

PHYSICOCHEMICAL AND MORPHOLOGICAL CHARACTERISATION STUDIES OF FABRICS TREATED WITH LAVENDER ESSENTIAL OIL-BASED NANOEMULSION

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Abstract. Lavender essential oil is a concentrated plant extract obtained through steam distillation. It is well known for its properties as a good anxiety and stress reliever, for wound healing, for insect repellency and as an insomnia fighter. However, there are many volatile chemical compounds in the essential oil. Hence, formulating a lavender essential oil nanoemulsion is a potential solution to prolong those exclusive applications of the essential oil. Lavender essential oil-based nanoemulsion (LEONE) was prepared with tragacanth gum and Triton X-100 by a combined method of ultrasonic and high-speed homogenisation. This study focused on the physicochemical and morphological characterisation effects of fabrics treated with LEONE. Morphological and physicochemical evaluation of nanoemulsions were conducted by optical microscopy (OM), high resolution transmission electron microscopy (HR-TEM) and Fourier transform infrared (FTIR) spectroscopy. Ultraviolet-visible (UV-Vis) spectroscopy was utilised to evaluate the encapsulation efficiency of the nanoemulsion. Surface morphologies of LEONE on cotton, polyester and tetoron were observed using field emission scanning electron microscopy (FESEM). OPM images demonstrated fine and reasonably monodispersed distributions of nanoemulsion. In TEM images, LEONE exhibited spherical shapes and in the size range of 55-90 nm. The successful blending of the materials was identified via FTIR spectroscopic functional group analyses. The successful encapsulation of essential oil into the nanoemulsion was proven by the high encapsulation efficiency of close to 90 %. Deposition of LEONE affected the surface morphologies and total porosity of fabrics slightly. The results obtained are useful for applications in household and medical fields, especially in relation to sleep deprivation.

Keywords: Cotton, polyester, tetoron, lavender essential oil, nanoemulsion.

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1. INTRODUCTION

The word “textile” originates from the Latin words, “*textilis*” and “*texere*”, which means woven and to weave. A textile is a flexible material which is made of a network of natural or artificial fibres through weaving, knitting and felting [1]. The use of clothing is an exclusive privilege of human society; and therefore clothing and textiles are informative in relation to be the materials and technologies available for different civilisations. Furthermore, the variety and distribution of textiles is revealing in terms of historical social customs and cultures; nevertheless, clothing plays important role in protecting human beings from heat and cold regardless of the type of textile [2]. For instance, farmers wear protective clothing when spraying their crops with insecticides. Fruits and trees are provided with protection from weather and insects with polyester sheets that have the properties of high strength and durability. However, it is important to pay attention to the nature and the properties of the fabric which is used to make the clothing in terms of the context of its use; for example, polyester based clothing retains sweat and body odour inside. Hence, a suitable textile must be chosen to meet the requirements of users under every situation.

Essential oils are oils that are extracted from plants and comprise volatile compounds that possess various biological effects, such as antibacterial, antifungal, anti-inflammation and mood boosting properties. With all these advantages, various essential oils have been introduced as additives in cosmetic and food or as aromatherapy agents. The direct application of essential oils has been limited because they are highly volatile and unstable. Essential oils are complicated mixtures that contain lots of differently structured compounds and phytochemicals and most of them have a significant vapour component at room temperature because they have high vapour pressure under conditions of atmospheric pressure and room temperature [3]. Additionally, a very large amount of plant material is required to produce essential oils. For example, 250 pounds of lavender flower is needed to produce 1 pound of lavender essential oil. Hence, it is of interest to further enhance the utilisation of essential oil could employing carrier agents.

In this study, lavender essential oil is the active ingredient to be sprayed on textile. Among lavender species used to produce essential oils, *Lavandula angustifolia* and *Lavandula hybrida* are the most popular species. *Lavandula angustifolia* is also known as ‘true lavender’ and produces the most recognised lavender scent. In Malaysia, *Lavandula dentata* and *Lavandula galanga* species are prevalent. Optimum growing conditions for lavender are in full sun and in cool temperate, dry, well-drained soils, with a sandy or gravelly consistency [4]. Lavender essential oil is obtained through steam distillation of flowers which involves boiling water. The distillation part of the production process determines the oil’s value, and the pressure or temperature applied to the flowers during the distillation process can potentially alter the structure of the fragrance molecules and thus change the oil’s chemical constituents. It is believed that the first distillation produces an oil that is clear and of the highest quality. The key constituents of lavender essential oil are linalool, linalyl acetate, camphor, 1,8-cineole, lavandulyl acetate, terpinene-4-ol, and β -caryophyllene. The quality of lavender oil is determined by its camphor content and the lower the camphor content, the sweeter smell of lavender essential oil.

