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Microencapsulation of Lemongrass (*Cymbopogon citratus*) Essential Oil and Salicylic Acid via in Situ Polymerization and Their Immobilization onto a Cellulosic Substrate

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ABSTRACT

Lemongrass essential oil (*Cymbopogon citratus*) and salicylic acid are dermatological active compounds with unique antimicrobial and anti-inflammatory properties. However, their high volatility and poor aqueous solubility limit their practical use. We characterize the microcapsules via scanning electron microscopy (SEM), dynamic light scattering (DLS), fourier-transform infrared (FTIR) spectroscopy, and thermogravimetric analysis (TGA). The spherical microcapsules, with an average diameter of $4.59 \pm 1.56 \mu\text{m}$, were applied to a 100% cotton fabric. The coated fabric showed potential for applications in therapeutic textiles for acne treatment, antimicrobial wound dressings, as well as skincare applications as cosmetic textiles with controlled release of dermatological actives.

1 | Introduction

Dermatological products play several roles, such as enhancing appearance, preventing and treating disorders of the skin. Many of these dermatological products contain essential oils (EOs), a complex mixture of organic compounds with antimicrobial, anti-inflammatory, and antioxidant properties [1, 2]. These properties confer high applicability to various industrial sectors, including cosmetic, medicinal, agricultural, textile, and food industries [3–7]. The capacity of EOs to interact with cellular structures and their lipophilic nature enable them to act on enzymes, DNA, and affect mitochondrial metabolism [2]. Lemongrass essential oil (LEO) (*Cymbopogon citratus*) has a high concentration of citral, a compound associated with significant antimicrobial, anti-inflammatory, and antioxidant properties [8–13].

Salicylic acid (SA), 2-hydroxybenzoic acid, is actively used in the pharmaceutical and cosmetic industries, owing to its keratolytic, antimicrobial, and anti-inflammatory properties [14–17]. SA is used particularly in the treatment of acne, excessive sebum production, and other cutaneous conditions [18, 19].

Since SA has poor water solubility, which compromises its bioavailability, it represents a good candidate for microencapsulation [20]. Traditional encapsulation methods often result in low efficiency for hydrophobic compounds [21], premature release during storage, and limited thermal stability [22]. Moreover, conventional techniques struggle to simultaneously encapsulate compounds with different polarities, often resulting in phase separation or heterogeneous distribution within the capsule core. LEO is hydrophobic, and SA is amphiphilic [23, 24].

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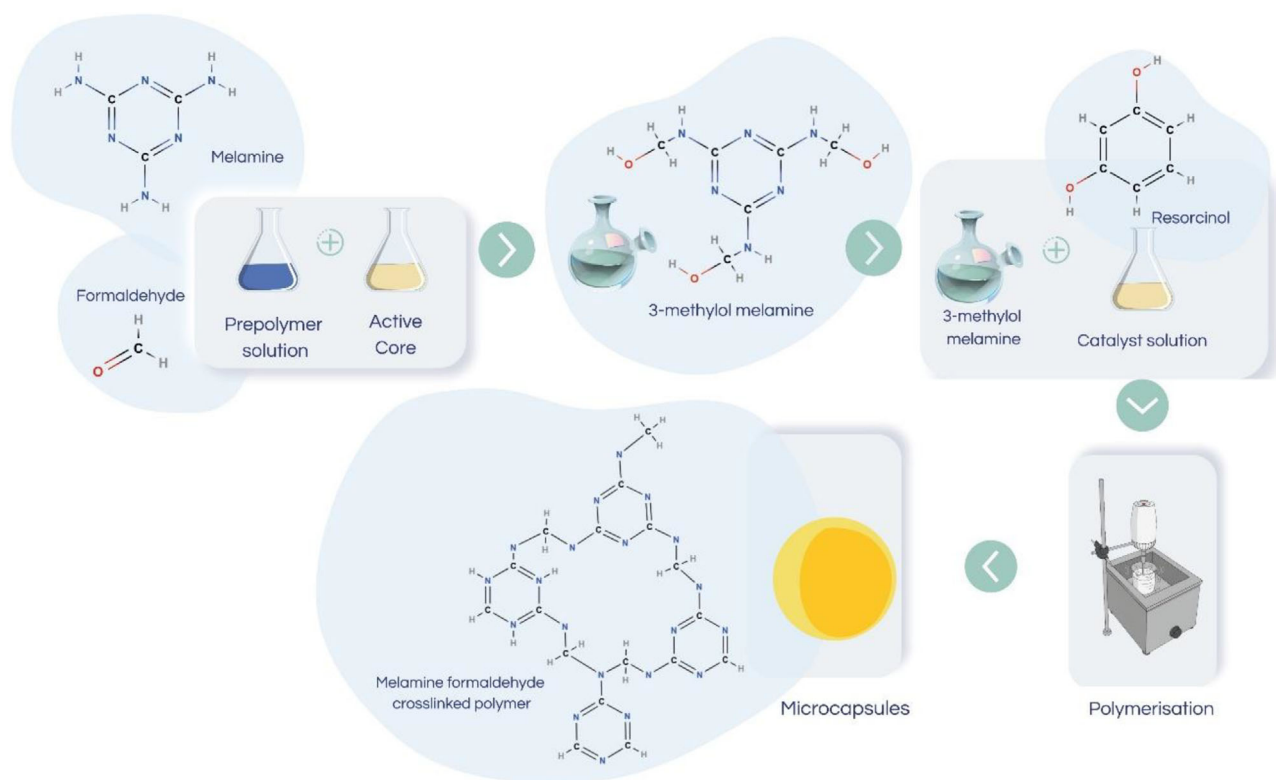


