

# A comparison between padding and bath exhaustion to apply microcapsules onto cotton

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Received: 21 October 2014 / Accepted: 7 March 2015 / Published online: 14 March 2015  
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**Abstract** The use of Microcapsules has increased in the textile sector. They have been applied as a possible means of introducing new products to textiles, such as insect repellents, antibiotics, skin moisturizers, etc. Microencapsulation technology has improved the fragrance durability on fabrics. Historically, the durability of the fragrance was poor, especially once the fabric had been washed. Microcapsules have been used in textiles for many years, however their previous characterization, adhesion behaviour and permanence on the fabrics are not well known. Nowadays the majority of textile industries are not able to characterize commercial products, or to study the process of adhering the microcapsule to the fibre's surface nor their functionality. Thus, the characterization of microencapsulated fabrics with different active core and the knowledge of the various application processes becomes a major challenge in the field of microcapsules use. There are various industrial processes to apply microcapsules, but determining optimal amounts of products, temperature, conditions and other process variables are an important challenge for the textile sector in order to achieve the highest depositions and retention of microcapsules. This work is focused on

determining and quantifying presence fragrance microcapsules when applied onto fabrics by padding and by bath exhaustion and determining which method is the most effective. Consequently, diverse analysis techniques such as microscopy (SEM), spectroscopy FTIR and XPS have been used. We concluded that proposed techniques seem to be useful to compare fabrics treated with microcapsules. Results demonstrate that padding application gives better yields than bath exhaustion.

**Keywords** Microcapsule · Padding · Bath exhaustion · Cotton

## Introduction

Textile fibres are commonly used everyday not only in fashion but they are the basis of home textiles and other technical applications such as medical care, thermal protection, etc. The modification of fibres or fabrics is an important method to provide textiles with improved properties. It is becoming increasingly difficult to ignore the benefits of functionalizing the fibres.

The more developed countries use microcapsules in different fields, including textiles. Their purpose is to confer new properties and added value to the fabrics, for example antibiotics in medical fabrics and phase change materials (PCM's) for technical textiles. It has

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encouraged the industry to use microencapsulation processes as a means of imparting finishing and properties to textiles which were not possible or cost-effective using other technologies (Nelson 2002).

Textile manufacturers are concerned about the increasing interest in the application of new properties such as durable fragrances and skin softeners to textiles. Other potential applications include insect repellents, dyes, vitamins, antimicrobials, phase change materials and specific medical applications, antibiotics, hormones, and other drugs (Nelson 1991, 2001, 2002; Monllor et al. 2007; Miro Specos et al. 2010; Gisbert et al. 2009).

Microcapsules present an active core, which is protected by means of an external polymer. The composition of microencapsulated products can be different because they can be made of different shell materials and diverse core materials. The core material will define the main property to add and consequently the use, i.e. medicine, food, etc. There are many types of shell, however, it is not common to find reactive polymers which can react with a fibre's surface.

Microencapsulated products can be applied to fabrics by impregnation, bath exhaustion, foaming, spraying and coating. The most extended industrial application is padding. In order to adhere microcapsules to fabrics, they should be in contact with a bath, which contains microcapsules, resin and water. The resin allows the microcapsules to be adhered to the fabrics' fibres.

The presence of microcapsules is usually measured by the existence of a property such as odour measurements when flavours are encapsulated. Nevertheless, some properties such as hydration, insect repellence, antibacterial effect, etc., cannot be tested without analytical methods. Some characterization techniques have been applied in order to study microcapsule products, presence and state (Monllor et al. 2007, 2009, 2010; Gisbert et al. 2009; Hong and Park 1999; Rodrigues et al. 2008, 2009; Jing et al. 2011; Bonet et al. 2012).

Scanning electron microscopy (SEM) has been commonly used for many years to characterise the microcapsules state and presence on fabrics (Nelson 1991, 2001, 2002; Hong and Park 1999). The Fourier transform infrared spectroscopy (FTIR) is used as a method of quantifying the microcapsules presence on the fabrics' surface (Monllor et al. 2007, 2009). And X-ray photoelectronic spectroscopy (XPS) is a surface

chemical analysis technique that allows to detect microcapsules on fabric's surface when a shell is composed of a polymer with nitrogen in its structure (Bonet et al. 2012). Both techniques (FTIR and XPS) seem to be complementary to microscopy.

In this work microcapsules have been applied by two different methods; padding and bath exhaustion. SEM, FTIR and XPS techniques have been used in order to determine microcapsules presence on the fabrics' surface and the effectiveness of the process used to apply microcapsules onto fabrics.