Fragrance finishing is a recently emerged field in the textile industry which involves the incorporation of various aromas into fabrics. By adding the fragrance, the value of the textile can be increased. Hence, numerous functional agents are tested on fabrics to maintain the durability and functionality after incorporation of the aroma bearing agents. In addition, extending the durability of scents on textiles is an interest to in the field. Hence, tragacanth

gum (TG) has been proposed to be utilised as a natural binder for textiles with the protective agent of lavender essential oil. TG is a natural gum obtained from the dried sap of *Astragalus* species and it is mainly applied as a food thickener in jellies [5]. Spraying, printing, padding, grafting, surface coating, impregnation are common methods of imparting active ingredients into textiles. Up to date, synthetic polymeric binders are used in most of the studies. For example, Chavan et al. [6] used Pidicryl Binder SUN (an acrylate binder) to impart chrysanthemum oil emulsion on cotton through pad-dry-cure method. In this study, TG is not only a natural binder on the fabrics, but it is also able to form double-layer micelles to prolong release of active ingredients. Overall, the objectives of this study are: (1) to introduce lavender essential oil-based nanoemulsion onto three types of fabric finishes, namely cotton, polyester and tetoron, and (2) to compare the durability between non-treated and treated fabrics.

2. MATERIALS AND METHODS

2.1 Materials

Food grade TG and lavender essential oil were purchased from a local market in Tanjung Malim, Perak. Triton X-100 and sodium hydroxide (NaOH) were supplied by Acros Organic (Belgium). Plain cotton, polyester and tetoron fabrics were purchased from a textile store in Kuala Lumpur. Phosphotungstic acid hydrate was obtained from Sigma-Aldrich (St. Louis, MI, USA).

2.2 Preparation of LEONE

Lavender essential oil (1.0 mL) was added into a 1.0 mL of Triton X-100 solution and then mixed on a stirring hotplate (Favorit HS070V2, Italy) for two hours. TG solution (1.0% w/v) was prepared with deionized water. An exact amount of 15.0 mL of TG solution was added dropwise into the mixture and left to stir for another hour. The emulsion was then homogenized by a high-speed homogenizer for 15 minutes followed by an ultrasonic homogenizer (Biosafe, China) for 5 minutes.

2.3 Preparation of LEONE-treated Fabrics

The fabrics chosen in this study were cotton, polyester and tetoron. The fabrics were scoured with 2% (w/v) NaOH solution and air-dried prior to the impartation of LEONE. Scouring is a textile-cleaning process before dyeing and mordanting. Briefly, scouring procedures were as follows: NaOH pellets were weighed and then dissolved in hot water. The fabrics were boiled for 30 minutes and then hung to be air-dried at room temperature. The fabrics were then immersed in LEONE for a minute. The fabrics were left to dry at room temperature.

2.4 Characterisation Studies of LEONE

The physicochemical properties of LEONE were characterised by optical microscopy (OM), high resolution transmission electron microscopy (HR-TEM) and Fourier transform infrared (FTIR) spectroscopy.

a) Optical Microscopy (OM)

OM is the simplest method to examine the microstructure of nanoemulsion. Briefly, a glass slide was cleaned with alcohol prior to investigation of LEONE. A drop of LEONE was absorbed and covered on the glass slide. The nanoemulsion was observed and recorded with the optical microscope (UB203i-LCD-2, China) at 40× magnification.

b) High Resolution Transmission Electron Microscopy (HR-TEM)

HR-TEM (Jeol JEM-2100F, Tokyo, Japan) was used to observe LEONE. The LEONE was vortexed for 5 minutes. Two drops of sample solution were dropped on the 300-mesh-carbon coated copper grid. The sample was negatively stained using 1% (w/v) phosphotungstic acid. After that, the stained piece was allowed to dry on a piece of filter paper at room temperature (26 ± 2 °C) and was kept for storage in a dry cabinet (AIPO Digital Series, AP-88EX; 35% RH) until further analysis. In the analysis, a tungsten filament was used to produce electrons using an acceleration voltage of 200 kV.

c) Fourier Transform Infrared (FTIR) Spectroscopy

The transmittance spectra of the precursors and nanoemulsion were obtained with a Perkin Elmer (Waltham, MA) ATR-FTIR Spectrum 3 instrument employing Perkin Elmer Spectrum software (version 10.4.3.279). The spectral range measured was 4000-400 cm^{-1} with a resolution of 4 cm^{-1} and 64 scans. The spectrum of air was employed for background subtraction.

