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Article

Development of Mosquito-Repellent Camouflage Fabric Using Eucalyptus Oil with *Moringa oleifera* Gum

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Abstract: Military personnel are exposed to several harsh conditions and mosquitos in mountains and wild forests. Mosquito-repellent textiles can help them to cope with such conditions. The present research work established a sustainable approach for fabricating microcapsules from Eucalyptus oil, *Moringa oleifera*, and Arabic gum via a complex coacervation method. *Moringa oleifera* and Arabic gums were utilized as the outer shell of the microcapsules, whereas the core part was made of Eucalyptus oil in different concentrations. The military camouflage-printed polyester/cotton (PC) blended fabric was coated with the as-prepared microcapsules using the pad–dry–cure technique. The surface morphology of the microcapsules was examined using an optical microscope and scanning electron microscope (SEM), and the coated fabric's mosquito-repellent property was investigated using a specified cage test according to a standard testing protocol. The water absorbency and air permeability of the treated samples were also evaluated in order to learn about the comfort properties. The cage test results revealed that the coated fabric had a good tendency to repel the mosquitoes used in the cage test. In addition, the coated fabric showed significant durability even after several rigorous washing cycles. However, the application of microcapsules to the fabric slightly affected the water absorbency and air permeability of the fabric. This study presents a novel sustainable approach for fabricating microcapsules from the mentioned precursors and their application in the field of textiles, particularly for military purposes.

Keywords: microcapsules; Eucalyptus oil; *Moringa oleifera* gum; mosquito repellency; protective textiles

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1. Introduction

With the advancement of technology, functionality, along with aesthetics, have become an important focus of the textile industry and daily life apparel [1,2]. Protective textiles are textile products that provide protection for humans, and insect repellent textiles protect humans from vector-borne diseases like dengue and malaria [3,4]. These insect repellent and protective textiles provide safety features not only for daily routines, but they can also have a large impact on military uniform applications [5]. Many factors, such as fast urbanization, as well as a changing climate, are responsible for the rapid increase in the growth of mosquitos [6]. According to the World Health Organization (WHO), malaria is one of the six major communicable diseases that cause millions of deaths annually. Insect-repellent cloths are considered to be protective textiles that support safety features [7].

Therefore, to ensure a safe barrier between humans and vector-borne diseases, new technologies need to be developed. Existing repellents are synthetic/chemical, which are not considered to be eco-friendly [8]. Chemicals, such as N,N-diethyl-meta-toluamide (DEET), used as mosquito-repellent functional finishing materials can cause severe and diverse effects, including allergies, rashes, and the redness of the skin, when they are used for long durations and in high concentrations [9]. On the other hand, finishing materials prepared from essential oils, such as rosemary, clove oil, lemon grass oil, peppermint oil, and castor oil, as well Eucalyptus oil, are well reported to have good repellence properties and a minimal impact or no impact on the atmosphere and non-targeted species [10–12]. Essential oil-based repellents and insecticides have good potential because of their organic properties for protection, and Eucalyptus oil is known for its significant use as an antifungal, antibacterial, and antiseptic agent. Because of its multipurpose ability, it is far superior to other essential oils [13,14]. *Moringa oleifera* is a type of organic gum which is considered as a sensational plant because of its many exceptional properties. Almost every element of this tree has nutritional, pharmaceutical, and industrial functions [15]. It has antioxidant, anti-inflammatory, and antimicrobial properties, and it can be used for ultraviolet ray protection [16,17]. Arabic gum is also considered to be one of the best antimicrobial and antioxidant natural materials used in the textile industry [18,19]. Organic essential oils and gums have been utilized individually or in combination using the simple pad–dry–cure method to develop functional textiles. However, the biggest challenge is the durability of their special properties after commercial or domestic laundering. In textile finishing, the shelf life can be improved by using the encapsulation technique. The durability of the treated fabric is enhanced to be flame-retardant, insect-repellent, thermal-regulated, thermal and photo-chronic, and aromatic. Here, in this research, a similar strategy was employed to encapsulate mosquito-repellent materials and enhance their durability.

The purpose of this study was to develop protective finishes through microencapsulation using green chemistry and natural products that are sustainable, environmentally friendly, and have no impact on other non-targeted products. For this purpose, microcapsules were fabricated from Moringa and Arabic gums (as the outer shell) with Eucalyptus oil (inside the core). The as-prepared microcapsules were applied to polyester/cotton (PC) blended fabric to enhance the mosquito-repellent property of fabrics used by security and armed personnel.

2. Materials and Methods

2.1. Substrate and Materials

A fabric made of cotton blended with polyester (PC) printed with a camouflage design was used as the substrate. A 2/1 twill ripstop-type woven fabric of 250 g/m^2 was composed of warp and weft yarns, both being made of 60% cotton and 40% polyester. The warp yarn (count: 20/1) had 18 twists per inch (TPI) and 122 ends/inch, while the weft yarn (count: 16/1) had 16 TPI and 65 picks/inch.

