic salts and softening agents including various cationic surfactants.

Tests have now been developed in an attempt to quantify the effectiveness of the cleaning compositions provided by the present invention in comparison with known cleaning techniques and compositions. First of all artificial soil has been developed which comprises vacuum cleaner dust, triolein and carbon black in the ratio 65:25:10 by weight. This is applied to a carpet sample as a spray using, as a volatile solvent, isopropanol (IPA). After drying the carpet is treated with a vacuum cleaner to remove loose dust. To obtain a consistent cleaning pattern, a textile abrasion tester has been

adapted to brush the carpet's surface in a reproducible and predetermined pattern under various weights of brushing pressure. The cleaning effect has been measured using the Hunter colour co-ordinates L,a,b. The results are quoted as a percentage of the total colour regain (TCR):

$$\% \text{ TCR} = \frac{(L_c-L_s)^2 + (a_c-a_s)^2 + (b_c-b_s)^2}{(L_u-L_s)^2 + (a_u-a_s)^2 + (b_u-b_s)^2} \times 100$$

where the subscripts refer to the samples being unsoiled (u), soiled (s) or cleaned (c).

In order to demonstrate the outstanding effectiveness of the preferred solvent known as Diglyme (diethylene glycol dimethyl ether), experiments were carried out using two different grades of silica hydrogel, bentonite, woodflour, a silica xerogel and a porous polymer. Compositions were prepared and comparisons made between Diglyme and isopropanol (IPA)in the various solid carriers. The tests were carried out by applying, at a rate of 40 g/m², the various powder compositions containing the solvents to carpet pieces which had been treated with the artificial soil. The results expressed in terms of TCR% are shown in Table I.

Table I

Carrier	Solvent Content % by Weight Composition		Water Content % by Weight Composition	% TCR
Silica hydrogel (1)	Diglyme	62	6	79.9
Silica hydrogel (1)	IPA	62	7	42.6
Bentonite	Diglyme	34	0	44.3
	IPA	34	0	27.4
Woodflour	Diglyme	41	0	63.1
	IPA	41	0	31.9
Silica hydrogel (2)	Diglyme	62	6	67.1
	IPA	62	6	30.1
Silica xerogel	Diglyme	30	6	69.7
	IPA	30	6	49.0
Accurel PA	Diglyme	60	0	81.0
	IPA	60	0	52.0

Details of the carriers used are set out in Table II below:

Table II

Silica	Surface Area (m^2/g)	Pore Volume (ml/g)	Particle Size (μm)
Silica xerogel	750	0.4	4.5
Silica hydrogel (1)	850	2.0	15.0
Silica hydrogel (2)	850	2.0	30-55
Accurel PA (A porous polyamide resin)	voids space 75%	-	100-200
Bentonite	-	0.5	<76
Woodflour	-	0.6	>200

Further experiments were carried out to compare diglyme with chemically similar solvents. Using a preferred carrier - a silica hydrogel - approximately 60% by weight of each solvent was exchanged into the hydrogel. Suitable silica hydrogels for use in the compositions provided by this invention can be obtained from Joseph Crosfield & Sons Limited at Warrington, England, and are usually sold in a form containing about 68% by weight water. The diglyme can conveniently be introduced into the hydrogel in a simple counter-current reactor in which the diglyme is fed counter-current into the container to displace water from the hydrogel.

The cleaning tests previously described were repeated for three cycles and the results are set out in Table III below:

Table III

Composition	% TCR Cycle 1	Cycle 2	Cycle 3
Silica hydrogel (1)/ethylene glycol	39.9	5.5	1.0
Silica hydrogel (1)/ethylene glycol monomethyl ether	44.8	36.7	38.8
Silica hydrogel (1)/ethylene glycol dimethyl ether	26.4	23.1	23.8
Silica hydrogel (1)/diethylene glycol	16.8	1.0	1.5
Silica hydrogel (1)/diethylene glycol monomethyl ether	35.5	9.8	10.0
Silica hydrogel (1)/diethylene glycol dimethyl ether (diglyme)	85.5	68.9	60.2
Silica hydrogel (1)/IPA	37.2	43.1	22.1
A commercial product based on a porous urea formaldehyde resin (17%) carrying IPA (2.4%) and polyol (5.6%) and water (to 100% by weight)	26.3	11.9	10.7

A further comparison was made between a mixture of solvents and diglyme on various carriers. A mixture (A) was prepared of the solvents:

D-limonene
Ethylene glycol monomethyl ether
Ethylene glycol monomethyl ether acetate

and a nonionic surfactant. The levels were respectively 50%, 20%, 10%, 20%. This mixture was used to fill three carriers. The same carriers were also filled with a similar mix (B) in which diglyme (80%) had replaced all the above solvents. There was no added water.

