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Study on the Possibilities to Graft Microencapsulated Essential Oil on Natural Fibres and Terry Fabrics

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Abstract
This research studied the influence of thermo-fixing temperature and binder concentration on the fixation of microcapsules with Eucalyptus essential oil to yarns and terry fabrics as well as physical properties of textiles with microcapsules. SEM micrographs confirmed that the grafting of microcapsules onto yarns spun from cotton, grey linen, bleached linen, ramie and bamboo fibres as well as onto ramie-cotton terry fabrics was effective. We conclude that a thermo-fixing temperature of 160 °C and higher caused the deformation of microcapsules or the partial or entire loss of an active product of the majority of microcapsules. Larger microcapsules were commonly damaged faster and were worse than smaller ones. It was determined that variation of the binder concentration from 20 to 95 g/l increased the area density of ramie/cotton terry fabric 1.7 times. The area density of terry fabric grafted with microcapsules in relation to the weft density could be described by linear equations.

Key words: *aroma microcapsules, binder concentration, natural fibres, terry fabric.*

Introduction

Micro-encapsulation is the method of incorporation of various non-toxic, biocompatible and biodegradable substances, which can be liquid or solid in nature, within shell material, thus forming microcapsules (MC) with the aim to produce bio-functional textiles fixing microcapsules onto fabrics and holding them in place during washing and wearing [1 - 3].

Microencapsulated fragrances can be used for textiles to prepare products with additional qualities like energising, perfuming, refreshing, relaxing and vitalising [4]. Other potential applications of MC include skin softeners, vitamins, antimicrobials, insect repellents, dyes, phase change materials and specific medical applications, for example drugs or scientific prevention of burn scars [5 - 9].

Having in the mind that the exploitation of microcapsules in textile has been limited owing to a lack of awareness across the industry and the relative high cost of global processing [10, 11], investigations in this field are particularly required and purposeful. Aromatherapy and aromatherapic textiles are the first choice for anyone who wants to keep healthy in their daily life, and these textiles will become a fashion in the near future [12]. There are various essential oils which have found their place in aroma therapy. The prominent essential oils are lavender, thyme, eucalyptus, peppermint, camomile, etc. [4].

Authors [13] found that the impregnation of knitted fabrics with microencapsulated neroline fragrance and bio-based polyurethane as wall material was successful, and it slowly released the microcapsule content, with neroline remaining until the 20th washing cycle. However, over 50% of polysulfone/vanillin microcapsules were lost after two washings; however, capsules with a diameter of 10 μm remained longer because small capsules got stuck into gaps among the fibres. Besides this it was observed that the loss of the mass of limonene was 46% after one wash and 97% after 20 washes. The main weight changes are due to the binder because a thin layer of binder, which is in direct contact with the textile, may remain for a longer time, retaining some microcapsules impregnated in the fabrics [14, 15]. During the dry cleaning of lab-scale impregnated wool/polyester fabrics, the loss of perfume was 38% in the first cycle and up to 87% after five dry cleaning cycles [16].

For microcapsule adhesion to fabrics, the binder is most important because this material holds capsules on the fabric surface, and the higher the quantity of resin, the more capsules remain on the fabric [13, 17]. The add-ons [11] for cotton fabrics with microcapsules were calculated as a ratio of the specimen after and before treatment. It was determined that as the add-on gets higher, the thermal resistance of the fabrics treated increases, whereas the thermal conductivity decreases. Sh. Li and co-authors [18] established that a sample cured with a conventional hot air tenter frame has high fabric strength retention, low fabric stiffness, and excellent abrasion resistance. Therefore conventional hot air curing is a suitable process for encapsulating a fragrance on cotton. To create a range of conditions that might yield acceptable fixing of capsules with minimum fragrance loss, different combinations of microcapsule treatment need to be investigated, varying such parameters as the binder concentration, curing time, fixing time and temperature. Meanwhile, in other research [19], no resin was used to promote the adhesion of MC to the fabric because the use of resins could interfere in the release of the active agent.

Research [20] confirmed that when different concentrations of microcapsules were used in the padding bath, the quantity of MC that remained on the cotton twill fabric was different. Furthermore the authors detected some deflated microcapsules by SEM analysing the fabric heated at 140 °C. Whereas in the fabric heated to 160 °C MC remained on the fibre, the fragrance had partially gone. The results obtained in [3] indicate that the amount of triclosan introduced onto viscose nonwoven depends on the product of triclosan concentrations in microspheres and on the micro-sphere concentration in dispersion. The physical properties [21] of knitted textiles containing MC with Jojoba oil were investigated. A slight increase in the surface mass was estimated for treated knits, caused by the addition of MC and acrylic resin. The characteristics of cotton textiles with microcapsules [22] depend on the amount of MC deposited onto them in relation to the MC/binder composite ratio. At a low binder amount, most of the MC are coated by the PU binder to form an opened structure at the surface resulting from water evaporation during the cross linking. An increase in the binder amount allows to obtain a smoother film surface with an amount of air still entrapped in the inner structure. Besides this the weft yarn density, weft yarn count as well as cover factors were considered as significant factors that have an important effect on the MC content of woven textile.