Moringa gum, Arabic gum, and Eucalyptus oil were sourced from VWR Chemicals (Techtron Technologies, Delhi, India), while other chemicals, like gelatin, dilute acetic acid, acrylic binder, and Tween 20, were obtained from Sigma Aldrich (Burlington, MA, USA) and a local supplier. The printed camouflage cotton/polyester blended fabric was obtained from Nishat Textile Mills, Lahore, Pakistan. All chemicals were used as received without further purification and processing. Distilled water was used throughout this research work.

2.2. Methods

Two different types of finishes were prepared using Moringa gum with Eucalyptus oil and with Arabic gum and Eucalyptus oil. In both types of microcapsules, Eucalyptus oil was used as the core material with different shell materials.

A complex coacervation method was adopted for the preparation of the microcapsules, as reported in the literature [17]. In solution 1: 12 g gelatin was dissolved in distilled water at 40 °C and stirred at 250 rpm for 10 min (Figure 1a). In solution 2: 3, 6, and 12 g of shell material gum (Arabic gum and Moringa gum) were dissolved separately in distilled water under continuous stirring at 250 rpm. Four mL and five mL core material (Eucalyptus oil) were added in solution 2 one by one at 50 °C and 500 rpm stirring speed (Figure 1b). Then, 30 mL of solution 1 was added to solution 2 under vigorous stirring (250 rpm) for 20 min. Subsequently, the stirring speed was increased to 500 rpm, and 190 mL distilled water was added to the solution for 30 min at 50 °C. To escalate the coacervation process, the pH of the solution was sustained at 4.5 via the addition of 10% acetic acid in dropwise manner. The temperature of the solution was lowered to 10 °C, and 210 mL of distilled water was added again and stirred for 60 min at 250 rpm. Later, 25% of glutaraldehyde was added as a crosslinking agent (Figure 1c). The pH was increased to 8.5 to stabilize the formation of the microcapsules. The solution was neutralized for 8–9 h in a separate funnel. In the last step, a stabilizing agent Tween 20 was added at 25 °C and stirred well at 700 rpm for approximately 15 min to acquire the microcapsules (Figure 1d).

