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- **Bou, Belda, Eva**
03801 Alcoy (Alicante) (ES)
- **Monllor Pérez, Pablo**
03803 Alcoy (Alicante) (ES)
- **Bonet Aracil, M Angeles**
03803 Alcoy (Alicante) (ES)

(71) Applicant: **InnovaTec Sensorización y Communication S.L.**
03801 Alcoy Alicante (ES)

(74) Representative: **Torner Lasalle, Elisabet et al Torner, Juncosa I Associats, S.L.**
C/Gran Via de les Corts Catalanes, 669bis 1è 2º
08013 Barcelona (ES)

(72) Inventors:

- **Jaime, Gisbert, Paya**
03803 Alcoy (Alicante) (ES)

(54) **A method and composition to infuse an active ingredient into clothes and use of a binder agent for microcapsules of said composition**

(57) The method comprises microencapsulation of an active ingredient in microcapsules and putting in contact said ingredient with the clothes including microcapsules in the washing water when washing and delivering the active ingredient through the wall of the microcapsules providing a gradual release of active ingredient particles as a result of the rupture of rubbed microcapsules while the clothes are being used wherein succinic acid

is blended with the microcapsules to adhere them to the clothes, then the clothes with microcapsules are submitted to a thermal treatment.

A composition is obtained by blending together microencapsulated active ingredient and succinic acid as a binder agent.

DescriptionField of the art

- 5 **[0001]** The present invention relates in general in a first aspect, to a method to infuse an active ingredient into textiles by means of microencapsulation, and in particular, to a method to infuse fragrances into clothes.
- [0002]** A second aspect of the invention relates to a composition containing a microencapsulated active ingredient (such a fragrance) prepared for gradual release of the active ingredient on laundered clothes either in a washing machine or in hand washing.
- 10 **[0003]** The invention also proposes the use of a binder agent for applying microcapsules containing an active ingredient on laundry either in a washing machine or in hand washing.

Prior State of the Art

- 15 **[0004]** There is a market demand for detergents or similar products with additives to wash laundry and infuse it with an attractive and persisting fragrance. The perfume-based additives make washed garments more appealing to the consumer. For instance, it is desired to have a product that could add a subtle fragrance to their clothes with a long-lasting and stable effect: scented detergents that can remain on washed fabrics.
- [0005]** There are currently numerous formulas with value-added functions for the care and treatment of clothes, some of them with perfumes.
- 20 **[0006]** Different techniques have been developed to hinder or delay the release of aroma, making it possible for the textile to retain the perfume, but to date few products and procedures can provide long-term release of perfume after prolonged storage.
- [0007]** In addition, there is on-going search for methods and compositions that can effectively and efficiently provide aromas to textile articles during a washing bath. As it can be seen from the following descriptions, various methods of perfume supply have been developed, involving perfume protection during the wash cycles. U.S. 2008/0103265, outlined the development of microcapsules dispersed in hydrophobic solutions where these microcapsules may contain different active ingredients, including fragrances of different composition. PCT patent publication, WO 01/23512 discloses the application of a washing detergent consisting of copolyester and a sulfo-functional group aimed at retaining fragrances on the surface of the cloth fibres, to be applied mainly by washing. EP 2342628, uses aqueous emulsions in the shape of foam for recharging textiles. Cognis uses oil mixtures in aqueous solutions to treat textiles in order to obtain beneficial effects for the human skin, but after the first wash the effect decreases. WO 2006/113091 includes the fragrance in a dispenser to be added in the tumble dryer; the product is mixed with a carrier that reacts at a certain temperature (170° F) infusing the fragrance onto the textile.
- 25 **[0008]** US 5066419, describes the encapsulation of a perfume dispersed with a non-polymeric, water-insoluble carrier material, which is itself encased in a protective, water-insoluble coating. Likewise EP 0701600 (B1) covers a perfume delivery system comprising zeolites. This system faces the problem that the perfume is absorbed by the porous surface of the zeolite and the scent disappears over a short period of time.
- [0009]** Industry efforts concentrate on three main research lines: finding the right formula of a washing product that will keep for longer storage times without significant loss of perfume properties; odour intensity or fragrance level supplied; and the duration of the perfume on the surface of the treated fabric.
- 30 **[0010]** Another problem found in the manufacturing of perfumed washing products is the odour intensity, particularly on granular detergent compositions of high density. When density and concentration levels of a detergent are increased, the smell of the perfumed components may become undesirable due to its intensity.
- [0011]** The most extended microcapsules applied to textiles are based on non reactive shells. In order to improve durability to washing or handle some auxiliary products are required to join microcapsules to the fibre surface, see Nelson G., 2001 "Microencapsulates in textile finishing", Rev. Prog Color 31, 57-64; they are usually based on acrylic, polyurethanes, or silicone resins (Li et al, 2008 "Effect of finishing methods on washing durability of microencapsulated aroma finishing". Journal of the textile institute 99.2.177-183, Rodrigues et al, 2009 "Microencapsulated perfumes for textile application", Chemical Engineering Journal 149.2009.463-472). Some studies show how polycarboxylic acids can be used in encapsulation process (Pascu et al, 2008 "Interfacial polymerization of an epoxy resin and carboxylic acids for the synthesis of microcapsules" M Polymer international 57.995-1006) or 1,2,3,4-butanetetracarboxylic acid (BTCA) as a binder to join microcapsules to fabrics as disclosed in Voncina , Badulescu et al. "Grafting of ethylcellulose microcapsules onto cotton fibres" Carbohydrate polymers 71.2008.85-91.
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Summary of the Invention