## Materials and methods

### Materials

Microcapsules (CENTERFINISH 164/02 LAVANDA) were supplied by COLOR CENTER (Tarrasa, Spain). According to the provider specifications the wall material was melamine formaldehyde, and the microcapsules contained lavender fragrance. The dry weight of the microcapsules slurry was 42 %. In order to bond the microcapsules to fabrics, an acrylic resin, Color Center BC, was applied, also supplied by COLOR CENTER.

The fabric was a 100 % cotton twill, with 210 g/m<sup>2</sup>, which had been chemically bleached with peroxide in an industrial process.

### Microcapsule application procedure

Commercial microcapsules were applied to the surface of the fabric by padding or by bath exhaustion. A resin was used as a binder. For this reason thermal treatment in the form of hot air was applied to cure the resin and to induce adhesion of the microcapsules to the fabric.

For padding, we used a 2608 TEPA horizontal foulard of 1 kW, its work was performed at a speed of 2 m/min and cylinder pressure of 1.5 kg/cm<sup>2</sup>, it allowed to guarantee a pick up between 85 and 90 %. Bath treatments had been studied previously (Monllor et al. 2007, 2010), and they were composed of different concentrations of microcapsules and resin. The use of a resin as a binder implied a thermal treatment in the form of hot air in order to cure the resin and adhere the microcapsules to the fibres' surface. Samples were thermally fixed in a scale pin

stenter and dried at 110 °C for 10 min in WTC BINDER 030. Previously, it has been demonstrated that this temperature was high enough to polymerise the binder without evaporating the fragrance (Monllor et al. 2010). Table 1 summarises the padding conditions.

In the bath exhaustion application, some cotton samples (20 g) were treated for 40 min in an opened reactor with liquor ratio of 1/20. Different concentrations of microcapsules and resin were used. Influence of the baths' temperature was tested (60 and 80 °C). Samples were thermally dried in the same conditions than padded samples.

#### Gravimetric analysis

In order to determine if the application of microcapsules increase the weight of the treated samples, they were weighted in a Mettler Toledo balance. Cotton fibres are known because of its higroscopicity, in other words, they can absorb moisture from the environment. Consequently, the weight of each sample can vary depending on the temperature and moisture in the laboratory. Samples were introduced in flasks and dried for 120 min in a heater at 100 °C, to evaporate all the moisture from the fibres. Then while the flasks were in the heater, they were closed and weighted ( $W_1$ ). Afterwards flasks without the fabric were weighted ( $W_0$ ) and the difference in weight was attributed to the weight of the treatment.

$$T_x = W_1 - W_0$$

where  $T_x$  is the sample from the test X, according to Tables 1 and 2.

#### Scanning electron microscopy (SEM)

For surface observation, a scanning electron microscope, Phenom microscope, was used (FEI Company, Hillsboro, OR, USA). 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.

#### Fourier transform infrared spectroscopy (FTIR)

BRUKER IFS 66/S FTIR spectrometer was used to analyse the spectrum. Resolution for the infrared spectra was  $4 \text{ cm}^{-1}$ , and there were 64 scans for each spectrum. Spectra were collected in ATR mode.

#### X-ray photoelectric spectroscopy (XPS)

The XPS spectra have been obtained with a VG-Microtech Multilab Electron Spectrometer, by using the Mg/K//a/ (1253.6 eV) radiation of twin anode in the constant analyser energy mode with pass energy of 50 eV. The Pressure of the analysis chamber was maintained at  $5 \times 10^{-10}$  mB. The binding energy (BE) and the Auger kinetic energy (KE) scales were regulated by setting the C1s transition at 284.6 eV. The accuracy of BE and KE values was  $\pm 0.2$  and  $\pm 0.3$  eV, respectively. The BE and KE values were obtained by using the Peak-fit Program implemented in the control software of the spectrometer. XPS measurements were collected from five different zones of the fabric.