2.5 Characterisation Studies of LEONE-treated Fabrics

There were three types of fabrics tested in this study: cotton, polyester and tetoron. The efficacy of the treated fabrics was studied by washing all the finished samples with 5 washing cycles in launder-o-meter using a standard method AATCC 43-1995 recommended by American Association of Textile Chemists and Colorists. The physicochemical properties of untreated fabrics, LEONE-treated fabrics before and after 5 washing cycles were analysed by field emission electron scanning microscopy (FESEM) and the tensile test outlined below. Additionally, encapsulation efficiency (EE) of LEONE-treated fabrics was evaluated by using a UV-Vis spectroscopic method.

a) Field Emission Scanning Electron Microscopy (FESEM)

The morphologies of untreated fabrics, LEONE-treated fabrics before and after 5 washing cycles were examined using field emission scanning electron microscope (FESEM; Hitachi SU 8020 UHR, Japan). The samples were placed and dispersed on a circular platform and coated with platinum. All samples were observed with an accelerating voltage ranging between 0.5 kV and 1 kV at 1,000× magnification.

b) Tensile Test

The tensile tests of untreated fabrics and LEONE-treated fabrics before and after 5 washing cycles were conducted based on ISO 13934-1 with some modifications. Three samples of each fabric were cut with dimensions of 4 cm × 15 cm. Each sample was tested using a Universal Testing Machine (Instron 5967, Norwood, Massachusetts). The test was

carried out using a 20 N load cell with an extension rate of 150 mm/min. The results were expressed as mean $\pm$ SD, $n=3$.

c) Encapsulation Efficiency (EE) of LEONE-treated Fabrics

An ultraviolet-visible (UV-Vis) spectrophotometer was used to determine the encapsulation efficiency of LEONE-treated fabrics. The treated fabrics were centrifuged and the supernatant was filtered and analysed in a UV-Vis spectrophotometer (Agilent Cary 60, Santa Clara, California). The EE is defined as the ratio and calculated using Equation 1:

$$\text{Encapsulation efficiency (\%)} = \frac{\text{Essential oil released from the fabric}}{\text{Total amount of essential oil used for nanoemulsion preparation}} \quad (1)$$

3. RESULTS AND DISCUSSION

3.1 Characterisation Studies of LEONE Nanoemulsion

a) Optical Microscopy Analysis

OM is a simple method to study the microstructure of nanoemulsions. An optical microscope can be used to magnify small features in the samples after the interaction with visible light such as absorption, reflection and scattering. It detects any large aggregates, droplets or undissolved crystals in the formulation other than observing the background movement which is Brownian motion displayed by those droplets exist in the nanoemulsion [7]. Figure 1 shows optical microscopy image of LEONE.