- [0012]** It is necessary to offer an alternative to the state of the art which covers the gaps found therein, particularly

related to the lack of proposals that consider the effectivity of binding agents used when scented microcapsules are applied in a washing machine while laundry, as well as binding effect and its impact in the number of laundry cycles the microcapsules remain on the fabric considering laundry behaviour.

[0013] For this purpose the invention develops a method to infuse an active ingredient into textiles by means of microencapsulation, and in particular, to a method to infuse fragrances into clothes.

[0014] The invention also proposes a composition formed by scented microcapsules and a binder agent, suitable for household washing machines, that reloads microcapsules on textiles. The perfume durability linked to several textile compositions are also determined, as well as its effectiveness depending on the final use of the garment.

[0015] The composition is used while laundering, so that the microcapsules are introduced into fabric yarns. The resulting laundered textiles can be used as normal and the active ingredient will be released gradually from friction in everyday use of the garment.

[0016] The method of the invention is intended for gradual release of an active ingredient (such as a fragrance or scented material) on laundered cloths either in a washing machine or by hand washing comprising as already known in the art:

- microencapsulation of said active ingredient in microcapsules;
- putting in contact said ingredient with the cloths including microcapsules in the washing water when washing; and
- delivering the active ingredient through the wall of the microcapsules providing a gradual release of active ingredient particles as a result of the rupture of rubbed microcapsules while the clothes are being used.

[0017] According to the proposal of this invention succinic acid as a binder agent has been added blended with the microcapsules to bond the microcapsules to the clothes and then said clothes with microcapsules are submitted to a thermal treatment in order to provide a suitable curing of the binder agent.

[0018] In a suitable embodiment thermal treatment of the binder agent comprises curing it at about 150°C by ironing the clothes. Curing can be provided alternatively with tumble dryer.

[0019] The active ingredient is selected among a fragrance, an anti-bacterial, an anti-mosquito, a moisturizer or a blend thereof.

[0020] The microencapsulation is carried on by an interfacial polymerization process (well known in the art) obtaining microcapsules with a membrane is formed using two monomers by preparing a pre-condensed using melamine and formaldehyde in aqueous phase.

[0021] As known in the art the wall of the microcapsule is a membrane allowing evaporation through it of said active ingredient.

[0022] According to an embodiment the size of said membrane ranges from 0.5 to 150 nanometres.

[0023] The composition of the invention to infuse an active ingredient into clothes has been prepared to be used in domestic laundry either in a washing machine or in hand washing for gradual release of the active ingredient on laundered cloths and comprises microcapsules containing said active ingredient (selected among a fragrance, an anti-bacterial, an anti-mosquito, a cosmetic, a lavender oil or blend thereof) and a binder agent to adhere said microcapsules to the cloth fibres, so that said active ingredient is gradually delivered to the clothes through the capsule wall, the composition comprising a microencapsulated active ingredient and succinic acid as a binder agent blended together.

[0024] In order that the resulting effect on clothes will endure prolonged washing and wear the binder agent should be cured at about 150°C for example by ironing.

[0025] According to an embodiment of the invention the microcapsules have a size between 2 to 20 microns.

