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(54) **Textile treatment.**

(57) A method of treating textile fibres in a centrifuge with a substantive treatment agent comprises supplying the substantive treatment agent to the centrifuge whereby it passes through the fibres during the process of centrifuging characterised in that the treatment agent is in the form of a microemulsion. The fibres are preferably wool or wool-blend fibres, and the treatment agent is a preferably a mothproofing agent such as a synthetic pyrethroid. The use of a microemulsion ensures level treatment even though the agent is substantive to the fibres.

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TEXTILE TREATMENT

This invention relates to a method of treatment of textiles, and in particular relates to a method of applying substantive treatment agents in a centrifuge.

It has been proposed to use a centrifuge or hydroextractor for the application of certain finishes to textile materials, for example spin finishes for polyester fibre materials. However, such applications can only successfully be carried out where the treatment agent is non-substantive to the textile fibre. This employs the principle of application by impregnation, whereby a small volume of highly concentrated liquor is retained by the load following hydroextraction. The amount of finishing agent present in the treated load is simply a function of the wet pickup of the fibre together with the liquor concentration. Where, on the other hand, the treatment agent is substantive, very unlevel treatment results since a unidirectional liquor flow occurs radially outwardly through the textile pack resulting in fibres situated on the inside of the centrifuge pack being consistently exposed to the highest concentration of treatment agent. Absorbtion of the agent onto the fibres progressively reduces the liquor concentration in its passage through the fibre pack until fibres close to the centrifuge wall are exposed to the lowest concentration and thus receive the lowest degree of treatment. Indeed, it is not uncommon for the fibres closest to the liquor source to absorb ten times the amount of treatment agent than the fibres furthest away.

The invention seeks to provide a method of treating fibres with a substantive finishing agent in a centrifuge or hydroextractor which overcomes or reduces the above disadvantages.

According to the present invention there is provided a method of treating textile fibres in a centrifuge with a substantive treatment agent which comprises supplying the substantive treatment agent to the centrifuge whereby it passes through the fibres during the process of centrifuging characterised in that the treatment agent is in the form of a microemulsion.

We have surprisingly found where the treatment agent is in the form of a microemulsion very level results can be obtained even though the treatment agent is substantive to the fibres. An ordinary, or "macroemulsion", does not give the same effect and treatments with macroemulsions are unlevel as one would have expected with substantive treatments.

While the method of the invention is broadly applicable to treatment agents substantive to textile materials and available in microemuslion form, we have found that it is of particular utility in the treatment of wool or woolblend textiles with mothproofing agents. A particular advantage in respect of the latter is that effluents containing unused mothproofer, which are environmentally harmful, are reduced to a minimum and may be isolated from other effluents in the finishing routine. indeed the effluent may be reduced by more than an order of magnitude compared to some current moth proofing application methods.

Preferably, in the method of the invention, the treatment agent is applied at elevated temperature and to this end heating means, such as a heat exchanger, may be provided in the input liquor line.

Application of agents by exhaustion rather than by impregnation offers the advantage of working with relatively dilute solutions, and the quantity of finishing agent, especially mothproofing agent, to be removed from the spent liquor and disposed of safely is therefore significantly reduced. Moreover, having the facility to heat the circulating treatment liquor enhances exhaustion of the agent and again facilitates the use of environmentally desirable liquor concetrations which result in low residues.

The invention will be describe further, by way of example, with reference to the accompanying drawing, in which the sole figure is a diagrammatic view of a centrifuge apparatus modified in accordance with the invention.

Referring to the drawing, it can be seen that a textile centrifuge or hydroextractor 10 contains a pack 12 of textile fibrous material, for example wool fibres. The centrifuge 10 is modified by the provision of a spray head 14 connected via a heat exchanger 16 and a pump 18 to a tank 20 containing the treatment solution or "liquor". The tank 20 is connected via a two way valve 22 to the base of the centrifuge 10.

