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RESEARCH ARTICLE
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Solvent-Free Microwave Extraction and Microencapsulation of Kaffir Lime Essential Oil Using Gelatin and Gum Arabic as Fragrances in Textiles

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ABSTRACT

In this study, the hydrodistillation (HD) method and solvent-free microwave extraction (SFME) have been used for extraction of essential oil from kaffir lime peel. The HD method was able to extract 0.650±0.020% of kaffir lime essential oil in 3h. While SFME provided an extraction yield of 0.615±0.021% under a microwave irradiation power of 800 W for shorter time (10min). The chemical components were investigated by gas chromatography-mass spectrometry (GC-MS). The peak area revealed that the main components were β-phellandrene (24.38%), D-limonene (17.03%), β-pinene (15.69%), citronellal (12.5%), and citronellol (4.16%). The microcapsules were prepared by complex coacervation method using Arabic gum and gelatin as the core material. The encapsulation yield, loading capacity, and encapsulation efficiency of the microcapsules were 76.02, 28.4, and 62.07%, respectively. The scanning electron microscopy (SEM) and optical microscopy showed spherical-shaped microcapsules with a particle size of 4-7µm. Fourier-transform infrared spectroscopy supported gum Arabic, gelatin, and kaffir lime oil components into the microcapsules. Last of all, the scented microcapsules were impregnated into textile materials. Further SEM analysis confirmed the finding to reveal that microcapsules anchored themselves onto fibrous textile surface. Thus, this study has provided the revelation that kaffir lime essential oil can be efficiently extracted by both HD and SFME. High-efficiency microcapsules in the form of spherical microcapsules composed of gum Arabic and gelatin with reasonable efficiency of the essential oil were used. The fabrication of the essential oil microcapsules was accomplished, and the microcapsules were immobilized onto textiles to be used as fragrance delivery systems.

Keywords: Solvent-free Microwave Extraction, Microcapsule, Encapsulation, Kaffir Lime Oil, *Citrus hystrix* DC.

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INTRODUCTION

Essential oils are extracted from various plants and they are in liquid form, more specifically, these contain oils that do not dissolve in water due to the volatile contents derived from the odoriferous part of the plant (He et al., 2025). They have been used for centuries in various cultures around the world for their aromatic, perfumery, cosmetic, and traditional medicinal and therapeutic properties (Zhao et al., 2020). The genus *Citrus* belongs to the family Rutaceae and includes a diverse group of flowering plants known for their edible fruits, including oranges, lemons, limes, grapefruits, and many others (Srifuengfung et al., 2020). These plants are widely distributed throughout the world and are cultivated in many regions for their fruits, which are valued for their flavour, aroma, and nutritional content (Hien et al., 2020). Kaffir lime (*Citrus hystrix* DC), a member of the Citrus family, is a tropical plant that is widespread in many countries, especially Southeast Asia. Kaffir lime essential oil is a versatile aromatic additive for aromatherapy, skincare, and culinary applications, providing sensory enjoyment and potential therapeutic benefits (An et al., 2021; Husni et al., 2021). Essential oils are extracted from plant materials using various methods, which are suitable for different plant types and extraction goals. Essential oil extraction techniques include steam distillation, hydrodistillation (HD), solvent extraction, enfleurage, microwave-assisted extraction (MAE) and solvent-free microwave extraction (SFME) (Wei et al., 2023; Li et al., 2023). SFME uses

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microwave energy to extract compounds from plant materials without using solvents (Wei et al., 2022; Wang et al., 2023). This method has attracted attention in the field of natural product extraction because of its potential advantages, including shorter extraction time, lower environmental impact, and potentially higher extraction yields (Peng et al., 2021; Yingngam et al., 2021).