Despite the existence of commercial aroma finishing products on the market and tremendous interest of the industry in smart products, there is a lack of research on textiles of more rare fibres and complex structure textiles grafted with microcapsules.

The aim of this work was to investigate the possibilities to graft microcapsules with Eucalyptus essential oil, embedding them into different yarns and varying the thermo-fixing temperature, as well as to research the influence of the binder concentration on the fixation of MC on terry fabrics and their area density.

Experimental

Materials and methods

Spun yarns of five different fibres were selected for impregnation with MC: cotton (50 tex), bleached linen (50 tex), grey linen (68 tex), bamboo (74 tex), and ramie (67 tex). Terry fabrics (***Table 1***) analysed in the experimental work were made from ramie/cotton yarns. The impregnation of yarns and terry fabrics was performed using a concentration of commercial MC containing Eucalyptus essential oil (LJ Specialities, UK). The producer presents general approximate characteristics of MC: solid content – 50%, pH – 6 - 7. The destruction of capsules follows mechanical impacts such as rubbing, scrubbing, etc. The contents of microcapsules of LJ Specialities [23] are released with light abrasion, as would take place during day-to-day wear. The capsules survive repeated washing and can be applied to various fibres.

The impregnation method was applied, as the producer suggests, as well as considering peculiarities of the textile investigated: yarns in strands and terry fabrics in pieces. The treatment protocol consisted in the dispersion of the microcapsules into an aqueous solution containing a dispersant agent and fixing agent. The dispersant agent enabled the obtaining of MC dispersed among the fixing agent. A magnetic stirrer was used for preparation of the solution. Next the strands of yarns and pieces of terry fabrics were soaked in a bath of the aqueous solution for 10 min. The bath treatment for impregnation was a compound of 30 g/l of microcapsules and the binder (ITOBINDER AG, LJ Specialities, UK). A binder concentration of 80 g/l was used for preparation of the yarns treated, and binder concentrations of 20, 35, 50, 65, 80, and 95 g/l were used in the preparation of the terry fabrics treated.

The impregnated samples: yarns in strands and terry fabrics in pieces were pre-dried in a loose shape at 20 ± 2 °C temperature, dried at 105 - 110 °C (for 1 min) and thermo-fixed. Different fixation temperatures of 120, 130, 140, 150, 160 and 170 °C (for 2 min) for yarns and 150 °C (for 2 min) for terry fabrics were used. Heating was performed in a laboratory oven - SNOL 20/300 LFN (Lithuania).

All the samples were conditioned under standard atmospheric conditions: 20 ± 2 °C temperature and 65 ± 4% relative humidity for 24 h before the experiments [24]. The mass and linear density of the yarns [25] as well as the area density of the terry fabrics [26] were determined using electronic scales - KERN EW 150-3M (KERN & Sohn GmbH, Germany) at an accuracy of 0.001 g. The surface and morphology of microcapsules as well as the yarns and terry fabrics treated were examined by scanning electron microscopy (SEM) - Hitachi S-3400N (USA) and Quanta 200 FEG (USA), at an acceleration voltage of 5 - 20 kV.

Results and discussion

The capsules containing the natural aroma of flowers, fruit, herbs, perfumes, etc., break on the movement of the wearer, releasing the aroma.

Traditionally the fixation of MC onto textiles is achieved by applying a binder and the thermal curing procedure, during which the binder is converted into a tough polymer that forms a network on the textile to hold the microcapsules. The capsules continue to emit the active agent for up to 10 - 25 washing cycles [22, 23]. The neroline fragrance component decreased slowly after washing of the cosmeto-textile developed, with up to 30% of neroline remaining on the fabrics after the 20th washing cycle [13]. Besides this with a proper amount of the binder, it is possible to hold microcapsules containing the aloa vera agent after 20 washing cycles [27]. In general, the release rates of *citrus unshiu* oil [28] from cotton textile were low enough, hence the textile could be assumed to let little fragrance out despite repeated launderings. Though the fixation of MC is necessary, at the same time it is possible that high thermo-fixation temperature can damage the shell of the microcapsule, with the result that its functionality can decline.

The yarn samples grafted with Eucalyptus microcapsules were analysed by SEM, which was a good technique for other qualitative research [3, 20, 29]. Researches in [3] confirmed that the effect of changing the concentration of micro-spheres in dispersion can be easily observed from SEM micrographs. Moreover at concentrations above 5% the phenomenon of micro-sphere aggregation occurs, which causes weaker bonds amongst micro-spheres with fibres. When the curing temperature was 70 °C [30], the break phenomena of MicroPCMs and adhesion increased. SEM images of impregnated textile [31, 32] also confirmed the adhesion between fibres and microcapsules.