[0026] In an alternative embodiment the microcapsules have a size between 3-8 microns.

[0027] The invention also teaches the use of succinic acid as a binder agent for microcapsules containing an active ingredient for applying said microcapsules on laundered cloths either in a washing machine or by hand washing.

Brief Description of the Drawings

[0028] The previous and other advantages and features will be more fully understood from the following detailed description of embodiments, with reference to the attached drawings, which must be considered in an illustrative and non-limiting manner, in which:

Figure 1 shows the whiteness index depending on the binder used and the temperature of treatment or curing temperature.

Figure 2 is a microphotograph showing microcapsules applied on fabrics by washing machine process with BTCA binder, cured at 150°C (sample BTCA-150) and without any laundry.

Figures 3a to 3f show fabrics treated by ISO 105 C10 after 5 cycles: a) 25M; b) RES; c) BTCA; d) BTCA-150; e) SUC; f) SUC-150.

Figure 4 shows the behaviour of the microcapsules while washing the treated fabrics indicating the microcapsules presence on the fabrics (mics/m2) after different laundry cycles.

Figures 5a to 5c shows fabrics treated by ironing and washed once: a) ironed at 110°C; b) ironed at 150°C; c) ironed at 200°C.

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Detailed Description of Several Embodiments

[0029] The present invention shows that scented perfume microcapsules developed by interfacial polymerization with an average size of 8 microns blended together with a binder agent are capable to bond to the textile surface and may last for several washes, releasing the microencapsulated fragrance gradually.

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[0030] Embodiments of the invention solve the problem of fragrance durability on clothes by creating a microencapsulated product that can be incorporated in domestic washing machines, making it possible for customers to recharge them for prolonging the fragrance or functional effects.

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[0031] This invention is the result of a search directed to find a suitable binder agent for microcapsules containing an active ingredient for applying said microcapsules on laundered cloths either in a washing machine or by hand washing, providing a good adhesion and permanence of the microcapsules bonded to the fibres when they are pasted onto the fabric while clothes are washed and resisting a high number of laundry cycles with the microcapsules remaining on the fabric.

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[0032] To this aim the effectiveness of different resins and bonding agents was evaluated in order to find the optimal formula in both composition and concentration of the binder agent, so that the resulting effect on clothes will endure prolonged washing and wear.

[0033] Firstly laundry behaviour and lately, change in colour was appraised to determine if the procedure has secondary effects as yellowing.

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Materials and methods

[0034] Microcapsules (CENTER FINISH 164/02 LAVANDA) were supplied by COLOR CENTER (Tarrasa, Spain). The wall material was melamine formaldehyde, and the microcapsules contained lavender fragrance. No further information was supplied by the provider. In order to bond the microcapsules to the fabric, an acrylic resin was applied, also supplied by Color Center and two polycarboxilic acids, butanetetracarboxilic acid (BTCA) from Alfa Aesar and Succinic acid (SUC) from Panreac.

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[0035] The fabric used was a 100% cotton twill fabric with 210 g/m2, which had been chemically bleached with peroxide in an industrial process.

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Adhering microcapsules to fabrics.

[0036] The equipment used to apply microcapsules was a domestic washing machine. Its capacity was 5 Kg and its maximum volume of water was 11 L.

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[0037] Microcapsules dispersion was made in a glass vessel. Four tests were conducted. Different samples were prepared depending on the binder nature. All the samples contained 5% owf (over weight of fibre) of microcapsules. One of the baths was comprised of microcapsules only. The recipe for samples with polycarboxilic acids (BTCA and SUC) as binder was formulated with 5% owf of microcapsules 4% owf of binder and 2% owf of catalyst. The test with acrylic binder (RES) was based on 5% owf of microcapsules and 12,5 % owf of binder. As soon as the washing machine finished, the fabrics were dried in an IR dryer at 100°C until the fabric was completely dry. Lately, fabrics should be thermally treated so as to polymerize the binder. Commercial brands suggest different temperatures but in this study the curing temperature has been selected in order to get the minimum yellowness on the cotton fibre and different curing temperatures have been evaluated.

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[0038] Table 1 summarises the treatment conditions herein studied.