Essential oil encapsulation involves volatile and sensitive compounds. The encapsulation of essential oils offers several advantages, including protection of volatile compounds from degradation, controlled release of fragrance or active ingredients, and an increase in stability during storage and handling (Carpentier et al., 2022; Raj & Dash, 2022). This technology has several applications beyond textiles, including cosmetics, personal care products, pharmaceuticals, and food products (de Araújo et al., 2020). Various encapsulation methods can be used to encapsulate essential oils, such as solvent evaporation, coacervation, spray drying, extrusion, and complex coacervation (Ikutegbe et al., 2022; Wongchompoo & Buntem, 2022). Impregnating essential oil microcapsules onto textiles can offer various benefits, especially in terms of fragrance, antimicrobial properties, and even therapeutic effects (Xiao et al., 2022; Verma et al., 2024). Natural kaffir lime essential oil can be used as a fragrance for application in textiles.

In this study, we extracted essential oils from kaffir lime fruit peels using a green technique by SFME. The chemical components in the essential oil from SFME were identified by gas chromatography-mass spectrometry (GC-MS). Additionally, the kaffir lime essential oil was encapsulated by a complex coacervation process to prevent volatilization and degradation before being used to impregnate textiles. The characteristics of the microcapsules were investigated by scanning electron microscopy (SEM) and Fourier-transform infrared spectroscopy (FTIR).

MATERIALS & METHODS

Materials and Chemicals

Mature kaffir lime fruits were collected from Buriram Province, Thailand. The kaffir lime peel was prepared by rough cutting (1×1cm), including white and green peels. Sodium sulfate anhydrous, glutaraldehyde, glacial acetic acid, and sodium hydroxide were purchased from RCI Labscan. Gelatin and gum Arabic were purchased from KemAus. Commercial acrylic binder was purchased from Ratchada Chemicals, Thailand. All chemicals were used without further purification.

HD and SFME Methods

For kaffir lime oil extraction, the method was adjusted from our previous report (Tangtubtim et al., 2024). Briefly, the HD method was conducted in a simple distillation apparatus for 3h using 200g of kaffir lime peel in 200mL of distilled water with a hot plate as a heating source. The distillate was separated and dried over anhydrous sodium sulfate. The essential oil was stored at 4°C in a brown glass vial.

The SFME technique was similar to HD except for the use of a heating source without solvent for 10min under a microwave radiation power of 800W and stirring with an overhead stirrer (Fig. 1). The essential oil (EO) yield was calculated as follows (Zhang et al., 2024):

$$EO\,(\%) = \frac{\text{amount of essential oil (g)}}{\text{amount of fresh matter (g)}} \times 100$$

Fig. 1: Solvent-free microwave extraction (SFME) apparatus with an overhead stirrer.

Preparation of Kaffir Lime Oil Microcapsules

The complex coacervation technique was used to prepare a microcapsule of kaffir lime oil (Tariq et al., 2022). In this method, 30mL of 12.5% gum Arabic and 4 g of kaffir lime oil were mixed in 5mL of distilled water under constant stirring (500rpm) at 50°C for 20min. Then, 30mL of 12.5% gelatin was added and stirred (500rpm) at room temperature for 20min, followed by the addition of 195mL of distilled water. The mixture was warmed to 50°C, and stirring was continued at 500rpm for 30min. The temperature of the mixture was decreased to room temperature, and the pH of the mixture was adjusted to 4.5 using 10% acetic acid and 10% NaOH. For crosslinking, the mixture was kept at a temperature below 10°C and stirred at 250rpm for 60min. Next, 20mL of 25% glutaraldehyde was added, and the mixture was stirred for 30min. The mixture was kept at room temperature overnight to separate the microcapsule layer and water. The suspension of microcapsule (SM) was filtered and dried using a freeze dryer (Flexi-Dry MP Freeze Dryer, FTS Systems FD-3-85A-MP) to obtain kaffir lime oil microcapsules (KLOMs). The encapsulation yield (EY) was calculated as follows (Aldana-Heredia et al., 2024):

$$EY\,(\%) = \frac{\text{total amount of microcapsule (g)}}{\text{total amount of initial solid and oil (g)}} \times 100$$