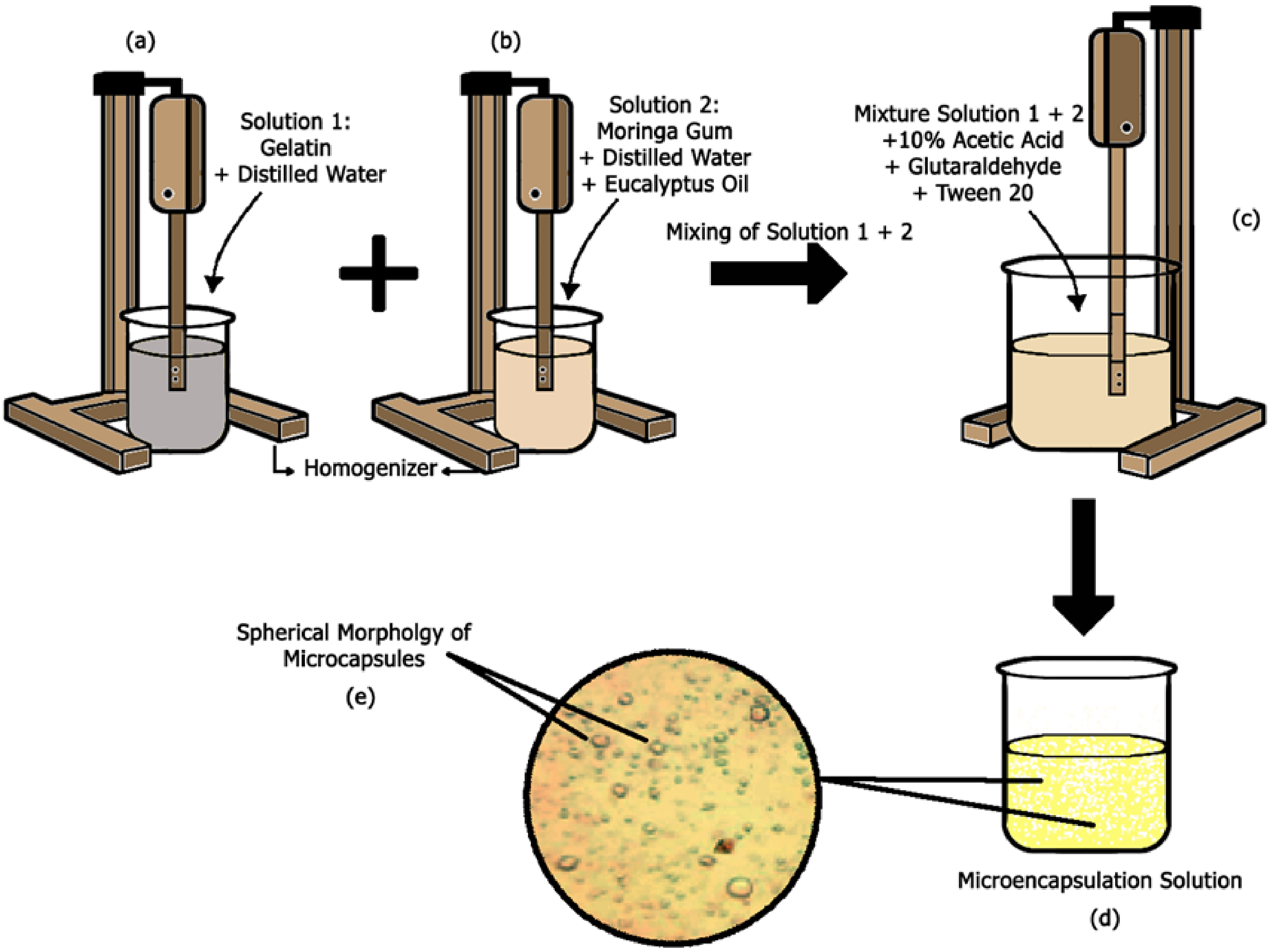

Figure 1. Schematic diagram of the synthesis of microcapsules: (**a**) solution 1, (**b**) solution 2, (**c**) mixing solution 1 and 2, (**d**) microcapsules in solution, and (**e**) optical microscopic image of the prepared microcapsules.

Three different solutions were prepared with different concentrations of shell materials (Moringa gum and Arabic gum) and core material (Eucalyptus oil), as shown in Table 1. Four fabric swatches were cut out with same dimensions, i.e., of 8 × 12 inches, and each swatch was marked as S-1, S-2, S-3, and S-0, respectively (Table 1). Different dispersions of microcapsules were made at a liquor-to-goods ratio of 50:1 for each printed fabric sample. Each dispersion was stirred and homogenized for 20 min at room temperature to make a uniform, finished solution.

Table 1. Composition difference of microcapsules samples in terms of shell and core materials.

Sample No.	Shell (Material + Conc.)	Core (Material + Conc.)	Fabric Weight	Disperse Solution (1:50)	Acrylic Binder
S0	Untreated	Untreated		----	----
S1	3% Moringa gum	4 mL Eucalyptus oil			
S2	6% Moringa gum	5 mL Eucalyptus oil	18 g	1000 mL	20 g/L
S3	12% Arabic gum	5 mL Eucalyptus oil			

2.3. Application on the Blended Polyester Cotton Fabric

Microcapsules dispersions were applied to the substrate fabric using the pad–dry–cure technique. PC blended fabric with 60% cotton and 40% polyester was cut to a dimension of 8 × 12 inches. The dispersions were prepared using microcapsules (30 g/L) and acrylic binder (20 g/L), and the liquor ratio was kept at 1:50.

Then, the PC blended fabric was dipped (Figure 2c) into the beaker containing microcapsule dispersion for 30 min. All the samples were padded 5 times using a lab-scale padder with 2.7 kg pressure rollers (Figure 2d). Then, they were dried out in stenter frame that was preheated at 100 °C for 3 min (Figure 2e) and cured at 120 °C for about 60 s (Figure 2f). The finished fabrics were then tested for mosquito repellency and washing durability and comfort (air permeability and water absorbency). SEM images were taken before and after the application of the microcapsules.

