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A COMPARISON BETWEEN PADDING AND BATH EXHAUSTION TO APPLY MICROCAPSULES ONTO COTTON.

ABSTRACT (300 words)

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 know. Nowadays the majority of textile industries are not able to characterize commercial products, nor study the process of adhering the microcapsule to the fibre 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 for qualitative and quantitative characterization of microcapsules on fabrics. Results demonstrate that padding application gives better yields than bath exhaustion.

KEY WORDS (up to 10)

Fragrance microcapsules

Padding

Bath exhaustion

Cotton

Resin

Treatment Temperature

SEM

FTIR-ATR

XPS

1. - 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. It is becoming increasingly difficult to ignore the benefits of functionalizing the fibres. The modification of fibres or fabrics is an important method to provide textiles with improved properties.

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 PCM´s for technical textiles. It has encouraged the industry to use microencapsulation processes as a means of imparting finishings and properties to textiles which were not possible or cost-effective using other technologies (Nelson G, 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 G. 2002, Monllor P. et al. 2007, Nelson G. 1991, Nelson G. 2001, Miro Specos M et al. 2010, Gisbert G. 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. Ther are many types of shell however, it is not common to find reactive polymers that 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 by padding. In order to paste microcapsules to fabrics, they should be in contact with a bath, which contains microcapsules, resin and water. The resin allows the microcapsules to adhere 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 repellent, antibacterial, etc., cannot be tested without analytical methods. Some characterization techniques have been applied in order to study microcapsule products, presence and state (Monllor P. et al. 2007, Gisbert G. et al. 2009, Hong K. and Park S. 1999, Rodrigues SN. Et al 2008, Rodrigues SN. Et al 2009, Monllor P. et al. 2010, Jing H.U. et al. 2011, Bonet M. et al. 2012, Monllor P. et al. 2009) .

Scanning electron microscopy (SEM) has been commonly used for many tears to characterise the microcapsules state and presence on fabrics (Nelson G. 2002, Nelson G. 1991, Nelson G. 2001, Hong K. et al. 1999). The Fourier transform infrared spectroscopy (FTIR) is used as a method of quantifying the

microcapsules presence on the fabrics surface (Monllor P. et al. 2007, Monllor P. et al. 2009). And X-ray photoelectronic spectroscopy (XPS) is a surface chemical analysis technique that allows detection microcapsules on fabric surface when a shell is composed of a polymer with nitrogen in its structure (Bonet M. 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 application method effectiveness of different fabrics when microcapsules had been applied.

2.- MATERIALS AND METHODS

2.1.- 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. No further information was supplied by the provider, as companies disclose as little information as possible. 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^2, which had been chemically bleached with peroxide in an industrial process.

2.2. - Microcapsule application procedure.

Commercial microcapsules were applied to the surface of the fabric by padding or bath exhaustion. In the finishing process 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^2. Bath treatments had been studied previously (Monllor P et al. 2007, Monllor P et al.

2010), and they were composed of different concentrations of microcapsules and resin. Samples were thermally fixed in a scale pin stenter and dried at 110 ºC 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 [13]. Table 2 summarises the padding conditions.

Table 2. - Padding conditions

	Test 1	***Test 2***	***Test 3***
Microcapsules concentration (g/L)	60	60	-
Binder concentration (g/L)	-	10	10
Drying temperature (ºC)	110	110	110

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

Table 3. - 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 (ºC)	60	60	60	60	60	60	80
Drying temperature (ºC)	110	110	110	110	110	110	110

2.4. - 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

2.5. - Fourier transform infrared spectroscopy (FTIR)

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

2.6. - 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 analyzer energy mode with pass energy of 50 eV. The Pressure of the analysis chamber was maintained at $5 \cdot 10^{-10}$ 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 ± 0.2 and ± 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.

3. - RESULTS AND DISCUSSION

3.1. - 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, in bath exhaustion terms 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 (1a, test 2) and bath exhaustion application (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 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 fabrics' fibres. When figure 1 is analysed, it can be observed differences between figure 1a and figure 1b, what can be directly attributed to application process. The amount of microcapsules between cotton fibres differs according to the application process. In figure 1.b (bath exhaustion application) it can be observed more microcapsules and resin presence that in the figure 1 a. (padding application).

a) b)

Figure 1.- SEM micrographs of cotton fabrics with microcapsules.

(a) After padding treatment (60g/L microcapsules and 10g/L of resin).

(b) After bath exhaustion treatment (240% owf microcapsules and 40% owf resin) temperature process 60ºC.