## Results and discussion

#### Application procedure: scanning electron microscopy (SEM)

Microcapsules were applied onto fabrics by padding or by bath exhaustion. Both procedures were performed with the same recipe, 60 g/L of microcapsules and 10 g/L of binder. However, regarding to bath exhaustion treatments it is not commonly used the concentration in terms of g/L but in % over weight of fibre (% owf). Figure 1 compares SEM images after padding (Fig. 1a, test 2) and bath exhaustion application (Fig. 1b, test 9). It is well known that SEM allows detection microcapsule presence, location and condition. Figure 1 shows microcapsules, which have a spherical shape, which means the fragrance is inside the polymeric shell. It can be easily observed that for the same microencapsulated commercial product there are microcapsules with different sizes. It must be noticed microcapsules tend to be located in the

**Table 1** Padding conditions

	Test 1	Test 2	Test 3
Microcapsules concentration (g/L)	60	60	–
Binder concentration (g/L)	–	10	10
Drying temperature (°C)	110	110	110

**Table 2** Bath exhaustion conditions

	Test 4	Test 5	Test 6	Test 7	Test 8	Test 9	Test 10
Microcapsules concentration (owf)	12	12	24	24	240	240	240
Binder concentration (owf)	–	2	–	4	–	40	–
Bath temperature (°C)	60	60	60	60	60	60	80
Drying temperature (°C)	110	110	110	110	110	110	110



**Fig. 1** SEM micrographs of cotton fabrics with microcapsules. **a** After padding treatment (60 g/L microcapsules and 10 g/L of resin). **b** After bath exhaustion treatment (240 % owf microcapsules and 40 % owf resin) temperature process 60 °C

grooves formed by kidney section of the cotton fibres. The resin presence is significant in both micrographs, its presence allows the microcapsules adhesion to the fibres' surface. When Fig. 1 is analysed, differences between Fig. 1a and b can be observed. As the treatment has been conducted with the same recipe, the variation in the amount of microcapsules between cotton fabrics differs according to the application process. In Fig. 1b (bath exhaustion application) it can be observed more microcapsules and resin presence than in the Fig. 1a (padding application).

Figure 1 confirms the differences between the two processes used to apply microcapsules onto fabrics. The concentration for the treatments was 60 g/L of microcapsules and 10 g/L of resin what corresponds to 240 % owf of microcapsules and 40 % owf of resin. These amounts can be considered excessive for the bath exhaustion process. Commercial brand's recipe suggests using between 6 and 12 % of microcapsules and 1–2 % of resin with a temperature process below

70 °C. When working with microcapsules temperature treatments should be as lower as possible; otherwise active product can be damaged however, some researchers demonstrated that for the fragrances used in this study microcapsules could bear up to 110 °C. (Bonet et al. 2012; Monllor et al. 2009).

#### Gravimetric characterization

In order to determine the effect of the treatment in the weight of the samples, they were analysed in two groups depending on the recipe. The first group was comprised of the cotton fabrics treated with microcapsules but without any binder (tests 4, 6, 8, 10). The second one included the binder in its composition (tests 5, 7, 9). Each sample was compared with the weight of the cotton fabric without any treatment. Table 3 shows the results for the ratio  $W_x/W_{\text{cotton}}$ . Where  $W_{\text{cotton}}$  corresponds to the weight of the cotton fabric without any treatment.

**Table 3** Weight variation

Samples X	Test 4	Test 6	Test 8	Test 10	Test 5	Test 7	Test 9
$W_x/W_{\text{cotton}}$	1.0749	1.1142	1.3016	1.2111	1.1476	1.1892	1.2804

The established ratio shows values higher than 1. This implies the applied process adds weight to the cotton fabric, as it should be expected. When samples for the first group (T4, T6, and T8), or the samples for the second one (T5, T7, and T9) are observed, the weight increases as the quantity of microcapsules (first group) or microcapsules and resin (second group) increases in the recipe.

However, when the ratio is studied for samples T8 and T10 it should not have changed as the recipe is the same and the difference is the temperature in the treatment. The ratio for sample 10 is lower than the one for sample 8. Having the sample dried at 80 °C reduces the weight in the sample what can be attributed to the degradation in the microcapsules because of the temperature, what contradicts what had been previously suggested (Bonet et al. 2012; Monllor et al. 2009). Consequently, drying samples can vary the samples' weight and this process is not accurate enough to establish a comparison between samples.

#### Fourier transform infrared spectroscopy (FTIR-ATR)

##### *Microencapsulated commercial products characterization*

Commercial brand fixed the shell composition as melamine formalin. Figure 2 shows the FTIR-ATR spectrum of the commercial microencapsulated product with lavender fragrance. It presents two characteristics broad bands, one centred on 3700–3000  $\text{cm}^{-1}$  attributed to the stretching vibration of the hydroxyl and amine groups (Monllor et al. 2007, 2009; Rodrigues et al. 2009; Sócrates 1997; Wilson and Pffol 2000; Zhang and Wang 2009), and the other centred on 1650  $\text{cm}^{-1}$ , it can be related to CO stretching vibration in the amide group (Monllor et al. 2007; Rodrigues et al. 2009; Sócrates 1997; Wilson and Pffol 2000; Zhang and Wang 2009).