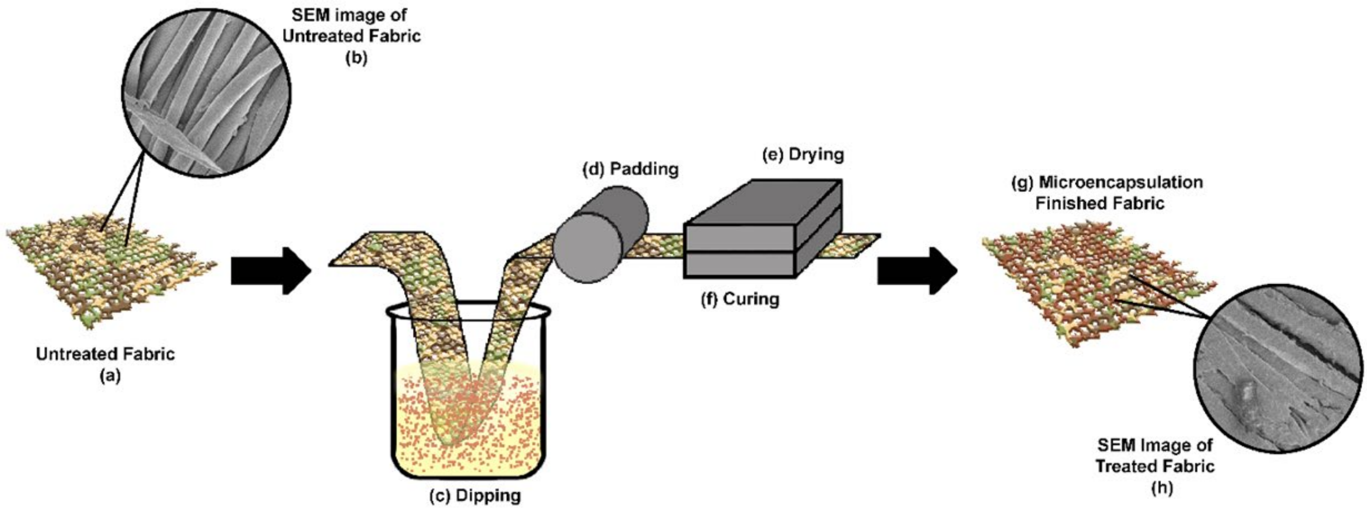

Figure 2. Schematic diagram of the application of microcapsules to the PC fabric: (**a**) untreated fabric, (**b**) SEM image of untreated fabric, (**c**) dipped microcapsules, (**d**) padding, (**e**) drying, (**f**) curing, (**g**) finished fabric, and (**h**) SEM image of finished fabric.

2.4. Testing

2.4.1. Mosquito-Repellent Testing

The untreated fabric and treated fabrics were tested for mosquito repellency in a cage test according to the standard protocol (Figure 3). From 25 to 30 mosquitos were captured in a flask for one sample test. A wooden cage was built with a cubical shape with dimensions of 35 cm on all sides. The sides of the cage were covered with mesh/net material so that a sufficient amount of air could pass through the cage.

Aedes Aegypti-type mosquitoes were used for the mosquito repellency test, which were bred in a laboratory at the University of Veterinary and Animal Sciences (UVAS), Lahore. The adult mosquitoes were then released in this cage so they would attack the forearm covered with the substrate. A fresh batch of mosquito were used for every single test, and each test was repeated three times for each sample.

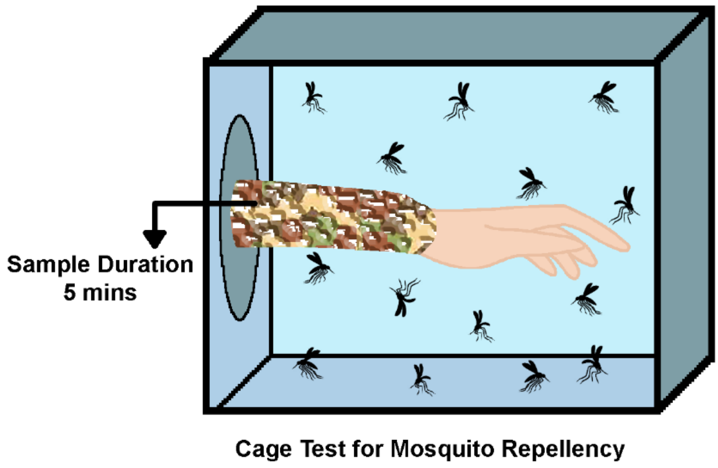

Figure 3. Schematic diagram of cage test for mosquito repellency of textile fabric.

A human hand and forearm protected with gloves and the treated fabric was introduced through a pipe hole inside the chamber to check the repellency property of the fabric against the mosquitoes. The fabric wound on the arm was tested for about 5 min each time inside the chamber. Two observers keenly observed the reaction of mosquitoes towards the fabric substrate so that the number of mosquitoes that landed on the forearm could be counted and recorded [18].

All tests were carried out under safe specific observation and testing standards at the University of Veterinary and Animal Sciences, Lahore, Pakistan.

The evaluation percentage of mosquito repellency (MR) of the surface of the fabric was calculated by the formula (Equation (1)).

$$\%\ \mathrm{MR} = (\mathrm{U} - \mathrm{T})/\mathrm{U} \times 100 \quad (1)$$

Here,

MR: Mosquito repellency;
U: Mosquitoes landed on untreated fabric;
T: Mosquitoes landed on treated fabric.

2.4.2. Scanning Electron Microscope and Optical Microscope Analyses

The morphology of the surfaces of the treated and untreated samples was analyzed using a scanning electron microscope (SEM). The samples were evaluated using an SEM FEI Quanta 250 (Thermo Fisher Scientific, Hillsboro, OR, USA). In addition, the samples were observed with a two-edge masking tape in front of the lens to investigate the shape of the microcapsules with an optical light microscope (MODEL XSZ-2101, Ningbo Shengheng Optics & Electronics Co., Ltd., Ningbo, China).

2.4.3. Washing Durability Test

The washing durability of the samples was investigated according to the AATCC standard LP1 used for domestic and home laundry. An LG front-loaded washing machine with capacity of 7 kg was used for the washing of samples using certain washing parameters. The washing process was carried for four different washes, such as 10, 20, 30, and 40 cycles, for each sample separately. The mosquito repellency test was performed after washing the samples, and the results before and after washing were compared [19]. The washing parameters are discussed in Table 2, which includes the water capacity (60 L), cycle time (16 min,) cycle type (normal flat drying), spinning speed (660 rpm), spinning time (5 min), and temperature (30 °C). Washing was performed in using a standard detergent mostly used for home laundry.