FIGURE 1 | Schematic representation of the in situ polymerization route used for the synthesis of melamine–formaldehyde microcapsules to encapsulate lemongrass essential oil and salicylic acid.

Microencapsulation can be defined as a technique that aims to entrap active principles within a polymeric matrix, enabling controlled release and protection of sensitive compounds [25–27]. The global market for this technology demonstrates accelerated growth, estimated at US\$11.9 billion in 2022, with projected expansion at an annual rate of 10.3% through 2030 [28]. This advancement reflects growing interest in applications ranging from fragrances and foods to biofunctional textile materials capable of incorporating cosmetics and pharmaceuticals into everyday products [24, 29]. The incorporation of microcapsules containing EOs and pharmaceutical actives into textile materials allows for the controlled and sustained release of these compounds, leading to the formation of smart textiles, cosmeo-textiles, or biofunctional textiles with therapeutic potential [30]. Among various microencapsulation methods, microencapsulation via in situ polymerization with amino-plastic resins, such as melamine-formaldehyde (MF), is widely utilized as it yields high encapsulation efficiencies [31, 32].

While the microencapsulation of EOs has been reported [22], the simultaneous encapsulation of LEO and SA has not yet been pursued. This dual-active system offers complementary mechanisms of action: LEO provides broad-spectrum antimicrobial activity and antioxidant protection, while SA enhances skin penetration and exhibits anti-inflammatory effects.

The use of an MF resin as the shell material for microcapsules has been reported as an effective approach for encapsulating multicomponent cores with contrasting polarities [7]. MF polymers undergo condensation at the oil-water interface, forming a dense

crosslinked shell [31, 33]. We expected that this MF shell, at the oil-water interface, would be able to entrap the hydrophobic LEO and the amphiphilic SA within a unified core. The encapsulation mechanism relies on two complementary interactions: (i) interfacial condensation, which creates a mechanical barrier around the oil phase, and (ii) hydrogen bonding between MF polar groups ($-\text{NH}$, $-\text{CH}_2-\text{O}-$, $-\text{OH}$) and carboxyl and phenolic moieties of the trapped compound. This association not only potentiates therapeutic and cosmetic effects but also expands application prospects in emerging sectors.

2 | Experimental Section

2.1 | Materials

The reagents utilized in the experiments include melamine (Sigma-Aldrich, USA), aqueous formaldehyde solution 37% (Seiko, Brazil), *Cymbopogon citratus* essential oil (lemongrass, Quinarí, Brazil), SA (Bottica Botanika, Brazil), sorbitan monooleate (Span 80, Sigma-Aldrich, USA), and Arabic gum (Sigma-Aldrich, USA). Additional reagents such as ammonium chloride, resorcinol, sodium hydroxide, and glacial acetic acid were supplied by Neon, Brazil. For textile functionalization experiments, a bleached 100% cotton fabric (style 400, $100 \text{ g}\cdot\text{m}^{-2}$, ISO 105-F02, was obtained from Test Fabrics Inc., USA). The microcapsule application onto the textile was performed using a pre-condensed melamine-formaldehyde resin (Golden Easy-Care Lem) and a nonionic softener (Goldsoft CLN), both supplied by Golden Technology (Brazil).