The information found in the libraries of the FTIR equipment relating to melamine formaldehyde spectra

as well as that provided by previous studies (Monllor et al. 2007, 2009; Rodrigues et al. 2009; Sócrates 1997; Wilson and Pffol 2000; Zhang and Wang 2009) confirm that FTIR is similar which corroborates that the microcapsules wall is melamine formalin.

##### *Microcapsules concentration on fabrics*

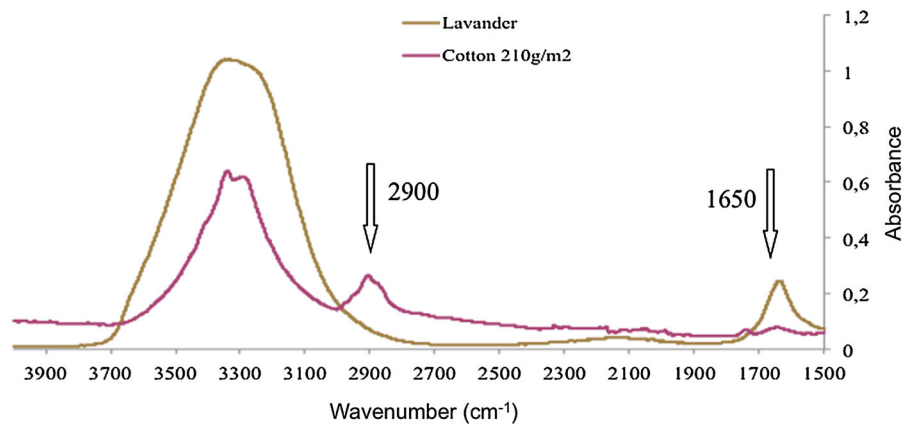
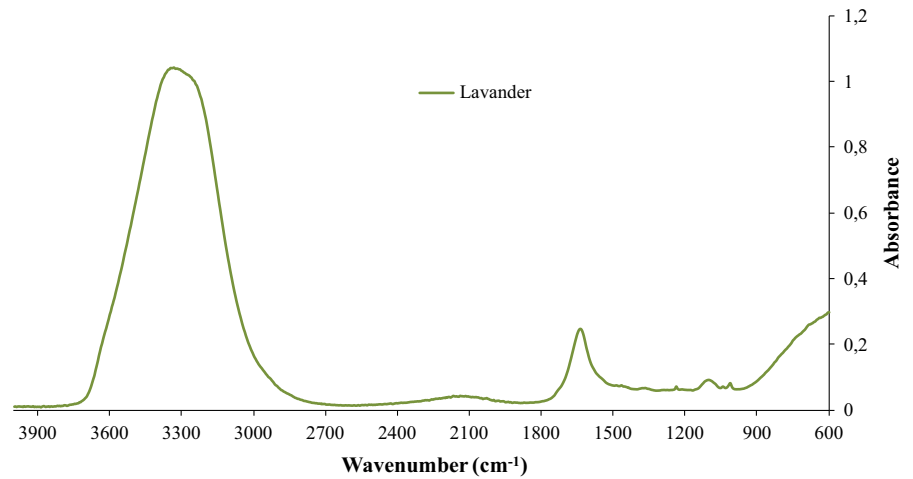
In order to observe differences between cotton and microcapsules, Fig. 3 compares the infrared spectra of the commercial microencapsulated product and the one from the fabric before applying microcapsules.

Cotton fabric shows two broad bands, one at 3300  $\text{cm}^{-1}$  attributed to the stretching vibration of the hydroxyl group of cellulose (Monllor et al. 2007, 2009; Sócrates 1997; Kokot et al. 2002; Bonet et al. 2004; Kondo et al. 1994), and the other at 2900  $\text{cm}^{-1}$ , it can be assigned to the CH stretching vibration (Sócrates 1997; Kokot et al. 2002). Comparing both spectra it is observed that both the fabric and the commercial products have OH groups, 3700–3000  $\text{cm}^{-1}$  region, so that when applying microcapsules on the fabric they provide more OH functional groups (Monllor et al. 2007).

There are two bands, which do not present the same behaviour. The band centred at 2900  $\text{cm}^{-1}$  (CH stretching vibration) from cotton spectrum and the one centred at 1650  $\text{cm}^{-1}$  (CO stretching vibration) from lavender microcapsules spectrum. Thus, the fabric presents a characteristic band in the 2900  $\text{cm}^{-1}$  region while the commercial product shows no variation at this region, on the other hand, the commercial product at 1650  $\text{cm}^{-1}$  has a characteristic band whereas the cotton fabric reaches the baseline.