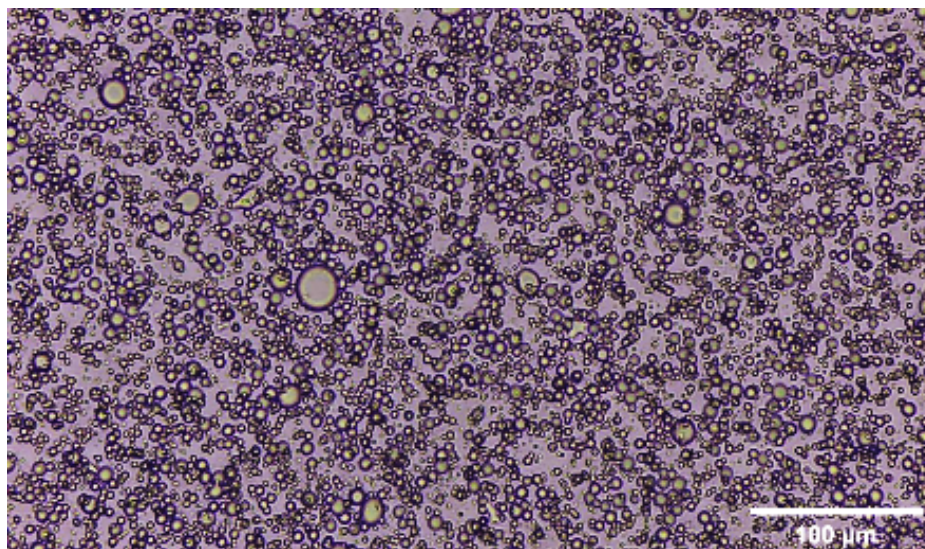


Figure 1: Optical microscopy image of LEONE

As shown in Figure 1, the LEONE presented spherical morphology and “core-shell” structures. Tragacanth gum was absorbed on the interfaces of nanoemulsions and the successful encapsulation of lavender essential oil into the nanoemulsion was represented by the green yellowish colour in the micrograph. The particles of LEONE are not uniform and

large particles are apparent in the image although the spread of dispersion seems relatively small. Nevertheless, OM is not suitable for nanoemulsions below 500 nm because of limitation relating to the observation on destabilisation phenomena such as coalescence or Ostwald ripening [8].

b) HR-TEM Analysis

Transmission electron microscopy (TEM) can be employed to monitor changes in the droplet size and stability of a formulation. Coexisting structure and microstructural transitions can be examined under a TEM. A TEM image of the produced LEONE is shown in Figure 2. The structure of colloidal particles can be seen to be spherical with some irregularities which may be due to aggregation phenomena. The similar shapes of nanoemulsions were obtained by Gupta et al. [9] in their thyme oil nanoemulsion that encapsulated by chitosan. It is interesting to note that their droplets presented uniform clumps and aligned in tandem.

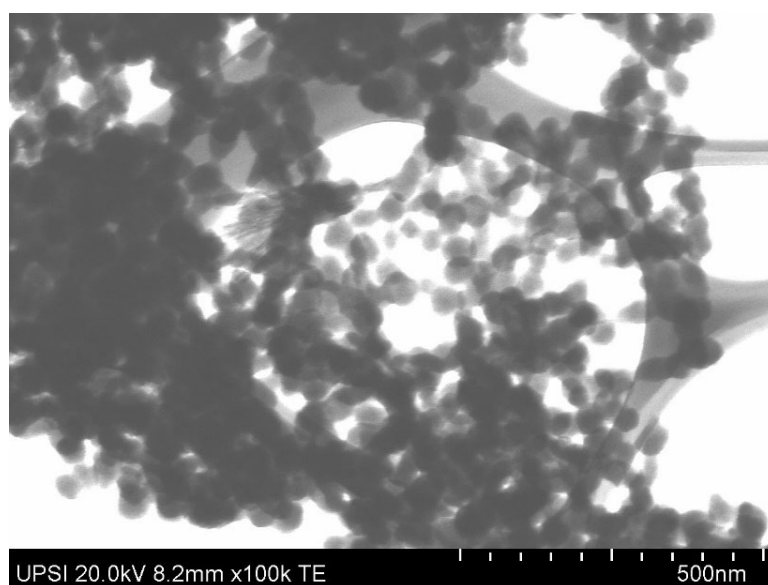


Figure 2: TEM image of LEONE

Cryo preparation is one of the most suitable preparation methods to study nanoemulsions. However, negative staining analysis with or without dilution can be used as an alternative when cryo TEM is not available [10]. Other than TEM, the mean particle size and the particle size distribution are the common techniques that have been employed to characterise the stability of a colloidal system. These parameters can be determined through optical light scattering techniques. Dynamic light scattering (DLS) is also known as photon correlation spectroscopy and the sizes of oil droplets within the nanoemulsion are presented in the form of intensity-weighted mean of the hydrodynamic diameter and the polydispersity index (PDI). PDI is a measurement of particle size distribution and the homogeneity of nanoemulsion. The droplet size and PDI are the factors that contribute to the stability and physical properties of nanoemulsion [11].

c) FTIR Analysis

FTIR was utilised to characterise the potential interactions in the formulation. FTIR spectra of TG, lavender essential oil and LEONE were analysed and are presented in Figure 3.