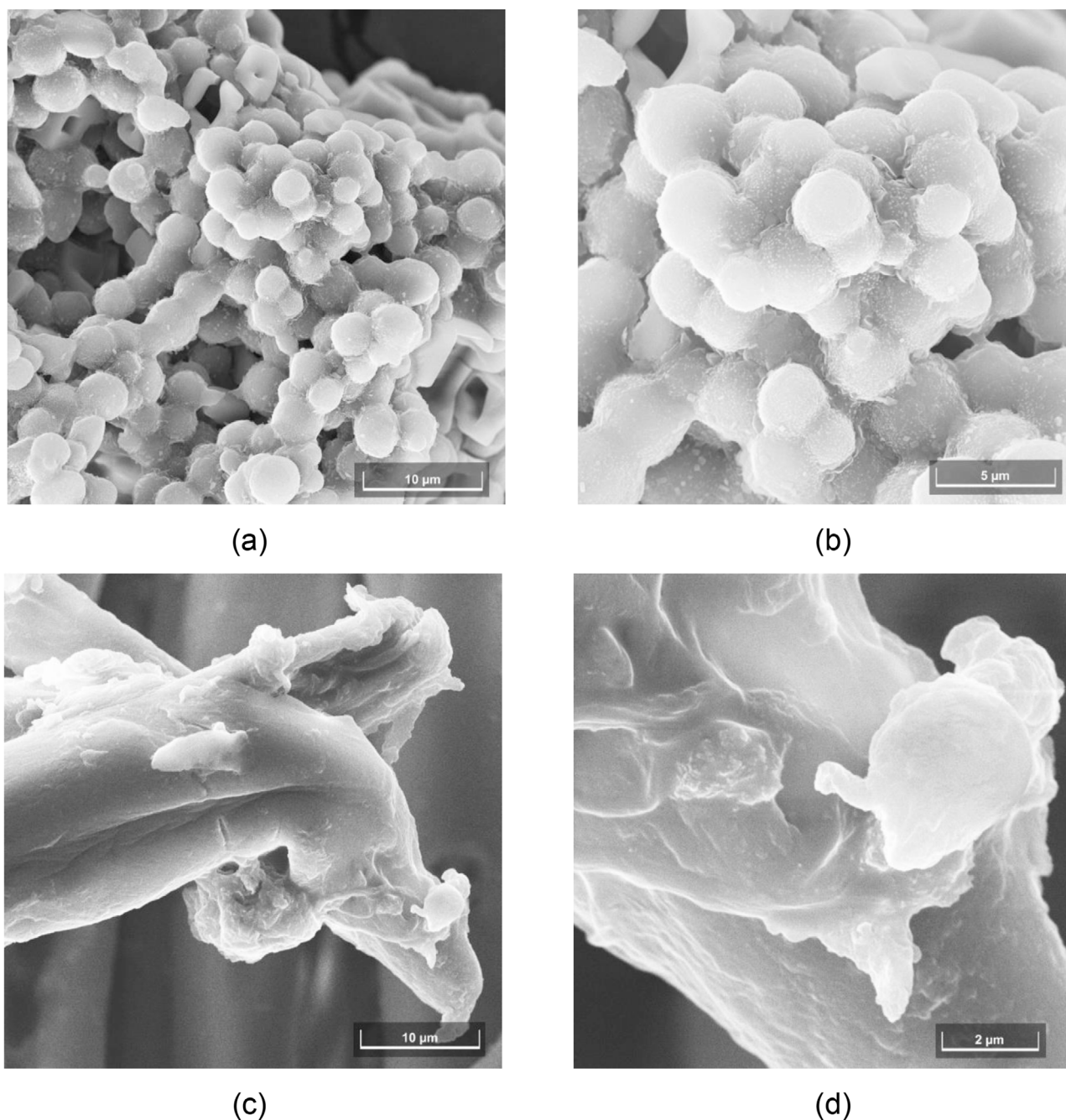


FIGURE 2 | Morphological characterization of lemongrass essential oil and salicylic acid microcapsules: (a) microcapsules overview; (b) surface morphology; (c) microcapsules on cotton fabric; (d) microcapsule-fiber interaction detail.

2.2 | Methods

2.2.1 | Microcapsule Synthesis via in Situ Polymerization

We used a protocol adapted from a previously established microencapsulation methodology [34–36]. The protocol requires three distinct solutions as shown in Figure 1.

Solution 1, a prepolymer solution preparation: 3.62 g of melamine, 7 mL of formaldehyde (37% w/v), and 10 mL of deionized water were combined in a 250-mL three-neck round-bottom flask.

The mixture was heated to 100°C under constant mechanical stirring (150 rpm) for 50 min. The pH was maintained within the range of 8–9 through controlled addition of sodium hydroxide (1 M).

Solution 2, active core (oil phase) solution: an oil-in-water (O/W) emulsion was formed by homogenizing 20 mL of LEO, 1 g of sorbitan monooleate, and 1 g of Arabic gum in 40 mL of deionized water at 10,000 rpm for 15 min using an Ultraturrax T-25 homogenizer (IKA, Germany). 1 g of SA was incorporated under continuous stirring (500 rpm) for an additional 15 min to ensure complete dissolution of SA within the oil droplets.

Solution 3, catalyst solution: 0.7 g of ammonium chloride and 0.36 g of resorcinol were dissolved in 20 mL of deionized water under magnetic stirring (300 rpm) for 10 min.

A 500 mL glass reactor was used to perform the polymerization by mixing the prepolymer solution with the oil phase solution at 50°C under mechanical stirring (340 rpm) for 15 min. Subsequently, the catalyst solution was added while the pH was adjusted to 4.0 using acetic acid. The polymerization reaction was conducted at 50°C for 5 h under continuous stirring.

After 5 h, the reaction mixture was cooled to room temperature. The microcapsules were collected by vacuum filtration and neutralized by washing them with sodium hydroxide (0.1 M, 3 × 100 mL), and then washed them again with ammonium chloride solution (1% w/v, 3 × 100 mL) to eliminate unreacted formaldehyde. A final rinse with deionized water (3 × 100 mL) was necessary to remove residual salts prior to being dried at 40°C for 12 h.