Figure 1 confirms the differences between both microcapsules application processes, baths composition of 240% owf of microcapsules and 40% owf of resin are excessive for the bath exhaustion process. Commercial brand´s recipe suggests using between 6 - 12% of microcapsules and 1 - 2% of resin with a temperature process below 70ºC. When working with microcapsules temperature treatments should be as lower as possible; otherwise active product can be damaged (Bonet M. et al. 2012, Monllor P. et al. 2009).

3.2. - 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 cm^{-1} attributed to the stretching vibration of the hydroxyl and amine groups (Monllor P. et al. 2007, Rodrigues SN et al. 2009, Monllor P et al 2009, Sócrates G. 1997, Wilson RC et al 2000, Zhang H and Wang X. 2009), and the other centred on 1650 cm^{-1}, it can be related to CO stretching vibration in the amide group (Monllor P. et al. 2007, Rodrigues SN et al. 2009, Sócrates G. 1997, Wilson RC et al 2000, Zhang H and Wang X. 2009)

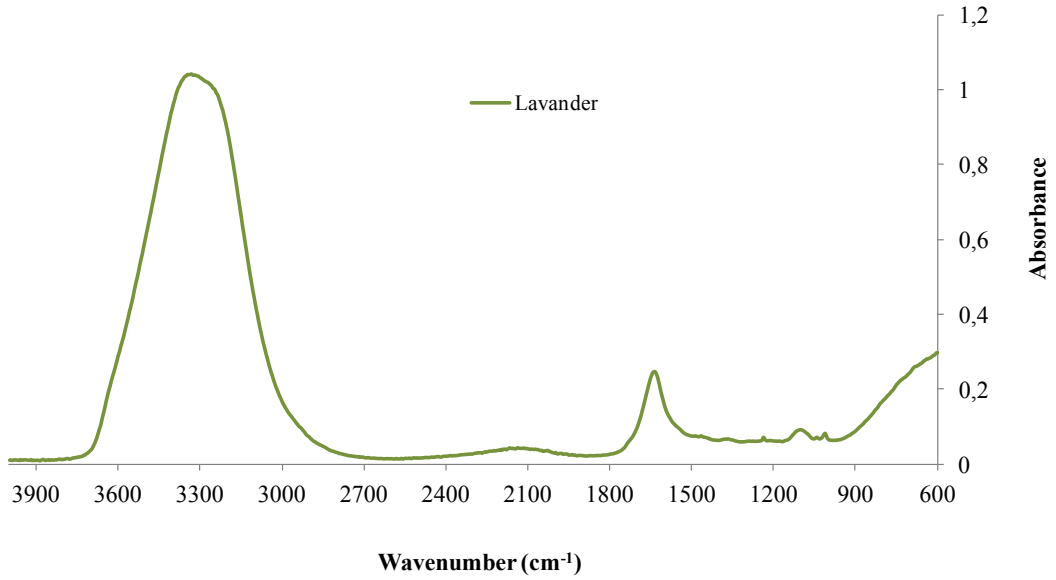


Figure 2.- Infrared spectra of microencapsulated product

The information found in the libraries of the FTIR equipment relating to melamine formaldehyde spectra as well as that provided by previous studies (Monllor P. et al. 2007, Rodrigues SN et al. 2009, Monllor P et al 2009, Sócrates G. 1997, Wilson RC et al 2000, Zhang H and Wang X. 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, figure 3 compares the infrared spectra of the commercial microencapsulated product and the one from the fabric before applying microcapsules.