In operation, wool or woolblend textile material in the form of loose wool, hanks, packages or fabric is loaded into the hydroextractor 10 and spun to remove excess water for a fixed period of time. Expressed water is allowed to run to drain via the two way valve 22 . It will be understood that the textile material 12 will already be wet with liquor from the previous processing stage, for example dyeing. The microemulsion of treatment agent, for example mothproofing agent, is prepared in the storage tank 20. The amount of agent added to the tank 20 is directly related to the weight of textile material 12 present in the centrifuge and will be expressed as a percentage on the weight of fibre (OWF). With the centrifuge spinning at normal extraction speed, the liquor is delivered by the pump 18 via the heat exchanger 1, where its temperature is raised to the required level, to the spray head 14 and hence into the textile pack 12. During this operation the two way valve 22 is adjusted so as to direct exhausted liquor back into the storage tank 20. The volume of the liquor is such that recirculation takes place a large number of times during the treatment process. On completion of the process the exhausted liqor is retained in the tank 20 for reuse when treating subsequent loads. Addition of further treatment agent to the tank 20 may then be effected for subsequent loads to be processed to replace that absorbed by the previous load.

It has been found that, for example, the application of mothproofing agents such as synthetic pyrethroid compounds, to wool takes place more effectively at elevated temperatures, for example 50-60°C. Accordingly the apparatus preferably embodies a heat exchanger such as the heat exchanger 16 capable of heating up the liquor to the required temperature.

Many of the biologically active substances found useful as mothproofing agents for wool have very limited solubility in water. Commercial mothproofing agents are, therefore, generally supplied to the user in the form of emulsifiable concentrates. Such emulsifiable concentrates readily form oil-in-water emulsions of the otherwise largely insoluble active substance when diluted to working strength for application to textile materials. However, in the practice of the present invention, not all emulsions are suitable. In the practice of the invention we have found that only microemulsions give sufficiently level results.

The more normal macroemulsions give unlevel results as would hitherto be expected with substantive compounds applied in a centrifuge. The differences between the two are explained further below.

MACROEMULSION SYSTEMS

These are oil-in-water emuslions containing oil, water, surfactant and active ingredient. They appear milky white and are characterised by the property of separating into the original liquid phases on standing. Particle sizes fall into the range 2,000-100,000 Angstrom units.

MICROEMULSION SYSTEMS

These systems contain oil, water, surfactant, co-surfactant and active ingredient. They appear clear and transparent to light and do not separate on standing. Particle sizes fall into the range 100-2,000 Angstrom units.

It is only emulsions of the latter sort which yield an even and level treatment in a centrifugal hydroextractor in accordance with the present invention.

The invention is illustrated further in the following examples.

EXAMPLE Commercial formulations of moth proofing agents, such as synthetic pyrethroid insecticides, serve to illustrate the advantages of the method of the invention.

Loads of wool carpet yarnof between 18-24 Kg were placed in centrifugal hydroextractor in accordance with the present invention. Treatment solution at 60-65°C was sprayed into the hydroextractor at a rate of 24 litres per minute whilst spinning, with the liquor being continuously recycled.

The results are shown in table 1 below which illustrate the wool load concentration profile given by treatment of the wool which each of five agents under similar conditions. It will be noted that examples 1 to 3 the agent was in the form of a microemulsion (in accordance with the invention) whereas examples 4 and 5 macroemulsion products are given by way of comparison.

TABLE 1

	AGENT				
Example	1	2	3	4	5
Position in Load	Eulan CA41039	Eulan WBP	Eulan SP	Mitin N	Perigen
1 Outer	0.15	0.19	0.22	0.28	0.14
2	0.10	0.12	0.12	0.28	0.56
3	0.21	0.19	0.17	0.09	0.29
4 Central	0.27	0.15	0.21	0.10	0.72
5	0.24	0.17	0.21	0.20	0.71
6	0.23	0.14	0.23	0.41	0.89
7 Inner	0.21	0.23	0.21	1.22	1.09
Coeff. Variation %	29.0	22.0	19.5	117.0	52.5
Tot. Liquor Exh. %	70	75	72	90	78
Agent Type	Micro Emul.	Micro Emul.	Micro Emul.	Macro Emul.	Macro Emul.