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Table 1 (Laundry conditions)

	TEST 1	TEST 2	TEST 3	TEST 4	TEST 5	TEST 6
Microcapsules concentration (o.w.f)	5%	5%	5%	5%	5%	5%
Binder concentration (o.w.f)	0%	20%	20%	20%	20%	2,5%
Binder composition	--	BTCA	BTCA	SUC	SUC	RES
Catalyst (owf)	--	2%	2%	2%	2%	--
Bath Temperature (°C)	30	30	30	30	30	30

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(continued)

	TEST 1	TEST 2	TEST 3	TEST 4	TEST 5	TEST 6
Drying temperature (°C)	110	110	110	110	110	110
Curing temperature (°C)	--	--	150		150	110
Curing time (seconds)	--	--	120	120	120	120
Reference	25M	BTCA	BTCA-150	SUC-	SUC 150	RES

Scanning Electron Microscopy (SEM)

[0039] For surface observation, a scanning electron microscope (SEM) Phenom microscope (FEI Company) was used. Each sample was fixed on a standard sample holder and sputter coated with a gold -platinum mixture. Samples were then examined with suitable accelerating voltage and magnification.

Microcapsules permanence

[0040] To test the effectiveness of the binders some laundering tests were conducted. The specimens were treated on a short time program in a Heraeus Linitest for 45 min at 30 °C, in accordance with ISO Standard 105 C01. When a cycle was finished, samples were dried on a horizontal surface. All samples were examined after 1, 2, 3, 4 and 5 cycles.

Counting microcapsules

[0041] The particle size distribution of the microcapsules was measured by a Coulter® Counter apparatus (Multisizer Z2, Coulter Electronics, Northwell, UK). The particle size was expressed as the equivalent volume diameter and two replicates were performed for each batch of microcapsule, to reduce error an average curve was calculated and analysed.

[0042] This method was used to measure the microcapsules dispersion just immediately it was prepared and the wastewater from the washing machine. The number of microcapsules remaining on the fabric was calculated as the difference between the number of microcapsules on the prepared bath and the number of microcapsules in wastewater.

[0043] Furthermore, the wastewater from ISO laundry was also measured to observe the microcapsules behaviour in each laundry cycle.

Ironing samples

[0044] Some samples were ironed at different temperatures (110, 150 and 200°C). The ironing procedure was performed as UNE EN ISO 105 X11 standard indicated.

Curing temperature

[0045] Polycarboxilic acids should be cured at temperatures from 150°C in order to induce crosslinking. Knowing that thermal treatment on cellulosic fibres is responsible of yellowness, different curing temperatures have been evaluated. Figure 1 shows the whiteness index depending on the binder used and the temperature of treatment. The white colour decreases as the temperature increases, and it can be appreciated that if temperature is higher than 180°C cotton fibre whiteness considerably decreases. When fabrics are treated with binders the whiteness index is approximately the same than the fabric without treatment. As a result 150°C has been considered a good temperature for curing polycarboxilic acids.

Binder effectiveness

[0046] When microcapsules dispersion was added on washing machine it could be observed that all the samples showed some microcapsules on its surface, including the ones from test one (M25) where no binder was used. Images show not sensible differences between the samples obtained with different binder. Thus, only one fabric is shown in figure 2 where it is shown the sample BTCA-150.

[0047] In order to observe the binder effect images from washed samples were analysed as standard ISO 105 C10 indicates. When figure 2 is compared with figure 3 the laundering effect can be clearly seen, some microcapsules have been missed from the fabric surface. If the binder effect is analysed (see figure 3a and 3b) what can be appreciated is that binder plays its role helping to remain more number of microcapsules on fabric. This result is not surprising however, it is not only important the binder presence but the curing temperature for polycarboxilic acids too. The influence of curing

temperature has been demonstrated as fabrics cured at 150°C show a wider number of microcapsules on its surface than the ones without curing, compare figure 3c and 3d or 3e with 3f. Moreover, the samples treated with SUC are the ones that show the higher quantity of microcapsules after washing fabrics during 5 cycles when the sample has been cured 150°C, see figure 3e and 3f.

[0048] Despite the fact that results from SEM can be obvious, the analysis from wastewater can clarify those results as it is more objective and shows the results expressed in the quantity of particles that wastewater contained.

[0049] To begin with, the microcapsules deposition on fabric should be characterised. This was calculated as the difference in particles between the number of particles in the dispersion when it was just prepared to be put into the washing machine and the ones in the wastewater after treatment. Moreover, the number of particles in wastewater after each laundry was obtained. Figure 4 shows the behaviour of the microcapsules while washing the treated fabrics.

