

Research Article

Performance of Cotton, Cotton-Polyester, and Polyester-Elastane Fabrics Impregnated with Ultrafine Polymeric Nanoparticles Loaded with Lavender Oil

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Lavender oil- (LO-) loaded ultrafine nanoparticles (NP) (diameter < 50 nm) of a copolymer were used for impregnating fabrics of cotton, cotton polyester, and polyester-elastane. The copolymer was composed mainly of methyl methacrylate (MMA) units with a low proportion of methacrylic acid (MAA). Moreover, two kinds of NP were used: one containing uncrosslinked copolymer and another one composed of crosslinked material. All the fabrics impregnated with NP of uncrosslinked copolymer keep the aroma up to 25 washes. Under storage at ambient conditions, the fabrics impregnated with NP of either uncrosslinked or crosslinked copolymer emitted aroma for up to almost 60 days. The long duration of aroma emission from fabrics is attributed to ease of the small nanoparticles used in this study of penetrating and staying inside the pores of the fabric fibers.

1. Introduction

Incorporating nanomaterials into fabrics has emerged as an option to improve their conventional properties, as well as to give them new ones which greatly increases their value [1–5]. Within a great framework of possibilities, the preparation of fabrics that emit aromas even after a long time of use is a current research topic. The use of micro and nanocapsules containing essences and essential oils for impregnating fabrics has been widely reported in the specialized literature [5–13].

Among the reports on the use of micro and nanocapsules containing essences or essential oils to impregnate fabrics, the following could be highlighted. Aracil et al. [9] reported the impregnation of cotton twill fabrics with microcapsules, with diameters between 1 and 40 μm , containing lavender fragrance and a melamine-formaldehyde shell, which was carried out in a washing machine during a domestic laundry process. In order to increase the adherence to fabrics, either acrylic or succinic acid were added to the impregnation rec-

ipe. The microcapsules, at an initial concentration of 5% in the fabrics, remained more than 5 washes when succinic acid was used as binder. Generally speaking, there are evidences leading to assume that in the impregnation of fabrics with micro and nanoparticles containing essences, the smaller the particle size, the greater their permanence in fabrics after a given washing cycle [5–7, 12]. In this respect, Monllorpérez et al. [6] determined the permanence, in a cotton fabric, of melamine-formaldehyde microcapsules with diameters between 1 and 2.5 μm , containing peppermint essence. Using an acrylic resin to increase the adherence between the microcapsules and the fibers, they found that microcapsules 1 μm in diameter had the longest permanence after 20 washing cycles. Another study shows the results of the impregnation of cotton fabric with polyurethane nanocapsules loaded with rose essence and with two sizes: 51.4 and 532 nm in diameter [7]. The authors found that the fabric impregnated with the smaller nanocapsules showed a much better washing resistance than that containing the larger ones. Meanwhile, Liu et al. [12] carried out the impregnation of cotton fabrics with

nanocapsules and microcapsules, containing citronella oil. These authors reported that the fabrics impregnated with nanocapsules kept the aroma for a larger number of washes in comparison with those impregnated with microcapsules. This behavior was attributed to the nanocapsules penetrating and settling inside the cotton fibers pores, while the microcapsules, due to their larger size, are adsorbed on the fiber surface. More recently, Khedkar and Mallick [5] mention that the average pore size in the textile material is too small for most of the microcapsules to penetrate and remain inside the fabrics for a long time and suggest the use of nanocapsules containing essences to increase the service life of the scented fabrics.

In accordance with the specialized literature, the utilization of essences or essential oil-loaded polymeric NP to impregnate fabrics has not received much attention. This option could be interesting due to unlike with micro and nanocapsules where the active principle is concentrated in their center, in the case of solid polymeric NP would be distributed throughout the nanostructure, within interchain spaces. This feature could increase the permanence of the aroma, taking into account that when the shell is broken, the micro and nanocapsules release all their content at once, which would not occur in the case of polymeric NP. Moreover, the use of ultrafine polymeric NP would facilitate their penetration and permanence within the fabric pores.

Herein, we report the use of lavender oil- (LO-) loaded NP, with diameters less than 50 nm, for impregnation of cotton, cotton-polyester, and polyester-elastane fabrics. The material with which the NP were elaborated was the copolymer methyl methacrylate-*co*-methacrylic acid or P(MMA-*co*-MAA). The permanence of the aroma in the fabrics was followed during 25 washing cycles, as well as during storage where the fabrics were not washed.

2. Experimental Part

2.1. Materials. LO-loaded NP in aqueous dispersion (latex) were kindly provided by Nanoingredientes Bioactivos (Saltillo, México). They supplied two types of latexes: one with uncrosslinked copolymer in NP (B) and another one containing NP with crosslinked copolymer (C). Four types of fabrics, provided by Mas Branding (Monterrey, México), were used in this study: 100% cotton (T1), 90% cotton-10% polyester (T2), 50% cotton-50% polyester (T3), and 90% polyester-10% elastane (T4). Bidistilled and deionized water was employed for all dilutions of loaded NP.

2.2. Fabrics Impregnation. The LO-loaded NP latexes were diluted from its original concentration to 5 wt. % preserving pH at 5.5. Samples of approximately 7 g of each type of fabric were prepared and impregnated with a sufficient amount of diluted latex, approximately 14 g; so, it was enough to saturate the fabric, but without liquid runoff. As the materials were not drained, in theory, each fabric sample should contain an added 10 wt. % of LO-NP of B or C, as appropriate. The impregnation was carried out at room temperature, and the samples were allowed to stand for 30 min before

taking them to dry, also at room temperature for 24 h in a laboratory hood.