Table 2. Working parameters of AATCC washing protocol.

Sr. No.	1	2	3	4	5	6	7
Parameters	Water Capacity	Cycle Time	Cycle Type	Drying	Spinning Speed	Spinning Time	Temperature
Details	60 L	16 min	Normal	Flat	660 rpm	5 min	30 °C

2.4.4. Water Absorbency Test

The water absorbency test evaluates how much water a fabric can absorb in a specific period of time. The absorbency test was carried out using the AATCC 79 standard method via a drop test. A droplet of water was dropped on the fabric, and how much time it took to be absorbed by the fabric surface was carefully observed.

The water absorbency of the fabric was determined by comparing the time needed for a droplet to be absorbed by the untreated fabric S-0 that was not washed and treated fabric samples after 40 washes.

2.4.5. Air Permeability Test

The air permeability of the fabric was measured using the ISO standard ISO-9237 [20], which was calculated in SI units, i.e., mm/s at a pressure of 100 Pa. It evaluates the flow of air at a certain time under a specific pressure passing through the fabric. The sample size used for testing was 2 inches. The air permeability of the fabric was determined by comparing the results of untreated fabric S-0 that was not washed and treated fabric samples after 40 washes.

3. Results and Discussion

3.1. Characterization of Microcapsules

The microcapsules prepared from Moringa gum and Eucalyptus oil were observed under an optical microscope at 40× magnification and 100× magnification, respectively (Figure 4a,b). The morphology of the microcapsules under the microscope shows spherical shapes (Figure 4b) before it was applied to the surface of the fabric. It is evident that microcapsules formed, and they have a round-to-spherical shape. The microcapsules show homogeneous distribution in the solution. The Eucalyptus oil microcapsules with Moringa and Arabic gums (Figure 4c,d) show almost the same morphologies as those disclosed in the literature [21].

The surface morphologies of the treated and untreated fabrics were also determined at three different magnification levels 1000×, 25,000×, and 50,000×, respectively (Figure 5a–d). The untreated fabric (Figure 5a) clearly shows a smooth longitudinal fiber surface with no disturbance or deposition of layers on the fabric, as compared to that in Figure 5b, where a thin layer of microcapsules can be seen mixed with the acrylic binder deposited on the fibrous films of the fabric at a magnification power of 1000×. The attached residues on the surface of the fabric are a pattern of microcapsules with a binder used to enhance the durability of the finish when they were applied to the fabric. Mist film formation on the surface of the fabric has also been witnessed in the literature [22]. Figure 5c,d shows magnified characterizations at 25,000 and 50,000×, respectively. The microcapsules can be seen in a highly magnified and dispersed form on the fabric surface (Figure 5c,d). However, some disturbance in the shape of the microcapsules occurred during the high-pressure padding process.

3.2. Mosquito Repellency Test

The mosquito repellency test was carried out with the cage test, and the percentage of mosquitoes that made contact with the surface of the fabric was determined for both the treated and un-treated fabrics using Equation (1) derived above. Four different samples with different concentrations of Eucalyptus oil and gums were tested after four different wash cycles, 10, 20, 30, and 40 washes, respectively.