In Figure 3(a), the O-H stretch (at 3272 and 2888 cm^{-1}) is representative of the presence of carboxylic acids (tragacanthic acid or bassorin) and intermolecular O-H bonding; the C=C stretches at 1602 cm^{-1} represented conjugated alkenes; and the bands around 1020 cm^{-1} are attributed to its CO-O-CO stretching vibration. The spectrum of lavender essential oil is shown in Figure 3(b). The broad band at 3445 cm^{-1} corresponded to alcohols; and the peaks at the region between 2969 and 2926 cm^{-1} belonged to C-H of aldehyde or alkane of the compounds in the essential oil. The peaks at 1738 cm^{-1} relate to stretching vibrations of C=O from those compounds containing ester functional groups. The peaks observed at 1238 cm^{-1} were ascribed to C-O stretching from those aromatic esters.

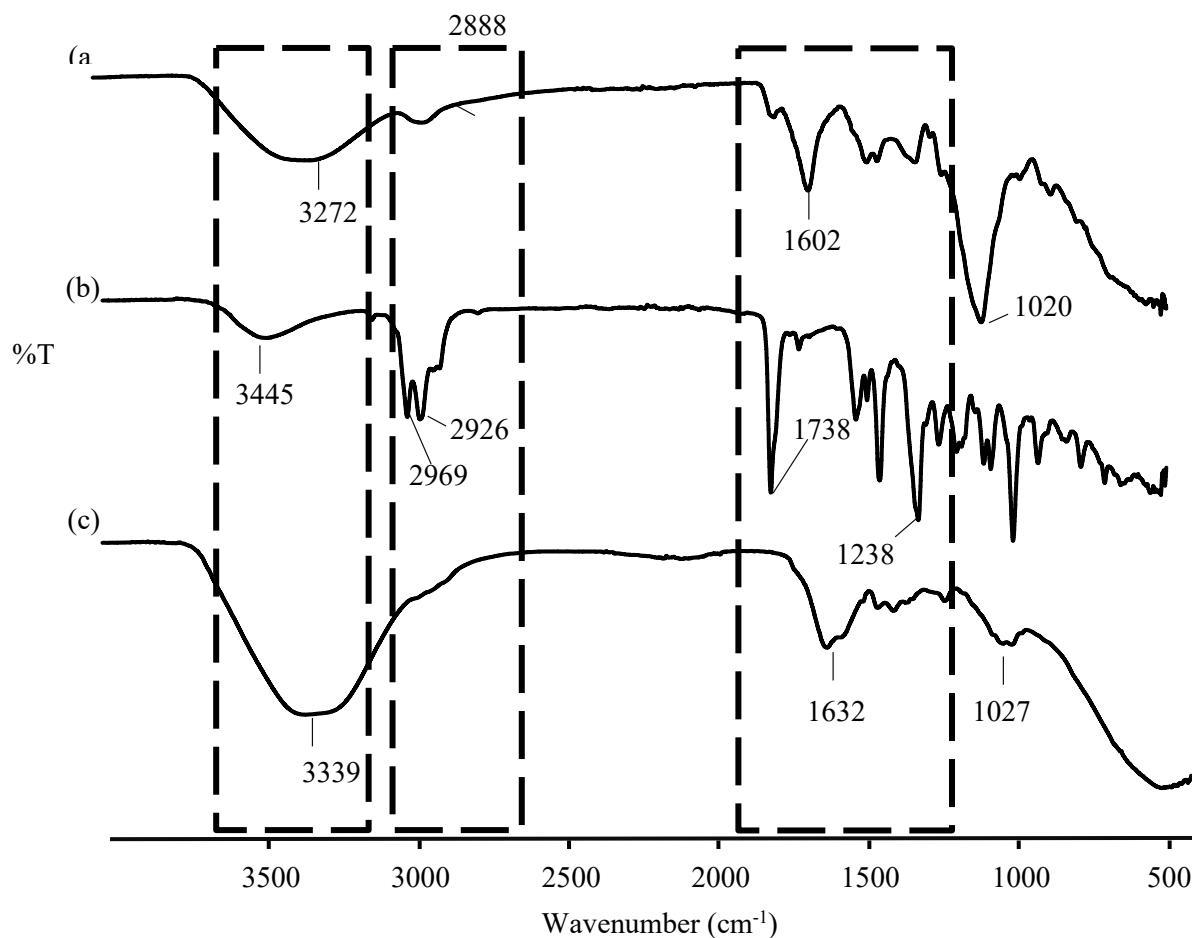


Figure 3: FTIR spectra of (a) TG, (b) lavender essential oil and (c) LEONE

In the FTIR spectrum of LEONE (Figure 3(c)), it is interesting to note that the peaks of lavender essential oil that appeared in region of 2900 cm^{-1} disappeared after the preparation of LEONE. This indicates the interaction between lavender essential oil and TG during the preparation process. The disappearance of peaks ranged between 2900 and 3000 cm^{-1} in Figure 3(c) can be attributed to the broader peak of OH in LEONE. This phenomenon can be explained by formation of more hydrogen bonding between TG and lavender essential oil [12]. The broad band of 3339 cm^{-1} became sharper and was ascribed to alcohols and the shift of the OH stretching peak to a lower wavenumber direction is ascribed to the formation of the hydrogen bonding interaction, which gradually changes the vibrational frequency of

this band. The peaks at 1632 cm^{-1} were caused by C=C stretches which showed the presence of alkenes. The bands around 1027 cm^{-1} were attributed to its CO-O-CO and C-O vibrations [12]. These results signalled an electrostatic interaction formed between the carboxylic groups of TG and lavender essential oil to form the polyelectrolyte complex.

3.2 Characterisation Studies of LEONE-treated Fabrics

a) FESEM Analysis

The morphological structures of untreated cotton, polyester and tetoron are presented in Figures 4(a) to (c). Figure 4(a) illustrated the presence of twisted ribbon-like fibres and this indicated the characteristic of untreated cotton fibres. After being treated with LEONE, the surface became highly rugged and there were small spheres adhered on the fibres, as shown in Figure 4(b). After the treated cotton