

Microencapsulation of *Zingiber cassumunar* Roxb. essential oil in poly-L-lactic acid: Effect of molecular weight on encapsulation efficiency and controlled release

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ABSTRACT: This study investigated the encapsulation of *Zingiber cassumunar* Roxb. essential oil using poly-L-lactic acid (PLLA). Shell capsules containing essential oil were prepared by solvent evaporation using PLLA with number-average molecular weight of 77,000, 140,000, and 200,000 g/mol, at PLLA:essential oil mass ratios of 90:10, 70:30, and 60:40. The molecular weight of PLLA plays a crucial role in the encapsulation efficiency and release behavior by influencing microcapsule formation, structural integrity, and the diffusion of the encapsulated oil. Among the formulations investigated, PLLA with a molecular weight of 140,000 g/mol at a 90:10 PLLA:essential oil mass ratio achieved the optimal balance between encapsulation efficiency and controlled-release performance. The microcapsules were characterized by optical microscopy, scanning electron microscopy, ultraviolet-visible spectrophotometry, and Fourier-transform infrared spectroscopy. The highest release rate was 0.54 mg for PLLA at 140,000 g/mol with a 60:40 PLLA:essential oil mass ratio at 120 min. These findings are applicable to preserving essential oil properties over extended durations, including use in cosmetic, spa products, and controlled-release delivery products.

KEYWORDS: encapsulation, *Zingiber cassumunar*, poly-L-lactic acid (PLLA), essential oil, controlled-release

INTRODUCTION

Essential oils are widely used in textiles, cosmetics, food, and spa industries. They provide antiseptic effects, reduce inflammation and swelling, alleviate stress, and offer a pleasant aroma. Essential oils are organic compounds that plants produce and store naturally. *Zingiber cassumunar* Roxb. is a tuberous plant with large, fragrant, yellow-fleshed rhizomes, long pointed leaves, and inflorescences borne on a peduncle. Its rhizomes contain essential oils and other constituents with properties used to treat bruises, swelling, sprains, aches, flatulence, and asthma, among others [1–3]. The root oil of *Z. cassumunar* has also been characterized for its chemical composition and shown to possess *in vitro* cytotoxic activity supported by molecular docking studies [4]. The rhizome is the most useful part because it contains fragrant essential oils. Essential oils are widely incorporated into cosmetic products, perfumes, and household products due to their diverse properties, mainly their pleasant odor. However, they readily evaporate and degrade through oxidation in air. Similarly, phenylbutanoids in *Zingiber montanum* extract, a closely related species, undergo thermal degradation over time, and extraction as an oil rather than an aqueous extract has been shown to markedly improve their stability [5]. Encapsulation is a promising approach to reduce degradation and control evaporation. Techniques include coating, coacervation, monomer polymerization, precipitation,

and solvent evaporation [6–10]. Encapsulation not only protects the core from the external environment but also enables controlled release of the encapsulated substance.

Biodegradable polymers are studied for applications in agriculture, biomedicine, and pharmaceuticals [11]. They have been used as capsule shells in numerous encapsulation studies, including α -tocopherol loaded in poly(ϵ -caprolactone) prepared by solvent evaporation in an oil-in-water system and vitamin E encapsulated in poly-L-lactic acid (PLLA) using an evaporation technique [12]. PLLA is a well-known biodegradable polymer that is environmentally friendly and non-toxic. In addition, PLLA can be produced from sugarcane and corn starch, which are abundant in Thailand, improving availability. Multiple studies report the advantages of PLLA as a capsule shell for various materials using the solvent-evaporation method, which typically yields smooth, spherical microcapsules. Although PLLA has been widely reported as an encapsulating material for active compounds and essential oil, information regarding the effect of PLLA molecular weight on encapsulation efficiency, morphological characteristics, and release behavior remains limited. *Z. cassumunar* Roxb. essential oil is highly volatile and readily degrades during application. Understanding the effect of PLLA molecular weight is therefore important for improving encapsulation efficiency and controlled-release performance. To investigate this effect, *Z. cassumunar* Roxb. was

Table 1 Composition of PLLA microcapsules containing essential oil.