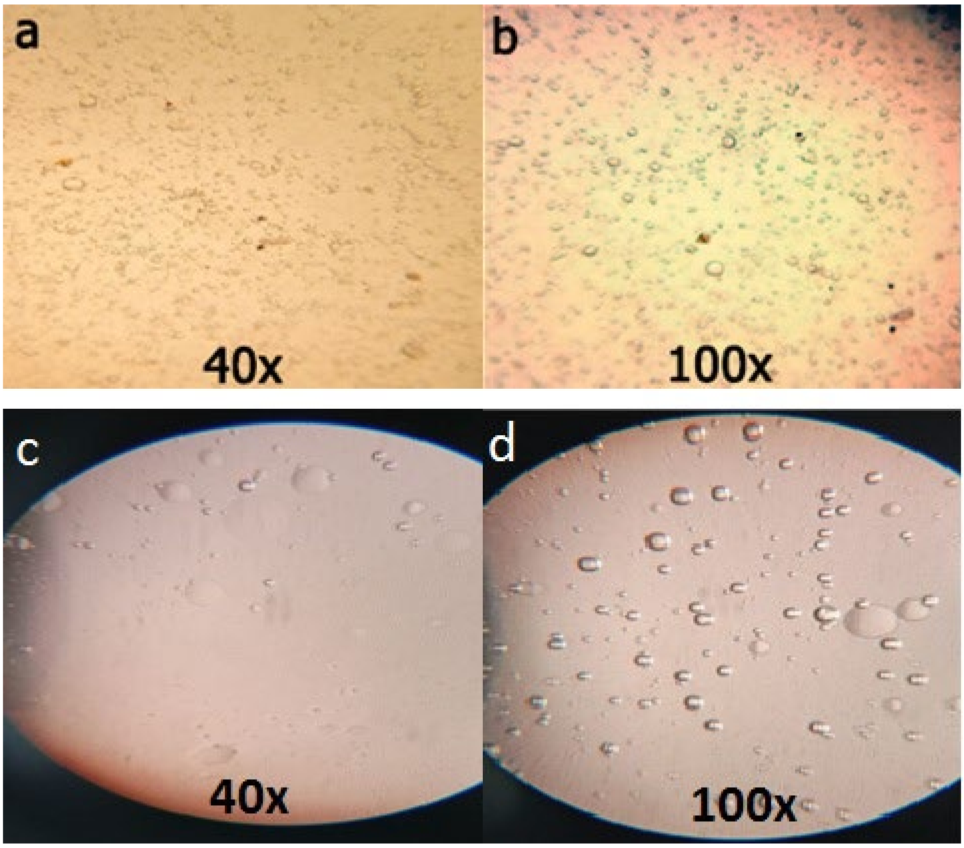

Figure 4. (**a**) Microcapsules of Moringa gum with eucalyptus oil at 40×, and (**b**) microcapsules at 100×; (**c**) microcapsules of Arabic gum with eucalyptus oil at 40×, and (**d**) microcapsules at 100×.

The fabric samples revealed positive results for mosquito repellency, as shown in Table 3. Interestingly, the results are in considerable agreement with the ones reported in the literature while using Coleus Aromaticus Leaf extracts [22]. The test results were compared individually for fabrics that underwent four different washing cycles. The sample S2, which was not washed and contained Moringa gum and Eucalyptus oil, shows the highest repellency rate, which is approximately 97% (Table 3). The concentration of Arabic gum doubled due to its lower viscosity as compared to that of Moringa gum. However, the concentration of Eucalyptus oil was kept at 5 mL in both types of microcapsules. The mosquito repellency rate of Moringa gum was higher than that of Arabic gum, which gradually reduced to 66.66% after 40 washes. In sample S2, significant repellency is shown after 0 washes, i.e., 96.66%, which reduced to 73.3% after washing. This phenomenon depicts that the durability of the microcapsule-finished fabric is high, and it has retained its position on the fabric surface. Due to rigorous washing, the mosquito repellency continuously declines as the number of washes increases because of the use of a detergent on the surface of the fabric, which makes the finish a little weak and chips it off. In sample S3, the repellency rate is 93.33%, and after 40 washes, it dropped to 60%. Hence, microencapsulation with 6% Moringa gum and 5 mL Eucalyptus oil has an enormous effect on the fabric surface of S2 in terms of mosquito repellency, providing a result of approximately 73% repellency even after 40 washes, which shows its significant durability as well.

A comparison of the microcapsules made from Moringa gum and Arabic gum with same concentration of 5 mL Eucalyptus oil is shown in Figure 6. The Moringa gum gives a higher mosquito repellency rate with a lower concentration than Arabic gum does. Moreover, it is economical in the case of mass production, as smaller quantities produce a higher repellency.