A mean size of 1.465 μm for Eucalyptus microcapsules was determined by SEM

Table 1. *Characteristic of terry fabrics.*

Fabric variant			Ramie/cotton				
			R_1	R_2	R_3	R_4	R_5
Characteristic	Fabric density, cm^{-1}	Pile and ground warp	25				
		weft	8	10	12	14	16
	Nominal linear density, tex	Pile warp	67, ramie				
		Ground warp	25x2, plied cotton yarn				
		Ground weft	50, cotton yarn				
	Pile high, mm		6				

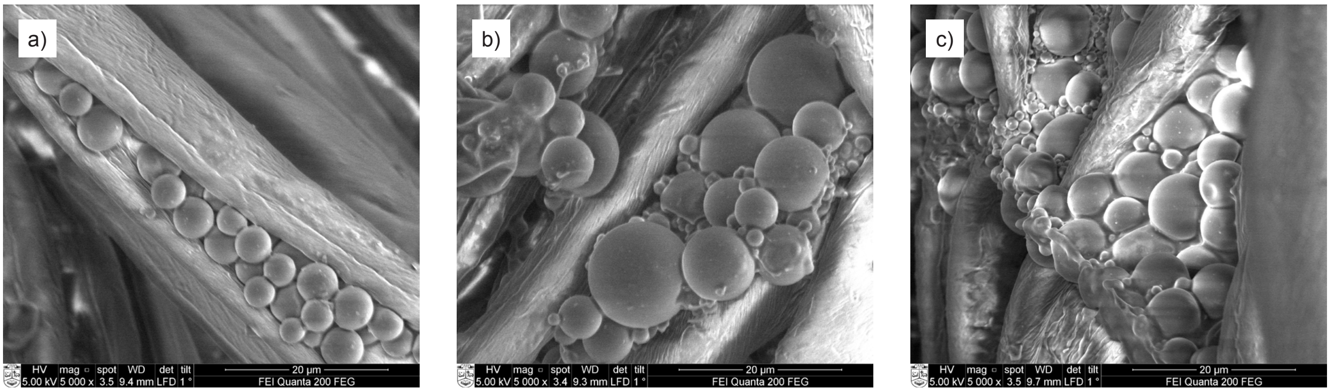

Figure 1. *SEM micrographs (5000× magnification) of cotton yarn after treatment with MC at thermo-fixing temperature: a) 120 °C, b) 150 °C, c) 170 °C.*

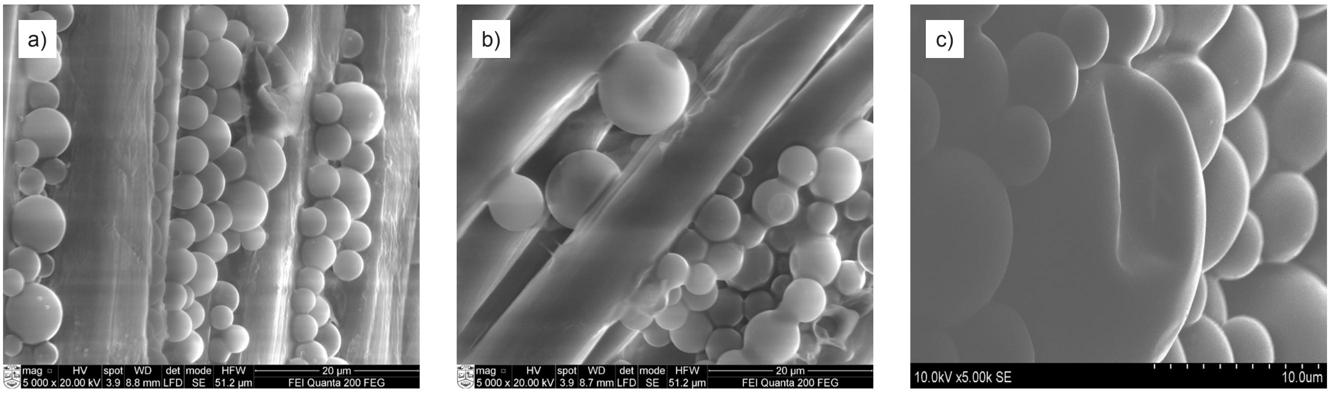

Figure 2. *SEM micrographs (5000× magnification) of bleached linen yarn after treatment with MC at thermo-fixing temperature: a) 120 °C, b) 150 °C, c) 170 °C.*