Once dry, the samples were weighed in order to know the weight of LO-NP impregnating the fabrics, and with this data and the weight of the added NP, the impregnation efficiency was calculated.

2.3. Evolution of Aroma Intensity in Impregnated Fabrics. To follow up the aroma intensity during the experimentation, three samples were taken of each type of impregnated fabric, to determine their behavior after washing and another three to follow them in storage, without washing them. To wash the fabrics, a bath with 7 L of tap water and 5 mL of neutral soap was used, at room temperature. The samples were immersed in the bath for 10 min while air was bubbled in to simulate the movement of a conventional washing machine. Afterwards, they were rinsed with clean water for another 10 min also with bubbling. Finally, they were squeezed manually and brought to dry at room temperature in a laboratory hood. The fabrics not washed during the experimentation were kept in a room temperature in another laboratory hood. The aroma measurement was carried out by a panel of 5 evaluators. To put the evaluation of the aroma in objective terms, it was assigned a score between 0 and 6, where the first value corresponded to a total absence of aroma, while the second was for a very evident one.

Follow-up with washes. The fabrics were washed one day in the morning, allowed to dry for 24 h to immediately evaluate the intensity of the aroma and then store them in the laboratory hood until the next day, when the evaluation process was undertaken again.

Follow-up without washes. The intensity of the aroma of the unwashed fabrics was evaluated 25 times distributed in a period of 58 days, for which the fabrics were removed from the hood, taken to the evaluation room, and then immediately stored in the laboratory hood.

2.4. Characterization

2.4.1. Particle Size Determinations. Measurements by quasi-light scattering (QLS) were conducted in a Malvern Zetasizer Nano-ZS90 apparatus at 25°C. To eliminate multiple scattering and particle interactions, the latexes samples were diluted 50 times with water. The particle size was also determined by high-resolution transmission electron microscopy (HRTEM) in a JEOL JEM 2100 equipment and by scanning electron microscopy (SEM) in a JEOL JSM-7401F field emission scanning electron microscope. The sample for HRTEM was prepared by dilution of latex to obtain a dispersion containing 0.15% LO-loaded NP in a 0.1% aqueous solution of trehalose (99%, Sigma-Aldrich, México). Then, one drop of this dispersion was deposited on a copper grid and allowed to dry. The purpose of trehalose was to prevent nanoparticle agglomeration. On the other hand, for SEM characterization, small samples of dry cotton-only fabric impregnated with LO-loaded NP were taken and directly observed under microscope.

2.4.2. Water Content and Solid Determination in Latexes. The determination of the water content was carried out by the

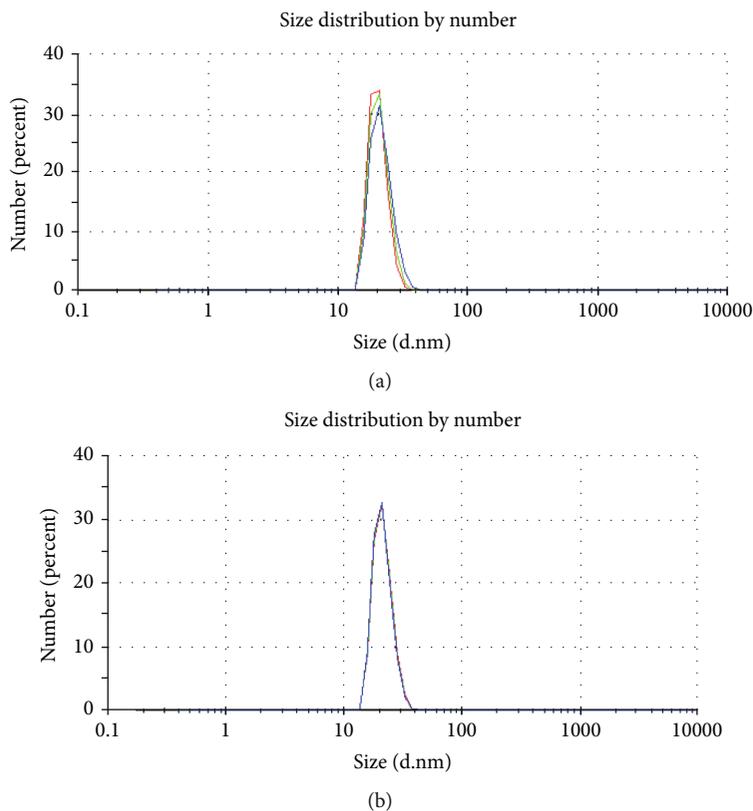


FIGURE 1: Latex particle size distribution by QLS: (a) NP of uncrosslinked copolymer and (b) NP of crosslinked copolymer.

Karl-Fisher method in a TitroLine 7500 KF volumetric titrator. For this measurement, a sample of 0.001 g was poured into the vessel of the equipment, which contained 20 mL anhydrous methanol (>99%, J.T. Baker, México). Then, automatic titration with Karl Fisher reagent (Fluka, México) was carried out. The solid content was determined in a Moisture Analyzer A&D MS-70, for which, 0.5 g of a latex sample was placed in the aluminum plate of the equipment. Then, the temperature was raised to 110°C until reaching constant weight.