Ingredient	PLLA:essential oil (%w/w)		
	90:10	70:30	60:40
PLLA (g)	9.0	3.5	3.0
Essential oil (g)	1.0	1.5	2.0
DCM (ml)	20.0	20.0	15.0
PVA solution 1 wt% (ml)	90.0	45.0	45.0

encapsulated in PLLA microcapsules prepared by the solvent-evaporation method using PLLA with different molecular weights. The effects of PLLA molecular weight and PLLA:essential oil mass ratio on microcapsule properties were investigated.

MATERIALS AND METHODS

Chemicals

PLLA, dichloromethane (DCM), and poly(vinyl alcohol) (PVA) were purchased from Sigma-Aldrich, Germany.

Extraction and chemical composition of essential oil

The essential oil was extracted by steam distillation. Fresh *Z. cassumunar* Roxb. rhizomes from Sakon Nakhon Province, Thailand, were rinsed, blended to a fine consistency, and placed in a 5 l round-bottom flask with 3 l of distilled water. The flask was connected to a condenser, and the mixture was boiled at 100 °C. The distillate was collected and transferred to a separatory funnel to isolate the essential oil.

The chemical composition of the essential oil was analyzed by gas chromatography-mass spectrometry (GC-MS) (Agilent Technologies, USA). An Agilent 6890N gas chromatograph equipped with a DB-5MS capillary column (30 m × 0.25 mm, 0.25 μm) and coupled to an Agilent 5973 Inert mass selective detector was used. The GC-MS conditions were: initial oven temperature 70 °C, ramped to 220 °C at 2 °C/min; total analysis time 85 min; injection mode split; column length 30 m; internal diameter 0.25 mm; film thickness 0.25 μm; carrier flow 1.0 ml/min; library Wiley7n. Compound identification was performed using the Wiley7n mass spectral library.

Preparation of polymer capsules encapsulating essential oils

Polymer capsules encapsulating essential oil were prepared in an oil-in-water emulsion using the solvent-evaporation method. PLLA and essential oil were dissolved in DCM at various mass ratios (PLLA with number-average molecular weights of 77,000, 140,000, and 200,000 g/mol; polymer:oil = 90:10, 70:30, and 60:40) to obtain homogeneous organic phases. Each organic phase was added to an aqueous

surfactant solution of PVA at 0.9 wt% and homogenized at 5,000 rpm for 5 min to obtain a milky emulsion. The dispersed phase consisted of organic droplets or polymer droplets containing PLLA/essential oil/DCM (Table 1). The solvent was then removed by gentle stirring until complete evaporation, yielding polymer capsules. The same procedure was applied to all PLLA samples with different molecular weights. The percentage loading and encapsulation efficiency were determined. However, the potential loss of essential oil during the encapsulation process was indirectly assessed through the determination of loading percentage and encapsulation efficiency, as these parameters reflect the amount of oil retained in the microcapsules after preparation.

Characterization of polymer capsules

Polymer capsules were characterized as follows. The morphology after solvent removal was examined by optical microscopy (iScope-LX97, Euromex, The Netherlands) by placing 1–2 drops of emulsion on a microscope slide. External surface features were examined by scanning electron microscopy (SEM, JSM-IT500, JEOL, Japan) by placing a drop of emulsion on a nickel SEM stub, drying at room temperature in a vacuum oven, and sputter-coating with gold. The amount of essential oil encapsulated in polymer capsules was determined by UV-Vis spectrophotometry (Evolution 350, Thermo Fisher Scientific, USA). Capsules containing essential oil were weighed (0.025 mg), dissolved in 25 g chloroform, diluted to 2, 4, 6, 8, and 10 ppm, and analyzed at 290 nm. The chemical structure of the microcapsules was characterized by Fourier-transform infrared spectroscopy (FT-IR, Spectrum 100, PerkinElmer, USA). The release behavior of encapsulated essential oil was studied by dispersing 20 mg of polymer capsules in phosphate-buffered saline (PBS, pH 5.6) at 34 °C. The UV-Vis absorbance of the supernatant was measured at fixed intervals every 10 s. PBS was prepared from NaCl, Na₂HPO₄ · 7 H₂O, KCl and KH₂PO₄ and adjusted to pH 5.6. All experiment performed in triplicate, and the results are expressed as mean ± standard deviation (SD).