The loading capacity (LC) and encapsulation efficiency (EE) were studied. KLOMs (1g) were extracted with 50mL of n-hexane using an ultrasonic bath for 30min. The kaffir lime oil content of the extract was determined by GC (PerkinElmer Clarus 690, Elite 5MS column, Temp. The temperature ranged from 80–150°C, and the temperature was increased at a rate of 7°C/min. The injection volume was 1.0µL, and the FID detector was used. The LC and EE of the microcapsules were calculated as follows (Aldana-Heredia et al., 2024):

$$LC\ (\%) = \frac{\text{amount of essential oils in microcapsule (g)}}{\text{total amount of microcapsule (g)}} \times 100$$

$$EE\ (\%) = \frac{\text{amount of essential oils in microcapsule (g)}}{\text{initial amount of essential oils (g)}} \times 100$$

Impregnation of Microcapsules onto Textiles

The microcapsules were finished onto polyester-cotton textiles using a modified process described in previous studies (Lim & Setthayanond, 2019; Singh & Sheikh, 2022). The polyester-cotton textiles were immersed in a solution containing microcapsules, acrylic binder, and distilled water (2g: 1g: 50mL) for 30min. Next, the textiles were padded such that about 75% of the liquid was wet-picked and dried at 110°C for 10min.

GC-MS Analysis

Gas chromatography-mass spectrometry (GC-MS) was conducted to identify the chemical composition and quality of the kaffir lime oil. The analysis was performed using GC-MS (GC: Agilent 7890A, MS: Agilent 7000B). An HP-5 column was used; length: 20m, internal diameter: 0.015mm, and film thickness: 0.18µm. The initial temperature was maintained at 40°C for 5min, followed by an increase from 40 to 80°C at a heating rate of 3°C/min and from 80 to 150°C at a heating rate of 10°C/min. The injection volume was 2.0µL, the injection temperature was 250°C, and the detector temperature was 250°C (Talebi et al., 2024).

FT-IR Analysis

The chemical structures of the essential oil, gelatin, gum Arabic and essential oil microcapsules were analyzed by an FT-IR spectrometer (PerkinElmer, spectrum GX-1) with an attenuated total reflectance (ATR) accessory. The FT-IR spectra (resolution of 4cm^{-1}) in transmittance mode were recorded from 550 to 4,000cm^{-1}.

Optical Microscope Analysis

The microcapsules were observed under an optical microscope (Nikon, E100). The microcapsule suspension was spread on microscope slides and observed. Images were captured at 400x magnification. The KLOMs were dispersed in ethanol and observed using a similar method. The obtained images were analyzed to estimate the particle size distribution using the ImageJ software.

SEM Analysis

The morphological characteristics of the microcapsules and textiles were observed by SEM (Hitachi, S3400N). The samples were placed on the SEM sample holder and sputter-coated with gold before measurement.

RESULTS & DISCUSSION

Kaffir Lime Oil Extraction

The content of kaffir lime oil (w/w) was 0.61±0.02% for HD at 180min and 0.63±0.03% for SFME after 10min of extraction under 800W microwave power. Both kaffir lime oils were colorless, clear and highly volatile, with no difference in smell. The SFME technique used in other studies required a small amount of sample, low microwave power (<500W) and a long extraction time (>60min) (Hien et al., 2020). In this study, SFME was performed for a shorter time and had a higher extraction yield; additionally, sample preparation for kaffir lime oil extraction was easy.