2.4.3. Nuclear Magnetic Resonance (NMR) Spectroscopy. Identification of copolymer functional groups. The solid material from the latexes was obtained by lyophilization and then analyzed by ^1H -NMR in a Bruker-400 MHz spectrometer, for which, 0.30 mg of each sample was dissolved in 0.5 mL of dimethyl sulfoxide (DMSO- d_6 , 99.5%, Aldrich, México). The analysis was carried out at room temperature, accumulating 32 scans. The spectra were processed in the MestReNova software.

3. Results and Discussion

In accordance with the results of characterization, the contents of water and solids in the latex containing OL-loaded NP with uncrosslinked copolymer were 68.5 and 24.6 wt. %, respectively, while the contents for the latex with crosslinked copolymer were 69.2 and 24.6 wt. %. From this information, contents of 6.0 and 6.2 wt. % of organic volatile, LO in this

case, for LO-loaded NP with uncrosslinked and crosslinked copolymer, respectively, were estimated. All the LO in the analyzed samples was considered to be contained within the nanoparticles for two reasons. First, the latex clearly shows a single macrophase; that is, there is no evidence of a separated oil phase; second, the particle size distribution determined by QLS in Figure 1 indicates that the dispersion is composed of a single population with a particle size distribution between 15 and 40 nm, approximately, which would correspond to the loaded nanoparticles.

Figure 2 shows the ^1H -NMR spectrum for a sample of solid material obtained from the latex containing OL-loaded NP with uncrosslinked copolymer, according to the supplier. As can be seen in this figure, the signal corresponding to the protons in the $-\text{O}-\text{CH}_3$ group of MMA units appears at 3.6 ppm, while that of the proton in the $-\text{COOH}$ group of MAA units becomes visible at 12.5 ppm. Despite the low signal intensity of the proton in the $-\text{COOH}$ group, the results of ^1H NMR demonstrate the presence of MAA units in the copolymer chains. Similar results (not shown) were obtained when analyzing a sample of the supplied latex containing LO-loaded NP with crosslinked copolymer. However, it is necessary to clarify that the material analyzed in the latter case corresponds to the fraction soluble in the solvent DMSO- d_6 , that is, the uncrosslinked fraction of the copolymer. This is because although the supplier calls the product a crosslinked copolymer latex, the reality is that a part of the chains remains uncrosslinked at the end of polymerization.

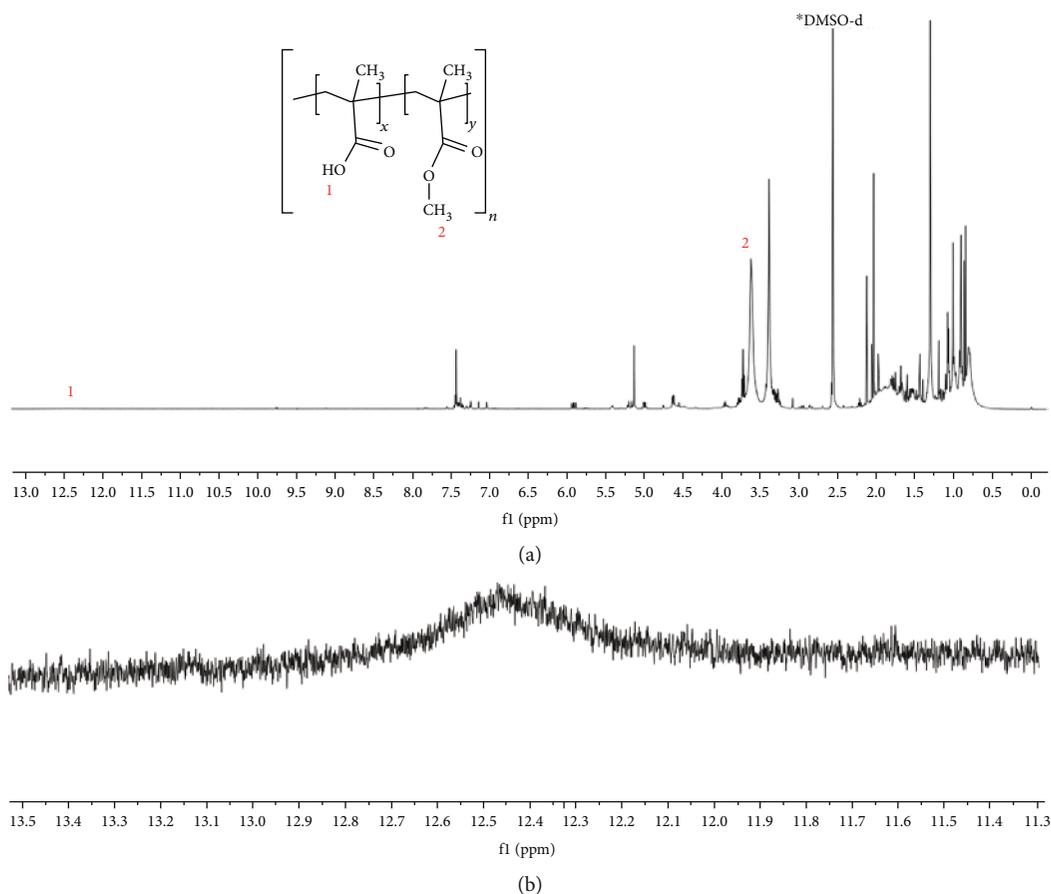


FIGURE 2: ¹H NMR spectrum for a sample of solid material in latex containing OL-loaded NP with uncrosslinked copolymer: (a) full spectrum and (b) magnified interval where the proton in the -OH group of MAA units appears.