RESULTS AND DISCUSSION

Essential oil extraction

Using steam distillation at 100 °C for 6 h, the rhizomes of *Z. cassumunar* Roxb. yielded 11.2 ml of essential oil per 1 kg of fresh material. Water distillation generally gives higher yields than conventional solvent extraction because volatile constituents are lost during solvent-removal steps in the latter [13].

The chemical composition of the essential oil, as determined by GC-MS, showed the following major constituents: methyl 3-phenyl-2-pentenoate (36.46% area), sabinene (34.47% area) [3,14], and 3-cyclohexane-1-ol, 4-methyl-1-(1-methylethyl)

Table 2 The active compounds of essential oil.

Retention time (min)	Chemical composition	Relative peak area (%)
4.22	Bicyclo[3.1.0] hex-2-ene,2-methyl-5-(1-methylethyl)-	0.33
4.40	Alpha-Pinene	1.21
5.36	Sabinene	34.47
5.47	2-Beta-Pinene	2.44
5.66	Beta-Myrcene	1.25
6.54	Alpha-Terpinene	1.06
6.95	dl-Limonene	0.39
7.95	gamma-Terpinene	2.09
8.42	trans-Sabinene hydrate	0.93
9.02	Alpha-Terpinolene	0.36
9.69	cis-Sabinene hydrate	0.88
13.54	3-Cyclohexane-1-ol, 4-methyl-1-(1-methylethyl)	12.02
14.28	3-Cyclohexane-1-mrthanol, alpha, alpha, 4-trimethyl	0.30
23.02	(+)-2-Carene	0.53
33.81	Beta-Sesquiphelladrene	1.43
35.14	3.Beta-ethoxy-2-oxabicyclo[3.5.0] deca-6, 8, 10-triene	0.30
37.95	2-Allyl-1, 4-dimethoxy-6-methylbenzene	1.85
40.31	Methyl 3-phenyl-2-pentenoate	36.46
47.60	3-(1-Camphenyl) propylsaure-methyl ester	0.26
50.25	Benzoic acid, 4-3-methoxy-3-oxo-1-propenyl-methyl ester	1.42

(12.02% area), among other components (Table 2). These results are consistent with previous reports indicating that *Z. cassumunar* Roxb. essential oil contains a high proportion of volatile compounds. Therefore, encapsulation using PLLA plays an important role in minimizing the loss of these volatile compounds and improving their stability. In addition, the hydrophobic characteristics of essential oil constituents are compatible with the PLLA matrix, resulting in high encapsulation efficiency and sustained release, as observed in this study.

Molecular weight and ratio of PLLA

PLLA was used as the shell polymer to encapsulate essential oil. PLLA and essential oil were dissolved in DCM and the organic phase was dispersed into an aqueous surfactant solution. Homogenization produced droplets of PLLA/essential oil/DCM that were stabilized against coalescence by the surfactant, after which solvent evaporation yielded polymer capsules. The effects of molecular weight (MW) and composition were evaluated for PLLA with MW of 77,000, 140,000, and 200,000 g/mol at PLLA:essential oil mass ratios of 90:10, 70:30, and 60:40. During capsule preparation, all systems showed suspended solids and high colloid density. Coagulation increased with polymer content. After settling for 24 h, capsules precipitated because PLLA is denser than water. After solvent evaporation, capsules were spherical, narrowly distributed, and opaque, likely due to phase separation and inhomogeneity within liquid droplets (Figs. 1–3). As solvent evaporated, PLLA separated from the organic phase and migrated outward toward the droplet surface, forming a polymer shell around the essential oil. Spherical capsules formed as polymer