[0050] It can be clearly observed a decreasing tendency in the number of microcapsules that remain on the fabric when the number of cycles increases. That behaviour evidences the necessity of any kind of binder if it is desired to increase the microcapsules presence on fabrics. Samples with no binder (25M) do not show any microcapsule between their fibres. This result is in accordance with the ones showed by SEM technique in figure 3. Furthermore, if resin has been added to act as a binder, thermal treatment should be performed otherwise microcapsules would be lost in the first or second cycle (see figure 4 SUC and SUC-150). However, when binder is added and thermal treatment is applied, the acrylic resin loses more quantity of microcapsules than succinic acid after the third cycle. This means that succinic acid if it is well cured shows the best results considering the permanence of microcapsules on fabrics. Samples treated with BTCA and washed could not be evaluated as some microcapsules stick together in the bath and it is not possible to measure them. This test has been performed twice and the same behaviour was observed. Moreover, when microcapsules dispersion was prepared with acrylic binder (RES) and non distilled water was used some aggregates could be observed. The aggregates presence can be due to some kind of ionic behaviour what makes them difficult to evaluate. If microcapsules have the property of getting stuck together when used with acrylic resin or BTCA binder, there is no possibility in obtaining an accurate procedure, as it is not possible to repeat it with the same result. This means that succinic acid is the most suitable for applying microcapsules on laundry in which current water is used.

Fabric yellowing

[0051] In order to determine the influence of the treatment on the fabrics the whiteness index was measured. Table 2 shows either the white index values for each sample and the difference in white compared with the cotton fabric without being treated.

Table 2 (White index values).

SAMPLE	WI CIE	Δ WI CIE
Cotton	80.99	--
25M	78.96	-2.03
BTCA	79.98	-1.01
BTCA-150	78.33	-2.66
SUC	79.09	-1.3
SUC-150	76.63	-4.36
RES	77.01	-3.98

[0052] It is interesting to note that microcapsules on fabric (25M) change the whiteness of the original fabric, but if we compare all the measurements slight differences between the different used binders can be appreciated. When measurements are referred to differences (Δ WI CIE) it can be observed that both BTCA-150 and SUC-150 are the ones with higher values. However, in order to evaluate if measured values would be observed by human eye, colorimetric values have been analysed and the difference between each sample and the untreated cotton have been calculated. One of the colour systems on which most chromatic studies are based is the CIE L*a*b* system [a,b]. When we observe chromatic values, we can appreciate that L* value (darkness/lightness) can be considered constant around the value 95. Thus, implies no changes because of the treatment have been induced in fabric lightness. When differences are calculated a clear correlation was found between Δ WI CIE and Δ Ea*b*, as it could be predicted. The single most striking observation to emerge from the data comparison is that samples with polycarboxylic acids (BTCA-150 and SUC-150), which include microcapsules show higher whiteness index than cotton treated at the same temperature without microcapsules and no polycarboxylic acids treatments (cotton 150). Colour differences from samples with microcapsules shown in Table 2 demonstrate that despite the fact that spectrophotometer measurements show objective differences, it can be stated that they cannot be appreciated by human eye as they are not higher than 1.

Ironing as thermal treatment for crosslinking

[0053] In order to determine if thermal treatment while ironing process would be able to induce crosslinking, samples after being treated in washing machine were ironed at different temperatures value as the Standard establishes. Later on, they were washed as UNE EN ISO 105 suggests so as to check if crosslinking by ironing is effective or not.

[0054] It can be clearly observed in Fig. 5 that after 1 laundry cycle, samples still show microcapsules however it is noticeable that the ones treated at 110°C have lost the majority of the microcapsules if compared with figure 2. By contrast, when samples ironed at temperatures of 150°C and 200°C are studied, results are really encouraging as a wide number of microcapsules still remain on fabric surface.

[0055] The above proves that a washing machine can be a suitable device to apply microcapsules onto fabrics binder agent.

[0056] First of all, the present study compares acrylic resin with two polycarboxylic acids and the thermal treatment to cure the binder agent. It is not surprising that microcapsules remain on fabric surface when the treatment includes some binder as it adheres microcapsules to fibre. It is noticeable that thermal treatment is required if the microcapsule effect should last more than one washing cycle.