In can be seen that those agents which are not macroemulsion formulations have very high values of coefficient of variation and are not uniformly distributed in the load. The coefficient of variation is a guide to the uniformity of treatment, the lower the value the more level is the treatment. All of the agents tested have a sufficient degree of exhaustion from the treatment liquor for commercial acceptability. Table 2 below shows the active ingredients corresponding to the trade names given in table 1.

TABLE 2

Commercial	Active
Eulan CA 41039	cis/trans Permethrin
Eulan SP	cis/trans Cyfluthrin
Eulan WBP	cis/trans Permethrin
Mitin N	Flucofuron
Perigen	cis/trans Permethrin

The treatment of example 2, with Eulan WBP, is repeated and samples of wool and liquor are tested at fixed time intervals. The results are given in Table 3 below.

TABLE 3
(Example 5)

Application Time (min.)	Liquor Concentration (Eulan WBP) (ml/L)	Agent Exhaustion (%)
0	1.82	-
1	0.93	49
2	0.68	63
3	0.58	68
4	0.52	71
5 (End)	0.47	75

This table shows how the agent is exhausted from the treatment liquor onto the wool as the process proceeds with time. A relatively large percentage of the agent is exhausted from the liquor during the first minute of application and the exhaustion rate then slows down. 100% exhaustion is probably not achievable in any reasonable time, but a 5 minute period generally provides a sufficient degree of exhaustion.

Examples 6, 7 and 8 show the uniformity of application to wool fabric and loose stock wool is comparable to that of yarn hanks. However, when the yarn is pre-acidified with acetic acid, the uniformity is lost. The remaining application conditions are similar to those outlined previously. It is believed that the presence of the acid destabilises the microemulsion and thus the advantages of the invention are lost. Accordingly the process of the invention should be carried out under pH conditions which do not destabilise the microemulsion.

TABLE 4

WOOL MATERIAL			
Example	6	7	8
Position in Load	Fabric	Loose Stock	Acidified Yarn
1 Outer	0.15	0.13	0.13
2			0.06
3			0.12
4 Central	0.10	0.12	0.15
5			0.26
6			0.26
7 inner	0.14	0.15	0.37
Coeff. Variation %	20.0	11.5	56.0
Tot. Liquor Exh. %	77	57	89
Agent	Eulan WBP	Eulan WBP	Eulan CA 41039
Agent Type	Microemul.	Microemul.	Microemul.

Losses of insect resist agent during the service life of carpets, rugs and upholstery fabrics are caused primarily by removal of agent during cleaning and by photodegradation of the active ingredient of the insect resist agent. The magnitude of these losses depends on the nature and chemistry of the insect resist agent, and on its method of application. The extent of loss may be determined by subjecting the treated textile to the fastness tests specified in IWS Test Method 28. The results are expressed as fastness factors, i.e. the fraction of original insect resist agent remaining after application of the appropriate fastness tests.

$$\frac{\text{Amount of agent after fastness test (\% oww)}}{\text{Amount of agent before test (\% oww)}} = \text{Fraction remaining on wool}$$

Fraction remaining for light x fraction remaining for shampoo = Fastness Factor

Table 5 below gives results of fastness testing on carpet made from yarn treated with Eulan WBP in the hydroextractor under standard conditions.

TABLE 5

Untested Carpet	Shampooed Carpet	Light Exposed Carpet
0.17	0.14	0.10
0.14	0.12	0.10
0.15	0.14	
0.13	0.12	
x 0.15	0.13	0.10

The fastness factor is calculated by taking the average amount (line x in the table 5) and applying to the calculation given above :

$$\text{Fastness Factor} = \frac{0.13}{0.15} \times \frac{0.10}{0.15} = 0.57$$

A value of 0.57 is regarded as at least equivalent to that attained with yarn treated by conventional scouring application of moth proofing agent.