GC Analysis of Kaffir Lime Oil

The chemical composition of kaffir lime oil was investigated by GC-MS (Fig. 2). The compounds were identified from the GC chromatograms by matching the data with those present in the NIST MS Search 2.0 library. The results revealed that kaffir lime essential oil from the HD method yielded 56 different compounds, including β-

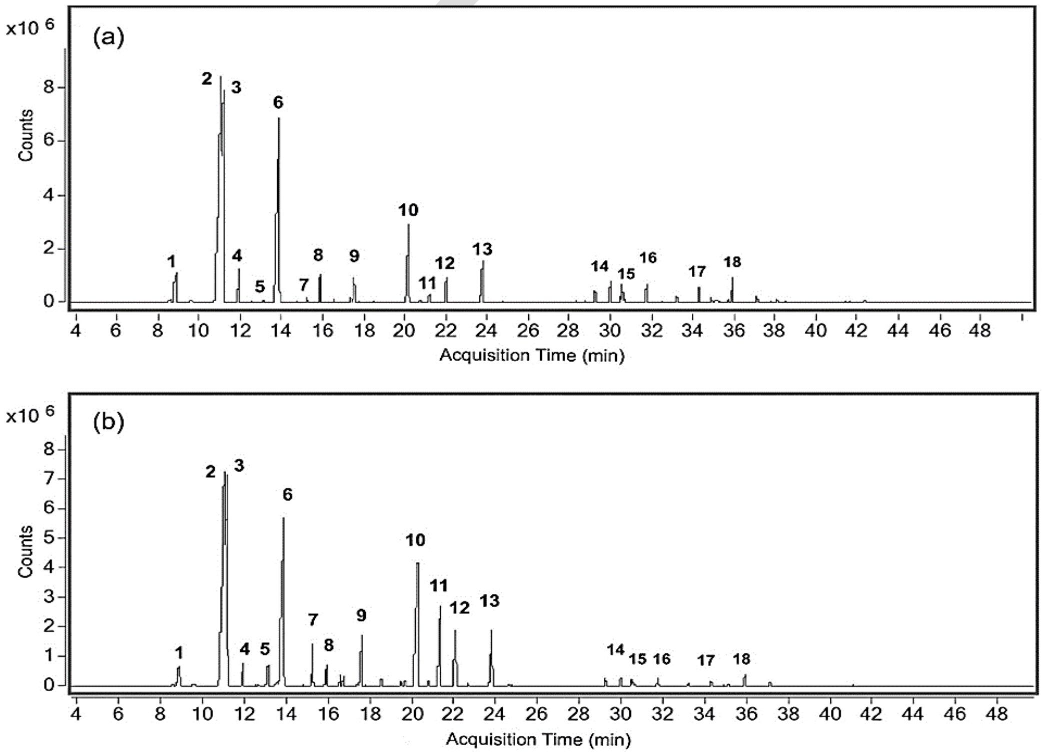

Fig. 2: GC Chromatogram of kaffir lime essential oils (a) SFME (b) HD.

phellandrene (24.38%), D-limonene (17.03%), β-pinene (15.69%), citronellal (12.5%), citronellol (4.16%), and 51 other compounds (Table 1). The SFME method yielded similar main compounds, including β-phellandrene (31.29%), D-limonene (23.63%), β-pinene (16.93), citronellal (6.25%), citronellol (3.03%) and 47 other compounds (Table 1). Thonglem et al. (2023) reported the chemical composition of kaffir lime peel essential oil from Lopburi Province, Thailand, using the HD method. The main compounds identified in their study were phellandrene, D-limonene, β-pinene, and citronellal. The results showed the same major component from the HD and SFME methods, according to this report. Hien et al. (2020) reported the components of kaffir lime oil (Peel) in Vietnam by SFME. They found 27 compounds, including β-pinene, α-pinene, D-limonene, citronellal, terpinen-4-ol, γ-terpinene, α-terpineol, β-citronellol, and otherminor compounds. Additionally, the chemical composition of the essential oil from kaffir lime peel in Vietnam extracted by the HD method revealed similar main compounds, including β-pinene, D-limonene, and citronellal (Le et al., 2020). Husni et al. (2021) reported the chemical composition of kaffir lime peel essential oil in Indonesia extracted by the HD method, the main components of which were D-limonene, 3-carene, γ-terpinene, and neoisoisopulegol. Therefore, the chemical composition of essential oils from kaffir lime peel depends on the extraction method, the source of cultivation, the species and the maturity of the fruit (Suresh et al., 2021).