had undergone 5 washing cycles, there was a dislocation in the number of microcapsules and the effect of leaching out can be noticed in Figure 4(c). The surface of the polyester fibres is very smooth, homogeneous and clear, as shown in Figure 4(d). Through observation on Figure 4(e), There were some aggregations of nanoemulsion deposited between the polyester fibres. This demonstrated the relevance of biopolymers in achieving uniform microcapsule dispersion and penetration on treated fabric surfaces. After five washes, it was found that the number of microcapsules per unit area did not decrease (Figure 4(f)). This indicates TG promotes good adhesion onto any fabric, either hydrophilic or hydrophilic. Figure 4(g), the surface of tetoron fibres was slightly rough and there was deposition observed on the fabric. The surface of the fibres became rougher and there were aggregations seen on the fibres. However, the aggregations had been washed away along the cycles, but the rough surface of the fibres was still can be spotted.

Overall, the FESEM analysis suggested that there was no fibre surface damage after the fibres were introduced with LEONE. The findings are in accordance with the findings from Chavan et al. [6]. They imparted chrysanthemum oil emulsion onto cotton by using Pidicryl Binder SUN and the deposition of emulsion was successfully observed. Furthermore, chitosan-gelatin microcapsules containing rosemary essential oil that prepared by Singh and Sheikh [13] have been displayed shapes of spherical, oval and irregular on the linen fibres and this is a common scenario for spray-dried microcapsules.

The presence of TG promoted adhesion between the fibres, causing the fibres to adhere along their length [14]. During preparation of LEONE, TG has been cationised by lavender essential oil. Hence, the ionic linkage occurred between cationised TG and negatively charged fibres, and this provides better washing durability for the treated fabrics [13]. Moreover, the deposition of the fine microcapsule was clearly observed on the fabrics. The deposition was found to affect the porosity of the fabrics due to the diffusion of LEONE in the fabrics. The porosity structure was found to be different from each other. It is because the fitness and nature of the threads of those three fabrics is different. Furthermore, the thread counts of the fabrics were different. Apparently, cotton has the lowest warp and weft yarn than polyester and tetoron. Thickness of the fabric is also a factor that contributes to the difference of porosity.