Taking into consideration the behaviour of the cotton fabric and the commercial products in these two regions, each sample was analysed by the absorbance values in these two regions, calculating the intensity ratio “ $I_{1650}/I_{2900}$ ”.

Table 4 shows the results of this ratio for fabrics under study in order to determine if there are differences between the application procedure (bath exhaustion–padding).

**Fig. 2** Infrared spectra of microencapsulated product**Fig. 3** Infrared spectra of microencapsulated product and a cotton fabric**Table 4** Intensity ratios: FTIR-ATR analysis

Reference	Process	Bath composition	$I_{1650}/I_{2900}$
Cotton sample		–	0.320
Test 1	Padding	60 g/L microcapsules	0.327
Test 2	Padding	60 g/L microcapsules 10 g/L resin	0.334
Test 3	Padding	10 g/L resin	0.328
Test 4	Bath exhaustion 60 °C	12 % owf microcapsules	0.319
Test 5	Bath exhaustion 60 °C	12 % owf microcapsules 2 % owf resin	0.324
Test 6	Bath exhaustion 60 °C	24 % owf microcapsules	0.315
Test 7	Bath exhaustion 60 °C	24 % owf microcapsules 4 % owf resin	0.334
Test 8	Bath exhaustion 60 °C	240 % owf microcapsules	0.329
Test 9	Bath exhaustion 60 °C	240 % owf microcapsules 40 % owf resin	0.334

The cotton sample without any treatment shows lower values as compared with other treated samples. However, apparently it seems there is not a clear tendency on the ratio values. When analysing the fabric bath composed only by resin (test 3), the value is higher than the untreated cotton fabric and than the fabric from the bath composed by microcapsules (test 1), confirming the contribution of functional groups to the fabric by the resin. A thorough analysis reveals that tests 1, 4, 6, 8 should be studied apart from tests 2, 5, 7, 9, as the baths' composition differs not only on the concentration of microcapsules but on the resin content as well. The first group is composed of microcapsules and the second one apart from microcapsules includes resin. The presence of microcapsules on fabrics increased the intensity ratios values in each fabric from the first group (test 1, 4, 6, 8), confirming the contribution of new functional groups on the textile substrate. When the resin is added to the bath analysis of the second group (test 2, 5, 7, 9) values are highly increased in both application processes due to the contribution of the resin functional groups which are not added in the first group (tests 1, 4, 6 and 8).

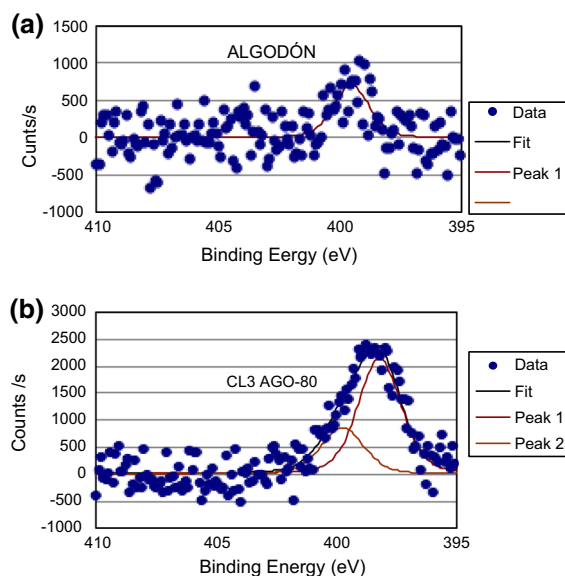
Comparing both application processes using the same quantities of products (test 2 and 9) it can be seen that the value is the same and coincides with the test 7 (24 % owf microcapsules and 4 % owf resin).

#### X-ray photoelectric spectroscopy (XPS)

Knowing the microcapsules shell is made of melamine formalin, this implies that the incorporation of that product to the fabrics' surface will result an increase in the presence of nitrogen.

If the cotton fabric would have been composed of pure cellulose, the XPS spectra would show only carbon and oxygen presence. The untreated cotton spectrum does not fit with the pure cellulose due to the presence of nitrogen, calcium and other elements (Buchert et al. 2001; Fras et al. 2005; Topalovic et al. 2007).