TABLE 1: Impregnation efficiency. Uncrosslinked P(MMA-co-MAA) NP loaded with LO.

Fabric	%
BT1	82.5
BT2	78.7
BT3	69.1
BT4	78.3

TABLE 2: Impregnation efficiency. Crosslinked P(MMA-co-MAA) NP loaded with LO.

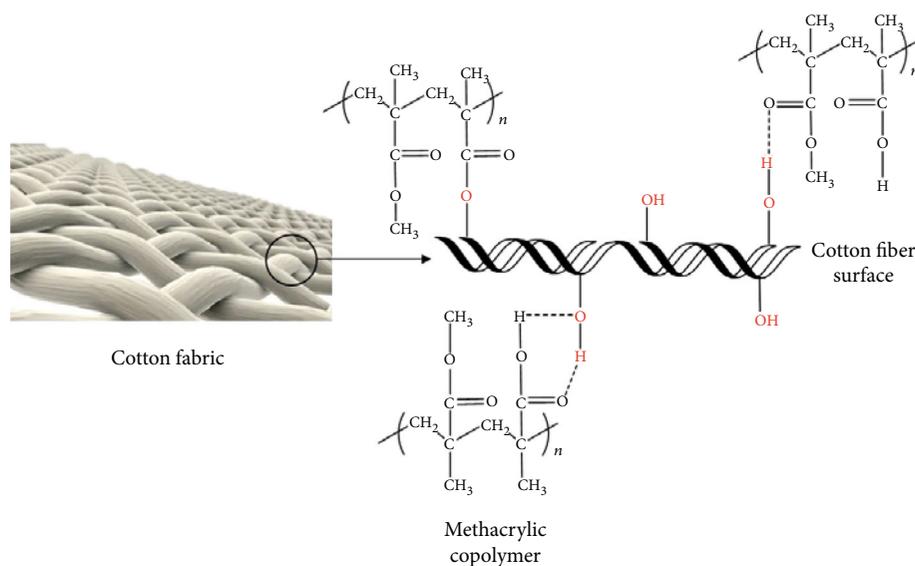
Fabric	%
CT1	82.7
CT2	80.3
CT3	61.0
CT4	70.8

The impregnation test results are shown in Tables 1 and 2. Cotton is seen to be more receptive to impregnation regardless of whether the copolymer in NP is crosslinked or not. To understand this behavior, it would be necessary

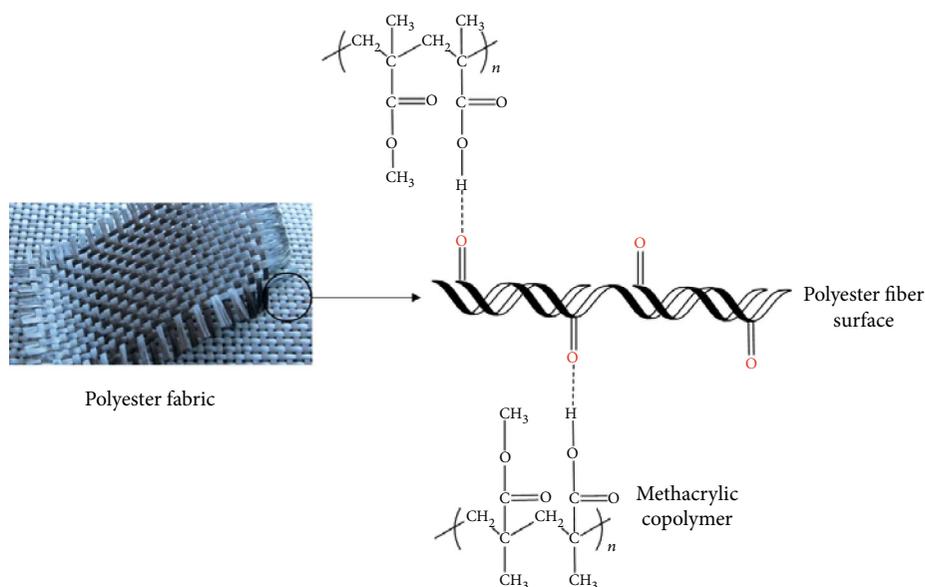
TABLE 3: Functional groups contained in the fabrics used in this study.