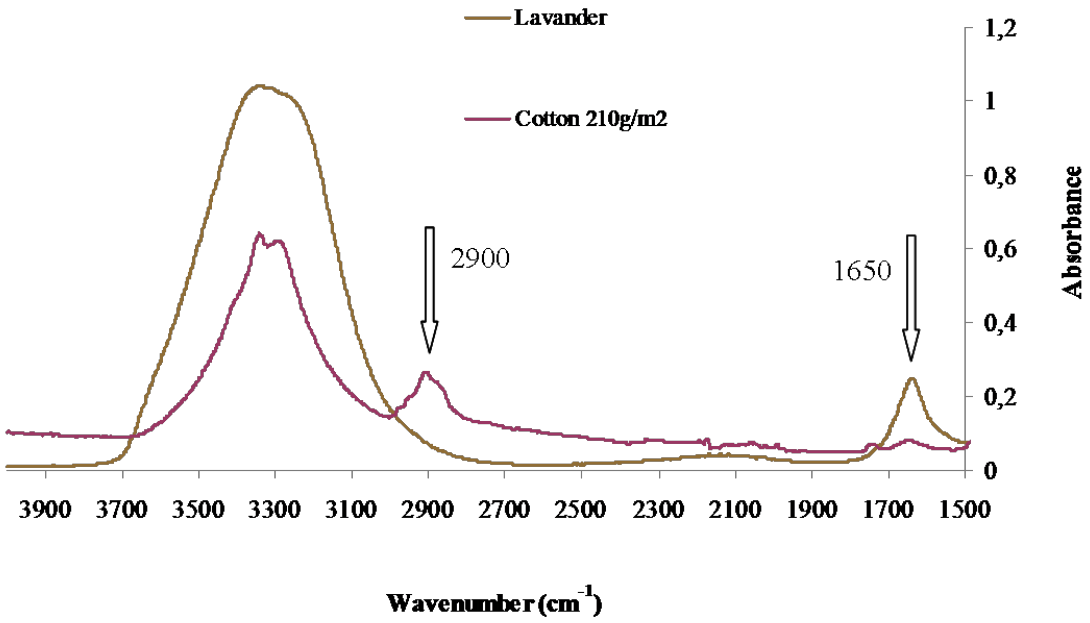


Figure 3.- Infrared spectra of microencapsulated product and a cotton fabric

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

There are two bands which do not present the same behaviour. The band centred at 2900 cm^{-1} (CH stretching vibration) from cotton pectrum and the one

centred at 1650 cm^{1} (CO stretching vibration) from lavender microcapsules spectrum. Thus, the fabric presents a characteristic band in the 2900 cm^{-1} region while the commercial product shows no variation at this region, on the other hand, the commercial product at 1650 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 analyzed 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.

Table 4. - Intensity ratios. FTIR - ATR analysis

Reference	***Process***	***Bath composition***	I_{1650} / I_{2900}
Cotton sample		-	*0,320*
Test 1	Padding	60g/L microcapsules	0,327
Test 2	Padding	60g/L microcapsules 10g/L resin	0,334
Test 3	Padding	10g/L resin	0,328
Test 4	Bath Exhaustion 60ºC	12% owf microcapsules	0,319
Test 5	Bath Exhaustion	12% owf microcapsules 2% owf resin	0,324

	60ºC		
Test 6	Bath Exhaustion 60ºC	24% owf microcapsules	0,315
Test 7	Bath Exhaustion 60ºC	24% owf microcapsules 4% owf resin	0,334
Test 8	Bath Exhaustion 60ºC	240% owf microcapsules	0,329
Test 9	Bath Exhaustion 60ºC	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 analyzing 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 I 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 grupo (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 micocapsules and 4% owf resin).

3.3.- X-ray photolelectric spectroscopy (XPS)

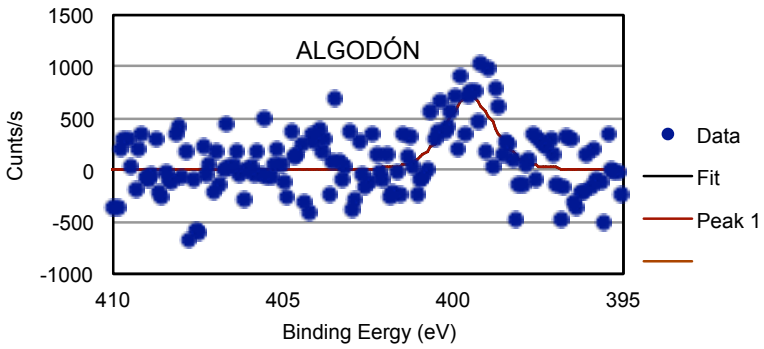
Knowing the microcapsules shell is made of melamine formalin, this implies that the incorporation of that product to the fabrics surface will result an increase in the presence of nitrogen.

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 J. et al. 2001, Fras L. et al. 2005, Topalovic et al. 2007)

Figure 4 shows high resolution XPS spectrum at the peak region 1Ns for untreated cotton fabric (4a) and cotton fabric with microcapsules applied by bath exhaustion at 80ºC (test 10, 240% owf microcapsules) (figure 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, Figure 4b, when

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.

a)



b)

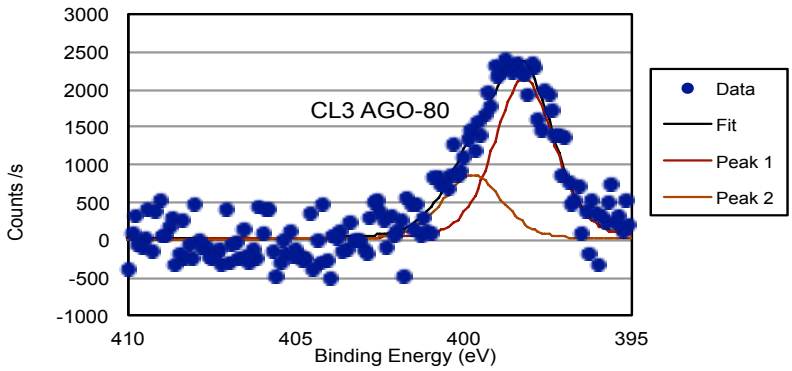


Figure 4. - XPS spectrum nitrogen content on different cotton fabrics (a) untreated cotton fabric, (b) cotton with microcapsules