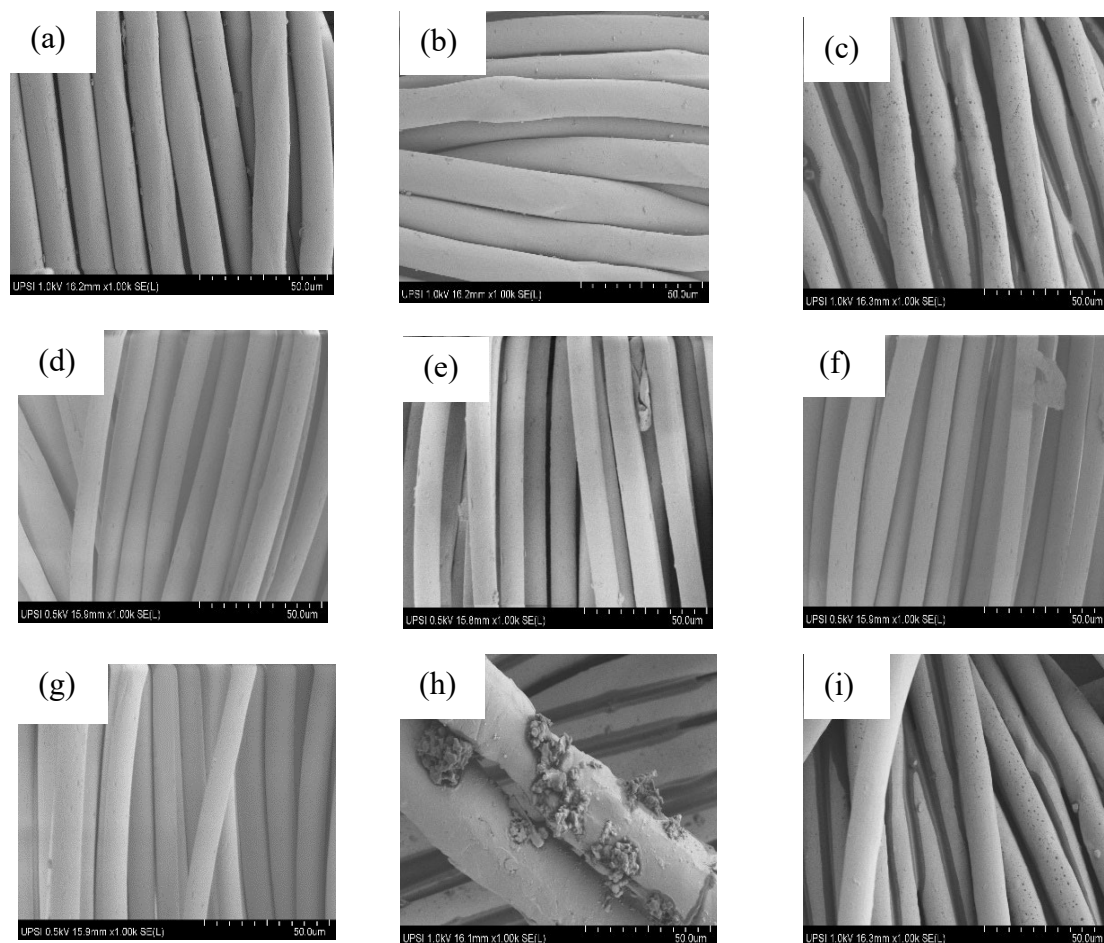


Figure 4: FESEM images of (a) untreated cotton, (b) LEONE-treated cotton, (c) LEONE-treated cotton after 5 cycles of wash, (d) untreated polyester, (e) LEONE-treated polyester, (f) LEONE-treated polyester after 5 cycles of wash, (g) untreated tetoron, (h) LEONE-treated tetoron and (i) LEONE-treated tetoron after 5 cycles of wash

b) Tensile strength Properties

Tensile testing measures the necessary force to elongate and break a sample. The tensile strength of a fabric is highly dependent on the polymer chain arrangement and the macrostructure [15]. In this study, the tensile strength of the fabrics was measured in the warp direction only because the tensile strength required during weaving is higher for warp yarn than weft yarn. During the weaving of the fabric, the warps are holding under high tension. Additionally, if the warp yarn does not meet the minimum requirement of tensile strength, the weft yarn has higher tendency to be broke faster because the tensile strength of weft yarn is smaller [16].