2.3 | Characterization

2.3.1 | Morphological and Dimensional Analysis

The morphological and size distribution of the microcapsules were examined using a Scanning electron microscope (TESCAN VEGA, Czech Republic). Samples were sputter-coated with gold prior to analysis. The particle size distribution was determined through Dynamic light scattering (DLS) (Litesizer 500, Anton Paar, Austria) following dispersion of the microcapsules in deionized water and ultrasound sonication (Cristófoli, Brazil).

2.3.2 | Immobilization of the Microcapsules onto a Cotton Fabric

Bleached 100% cotton fabric (style 400, 100 g·m⁻², ISO 105-F02) samples (10 cm × 10 cm) were dip-coated into an impregnation bath of microcapsules (40 g·L⁻¹), melamine resin (100 g·L⁻¹), nonionic softener (30 g·L⁻¹), and MgCl₂·6H₂O (12 g·L⁻¹) in distilled water. The cotton fabrics were immersed for 1 min at room temperature (25 ± 2°C) with a bath ratio of 50:1, v/w. Subsequently, the cotton swatches were padded using a laboratory padder (Texcontrol, Brazil) at 1 bar pressure, up to achieving 100% wet pickup. The treated fabrics were dried at 60°C for 5 min, and cured at 150°C for 3 min in a laboratory pin-frame stenter (Werner Mathis AG, Switzerland).

2.3.3 | Determining the Core Content of Microcapsules

The encapsulation efficiency of the active material was determined using a gravimetric solvent extraction method [37], to quantify the total core material (LEO + SA) entrapped within the polymeric shell. 1 g of dried microcapsules was used for this procedure and labeled as m_1 . These microcapsules were mechanically disrupted by maceration and subsequently subjected to Soxhlet extraction with acetone (50 mL) for 24 h. The suspension was subjected to vacuum filtration through Whatman Number 1 filter paper, followed by washing with acetone to ensure complete

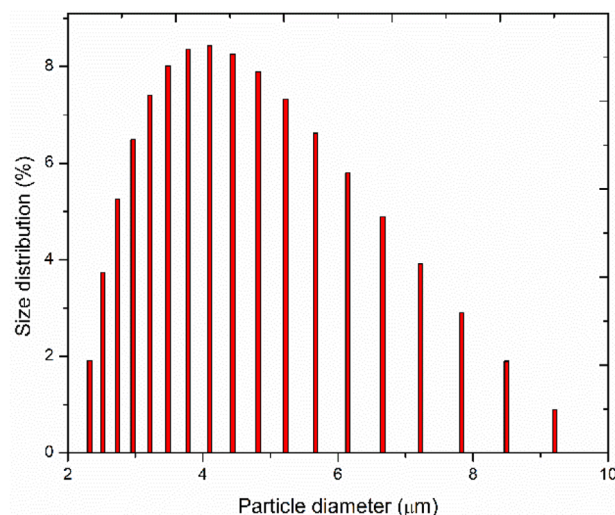


FIGURE 3 | Particle size distribution by DLS of microcapsules containing lemongrass essential oil (*Cymbopogon citratus*) and salicylic acid.

removal of the core material. The resulting solid residue from the shell was dried in an oven at 60°C for 6 h, and this mass was designated as m_2 . The core material loading percentage (%C) was calculated according to Equation (1):

$$\%C = \left[\left(\frac{m_1 - m_2}{m_1} \right) \times 100 \right] \quad (1)$$

All analyses were performed in triplicate.

2.3.4 | Chemical Characterization of the Core Compounds

Confirmation of the composition of the active compound was achieved through Fourier-transform infrared (FTIR) (Frontier Spectrometer, PerkinElmer, USA). Spectra were recorded in the wavenumber range of 4,000–400 cm⁻¹ with a resolution of 4 cm⁻¹ using the attenuated total reflectance (ATR) technique. For comparative analysis, control samples consisting of the empty MF shells of microcapsules containing only LEO were employed.

2.3.5 | Thermogravimetric Analysis

The thermal stability of the microcapsules of LEO+SA immobilized on the cotton fabrics was assessed by Thermogravimetric analysis TGA (TGA-50, Shimadzu, Japan). A sample of approximately 5 mg was heated from 30 to 500°C at a heating rate of 10°C·min⁻¹ under an inert nitrogen atmosphere (flow rate: 50 mL·min⁻¹). For comparative purposes, control samples of just MF reaction and capsules containing just LEO were analyzed under identical conditions.