Figure 4 shows high resolution XPS spectrum at the peak region 1Ns for untreated cotton fabric (Fig. 4a) and cotton fabric with microcapsules applied by bath exhaustion at 80 °C (test 10, 240 % owf microcapsules) (Fig. 4b). Certain differences can be observed. Figure 4a represents the spectrum for the cotton fabric when no microcapsules are on the fibre, nitrogen region shows a high dispersion of the experimental values. On the contrary, Fig. 4b, when microcapsules



**Fig. 4** XPS spectrum nitrogen content on different cotton fabrics. **a** Untreated cotton fabric, **b** cotton with microcapsules

are applied on cotton fabric there is a greater definition of the experimental values so the nitrogen presence can be easily detected and it can be attributed to the presence of microcapsules on cotton surface.

The variation in the fibres surface composition is examined by calculating the ratio C/N, due to fact that the microcapsules application incorporates nitrogen at the fabric surface (Fig. 4).

Focused on studying the reproducibility of XPS results two different measurements have been studied, both samples had been padded in different days. For both samples, the bath products were composed of 60 g/L microcapsules and 10 g/L of resin (test 2).

Table 5 evidences that differences between two measurements are negligible and may be due to the measurement process or the microcapsules irregularity deposition process.

XPS results for the padding application process are shown in Table 6. It can be observed a sharp reduction in the value of the ratio C/N due to the contribution of nitrogen added by the microcapsules and the resin. When adding resin to the application bath (test 3) the ratio value is higher compared to the value from the application bath with the same concentration of microcapsules without including resin (test 1). This is due to the contribution of both carbon and nitrogen in the resin composition. The higher value can be detected when there is only resin and there are no

**Table 5** XPS analysis reproducibility. Chemical composition (atomic contents of carbon, oxygen and nitrogen elements) of cotton fibre surface as measured by XPS

References	Bath composition	%C	%O	%N	Ratio C/N
Test 2 padding 2010	60 g/L microcapsules 10 g/L resin	74.86	23.74	1.39	53.86
Test 2 padding 2012	60 g/L microcapsules 10 g/L resin	72.52	26.14	1.35	53.72

**Table 6** Chemical composition (atomic contents of carbon, oxygen and nitrogen elements) of cotton fibre surfaces with microcapsules applied by padding as measured by XPS

Reference	Bath composition	%C	%O	%N	Ratio C/N
Cotton sample	–	67.34	32.37	0.29	232.21
Test 1 Padding	60 g/L microcapsules	73.53	25.12	2.35	30.86
Test 2 Padding	60 g/L microcapsules 10 g/L resin	74.86	23.74	1.39	53.86
Test 3 Padding	10 g/L resin	73.58	25.46	0.96	76.87

microcapsules. It can be attributed to the fact that carbon contribution is higher for resin products than for microcapsules.

When XPS results are studied for bath exhaustion application the same trend can be observed. Table 7 shows results for cotton fabrics treated with microcapsules by bath exhaustion.

It can be observed that when microcapsules concentration is higher, there is greater presence of nitrogen thus reducing the ratio C/N value. There is no linear relationship between the increase in the microcapsules concentration and the decrease ratio, though.

When resin is introduced into the bath the ratio values C/N increase as shown in Table 8.

These results confirm the higher contribution of carbon on the fabric surface attributed to the resin presence.

Some authors (Bonet et al. 2012; Monllor et al. 2009) demonstrated that when working with

microcapsules temperature treatments should be as lower possible; otherwise active product can be damaged. In this part of the research microcapsules have been applied by bath exhaustion using two working temperatures, 60 and 80 °C. In Table 9 it can be observed XPS analysis results.

When the bath temperature is 80 °C, process efficiency increases and more microcapsules are deposited on the fabric surface consequently, the ratio value decreases. However, this temperature affects the microcapsules stability. In Fig. 5 it can be appreciated the temperature effects in the microcapsules state. It compares two temperatures; 60 °C (Fig. 5a) and 80 °C (Fig. 5b) when baths were composed of 240 % owf microcapsules and 40 % owf of resin.

Previous research demonstrated (Monllor et al. 2007; Bonet et al. 2012) that the treatment of microcapsules by padding shows different behaviour than bath exhaustion. The process yield is higher in the

**Table 7** Chemical composition (atomic contents of carbon, oxygen and nitrogen elements) of cotton fibre surfaces with microcapsules applied by bath exhaustion at 60 °C as measured by XPS

Reference	Bath composition	%C	%O	%N	Ratio C/N
Cotton sample	–	67.34	32.37	0.29	232.21
Test 4 bath exhaustion	12 % owf microcapsules	70.80	28.66	0.54	131.11
Test 6 bath exhaustion	24 % owf microcapsules	0.315	33.25	0.67	98.6
Test 8 bath exhaustion	240 % owf microcapsules	73.14	24.90	1.96	37.32