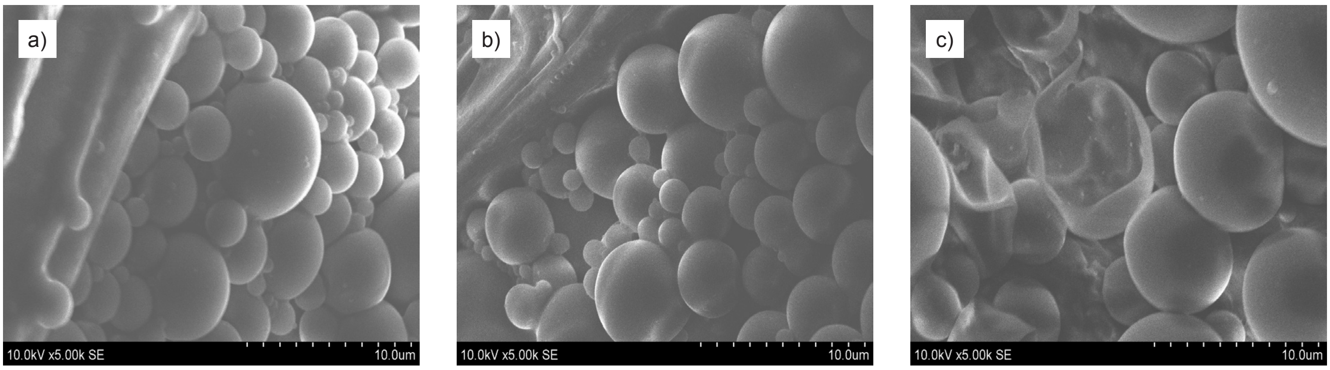

Figure 3. *SEM micrographs (5000× magnification) of grey linen yarn after treatment with MC at thermo-fixing temperature: a) 120 °C, b) 150 °C, c) 170 °C.*

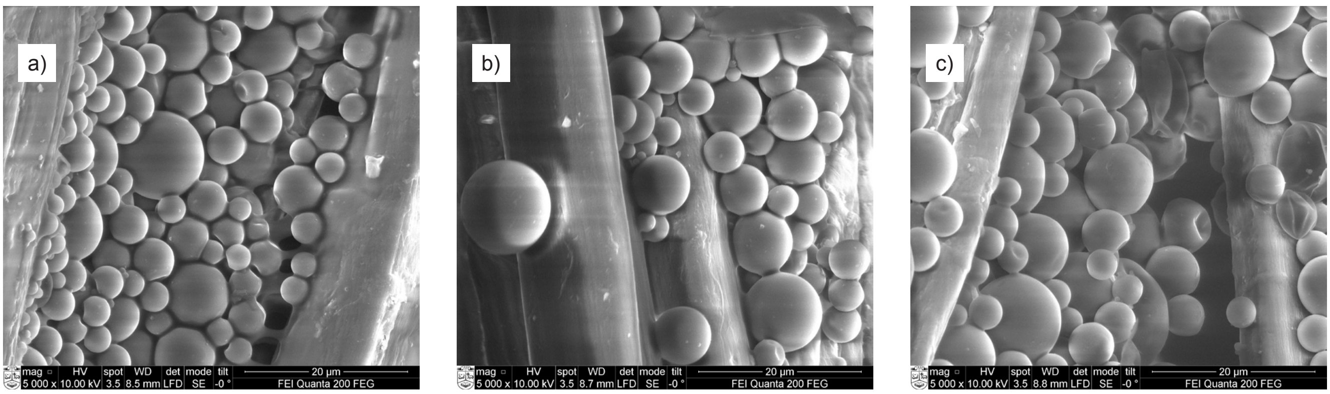

Figure 4. *SEM micrographs (5000× magnification) of ramie yarn after treatment with MC g at thermo-fixing temperature: a) 120 °C, b) 150 °C, c) 170 °C.*

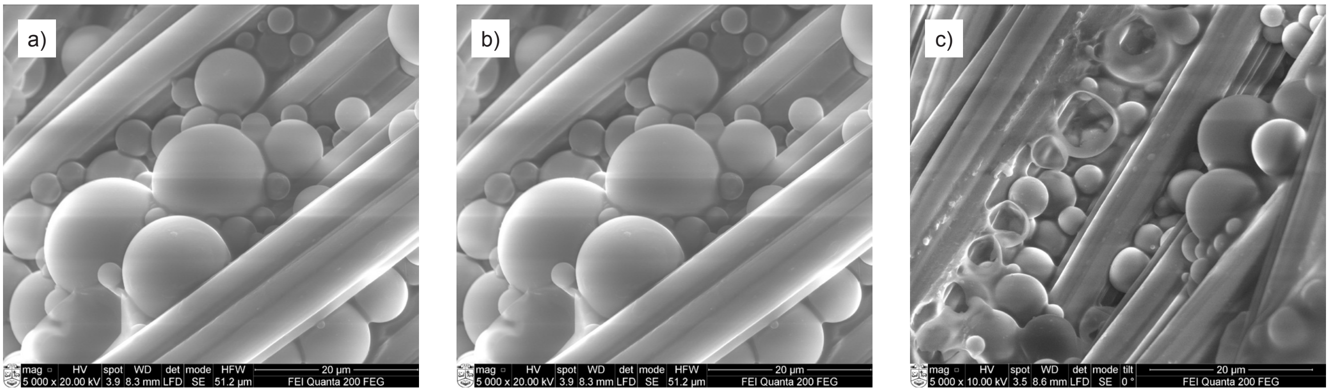

Figure 5. *SEM micrographs (5000× magnification) of bamboo yarn after treatment with MC at thermo-fixing temperature: a) 120 °C, b) 150 °C, c) 170 °C.*

using image analysis software LUCIA - VGA version 5.00 (Czech).