Table 1: Chemical compositions of kaffir lime oil from SFME and HD

No.	Compounds	SFME		HD	
		Time (min)	Area Sum (%)	Time (min)	Area Sum (%)
1	1R-α-Pinene	8.884	3.45	8.872	2.13
2	β-Phellandrene	11.053	31.29	11.024	24.38
3	β-Pinene	11.184	16.93	11.159	15.69
4	β-Myrcene	11.915	1.67	11.903	1.10
5	2-Carene	13.094	0.13	13.104	1.09
6	D-Limonene	13.890	23.63	13.855	17.03
7	γ-Terpinene	15.211	0.23	15.247	1.93
8	cis-β-Terpineol	15.872	1.46	15.910	1.11
9	β-Linalool	17.532	1.21	17.595	3.16
10	Citronellal	20.181	6.25	20.261	12.50
11	Terpinen-4-ol	21.194	0.38	21.332	5.52
12	α-Terpineol	21.993	1.27	22.075	3.54
13	Citronellol	23.781	3.03	23.822	4.16
14	Copaene	29.976	1.11	29.965	0.40
15	β-Cubebene	30.548	0.92	30.540	0.26
16	β-Caryophyllene	31.745	0.9	31.737	0.37
17	α-Cubebene	34.304	0.8	34.297	0.25
18	β-Cadinene	35.923	1.22	35.911	0.55
Total hydrocarbon monoterpenes			78.03		64.38
Total oxygenated monoterpenes			14.30		32.85
Total hydrocarbon sesquiterpenes			6.49		2.51
Other compounds			1.18		0.26

A comparison of the chemical compositions of the essential oils extracted by SFME and HD in this study showed that a greater proportion of hydrocarbon monoterpenes (Entries 1–7, Table 1) and hydrocarbon sesquiterpenes (Entries 15–19; Table 1) were present in the essential oils obtained using the SFME method. In the traditional extraction process, energy is transferred to kaffir lime peel by convection, conduction and radiation through the external surface of kaffir lime peel with a thermal gradient. During microwave extraction, microwave energy is directly delivered to kaffir lime peel through molecular interactions with electromagnetic fields due to the conversion of electromagnetic energy into heat energy. Hydrocarbon monoterpenes and hydrocarbon sesquiterpenes are nonpolar substances with a low dipole moment, and they receive less energy from microwave radiation. These reasons can decrease the decomposition process of hydrocarbon monoterpenes and hydrocarbon sesquiterpenes. Typically, oxygenated monoterpenes, which have a high dipole moment, can be interconnected through stronger interactions by microwave radiation. A lower proportion of essential oil from SFME was found in these samples, which occurred probably because the high power caused the decomposition of the compounds (Feng et al., 2024).

Microcapsules and Morphological Analysis

The microcapsules of kaffir lime oil were created by using gum Arabic and gelatin as shell materials, resulting in a yield of 76.02%. The microcapsules were extracted with n-hexane to separate kaffir lime oil from the microcapsules. The extract in hexane was determined by gas chromatography. The LC and EE were 28.2% and 62.07%, respectively. Optical microscopy images of SM and KLOM are shown in Fig. 3(a). The results showed that the microcapsule particles were spherical when dispersed in an aqueous solution. The microcapsules contained the inner core of the kaffir lime peel essential oil and appeared as thin shells around the oil droplets (Praveen et al., 2024). After freeze-drying, the SEM images indicated that the microcapsule particles were spherical and aggregated to form clusters (Fig. 3b and 3c) (Naziruddin et al., 2023; Azad et al., 2024). The KLOM images were analyzed using the ImageJ software to calculate the particle size; the size range was 4–7µm, and the average particle size was 6.54µm (Fig. 3d). The size of the dried microcapsule decreased due to their dehydration, while the essential oil remained unaffected. The advantage of freeze-dried ingredients includes greater porosity, which causes the inner core of essential oils to be exposed to the surrounding environment (Sharma et al., 2024).