2.3.6 | Qualitative Determination of Free Formaldehyde

Residual formaldehyde was assessed using the Tollens' test. Aqueous ammonia solution (NH₃, 2 mol·L⁻¹) was added to a

silver nitrate solution (AgNO_3 , $0.1 \text{ mol}\cdot\text{L}^{-1}$) until formation of a silver oxide (Ag_2O) precipitate. The precipitate was dissolved via dropwise addition of ammonia until complete solubilization was achieved, yielding $[\text{Ag}(\text{NH}_3)_2]^+$. The evaluated samples comprised: (i) standard formaldehyde solutions at concentrations of $32 \text{ mL}\cdot\text{L}^{-1}$ (positive control representing the maximum concentration employed in the process), (ii) 0.2% (positive control corresponding to the maximum limit established by Brazilian Health Regulatory Agency (ANVISA) for cosmetic applications); (iii) residual bath solutions containing microcapsules of LEO+SA. The assay was performed in test tubes by adding 10 mL of each solution and 10 mL of freshly prepared Tollens' reagent to individual tubes. The resulting mixtures were incubated at $40 \pm 2^\circ\text{C}$ for 30 min in an oven, and the reaction was monitored visually throughout the incubation period. Formation of a silver mirror on the tube shells or precipitation of dark solids was interpreted as a positive result for the presence of free formaldehyde.

3 | Results and Discussion

3.1 | Particle Morphology, Size Distribution, and Encapsulation Efficiency

Figure 2a,b shows that microcapsules of LEO+SA exhibit a predominantly spherical morphology with a mild agglomeration resulting from interparticle interactions during synthesis and drying. This effect was intensified by the presence of unencapsulated resin residue adhering to the microcapsules' surfaces, a phenomenon previously documented in microencapsulation systems [38].

When applied onto cotton fibers, Figure 2c,d, the microcapsules readily adhered to the textile surface, demonstrating effective immobilization. DLS measurements performed in aqueous medium, Figure 3, yielded a hydrodynamic diameter of $4.59 \pm 1.56 \mu\text{m}$, with the majority of the particle size population distributed in the $4.00\text{--}5.00 \mu\text{m}$ range.

The polydispersity index (PDI) of 0.12 indicates a relatively narrow size distribution, which is favorable for batch-to-batch reproducibility and predictable controlled release behavior. The encapsulation efficiency was determined to be $67.90 \pm 1.41\%$, which is consistent with values reported for similar polymeric systems [39].

3.2 | Evaluation by FTIR Spectroscopy

The FTIR spectra of the microcapsules and the reference MF were obtained by ATR-FTIR in the range of $4,000\text{--}400 \text{ cm}^{-1}$, Figure 4.

The pure spectrum of the MF resin (blue line, Figure 4a) exhibited characteristic bands attributable to its triazine network [39]. A broad absorption was observed at $3,310\text{--}3,200 \text{ cm}^{-1}$, corresponding to N–H stretching vibrations overlapped with residual O–H modes [40, 41]. The triazine ring vibrations were evidenced by intense absorptions between $1,650$ and $1,540 \text{ cm}^{-1}$, assigned to C=N stretching coupled with aromatic ring deformations [42]. Additionally, bands were detected at $1,460\text{--}1,450 \text{ cm}^{-1}$ (N–H bending vibrations), $1,360\text{--}1,340 \text{ cm}^{-1}$ (C–N

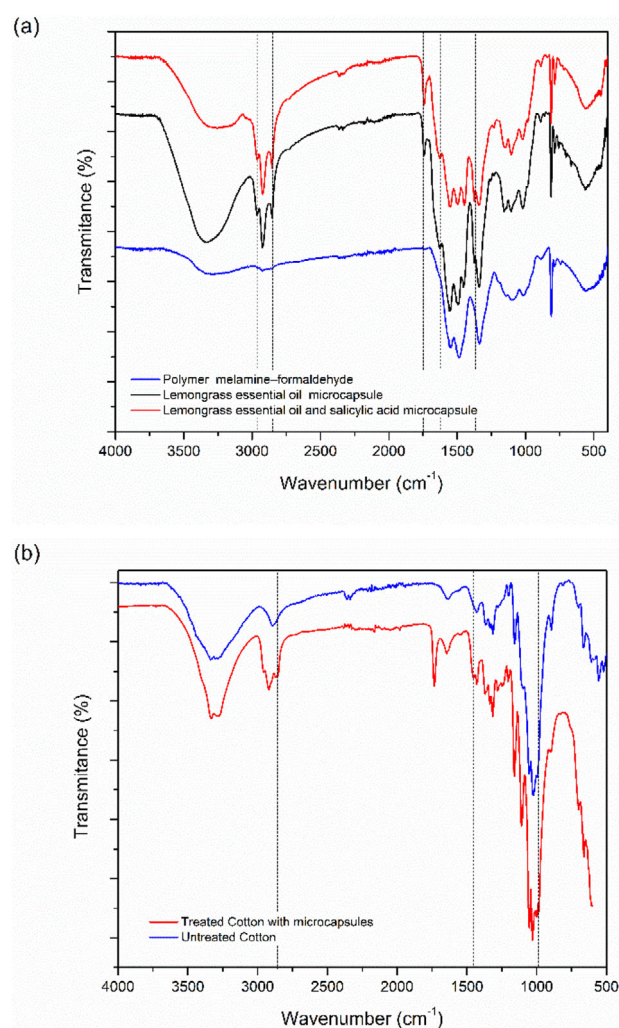


FIGURE 4 | FTIR spectra of the (a) reference polymer melamine-formaldehyde (blue line), Lemongrass essential oil microcapsule (black line), and Lemongrass essential oil and salicylic acid microcapsule (red line), and (b) Untreated cotton (blue line) and Lemongrass essential oil and salicylic acid microcapsule (red line).

stretching of melamine), and an intense absorption at 810 cm^{-1} , characteristic of out-of-plane bending deformation of the triazine ring, confirming the formation of the typical aminoplastic resin structure [31, 41].