Fabrics	=CO	-NH-	-OH
Cotton	No	No	Yes
Polyester	Yes	No	No
Elastane	Yes	Yes	No

to consider the following. P(MMA-co-MAA) contains two types of functional groups able to form hydrogen bonds: =CO and -OH. The first of these groups is present in the ester group of MMA as well as in the acidic group of MAA units, while the hydroxyl is present only in the acidic group of this last type of monomeric units. In this regard, it is interesting to note that Ghayempour and Mortazavi [14] reported the use of an acrylate, poly (vinyl acetate), and polyurethane as binders for increasing the graft of nanocapsules containing essential oils on cotton fabrics, showing that the acrylate renders the best results. Table 3 shows the different fiber's functional groups able to form hydrogen bonds with the functional groups in the copolymer. Clearly, the cotton -OH groups would form hydrogen bonds with the carbonyl as well as the hydroxyl groups of



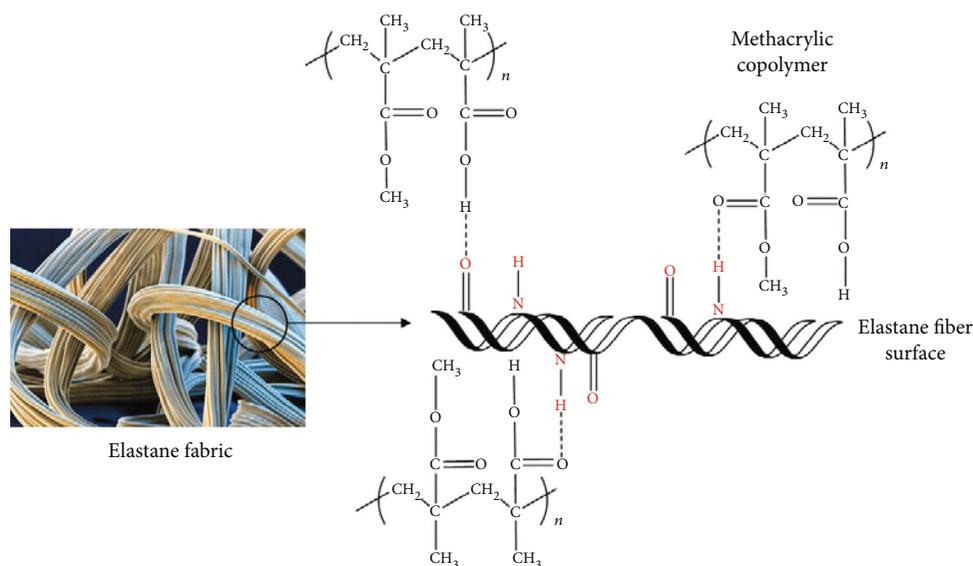
SCHEME 1: Cotton fibers and their hydrogen and esterification bonds with the copolymer in NP.



SCHEME 2: Polyester fibers and their hydrogen bonds with the copolymer in NP.

the copolymer (Scheme 1), while the polyester carbonyl would do so with the -OH of the acidic groups of the copolymer chain (Scheme 2). In the case of elastane, the hydrogen bonds would form between its carbonyl groups and the -OH of the acidic groups of the copolymer as well as between its -NH- groups and the carbonyl groups of the copolymer chains (Scheme 3). However, cotton has an advantage over polyester and elastane, since esterification can take place between its -OH groups and the acidic groups of the copolymer [15] (see Scheme 1). These links, in addition to the hydrogen bonds, could be the cause of the higher impregnation efficiency observed in Tables 1 and 2 for the cotton-only fabrics (BT1 and CT1) and those with 50% cotton (BT2 and CT2).

On the other hand, it seems that the fabric with lower cotton content (T3) as well as polyester-elastane blend (T4) is more receptive to NP of the uncrosslinked copolymer than those composed of crosslinked chains. A definitive explanation for this behavior cannot be offered at this time; however, the following justification may be proposed. Due to their polarity, the functional groups responsible for forming hydrogen bonds are attracted to the particle surface by the water molecules in the aqueous phase. Nevertheless, it is possible that crosslinking could modify the polymer chain arrangement inside the particles, preventing the free orientation of those groups towards the surface, lowering their surface density. The fabric with the highest content of cotton as well as that composed of only cotton would counteract



SCHEME 3: Elastane fibers and their hydrogen bonds with the copolymer in NP.

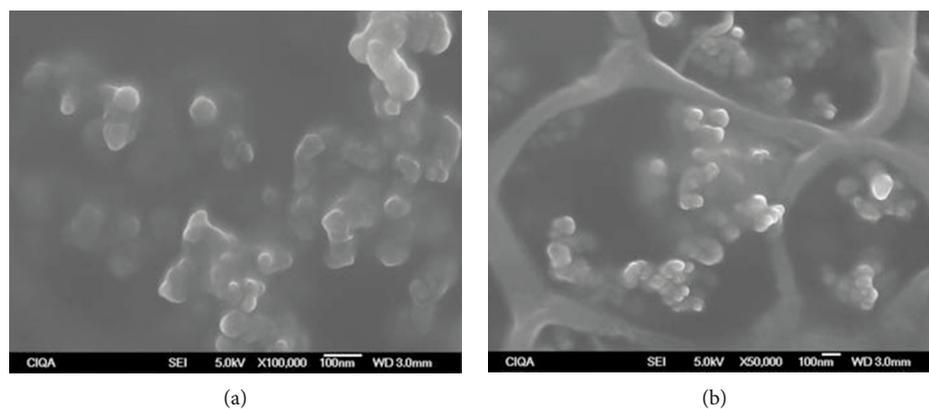


FIGURE 3: HTEM micrograph of LO-loaded NP: (a) NP of uncrosslinked copolymer and (b) NP of crosslinked copolymer.

the potential adverse effect caused by crosslinking by means of an esterification mechanism leading to stronger links. In accordance with literature, hydrogen bonds have a bond energy ranging 6 to 30 kJ/mol [16], while that of organic esters is 358 kJ/mol [17].