[0057] As a consequence of thermal treatment, colour changes can be induced on cotton fibre. Chromatic studies have been conducted so as to determine either the thermal influence or the presence of chemicals can produce on fabrics. The evidence from this study suggests that some changes occur, as it was predictable. However, either differences in witness's index or chromatic differences demonstrate they are not sensitive enough to be observed by human eye.

[0058] When acrylic resin is compared with polycarboxylic acids and considering thermal conditions for each one, the evidence shows that the later allow microcapsules to remain on the fabric the longer.

[0059] From the tests done succinic acid displays the higher number of microcapsules on the fibre. Furthermore, acrylic binder develops some aggregates of microcapsules and makes impossible to test the binder appropriately.

[0060] To sum up, when applying microcapsules to fabrics by washing machine, succinic acid if cured at 150°C shows the best results and treated fabric has not changed sensitively the colour. Ironing at 150°C is effective in the curing process and in the appropriate binding of microcapsules in fabrics.

[0061] From the above it has been demonstrated the possibility of applying microcapsules to fabrics in a domestic process using succinic acid in a conventional washing procedure and ironing with a domestic equipment.

Claims

1. A method to infuse an active ingredient into cloths for gradual release of the active ingredient on laundered cloths either in a washing machine or when hand washing comprising microencapsulation of said active ingredient in microcapsules, adding a binder agent to adhere the microcapsules to the cloths and putting in contact said microcapsules and binder agent with the cloths in the washing water when washing, ensuring that delivering of the active ingredient through the wall of the microcapsules occurs in the form of a gradual release of active ingredient particles as a result of the rupture of rubbed microcapsules while the clothes are being used, **characterized in that** succinic acid is used as a binder agent to bond the microcapsules to the clothes and then said clothes with microcapsules are submitted to a thermal treatment.
2. A method according to claim 1, wherein said thermal treatment of the binder agent comprises curing it at about 150°C by ironing the clothes.
3. A method according to claim 1 wherein said thermal treatment of the binder agent comprises curing it at about 150°C in a tumble dryer.
4. A method according to claim 1 or 2, wherein said active ingredient is a fragrance.
5. A method according to claim 1 or 2, wherein said active ingredient is selected among a fragrance, an anti-bacterial, an anti-mosquito, a cosmetic or a blend thereof.
6. A method according to claim 1 wherein said microencapsulation is carried on by an interfacial polymerization process obtaining microcapsules with a membrane is formed using two monomers by preparing a pre-condensed using melamine and formaldehyde in aqueous phase.
7. A composition to infuse an active ingredient into cloths prepared to be used in domestic laundry either in a washing machine or in hand washing for gradual release of the active ingredient on laundered cloths, comprising microcap-

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sules containing said active ingredient and a binder agent to adhere said microcapsules to the cloth fibres , so that said active ingredient is gradually delivered to the cloths trough the capsule wall **characterized in that** the composition comprises a microencapsulated active ingredient and succinic acid as a binder agent blended together.

- 5 **8.** A composition according to claim 7 , wherein said active ingredient is selected among a fragrance, an anti-bacterial, an anti-mosquito, a cosmetic or a blend thereof.
- 9.** A composition according to any of the claims 7 or 8, wherein said active ingredient is a lavender oil.
- 10 **10.** A composition according to any of the claims 7 to 9, wherein the microcapsules have a size between 2 to 20 microns.
- 11.** A composition according to claim 9, wherein the microcapsules have a size between 3-8 microns.
- 15 **12.** Use of succinic acid as a microcapsules binding agent containing an active ingredient for applying said microcapsules on laundered cloths either in a washing machine or by hand washing.