Analysis of LEO microcapsules (black line Figure 4a) also shows the typical MF matrix bands. New absorptions emerged between $1,735$ and $1,710 \text{ cm}^{-1}$, attributable to C=O stretching of carbonyl groups from citral [43], alongside a relative intensification of aliphatic bands at $2,963$, $2,924$, and $2,852 \text{ cm}^{-1}$, related to C–H stretching of methyl and methylene groups present in the essential oil's monoterpenes [31, 44–46]. The partial overlap of these bands with the MF spectrum evidenced efficient encapsulation. Bands at $1,375$ and $1,445 \text{ cm}^{-1}$, related to C–H bending of methylene groups, corroborated the effective condensation of melamine and formaldehyde during in situ polymerization [42].

For LEO+SA microcapsules (red line Figure 4a), a well-defined additional peak was detected at the region of $1,745 \text{ cm}^{-1}$, assigned to C=O stretching of the carboxylic acid carbonyl from the

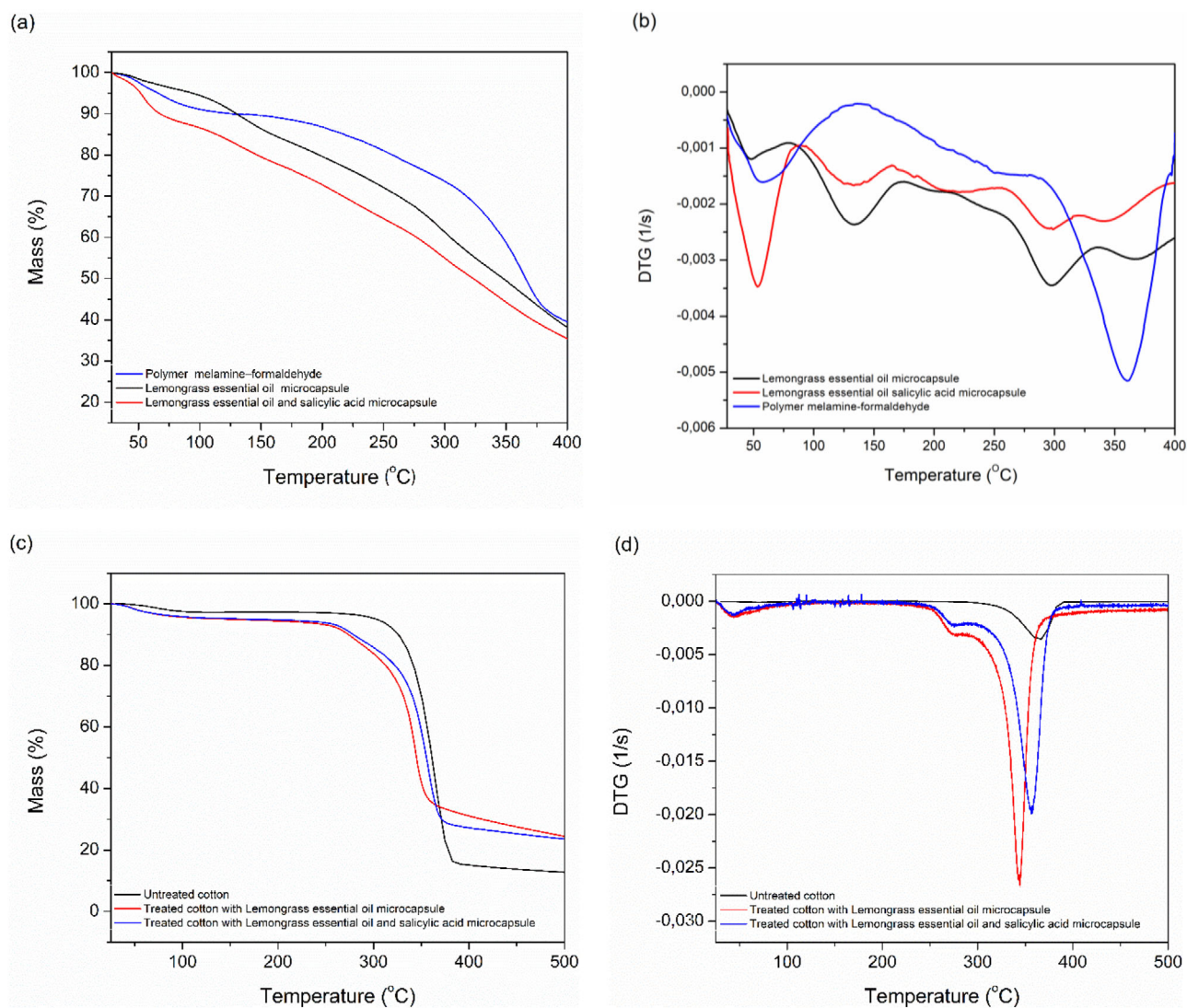


FIGURE 5 | Thermogravimetric analysis: (a, b) TG and DTG curves of melamine-formaldehyde reference polymer (blue), lemongrass essential oil microcapsules (black), and lemongrass essential oil and salicylic acid microcapsules (red); (c, d) TG and DTG curves of untreated cotton fabric (blue), cotton fabric treated with lemongrass essential oil microcapsules (black), and cotton fabric treated with lemongrass essential oil and salicylic acid microcapsules (red).