Figure 3 shows micrographs of samples from latexes containing LO-NP composed of uncrosslinked and crosslinked copolymer. Although they show some aggregation, it can be distinguished in this figure that the diameter of individual nanostructures is less than 50 nm, for both type of nanoparticles. These sizes match well with those obtained by QLS (see Figure 1). As far as we know, these sizes are the smallest ones reported in the specialized literature on particles loaded with essential oils. One of the great advantages of this feature is the large surface area per unit of volume of the nanoparticles, which gives them a greater number of contact points with the molecules of the fibers and, therefore, an increased probability of forming greater number of links. Moreover, according to Khedkar and Mallick [5], the very small size of the NP

would allow them to penetrate deeper into the pores of the fibers.

Figure 4 shows a micrograph of cotton fibers after impregnation with LO-loaded NP of both uncrosslinked and crosslinked copolymer. It is possible to distinguish here that, in general, nanoparticles are inside the fibers retaining their original size, although some aggregates are identified.

Figures 5 and 6 show the behavior of the intensity of the aroma emitted by the fabrics impregnated with NP of uncrosslinked and crosslinked copolymer, respectively, with the increase in the number of washes. As expected, in all cases, the decrease in the aroma intensity with the washes is evident. However, in the case of fabrics impregnated with NP of uncrosslinked copolymer, it seems that the evolution of the aroma intensity has two stages. During the first stage, comprising between the washes 1 and 12, all of the impregnated fabrics emit aroma with different intensity. Meanwhile, in the other stage which goes from wash 13 to 25, the aroma emission continues but with much lower intensity; in fact, the

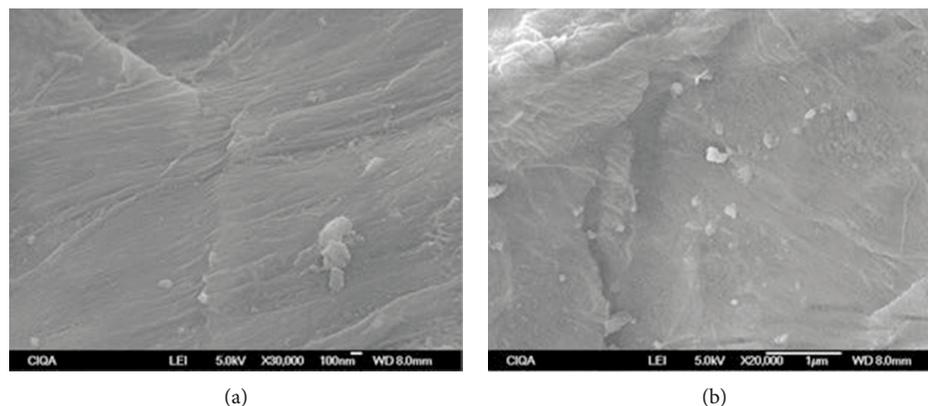


FIGURE 4: SEM micrograph of cotton fibers impregnated with LO-loaded NP: (a) NP of uncrosslinked copolymer and (b) NP of crosslinked copolymer.

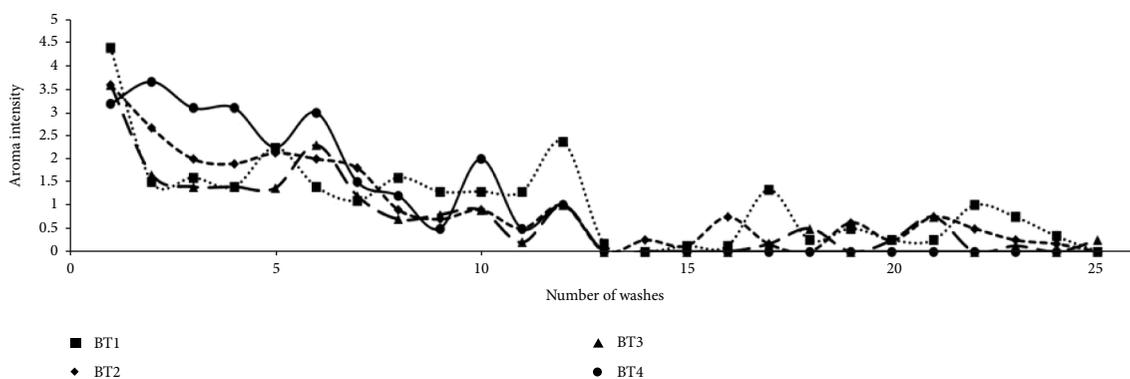


FIGURE 5: Variation in the aroma intensity with washes of the fabrics impregnated with nanoparticles of the uncrosslinked copolymer loaded with LO.