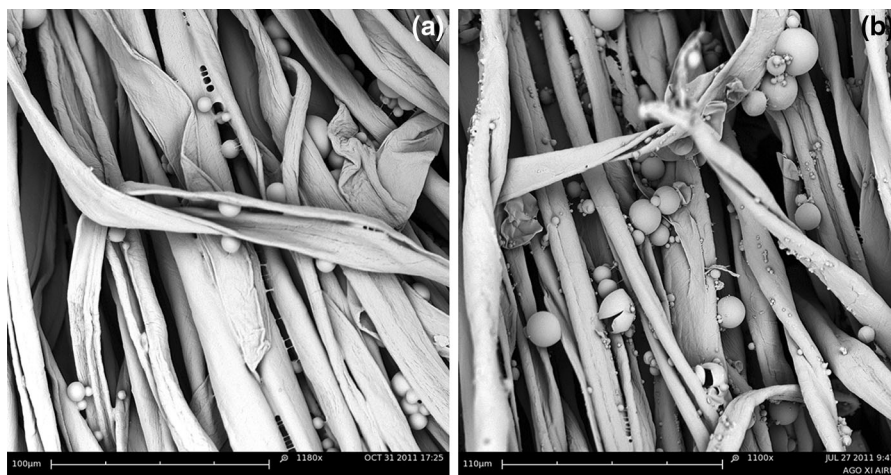


**Table 8** Chemical composition (atomic contents of carbon, oxygen and nitrogen elements) of cotton fibre surfaces with microcapsules applied by bath exhaustion at 60 °C as measured by XPS

Reference	Bath composition	%C	%O	%N	Ratio C/N
Cotton sample	–	67.34	32.37	0.29	232.21
Test 5 bath exhaustion	12 % owf microcapsules 1 % owf resin	69.27	30.34	0.39	177.62
Test 7 bath exhaustion	24 % owf microcapsules 4 % owf resin	66.78	32.78	0.44	151.77
Test 9 bath exhaustion	240 % owf microcapsules 40 % owf resin	72.99	25.83	1.18	61.86

**Table 9** Chemical composition (atomic contents of carbon, oxygen and nitrogen elements) of cotton fibre surfaces with microcapsules applied by bath exhaustion with different treatment temperature as measured by XPS

Reference	Bath composition	Application temperature (°C)	%C	%O	%N	Ratio C/N
Test 8	240 % owf microcapsules	60	73.14	24.90	1.96	37.32
Test 10	240 % owf microcapsules	80	74.56	21.09	4.34	17.18

**Fig. 5** SEM micrographs of cotton fabrics with microcapsules applied by bath exhaustion. **a** 60 °C. **b** 80 °C**Table 10** Chemical composition (atomic contents of carbon, oxygen and nitrogen elements) of cotton fibre surfaces with microcapsules applied by bath exhaustion at 60 °C and by padding as measured by XPS

Reference	Application process	Bath composition	%C	%O	%N	Ratio C/N
Cotton sample	No application	–	67.34	32.37	0.29	232.21
Test 8	Bath exhaustion	240 % owf microcapsules	73.14	24.90	1.96	37.32
Test 1	Padding	60 g/L microcapsules	73.53	25.12	2.35	30.86

padding treatment. The term yield means the percentage of microcapsules which have been transferred from the bat to the fabric because of the treatment, in

other words the effectiveness of the treatment. XPS results corroborate these conclusions showing that padding process offers better yields than bath

exhaustion. Table 10 compares ratio values for both processes.

Results showed that using the same quantities of products the ratio C/N is higher by bath exhaustion, what implies a lower deposition of MICS on fabric surface when it has been treated by bath exhaustion.

## Conclusions

Microcapsules have been used in textiles for a long time, however their previous characterization, adhesion behaviour and permanence on the fabrics are not well known. Commercial brands usually use them as the supplier recommends without any control or testing because it is not easy to characterize commercial products, how the microcapsules adhere to fabrics and their functionality.

Instrumental techniques used in this paper allow characterising the microcapsules presence on a fabric and knowing their state. SEM is a suitable technique analysis to detect the microcapsules presence, location and condition. The characteristic cross section of cotton fibres contributes to retain microcapsules in the grooves formed by the kidney shape. On the other hand, FTIR and XPS techniques allow comparing the presence of microcapsules on the fabric surface. Both demonstrate that microcapsules and the resin provide functional groups to the fabric.

Application procedure of microencapsulated products is an important factor to consider in the microcapsules application on textile sector. Process conditions and substantial quantities of products are also parameters to consider in order to ensure the stability and durability of microencapsulated.