SA [47, 48]. Furthermore, vibrations at 1,448 and 1,248 cm^{-1} , are attributed to aromatic ring deformation and phenolic C—O stretching, which are also characteristic of SA [49].

The shift of the MF triazine ring vibration from 1,550 cm^{-1} (pure MF) to 1,545 cm^{-1} (LEO+SA microcapsule) suggests electronic perturbation induced by proximity to SA's aromatic system. More significantly, the broadening of the N—H stretching region (3,310–3,200 cm^{-1}) in LEO+SA microcapsules indicates hydrogen bond formation between the shell amine groups and SA's carboxylic acid (—COOH) and phenolic hydroxyl (—OH). This interpretation is corroborated by the slight red-shifting of the SA carbonyl band from 1,760 cm^{-1} (free SA) to 1,745 cm^{-1} (LEO+SA microcapsules), characteristic of C=O groups engaged in hydrogen bonding. The simultaneous detection of both SA (1,745 cm^{-1}) and citral (1,715 cm^{-1}) carbonyl signatures confirms successful co-

encapsulation. These spectral features demonstrate that the MF shell provides a dual stabilization mechanism: hydrogen bonding with amphiphilic SA at the core-shell interface and hydrophobic sequestration of LEO components within the capsule's interior.

For the cotton fabrics (Figure 4b), the untreated cotton spectrum (blue line) exhibited characteristic cellulose bands: a broad O—H stretching vibration at 3,330–3,270 cm^{-1} , C—H stretching vibrations at 2,900 cm^{-1} , and a strong C—O—C glycosidic vibration at 1,050–1,020 cm^{-1} [50]. Following functionalization with the LEO+SA microcapsules (red line, Figure 4b), a slight broadening of the O—H region (3,400–3,200 cm^{-1}) was observed, similar to that seen in Figure 3a. Additionally, new bands appeared at 1,735 and 1,715 cm^{-1} , corresponding to C=O stretching from citral and SA, hence conforming to the encapsulation of both compounds [43, 47].

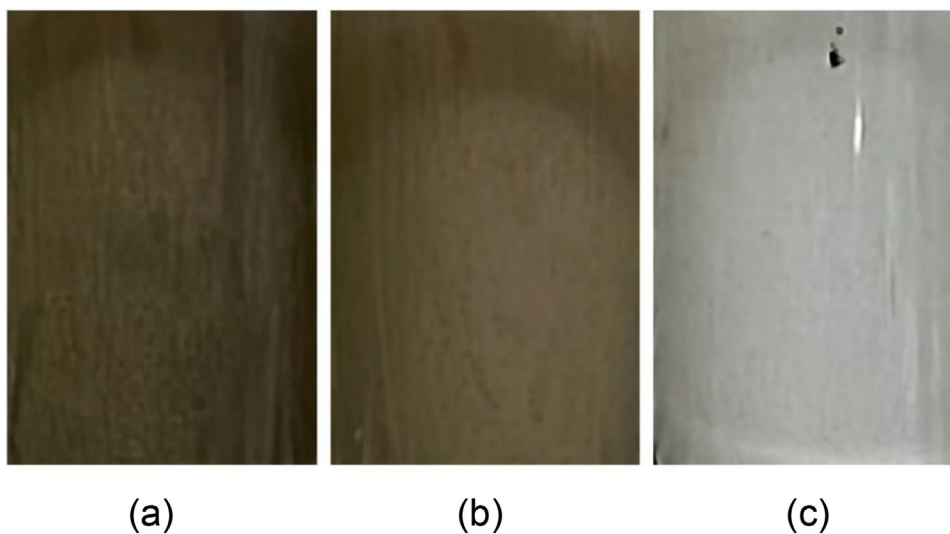


FIGURE 6 | Qualitative detection of free formaldehyde by Tollens' reagent in different solutions: (a) solution containing 32 mL·L⁻¹ formaldehyde (positive control – maximum concentration used in the process); (b) solution containing 0.2% formaldehyde (positive control – maximum concentration allowed by ANVISA in cosmetic products); (c) residual bath solution containing the microcapsules of LEO+SA.

3.3 | Thermogravimetric Analysis

Thermogravimetric analysis, Figure 5, shows distinct thermal profiles for the precursor materials, the LEO+SA microcapsules, and the functionalized fabrics.