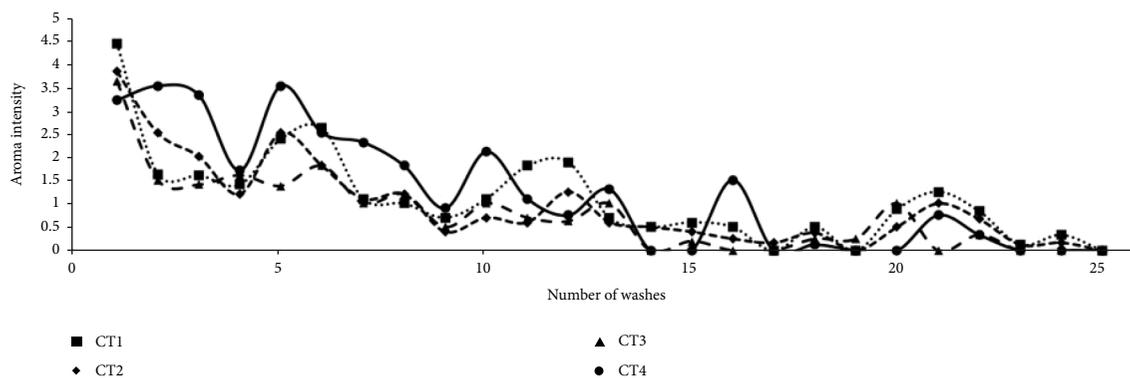


FIGURE 6: Variation in the aroma intensity with washes of the fabrics impregnated with nanoparticles of the crosslinked copolymer loaded with LO.

polyester-elastane fabric (BT4) stops emitting aroma. It is notorious that the cotton-only fabric (BT1) and that one containing 50% cotton (BT2) are those with the highest aroma emission throughout the trial period. The fabric with 10% cotton (BT3) also emits aroma throughout the trial, although with less intensity than BT1 and BT2.

The fabrics impregnated with NP of crosslinked copolymer (Figure 6) also show a stage where all of the fabrics

emit aroma with different intensities after the washes, in this case, between the numbers 1 and 13, followed by another one where the aroma intensity decreases appreciably. Here, it is observed that cotton-only fabric (CT1), that with 50% cotton (CT2) and that of polyester-elastane (CT4), has the highest aroma emission throughout the trial period, while the fabric with 10% cotton emits with less intensity throughout the trial.

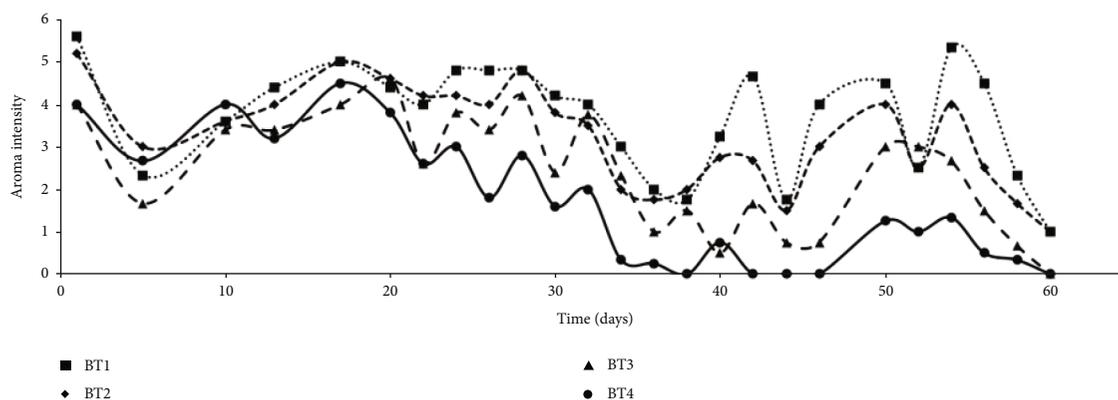


FIGURE 7: Variation in the aroma intensity over time of fabrics impregnated with nanoparticles of the uncrosslinked copolymer loaded with LO without being washed.

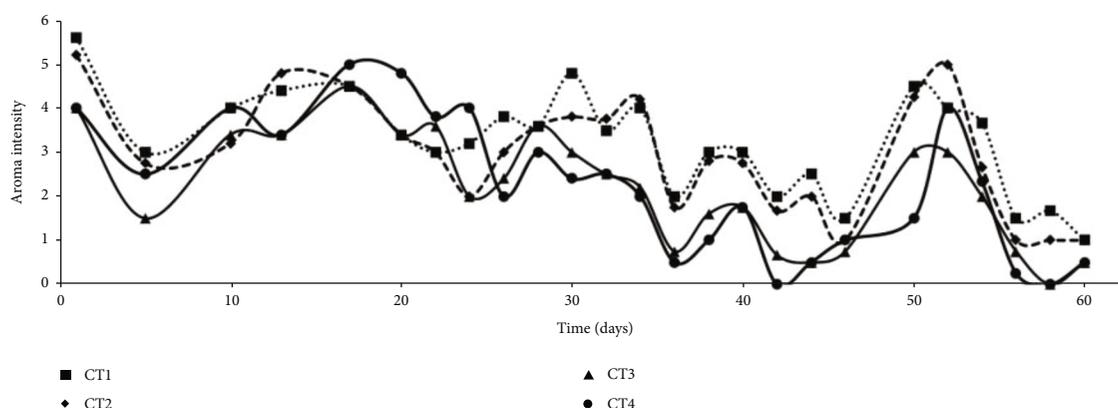


FIGURE 8: Variation in the aroma intensity over time of fabrics impregnated with nanoparticles of the crosslinked copolymer loaded with LO without being washed.