SEM micrographs (5000× magnification) with MC adhered on cotton, bleached linen, grey linen, ramie, and bamboo yarns are shown in ***Figures 1 - 5***. It could be seen that impregnation for all yarns and microcapsules investigated was effective on the surface of fibres and in the deeper layers between fibres as well.

The presence of apparently unaltered Eucalyptus MC could be seen on the yarns investigated when they were treated with a thermo-fixing temperature of 120 - 140 °C. As is evident from ***Figures 1.a - 5.a***, the microcapsules are of very regular spherical shape, without any deformations or changes in their shell. The authors in [20] also confirmed the fact that there were no transformations in peppermint microcapsules applied on cotton fabric heated by air at 120 °C. Additionally SEM analysis showed that with an increase in the temperature of up to 160 °C, the swelling was reduced considerably. We determined a similar phenomenon. A thermo-fixing temperature of 120 - 150 °C caused no metamorphosis in MC on yarns of various fibres since the soft and smooth surface of Eucalyptus microcapsules could be observed. Also it is noticed that generally the microcapsules are distributed individually without substantial agglomeration. As surfaces of linen (grey and bleached) and ramie fibre are not smooth enough, microcapsules are deposited on the specific contact surface. Because of their specific structure, cotton fibres adhered to MC not only on the surface and between fibres but also in the inner fibre cavities and sags (see ***Figure 1.a***). Bamboo fibres were also well fastened to the MC, but the smooth surface of the fibres conditioned the wrapping character of the fixation. ***Figures 1.b - 5.b*** show that few deformed MC could be seen when the thermo-fixing temperature was increased up to 150 °C. Meanwhile ***Figures 1.c - 5.c*** show yarns heated to 170 °C, in which surface damage to MC is clearly noticed, being a typical appearance for many items; some MC were deflated and cannot be observed as spheres. Besides this some microcapsules were broken entirely and became empty after such a procedure, confirming that MC remained on the fibres and even between them; but the fragrance had partly gone and in single cases even totally.

From the SEM analysis, it was also determined that with an increase in the thermo-fixing temperature, generally the larger microcapsules were damaged faster and than smaller ones hardly at all. It is very important to investigate what changes happen in the fibre mass after treatment with MC and the binder. The add-on as a significant index for textile impregnated with microcapsules was also analysed by other authors [11, 19]. Investigating the amount of capsules and active agents in fibres and quantifying them by the weight difference between untreated and treated fabrics, it was found in [19] that when liposomes were applied onto fabrics, there were differences of about 4% between the calculated amount of product impregnated (~22%) and the product found in the fabric after heating in a Stenter (~18%). Furthermore it seems that cotton fabric incorporates a lower percentage of liposomes, whereas polyamide, acrylic and polyester have similar percentages of product applied. While according to [29], results showed that regardless of the type of nonwoven defined by the technique of its making and the incorporation procedure, modification with micro-spheres does not change the thickness and surface mass of the textile. An insignificant increase in the fabric surface mass was observed in the case of increased sphere concentration in the modifying bath (spun bonded PET, 2.5 and 5.0% of micro spheres).

Our study shows that there is an increases in the mass (Δm) of cotton, grey linen, bleached linen, ramie and bamboo yarns after the grafting procedure at different thermo-fixing temperatures (***Figures 6 & 7, see page 52***). We found that the mass of cotton yarns after treatment changed by 2.7 - 3.9%. Meanwhile, the changes in the mass of bleached linen and ramie yarns are by even 7.1 - 17.3% and in 10.1 - 13.0%, respectively. The Δm of bleached linen and ramie yarns is distinguished by the highest increase at temperatures of the thermo-fixing of 160 and 140 ºC, respectively. Whereas the Δm for cotton yarns has the lowest value (2.7%) compared with other raw materials at a thermo-fixing temperature of 170 ºC. Additionally a substantial difference in Δm was found analysing both linen yarns: the Δm of bleached linen yarns was 11.1% higher compared with grey ones when the thermo-fixing temperature was 160 °C.

The microcapsule shell cannot react with the surface of the fibre, hence a binder is used to attach MC to the fibre surface, making a thin layer which holds capsules for a longer time. Since the adds-on of ramie yarns were found to be higher compared to grey linen, bleached linen, and bamboo yarns and bearing in the mind the well-known advantages of ramie for loop making (durable, highly absorbent, comfortable to wear, especially in a warm and damp environment, resistant to rotting and mildew, withstands high water temperatures during laundering, etc.) and the popularity of cotton for ground sets, the ramie/cotton fibre combination was used for weaving terry fabrics. In our experiment, the terry fab-