XPS results demonstrated that the best process to place microcapsules on fabrics is the padding process as the process yield is higher than the one from bath exhaustion. It needs fewer chemical products in the application bath and it can be used at room temperature.

## References

- Bonet M, Quijada C, Muñoz S, Cases F (2004) Characterization of ethylcellulose with different degrees of substitution (DS): a diffuse-reflectance infrared study. *Can J Anal Sci Spectrosc* 49(4):234–239
- Bonet M, Capablanca L, Monllor P, Díaz P, Montava I (2012) Studying bath exhaust as a method to apply microcapsules on fabrics. *J Text Inst* 103(6):629–635
- Buchert J, Pere LS, Johanson JM, Campbell J (2001) Analysis of surface chemistry of linen and cotton fabrics. *Text Res J* 71:626–629
- Fras L, Johanson LS, Stenius P, Laine P, Stana-Kleinscheck K, Ribitsch V (2005) Analysis of the reoxidation of cellulose by titration and XPS. *Colloids Surf A* 260:101–108
- Gisbert G, Ibañez F, Bonet M, Monllor P, Díaz P, Montava I (2009) Increasing hydration of the epidermis by microcapsules in sterilized products. *J Appl Polym Sci* 113(4):2282–2286
- Hong K, Park S (1999) Melamine resin microcapsules containing fragrant oil: synthesis and characterization. *J Appl Polym Sci* 58:128–131
- Jing HU, Zuobing X, Rujun Z, Shuangshuang M, Mingxi W, Zhen L (2011) Properties of aroma sustained-release cotton fabric with rose fragrance nanocapsule. *Chin J Chem Eng* 19(3):523–528
- Kokot S, Czarnik-Matusiewicz C, Ozaki Y (2002) Two-dimensional correlation spectroscopy and principal component analysis studies of temperature-dependent IR spectra of cotton-cellulose. *Biopolymers* 67:456–469
- Kondo T, Sawatari C, Manley RJ, Gray DG (1994) Characterization of hydrogen bonding in cellulose synthetic polymer blend systems with regioselectively substituted methylcellulose. *Macromolecules* 27(1):210–215
- Miró Specos MM, Escobar G, Marino P, Puggia C, Defain Tesoriero MV, Hermida L (2010) Aroma finishing of cotton fabrics by means of microencapsulation techniques. *J Ind Text* 40(1):13–32
- Monllor P, Bonet M, Cases F (2007) Characterization of the behaviour of flavour microcapsules in cotton fabrics. *Eur Polym J* 43:2481–2490
- Monllor P, Bonet M, Sánchez L, Cases F (2009) Thermal behaviour of microencapsulated flavours when applied to cellulose fabrics. *Text Res J* 79(4):365–380
- Monllor P, Capablanca L, Gisbert J, Díaz P, Bonet M (2010) Improvement of microcapsule adhesion to fabrics. *Text Res J* 80(7):631–635
- Nelson G (1991) Microencapsulates in textile coloration and finishing. *Rev Prog Color Relat Top* 21:72–85
- Nelson G (2001) Microencapsulation in textile finishing. *Rev Prog Color Relat Top* 321:57–64
- Nelson G (2002) Application of microencapsulation in textiles. *Int J Pharm* 242:55–62
- Rodrigues SN, Fernandes I, Martins IM, Mata VG, Barreiro F, Rodrigues AE (2008) Microencapsulation of limonene for textiles application. *Ind Eng Chem Res* 47:4142–4147
- Rodrigues SN, Martins IM, Fernandes IP, Gomes PB, Mata VG, Barreiro MF, Rodrigues AE (2009) Scentfashion®: microencapsulated perfumes for textile application. *Chem Eng J* 149(1–3):463–472. ISSN:1385-8947
- Sócrates G (1997) In: Infrared characteristic group frequencies. Tables and charts, 2nd edn
- Topalovic T, Nierstrasz VA, Bautista L, Jovic D, Navarro A, Warmoeskerken MMCG (2007) XPS and contact angle study of cotton surface oxidation by catalytic bleaching. *Colloids Surf A* 296:76–85

- Wilson RC, Pfhol WF (2000) Study of crosslinking reactions of melamine/formaldehyde resin with hydroxyl functional polyester by generalized 2-D infrared spectroscopy. *Vib Spectrosc* 23:13–22
- Zhang H, Wang X (2009) Fabrication and performances of microencapsulated phase change materials based on *n*-octadecane core and resorcinol-modified melamine-formaldehyde shell. *Colloids Surf A* 332:129–138