FT-IR Analysis

A Fourier Transform Infrared (FT-IR) spectroscopy was used to determine the chemical makeup of the microcapsules. The spectrum (Fig. 4) that was obtained for the microcapsules was analyzed and the presence of various vibrational bands corresponding to the functional groups of chemical bonds present in gum Arabic, gelatin and KL oil used for preparation of the microcapsules was identified. This analysis confirmed that gum Arabic, gelatin and kaffir lime oil were incorporated in the fabricated microcapsules. The spectrum showed O-H stretching ($3323 cm^{-1}$) of gum Arabic overlapped gelatin, C-H stretching ($2923 cm^{-1}$) of kaffir lime oil, C=O ($1638 cm^{-1}$) of gelatin overlapped kaffir lime oil and gum Arabic, C-C ($1433 cm^{-1}$) of gelatin overlapped kaffir lime oil, and

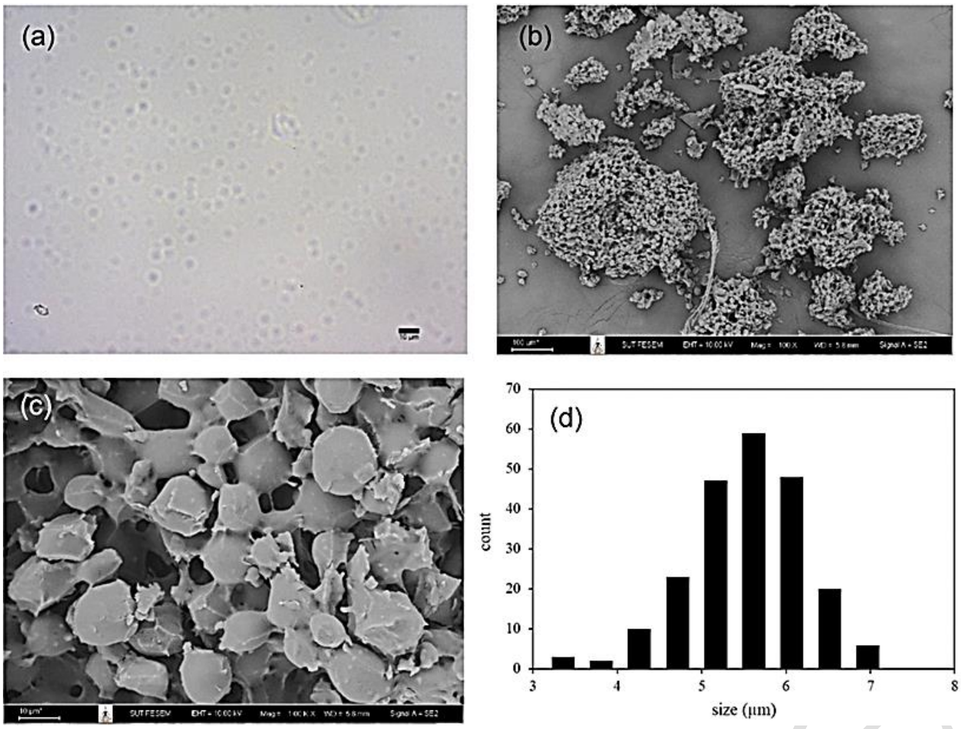

Fig. 3: Microscopic morphology of KLOMs: (a) optical microscope image, 400X (b) SEM image, 100X (c) SEM image, 1000X (d) particle size distribution.