Jointly observed Figures 5 and 6, it is noticeable that with the exception of the polyester-elastane fabric impregnated with NP of uncrosslinked copolymer (BT4), all of the fabrics impregnated with NP of either crosslinked or uncrosslinked copolymer emit aroma throughout the trial, although with decreasing intensity. Despite the differences in the impregnation and washing conditions between our work and that of Golja et al. [8], it is interesting to note that, in the last study, cotton fabrics impregnated with microcapsules ($1.6\ \mu\text{m}$ in diameter) containing different essential oils lost the aroma after 10 washes. These authors prepared microcapsules of melamine-formaldehyde superficially modified with an anionic polyelectrolyte based on polyacrylic acid. This modification was carried out trying to improve the adherence of the microcapsules to the fibers. However, this might not have been enough to keep the aroma on the fabric for a large number of washes. An interesting point to note is that a microcapsule with $1.6\ \mu\text{m}$ in diameter has an area to volume ratio of $0.0019\ \text{nm}^{-1}$, while a nanoparticle having $30\ \text{nm}$ in diameter has a ratio of $0.1\ \text{nm}^{-1}$; that is, for a similar total volume, the total surface of the nanoparticles used in our study is more than 50 times that of the microcapsules in the report of Golja et al. This fact would indicate that the nanoparticles would have a much greater number of linkage points with the

fibers than the microcapsules could have. However, there is another fact, mentioned by Khedkar and Mallick in a recent book on protection and functionalization of textiles [5], which establishes that, for fabrics impregnated with nanoparticles containing essences, the smaller the particle size, the longer the aroma remains in the fabrics. This behavior is explained as a consequence of the penetration of the smallest nanoparticles inside the pores of the fabric fibers. As far as our knowledge, the nanoparticles used in our work are the smallest ones reported in the articles on fabric impregnation with nanoparticles containing essences or essential oils.

A common practice to increase the adherence to the fabric fibers is the use of binders, either by the surface modification of the micro and nanocapsules or by adding the chemicals during the impregnation process. In accordance with the literature, acrylics are one of the most used binders [6, 9]. An additional factor that could help to explain the permanence of aroma emission during practically 25 washes is that the nanoparticles used in our study contain acrylic acid units as a part of the polymer chains that form them. Moreover, these acid units are oriented toward the surface of the nanoparticle as a consequence of their hydrophilicity.

Another interesting feature observed in Figures 5 and 6 is the fluctuation in the aroma emission; all of the impregnated

fabrics show ridges and valleys throughout the experimental period without following a clear pattern, other than the decreasing trend. Accordingly, it cannot be ruled out that the fabrics continue to emit aroma after wash number 25. The decreasing trend in the aroma emission is due to the loss of LO loaded in the NP as well as the loss of the loaded NP during the experimental procedure. A possible explanation for the aroma fluctuation is that the LO inside the NP is not homogeneously distributed, being instead present in the form of isolated reservoirs in the spaces between the copolymer chains. This establishes a difference with micro and nanocapsules in which the oil forms a continuous phase inside a container protected by a shell. As result of this morphology, the intensity of the aroma would decrease more evenly as the number of washes increases. In absence of reports in the specialized literature, it was not possible to make a comparison with other studies on the use of solid nanoparticles containing essences for fabric impregnation.

The change of the aroma intensity with the storage time of the NP-impregnated fabrics without being washed is shown in Figures 7 and 8, for those impregnated with NP of uncrosslinked and crosslinked copolymer, respectively. It is interesting how the aroma fluctuation pattern observed in Figures 5 and 6 is repeated; however, despite the general decrease in the aroma emission along the days, this is far less pronounced than in the tests where the fabrics were washed. Moreover, in accordance with that fluctuating behavior, it seems probable that the aroma emission will continue by several more days than the test duration.

Figures 7 and 8 also show that impregnated fabrics of cotton-only (BT1 and CT1) and those of 50% cotton (BT2 and CT2) emit the highest levels of aroma during the test period. On the other hand, the impregnated fabrics containing 10% cotton (BT3 and CT3) emit slightly less aroma, but impregnated fabrics of polyester-elastane (BT4 and CT4) show the lowest aroma emission of all fabrics.

4. Conclusions

Under the experimental conditions established in this study, the cotton-only fabric, as well as those with 50 and 10% cotton, impregnated with NP of either uncrosslinked or crosslinked copolymer, loaded with LO, maintains the aroma up to 25 washes. Polyester-elastane fabric retains its aroma even up to that number of washes, but only if it is impregnated with the NP of the crosslinked copolymer. If this fabric is impregnated with NP of uncrosslinked copolymer, it only withstands 13 washes. All fabrics impregnated with LO-loaded NP, of either uncrosslinked or crosslinked copolymer, but which were not subjected to wash, emit a fairly acceptable aroma after almost 60 days of being kept at room temperature. On the other hand, the polyester-elastane fabrics impregnated with either uncrosslinked or crosslinked copolymer NP loaded with LO show the poorest aroma emission during the trial period.

The aroma emission of the impregnated fabrics after 25 washes or after almost 60 days of being kept at room temperature without washing could be mainly caused by the greater ease of the small nanoparticles used in this study of penetrat-

ing and staying inside the pores of the fabric fibers. It is proposed that the fluctuating behavior of the aroma emitted by the fabrics throughout the washing and storage periods is due to the oil forming reservoirs within the spaces between the copolymer chains of the NP. This situation would slow down the emission of the aroma from the NP, which would be useful in certain applications.

Data Availability

The data used to support the findings of this study are included within the article.

Conflicts of Interest

The authors do not report conflict of interests whatsoever.

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