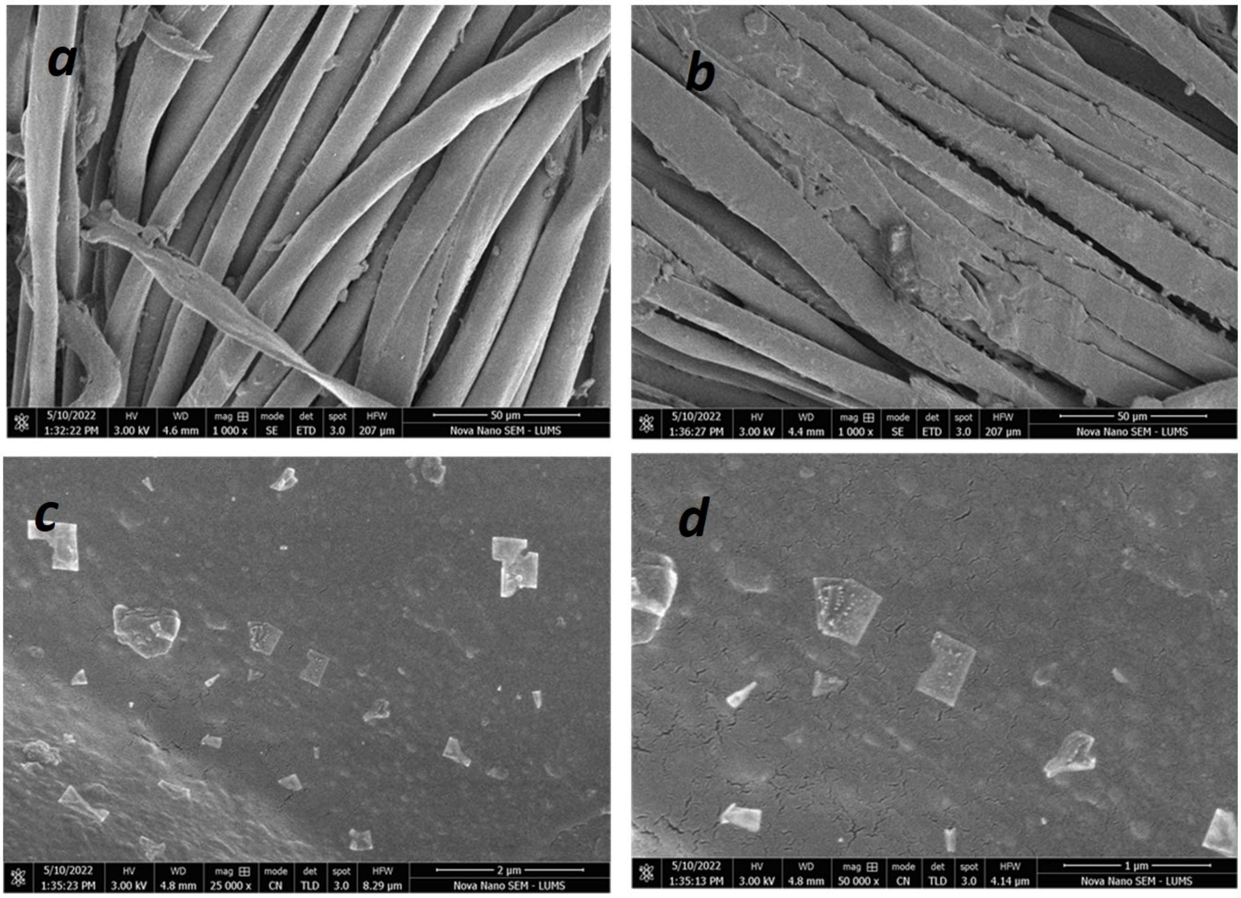

Figure 5. (**a**) Untreated fabric at 1000×, (**b**) treated fabric with microcapsules at 1000×, (**c**) treated fabric with microcapsules at 25,000×, and (**d**) treated fabric with microcapsules at 50,000×.

Table 3. Mosquito repellency results after 0, 10, 20, 30, and 40 washes of PC fabrics.

Sample No.	Microcapsules Application	No. of Mosquitoes Introduced	No. of Washing Cycles	No. of Mosquitoes Landed	Mosquito Repellency (%)
S0	Untreated	30	0	30	0
		30	10	30	0
		30	20	30	0
		30	30	30	0
		30	40	30	0
S1	3% Moringa Gum with 4 mL Eucalyptus oil	30	0	2	93.33
		30	10	3	90
		30	20	4	86.66
		30	30	5	83.33
		30	40	10	66.66
S2	6% Moringa Gum with 5 mL Eucalyptus oil	30	0	1	96.66
		30	10	2	93.33
		30	20	3	90
		30	30	4	86.66
		30	40	8	73.33
S3	12% Arabic Gum with 5 mL Eucalyptus oil	30	0	2	93.33
		30	10	4	86.66
		30	20	5	83.33
		30	30	6	80
		30	40	12	60

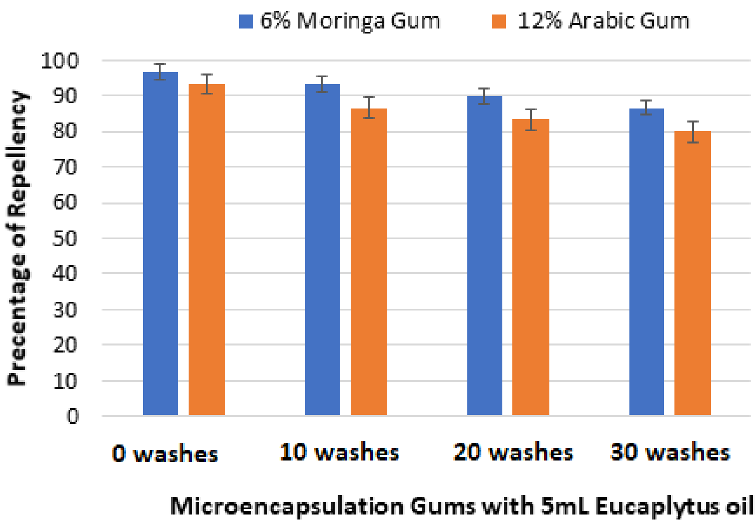

Figure 6. Comparison of percent repellency of microcapsules coated fabric made from Moringa and Arabic gums with same concentration of Eucalyptus oil in mosquito repellency tests.