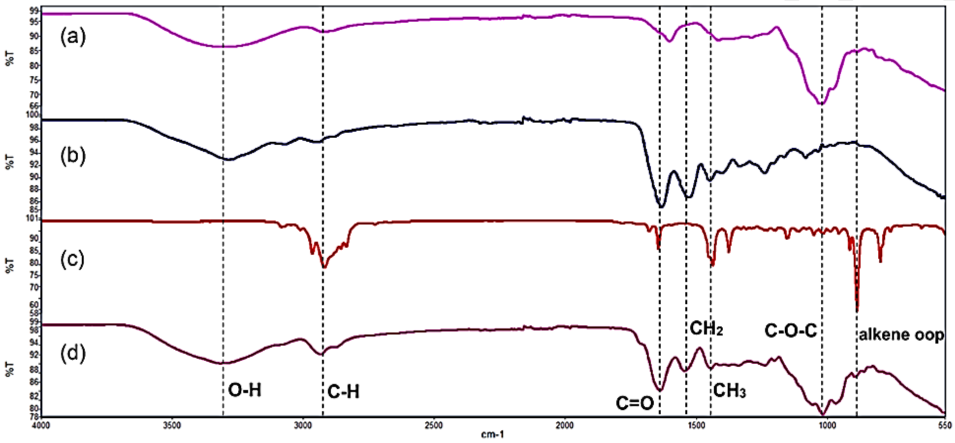

Fig. 4: FT-IR spectra of (a) Gum Arabic (b) Gelatin (c) Kaffir lime oil (d) Microcapsule.

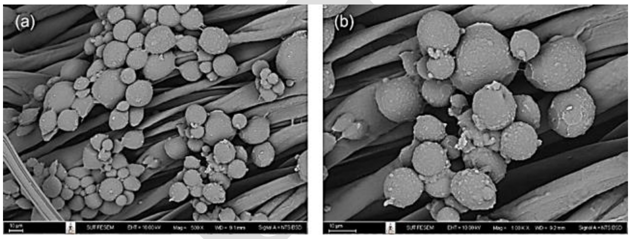

Fig. 5: Microscopic morphology of microcapsule on textile: (a) 500X (b) 1,000X.

characteristic peaks of gum Arabic (C-O-C, 1019cm^{-1}) and kaffir lime oil (alkene out-of-plane (oop), 892cm^{-1}) (Wang et al., 2021). The results showed the core materials and kaffir lime oil in the microcapsules.

Impregnation of Microcapsules onto Textiles

The micrographs of the surfaces and shapes of microcapsules with KLOM adhered to textiles are shown in SEM in Fig. 5. Observations made from the micrographs not only confirm the geometry of the microcapsules but also reveal their deposition on and clustering over the fabric material (Lim & Setthayanond, 2019). Additionally, the results showed that the impregnation process decreased the capsule particle size while still preserving the characteristics of the microcapsules. These results indicated that the impregnation method used in this study is an effective, simple, and cost-efficient approach.

Conclusion

In the extraction of essential oil from kaffir lime, Solvent-Free Microwave Extraction, SFME, was used, with a view of reducing the impact on the environment. In a short time, SFME proved to be effective regarding yield, and with low demands on sample preparation. Many of these compounds were confirmed using GC/MS and these included β-phellandrene, D-limonene, β-pinene, citronella and citronellol. Microwave irradiation significantly enhanced the contents of hydrocarbon monoterpenes and hydrocarbon sesquiterpenes. Gum Arabic and gelatin gave a successful encapsulation of the essential oil in the form of spherical microcapsules of 4–7µm size. Analysis by scanning electron microscopy established successful deposits of oil microcapsules on textile fabrics. Concisely, the techniques for kaffir lime essential oil extraction using SFME and microencapsulation for functionalized textiles have provided insights into the potential opportunities in the textiles industry. The approach was effective, cost-saving, biofriendly, and conducive to the encapsulation of essential oils onto the fabrics. Subsequent research could extend from these approaches to create functional textiles ready for commercialization.

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