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

Reefrences	Bath composition	%C	%O	%N	Ratio C/N
Test 2 Padding **2010**	60g/L microcapsules 10g/L resin	74,86	23,74	1,39	53,86
Test 2 Padding **2012**	60g/L microcapsules 10g/L resin	72,52	26,14	1,35	53,72

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

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	60g/L microcapsules	73,53	25,12	2,35	30,86
Test 2 Padding	60g/L microcapsules 10g/L resin	74,86	23,74	1,39	53,86
Test 3 Padding	10g/L resin	73,58	25,46	0,96	76,87

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.

Table 7. –Chemical composition (atomic contents of carbon, oxygen and nitrogen elements) of cotton fibre surfaces with microcapsules applied by bath exhaustion at 60 ºC 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

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 increase as shown in table 8.

Table 8. –Chemical composition (atomic contents of carbon, oxygen and nitrogen elements) of cotton fibre surfaces with microcapsules applied by bath exhaustion at 60 ºC 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

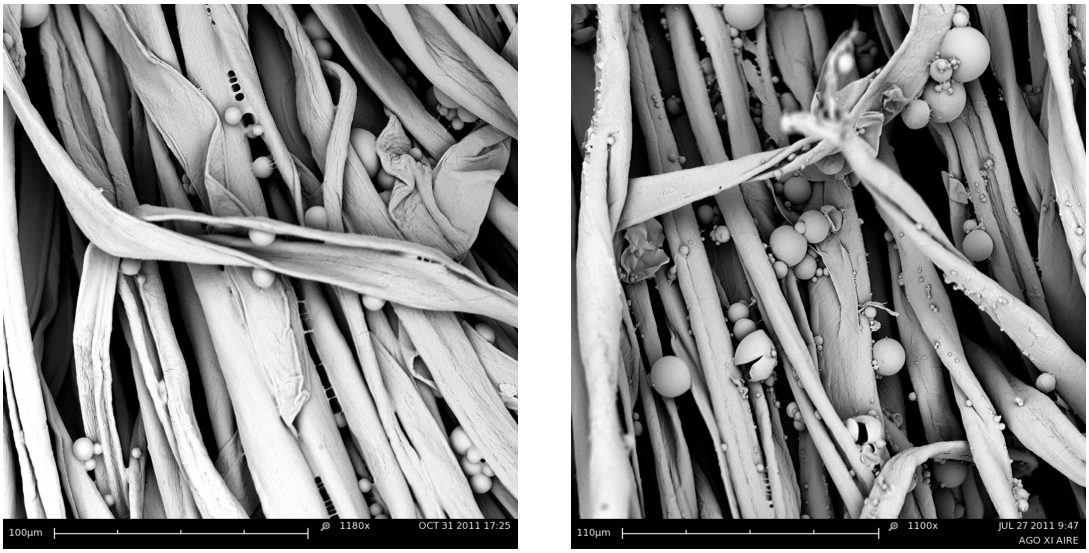
These results confirm the higher contribution of carbon on the fabric surface attributed to the resin presence.

Some authors [13] 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 ºC. In Table 9 it can be observed XPS analysis results.

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 (ºC)	%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

When the bath temperature is 80 ºC, 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; 60º C (figure 5a) and 80º C (figure 5b) when baths were composed of 240% owf microcapsules and 40% owf of resin.



a) b)

Figure 5.- SEM micrographs of cotton fabrics with microcapsules applied by bath exhaustion

(a) 60 ºC. (b) 80 ºC

Previous research demonstrated (Monllor P et al. 2007, Bonet M. et al. 2012) that the treatment of microcapsules by padding shows different behaviour than bath exhaustion. The process yield is higher in the padding treatment. XPS results corroborate these conclusions showing that padding process offers better yields than bath exhaustion. Table 10 compares ratio values for both processes.

Table 10. –Chemical composition (atomic contents of carbon, oxygen and nitrogen elements) of cotton fibre surfaces with microcapsules applied by bath exhaustion at 60 ºC 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	60g/L microcapsules	73,53	25,12	2,35	30,86

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.

4.- CONCLUSIONS

Microcapules have been used in textiles for a long time, however their previous characterization, adhesion behaviour and permanence on the fabrics are not well know. 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 to characterise the microcapsules presence on a fabric and know 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 to quantify 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 placemicrocapsules 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.

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