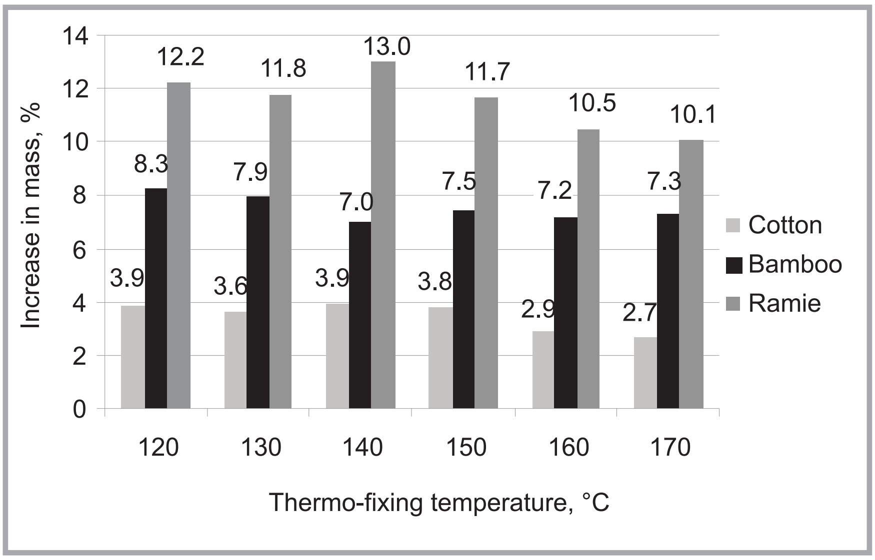

Figure 6. Increase in mass of cotton, bamboo and ramie yarn after treatment with MC at different thermo-fixing temperatures.

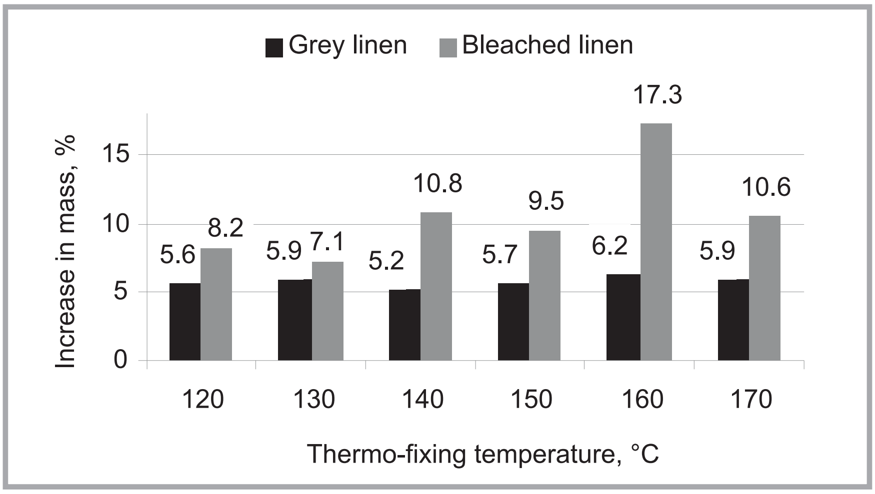

Figure 7. Increase in mass of grey linen and bleached linen yarn after treatment with MC at different thermo-fixing temperatures.

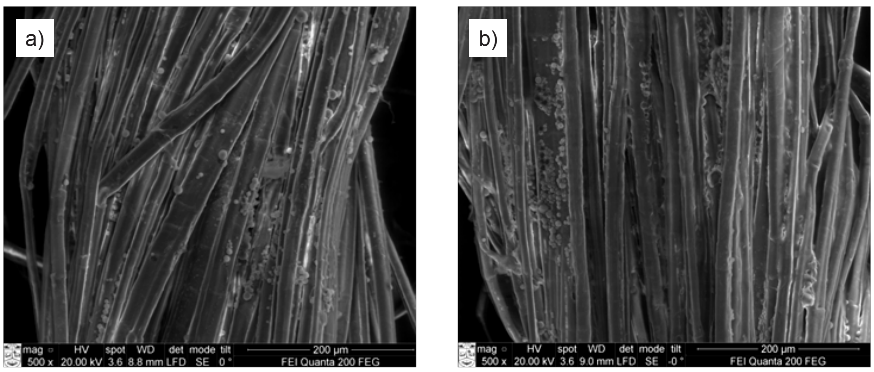

***Figure 8.** SEM (500× magnification) of terry fabrics with weft density of 16 cm-1 impregnated with MC using different binder concentrations: a) 20 g/l, b) 95 g/l.*