Cleaning results on soiled carpeting were as follows:

Table IV

Carrier	% Solvent in Carrier by Weight	Objective Result (%TCR)
Silica xerogel/(A)	44	59.6
Silica xerogel/(B)	42	78.3
Woodflour (>200μm)/(A)	60	11.2
Woodflour (>200μm)/(B)	54	60.9
Accurel PA/(A)	50	30.5
Accurel PA/(B)	53	84.4

To demonstrate the removal of the cleaning composition from carpet pieces two compositions described in Table I based on silica hydrogel (1) were prepared: one containing IPA and the other diglyme. Carpet pieces were treated with each of the compositions and then cleaned with a vacuum cleaner. The pile fibres were removed using a fine scalpel and analysed utilising X-ray fluorescence to determine the presence of silica on the

fibres in relation to set internal standards and the results are set out in Table V.

Table V

	Retention (% SiO_2)
Silica hydrogel (1)/IPA	0.47
Silica hydrogel (1)/diglyme	0.24

To demonstrate the effect of differing concentrations of diglyme in the compositions mixtures were prepared comprising silica hydrogel (1), $Na_2SO_4 10H_2O$ and diglyme and similar mixtures in which the silica hydrogel was replaced by Accurel PA resin powder. The results are set out in Table VI. The mixtures are expressed as % by weight of the composition and as before were applied at 40 g/m^2.

Table VI

Silica Hydrogel(1)	$Na_2SO_4 10H_2O$	Diglyme	Objective Result (%TCR)
40	-	60	92.9 ± 3.0
30	25	45	92.8 ± 1.7
20	50	30	95.2 ± 0.9
10	75	15	63.3 ± 1.5
5	87.5	7.5	16.4 ± 1.2
2.5	93.8	3.7	7.3 ± 0.8

Accurel (PA) Polymer	$Na_2SO_4 10H_2O$	Diglyme	Objective Result (%TCR)
40	-	60	85.1 ± 0.1
30	25	45	78.0 ± 2.2
20	50	30	86.2 ± 0.5
10	75	15	76.4 ± 1.4
5	87.5	7.5	62.6 ± 0.4
2.5	93.8	3.7	21.2 ± 0.7

It will be appreciated from the results in Table VI that a preferred form of the invention comprises a composition in which the carrier is a particulate porour polymeric material and the cleaning liquid comprises at least 7.5% by weight of the composition with diethylene glycol dimethyl ether.

A further preferred form of the invention comprises a composition in which the carrier material comprises a porous silica and the cleaning liquid comprises at least 15% by weight of the composition of diethylene glycol dimethyl ether.

CLAIMS

1. A fabric cleaning composition comprising a carrier material and as cleaning liquid at least 5% by weight of the composition of diethylene glycol dimethyl ether.

2. A fabric cleaning composition as claimed in claim 1 in which the liquid in the composition comprises up to 94% by weight of water.

3. A fabric cleaning composition as claimed in any one of the preceding claims comprising 1 to 5% by weight of the total composition of a surfactant.

4. A fabric cleaning composition as claimed in claim 1, 2 or 3 in which the carrier comprises particulate porous polymeric material and the cleaning liquid comprises at least 7.5% by weight of the composition of diethylene glycol dimethyl ether.

5. A fabric cleaning composition as claimed in claim 1, 2 or 3 in which the carrier material comprises a porous silica and the cleaning liquid comprises at least 15% by weight of the composition of diethylene glycol dimethyl ether.

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EUROPEAN SEARCH REPORT

Application number
EP 85 30 5136

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl.4)
D,A	GB-A-2 112 013 (FUJI KASEI CO. LTD.) * abstract, claims 1,2 *		C 11 D 3/00 C 11 D 3/43 C 11 D 7/50
D,A	GB-A-1 411 230 (DU PONT DE NEMOURS AND CO.) * page 1, lines 11-17, claims 1,2 *		
A	EP-A-0 062 536 (S.C. JOHNSON & SON, INC.) * abstract, example 1, claims 1,2 *		

TECHNICAL FIELDS SEARCHED (Int. Cl.4)

C 11 D 3/00
C 11 D 7/00

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
BERLIN	11-10-1985	SCHULTZE D

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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