3.3. Water Absorbency Test

The absorbency test results are given in Table 4, which reveals that the untreated printed PC fabric shows highest absorbency rate of 6 s, confirming that the fabric is moisture-absorbent and highly hydrophilic in nature mainly due to the presence of cotton. However, the finished fabric showed a decrease in the absorbency of water droplets to 31 s for 3% Moringa gum in S1, which shows an increase in absorbency after 40 washes to 18 s. In S2, the absorbency time was 80 s after 0 washes, which decreased to 23 s after 40 washes. In S3, the absorbency time was 110 s, which decreased to 47 s after 40 washes. In addition, the increase in the gum concentration increases the time needed for the absorption process to occur, ultimately affecting the properties of the textile materials. This phenomenon can be attributed to the clogging of small pores in the fabric structure by the finish applied. These results are in considerable agreement with the previously published literature [23].

Table 4. Water absorbency values of untreated and treated fabrics.

Sample No.	Microcapsule Finish	Absorbency Time (s)	
		0 Washes	40 Washes
S0	Untreated	6	6
S1	3% Moringa gum 4 mL Eucalyptus oil	31	18
S2	6% Moringa gum 5 mL Eucalyptus oil	80	23
S3	12% Arabic gum 5 mL Eucalyptus oil	110	47

3.4. Air Permeability Test

The air permeability test results concluded that the permeability of the untreated fabric is 45 mm/s, which is standard value for the PC blended fabric, and it remained the same even after 40 washes. The fabric with 3% Moringa gum and 4 mL Eucalyptus oil showed 13.4 mm/s air permeability after 0 washes, which increased to 30 mm/s after 40 washes. The fabric with 6% Moringa gum and 5 mL Eucalyptus oil showed 11.2 mm/s air permeability after 0 washes, which increased to 28 mm/s after 40 washes. The fabric with 12% Arabic gum and 5 mL Eucalyptus oil showed 11.9 mm/s air permeability, which increased to 29 mm/s after 40 washes (Table 5), and as the number of washes increased, the fabric pores tended to open due to the rigorous cycles of washing, which allowed more air to pass through the fabric. The air permeability was slightly affected by the application of the microcapsule coating, which is a common challenge in textile finishing and coating [24–27].

Table 5. Air permeability values of untreated and treated PC fabrics.

Sample No.	Microcapsule Finish	Air Permeability (mm/s)	
		0 Washes	40 Washes
S0	Untreated	45	45
S1	3% Moringa gum 4 mL Eucalyptus oil	13.4	30
S2	6% Moringa gum 5 mL Eucalyptus oil	11.2	28
S3	12% Arabic gum 5 mL Eucalyptus oil	11.9	29

4. Discussion

The development of mosquito-repellent finished fabrics by using Moringa and Arabic gums with Eucalyptus oil was the basic goal when we were conducting this study. Mosquito-repellant finished textiles help to protect people from mosquitoes without using any liquid bases on the skin. They can be used by military personnel and athletes during their long, hectic working shifts to protect them from mosquitoes. Due to their low toxicity and high customer acceptability, natural materials, such as essential oils, are becoming increasingly popular. As a result, developing long-lasting repellent textiles based on essential oil impregnation is critical. Therefore, the synthesis, characterization, and application of microcapsules to PC fabrics was performed, which helped to impart mosquito repellency. However, more studies based on chemical-based mosquito-repellent finishes have been conducted, as cited above in the Introduction Section. The major limitation is that natural finishes are not durable after rigorous washing. Here, in this study, a successful attempt has been made to achieve excellent durability even after 40 washing cycles. It is pertinent to mention that the air permeability and absorbency were slightly changed after the finishing treatments, which can be avoided by slightly decreasing the acrylic binder concentration used.

Research can be conducted for the purpose of producing bio/eco-friendly mosquito-repellent finished textiles. The substrate used for this research were military uniforms because the armed forces are more susceptible to vector-borne diseases because they spend more time in fields. The application of a mosquito-repellent finish during production is a more effective and suitable way to provide protection against mosquitos. Other approaches to avoid mosquitos, like lotions sprays, etc., can be less effective in terms of durability because a repellent finish applied to a textile substrate typically retains mosquito repellency longer than one applied to skin does.

5. Conclusions

The development of mosquito-repellent finished fabrics by using the microencapsulation of Moringa gum and Eucalyptus oil via a coacervation method were successful. The characterization confirmed the formation of spherical homogeneous microcapsules. These microcapsules were applied to printed camouflaged polyester cotton blended fabric used by army personnel and were tested against Ades Aegypti-type mosquitoes. Moringa gum shows a relatively higher repellency rate than Arabic gum does. Moringa gum, in comparison with Arabic gum, is used less often (6% instead of 12.5%) due to its firmer texture, but it provided better results in the washing durability test. Due to an increase in the concentration of the finish on the fabric, the air permeability and absorbency results were moderately affected. The finished fabric shows a very high durability result after 40 washes with a good mosquito repellency rate. This mosquito-repellent finished fabric will help to protect people from mosquitoes for long hours without using any liquid bases on their skin, which suits the hectic routine and long working shifts of military personnel and athletes. Because of their low toxicity and high customer acceptability, natural commodities like natural mosquito-repellent oils are becoming increasingly popular in the development of long-lasting mosquito-repellent textiles.

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Conflicts of Interest: The authors declare no conflict of interest.

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