rics were treated with MC using different binder concentrations. ***Figure 8*** presents the SEM of terry fabrics with a weft density of 16 cm^{-1} impregnated with MC using different binder concentrations. Photos of these fabrics show a difference in the MC applied when the binder concentration was 20 and 95 g/l, respectively. Generally it is notable that between the yarns of both fabrics there are no spaces without microcapsules. Nevertheless as more of the binder was put into the bath solution, the amount of microcapsules bound on the terry fabric tended to grow as well. Besides this, aggregations occur, which conditioned less secure bonds amongst microcapsules with fibres, and hence a not very uniform distribution of microcapsules in the terry fabric. Other authors [17] also investigated the influence of binder concentration on the adhesion of microcapsules with fragrance to cotton fabric. They concluded that with a higher quantity of resin, more microcapsules remain on the fabric surface. When 20 washing cycles were applied to the bleached cotton fabric, samples without a binder did not show MC in SEM micrographs. When either 5 or 10 g/l was used to paste the microcapsules, the fabric still presented MC after 20 washing cycles. Our results of the area density of terry fabrics before and after microencapsulating are presented in ***Figure 9.*** It was found that the highest increases in the area density of terry fabrics (in 23.08 and 27.11%) was estimated when the binder concentration was 80 g/l and 95 g/l, respectively, for samples with a weft density of 14 cm^{-1}. The minimum increase (8.18%) in the area density was determined for terry fabric with a weft density of 12 cm^{-1} and treated using a binder concentration of 35 g/l. The increase in the area density of terry fabrics with an increase in the binder concentration was determined as a typical tendency.

The experimental results of the area density were mathematically evaluated at a 95% confidence level. It was determined that the area density of terry fabrics increased till 282.9 - 486.3 g/m^2 while investigating terry fabrics of various weft densities and impregnated with a binder concentration of 20 g/l. During treatment with a binder concentration of 95 g/l, the area density of terry fabrics increased till 292.6 - 510.3 g/m^2. Regression analysis was performed and the informativity of the design was investigated for the area density of the terry fabrics treated in relation to the weft density and varying binder concentration. Using the criterion of informativity (criterion of R. A. Fisher), it was established that the mathematical models received that express the dependence between the area density of the terry fabrics treated and the weft density are informative for a binder concentration of 20 g/l and 95 g/l. To describe the results, various types of regressions were analysed. The equations with the highest determination coefficients were analysed and interpreted