The MF thermogram exhibited two main decomposition events as shown in Figure 5a,b. An initial mass loss (23–128°C; 10.1%) was attributed to water and volatile oligomer removal [51, 52]. MF network decomposition ($T_{\max} = 360.2^\circ\text{C}$) involves formaldehyde release and ether-to-methylene bridge conversion [53, 54], yielding a final residue of 39.5%.

The thermogram for the LEO microcapsules displays four decomposition stages shown in Figure 5a,b: (i) volatile evaporation up to 78°C (3.9%) [51]; (ii) initial shell decomposition with partial core release at 87–173°C (12.3%) [52]; (iii) polymer matrix decomposition at 261–335°C (17.3%) [44]; and (iv) final core release up to 397°C, yielding a final residue of 37.9%. The microcapsules of LEO+SA exhibited five stages, Figure 4a,b, including an additional event at 170–254°C (13.1%) associated with SA decomposition [55]. The main shell decomposition occurred at 262–319°C (11.6%), followed by final core release at 323–396°C (13.2%), hence yielding a final residue of 35.2%.

The presence of new thermal events below 250°C confirmed the presence of LEO+SA encapsulated cores. Systematic T_{onset} and $T_{\max}$ reductions indicated thermal stability modification by the organic load. The polymeric barrier delayed the volatilization of EO, and the addition of SA introduced an extra decomposition step [31, 56].

The thermogram for the unmodified cotton fabric, used as a control, is shown in Figure 5c,d, and it exhibits a thermal profile characteristic of pure cellulose. Dehydration occurred up to 114°C, followed by cellulose decomposition in the range of 300–370°C ($T_{\max} = 355^\circ\text{C}$), yielding a final residue of 14.63%.

The cotton fabric coated only with microcapsules of LEO exhibits an additional thermal event at 223°C, corresponding to an approximately 8% mass loss, which is attributed to polymer shell decomposition and partial release of the encapsulated LEO. Cellulose decomposition occurred at 348°C, yielding a final residue of 30.91%. The cotton fabric functionalized with microcapsules of LEO+SA displayed a similar thermal profile, exhibiting an intermediate event at 223°C and cellulose decomposition at 345°C, yielding a final residue of 27.17%.

The DTG peaks of the functionalized cotton fabrics shifted 7–10°C to lower temperatures relative to those in the unmodified control fabric, which is indicative of the interactions between the polymeric shell and the cellulose fibers.

3.4 | Detection of Free Formaldehyde

Figure 6 presents the qualitative determination of free formaldehyde using Tollens' reagent. The positive control containing 32 mL·L⁻¹ formaldehyde (Figure 6a) exhibited intense darkening, with a visible silver mirror formation, hence confirming complete reduction of $[\text{Ag}(\text{NH}_3)_2]^+$ complex. The 0.2% formaldehyde solution (Figure 6b), corresponding to the Brazilian Health Regulatory Agency (ANVISA) regulatory limit for cosmetics, yielded a moderate positive response, confirming the assay's sensitivity.

In contrast, the residual bath sample containing the microcapsules of LEO+SA (Figure 6c) shows no detectable silver mirror or precipitate formation, hence indicating no detectable free-formaldehyde after the polymerization and washing steps.

The mechanistic basis for formaldehyde depletion involves its incorporation into the polymer network through the hemiacetal and acetal linkages during urea-formaldehyde resin formation. FTIR analysis corroborates this mechanism, with bands at $\sim 1,030\text{--}1,150\text{ cm}^{-1}$ attributed to C–O–C stretching, confirming formaldehyde integration into stable covalent structures rather

than remaining as a free aldehyde. The concurrent evidence from Tollens' test (no free-aldehyde detection) and FTIR spectroscopy (presence of ether linkages) provides complementary validation of complete formaldehyde incorporation into the polymeric shell of the microcapsules.

4 | Conclusion

We report on a method to obtain microcapsules containing LEO and SA as via in situ polymerization and the immobilization of these microcapsules onto cotton fabrics. This method overcomes the large differences in polarities between LEO and SA. Morphological analyses revealed predominantly spherical particles with a mean diameter of $4.59 \pm 1.56 \mu\text{m}$ and relatively homogeneous distribution, while encapsulation efficiency reached $67.90\% \pm 1.40\%$. FTIR spectroscopy and TG analysis confirm the incorporation of SA and LEO. Scanning electron microscopy (SEM) analysis demonstrates an effective adhesion of the microcapsules onto the textile's surface. Thermogravimetric analysis of the functionalized cotton fabrics revealed additional thermal events and increased residual mass, hence confirming the immobilization of the microcapsules. The Tollens test and FTIR spectra corroborated the absence of residual free formaldehyde, indicating compliance with regulatory requirements and ensuring safety for the potential use of the microcapsules and the functionalized fabrics in cosmetic and dermatological applications. Additional studies on controlled release, wash fastness, skin contact performance, stability in final formulations, and biological evaluation are necessary to consolidate this potential.

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Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

Research data are not shared.

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