In order to illustrate how hydroextractor aplication of Permethrin - containing agents results in considerably less residual permethrin released to teh environment per Kg. of treated yarn, comparative tests were carried out with three conventional methods of applying this agent namely during stock dyeing, conventional scouring and hank dyeing. Table 6 below gives comparative amounts of residual permethrin in mg per kg of treated yarn, discharged to waste.

TABLE 6

Production Route	Residual Permethrin mg/kg Treated Yarn
Stock dye, hydro extract, continuous scour, hydroextract dry	200
Conventional scour, hydroextract, dry	50-100
Hank dyeing, hydroextract, dry	4-8
Hydroextract Application, dry	2

Additionally, as well as having an inherently low residual permethrin content, the small volume of liquor left at the end of the treatment process of the present application makes it feasible for the liquor to be chemically treated. In this case the residual figure may be reduced by a factor of up to 50 times further if such chemical treatment is carried out. This form of treatment is not commercially possible with the spent liquors from the other production routes owing to the large volumes concerned.

The method of the invention allows, in particular, mothproofing agents to be applied to wool or woolblend fibres at a late stage in the treatment cycle thereby generating a minumum of environmentally harmful effluent.

Claims

1. A method of treating textile fibres in a centrifuge with a substantive treatment agent which comprises supplying the substantive treatment agent to the centrifuge whereby it passes through the fibres during the process of centrifuging characterised in that the treatment agent is in the form of a microemulsion.

2. A method according to claim 1 wherein the fibres are wool or wool-blend fibres.

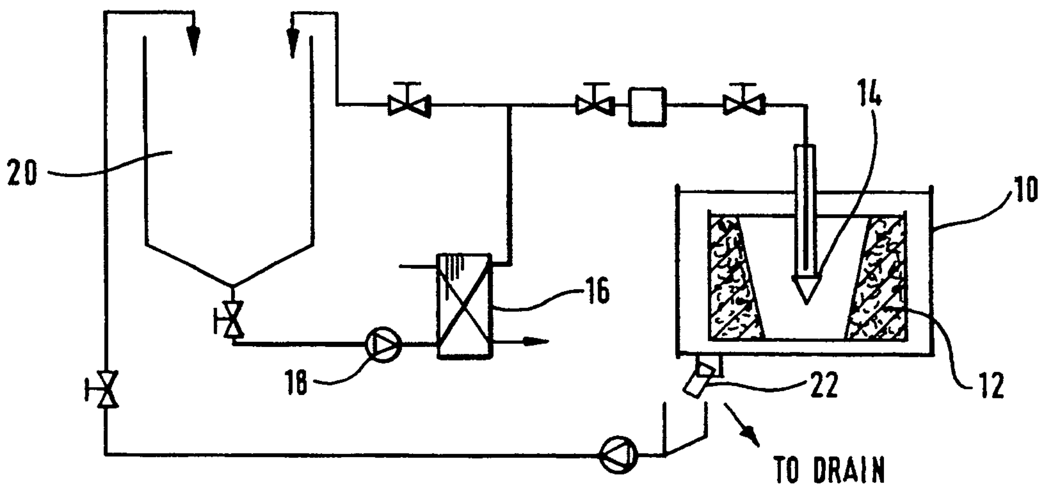
3. A method according to claim 2 wherein the treatment agent is a mothproofing agent.

4. A method according to any of claims 1 to 3 wherein the treatment agent is applied at an elevated temperature.

5. A method according to claim 4 wherein the temperature is about 60°C.

6. A method according to any of claims 3 to 5 wherein the mothproofing agent is a synthetic pyrethroid insecticide.

7. A method according to any of claims 1 to 6 in which the particlae size of the micro emulsion is in the range 100 to 2000 Angstrom units.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TO DRAIN