The tensile strengths of the untreated fabrics, the LEONE-treated fabrics and the LEONE-treated fabrics after 5 washing cycles are presented in Table 1. The LEONE increased the tensile strength of cotton, polyester and tetoron by about 24%, 30% and 79%, respectively. The LEONE formulation strengthens the fibre strength, causing the fabric to be able to elongate longer as the load increases. This suggests that LEONE binding to the fibres

resulting in their transformation. After 5 washing cycles, the treated fabrics became more fragile which might be caused by a reduction of number of nanoemulsion particles on the fibres of the textile. The decrease in the strength also can be attributed to poor wettability and interfacial bonding. The presence of hydroxide and polar groups contribute to moisture absorption in the fabrics causing weak interactions between the LEONE and fabrics. Although there is no cross-linking agent involved during impartation, Cross-linking does occur between the fabrics and LEONE. Every fabric has different crystallinity index. For instance, cotton has 65% crystalline and 35% amorphous. In this context, LEONE cross-links with free O-H group in the amorphous region, however it makes stiff of the fabrics, and this increases the tensile strength. Venkatraman et al. [17] treated cotton with 1-Karanja and 2-Gokhru nanoemulsions without using any cross-linking agent. However, the treated cotton exhibited reduced tensile strength, but the tensile strength increased after 10 washes. They deducted that the reduction was due to the simple mechanical adsorption of emulsion on the fabric structure, whereby the nano-emulsions remain between the voids of the fibres in the fabric.

Table 1: Tensile strength of untreated fabrics, LEONE-treated fabrics and LEONE-treated fabrics after 5 washes

Type of fabric	Force (N)		
	Untreated	LEONE-treated	LEONE-treated after 5 washes
Cotton	34.25 ± 3.12	42.55 ± 6.43	35.97 ± 6.45
Polyester	69.44 ± 1.34	90.26 ± 5.64	54.94 ± 2.36
Tetoron	36.67 ± 4.75	65.55 ± 1.31	43.47 ± 5.11

c) Encapsulation Efficiency (EE) of LEONE-treated Fabrics

Table 2 presents EE of LEONE-treated fabrics and the data showed good encapsulation abilities of those three fabrics. Among these three fabrics, cotton showed the best EE results, which was 71.03% and followed by tetoron (68.74%) and polyester (53.52%). These results suggested that tragacanth gum is a good material to encapsulate lavender essential oil to be nanoemulsions. There are many factors that can affect the EE such as the nature of the oil, biopolymer concentrations, oil polymer ratio, preparation method and so forth [18]. The encapsulation of oil in the nanoemulsion could prolong the efficacy of the essential oil through reduction of volatility and degradation rate.

Table 2: Encapsulation efficiency of LEONE-treated fabrics

Fabric	Encapsulation efficiency (%)
Cotton	71.03 ± 0.78
Polyester	53.52 ± 0.11
Tetoron	68.74 ± 0.35

4. CONCLUSIONS

A series of studies have been conducted related to the physicochemical properties of nanoemulsion and application of nanoemulsion as a formulation on aromatherapy textiles. This study has focused on physicochemical and morphological characterisation studies of

lavender essential oil-based nanoemulsion and three types of fabric (cotton, polyester and tetoron) treated with LEONE. A series of physicochemical and morphological evaluation of nanoemulsion has been conducted through OM, TEM and FTIR. OM and TEM images illustrated the spherical structures of LEONE and the successful encapsulation of LEONE. Furthermore, FTIR spectra showed the formation of electrostatic interaction between the carboxylic groups of TG and lavender essential oil.

The tensile strength of untreated, LEONE-treated fabrics before and after 5 washing cycles had been evaluated. According to the analyses, LEONE improved tensile strength of the fabric by 20%. However, the tensile strength of the fabrics decreased slightly after 5 cycles of washing. Therefore, the LEONE does not degrade the fabric. After the fabrics were treated with LEONE, the surface morphologies were observed under FESEM. It can be deduced that the morphological evaluation of the tested textile could be successfully performed using FESEM. The nanoemulsion was observed to be deposited on the fibres of those fabrics without damaging the properties of the textiles. In conclusion, LEONE-treated fabrics was effective and there is no significant problem on the properties of the textiles. The study can be further with more fabric types or other types of formulation.

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Author Contributions

Azlan Kamari conceived the study and received funding for this project. Susana Wong Siew Tin and Nurul Nabila Aini Abdullah designed methodology and implemented the project. Norjan Yusof, Hidayatulfathi Othman, Mohd Zobir Hussein and Justin S.J. Hargreaves contributed to data curation and writing reviews. All authors contributed toward data analysis, drafting and critically revising the paper and agree to be accountable for all aspects of the work.

Disclosure of Conflict of Interest

The authors have no disclosures to declare.

Compliance with Ethical Standards

The work is compliant with ethical standards.

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