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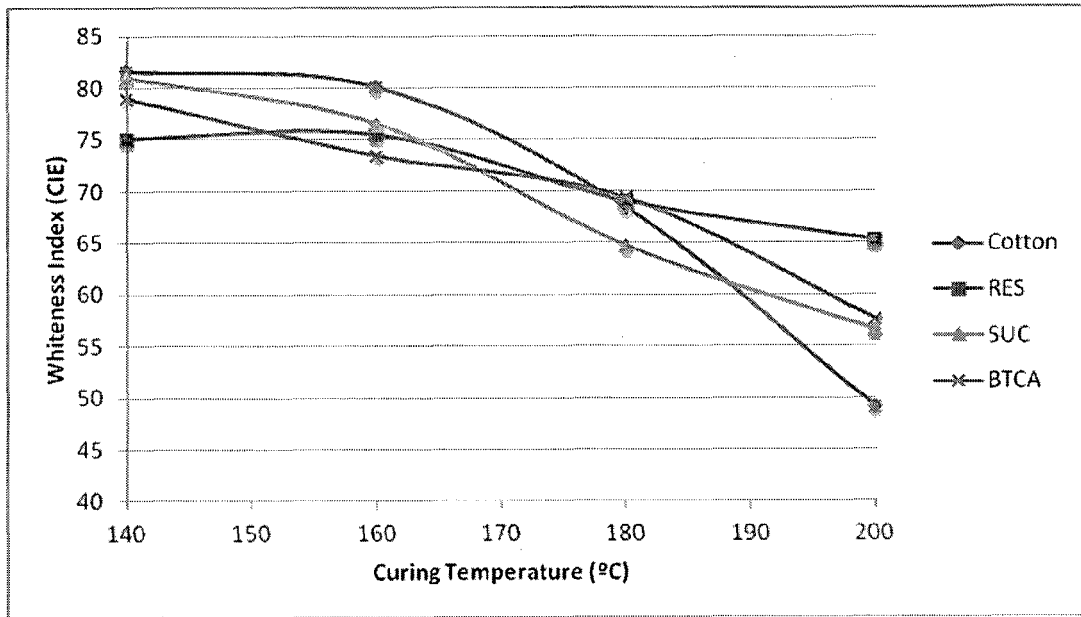


Fig. 1



Fig. 2

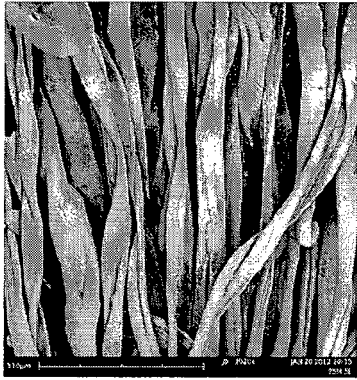


Fig. 3a



Fig. 3b



Fig. 3c



Fig. 3d



Fig. 3e



Fig. 3f

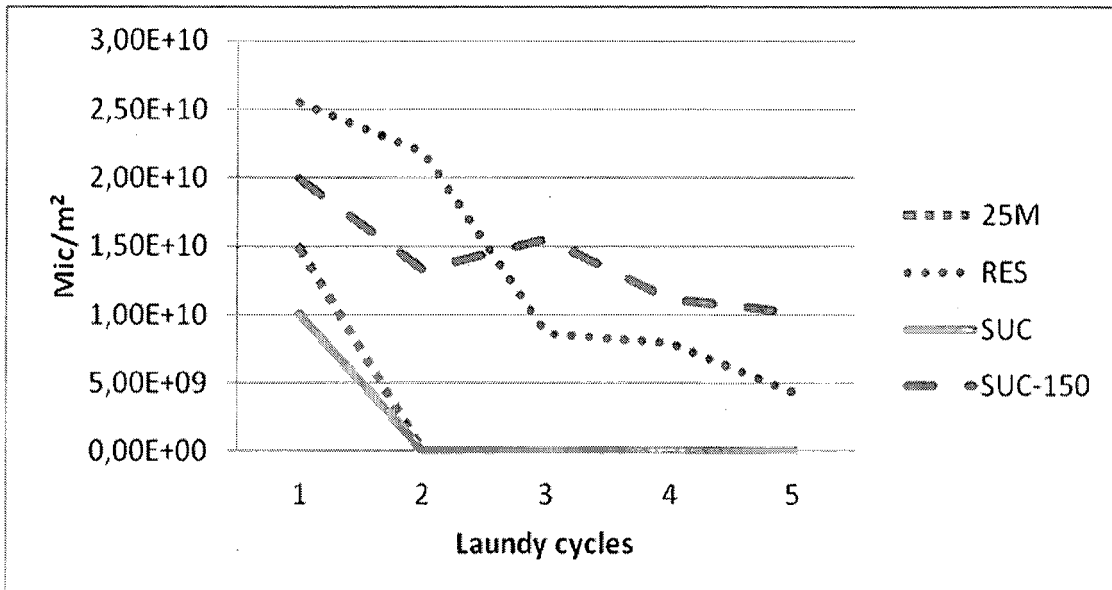


Fig. 4



Fig. 5a



Fig. 5b



Fig. 5c



EUROPEAN SEARCH REPORT

Application Number
EP 12 00 4957

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
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