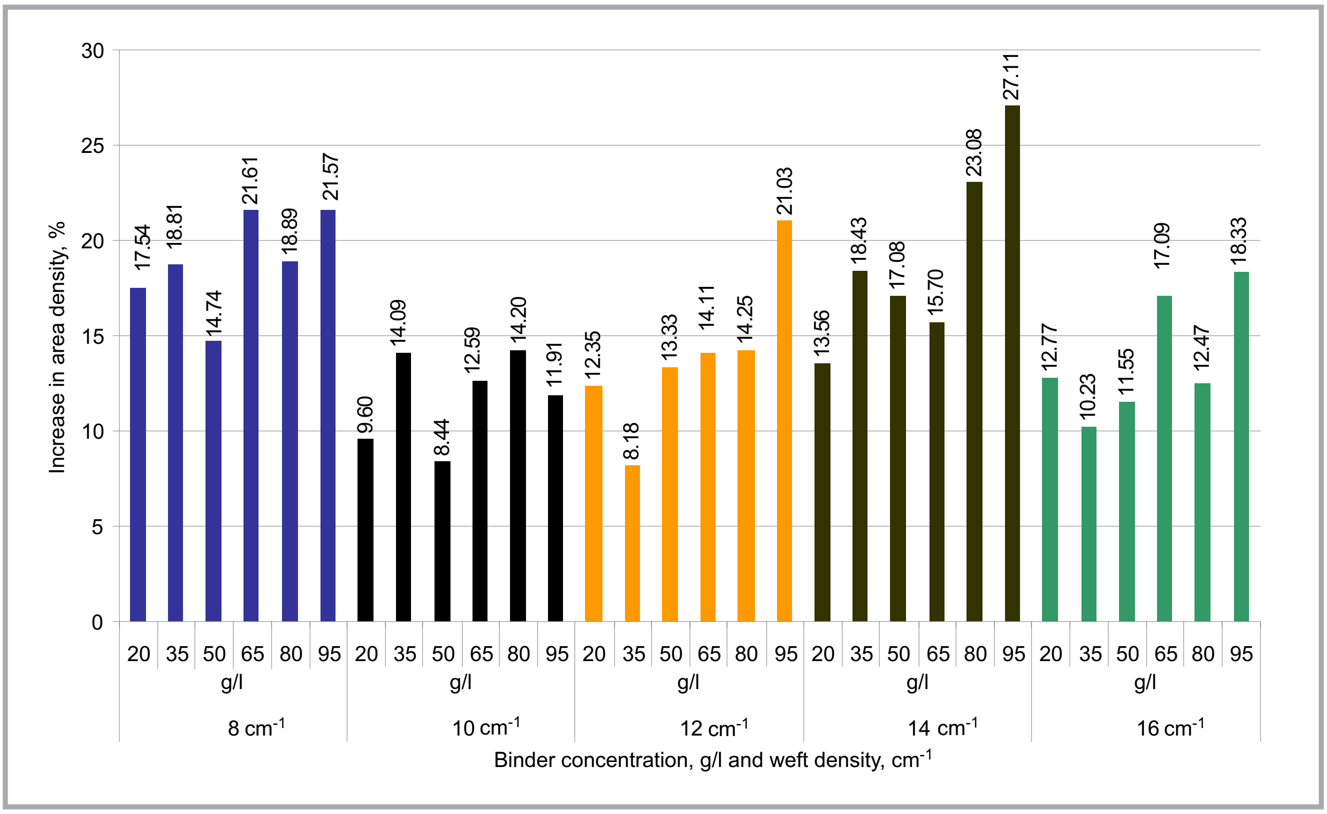

***Figure 9.** Increase in area density of terry fabrics after treatment with MC at different binder concentrations.*

further. The area density of the terry fabrics treated in relation to the weft density is shown in ***Figure 10*** as a linear type of regression. The determination coefficients (R^2 = 0.8819 and R^2 = 0.8937) showed that a very good relation between the parameters investigated exists.

Conclusion

1. Adding microcapsules with Eucalyptus essential oil to various yarns after treatment at different thermo-fixing temperatures results in effective impregnation and confirmed the spherical morphology and smooth surface of microcapsules. SEM images confirmed that microcapsules were deposited on the yarn's surface and into deeper levels of the terry fabric's structure.
2. The microcapsules were linked to the surface of the fibres, wrapped by the binder or by creating a linkage with the binder and fibres. The behaviour of the binder on the fibres investigated varied. On cotton the binder tended to coat the cavities of fibres, and microcapsules adhered not only to the surface and between fibres but also in the inner fibre cavities and sags, as the structure of cotton fibre has a very peculiar geometry of twisted flat tube. Grey and bleached linen fibres, because of their specific contact surface, also showed adhered microcapsules and their distribution between fibres. The smooth surface of bamboo fibres conditioned the wrapping character after treatment with microcapsules. On the ramie fibres, the binder with microcapsules enveloped the fibres homogeneously inside the fibres and between them.
3. Until 150 °C the thermo-fixing temperature conditioned the impregnation of microcapsules, which retained a very regular spherical shape, without any alterations. After the impact of higher thermo-fixing temperatures, the microcapsules that were partly or entirely damaged were the larger ones.
4. The bleached linen and ramie yarns grafted with microcapsules have the highest increase in mass (in 17.3 and 13.0%) at a thermo-fixing temperature

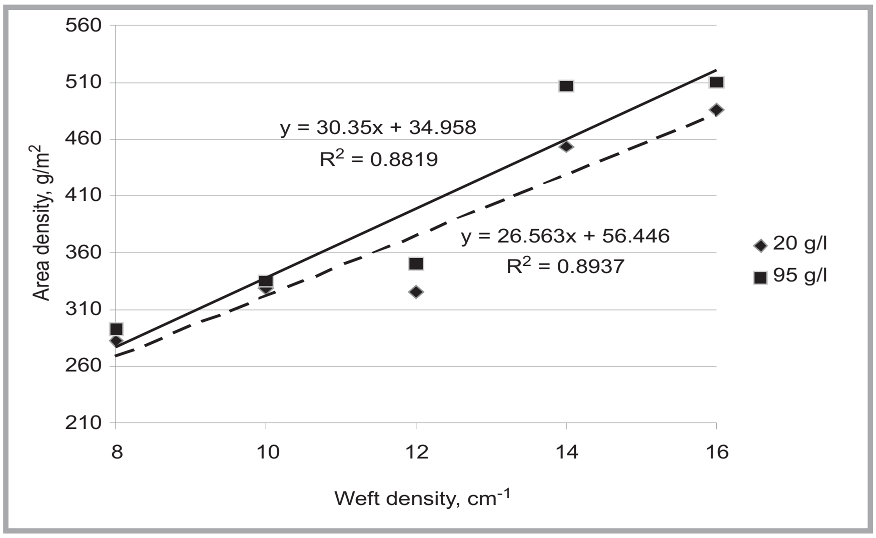

***Figure 10.** Area density of terry fabric samples after treatment with MC at different binder concentrations: 20 g/l and 95 g/l in relation to the weft density of the fabric.*

of 160 °C and 140 °C, respectively, while for cotton yarns this index has the lowest value of 2.7 - 3.9% compared with all other fibres investigated. The increase in mass of bleached linen yarns was even 3.8 - 11.1% higher compared with grey ones (at a thermo-fixing temperature of 140 - 170 °C), confirming the better impregnation of binder and microcapsules.
5. The highest increase in the area density of impregnated ramie-cotton terry fabrics of 27.11% was estimated after treatment with a binder concentration of 95 g/l for samples with a weft density of 14 cm^{-1}. It was demonstrated there exists the general tendency for almost all variants that the area density of ramie/cotton terry fabrics increases when the binder concentration increases as the binder homogeneously filled inter-yarns and inter-fibres spaces.
6. The area density of the terry fabrics treated in relation to the weft density could be described by linear equations with the existence of a very good relation between the parameters investigated .

Further studies of the binder/microcapsule/textile system are in progress to assess the behaviour of treated terry fabrics in contact with liquid.

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