chains had sufficient time to align and consolidate [15]. This is consistent with precipitation of PLLA from the volatile organic solvent and partitioning at the droplet interface, which left the essential oil within the core [16–18]. The particle size was estimated from the SEM micrographs using the scale bars provided by the instrument. The estimated average particle sizes of microcapsules prepared from PLLA with MW of 77,000, 140,000, and 200,000 g/mol were approximately 45 ± 13 , 58 ± 16 , and 69 ± 24 μm , respectively. The increase in particle size with increasing MW of PLLA is attributed to the higher viscosity of the polymer solution, which tends to generate the formation of larger emulsion droplets during homogenization.

The loading and encapsulation efficiencies are summarized in Table 3. The optimum was achieved with PLLA at 140,000 g/mol and a 90:10 polymer:oil mass ratio, giving an encapsulation efficiency of 97.03%. Across all three MWs, encapsulation decreased with increasing oil fraction, following 90:10 > 70:30 > 60:40. Lower polymer content may be insufficient to form stable shells and to encapsulate larger oil volumes [19, 20]. The high encapsulation efficiency obtained with PLLA at 140,000 g/mol resulted from an appropriate balance between solution viscosity and capsule formation. PLLA with a lower MW (77,000 g/mol) likely formed weaker capsule walls, whereas PLLA with a higher MW (200,000 g/mol) produced a more viscous solution that could hinder droplet dispersion during emulsification. As a result, PLLA at 140,000 g/mol provided more favorable conditions for capsule formation, resulting in higher loading capacity and encapsulation efficiency.

SEM observations were consistent with the encapsulation efficiency results. PLLA at 140,000 g/mol

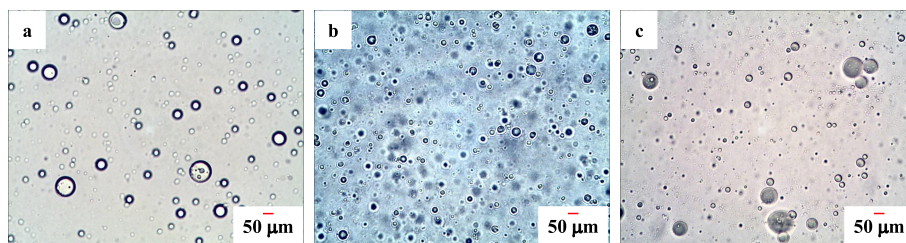


Fig. 1 Optical micrograph of PLLA (MW = 77,000 g/mol): essential oil after solvent evaporation at ratios: (a) 90:10, (b) 70:30, and (c) 60:40.

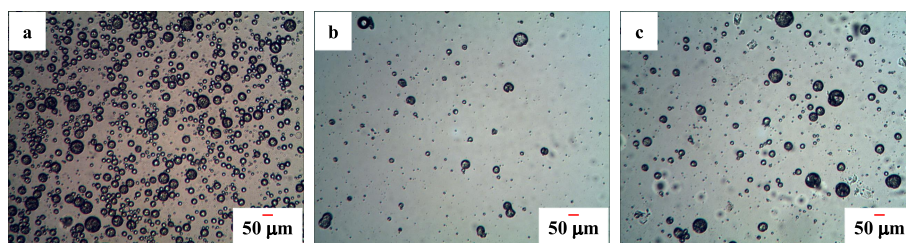


Fig. 2 Optical micrographs of PLLA (MW = 140,000 g/mol): essential oil after solvent evaporation at ratios: (a) 90:10, (b) 70:30, and (c) 60:40.

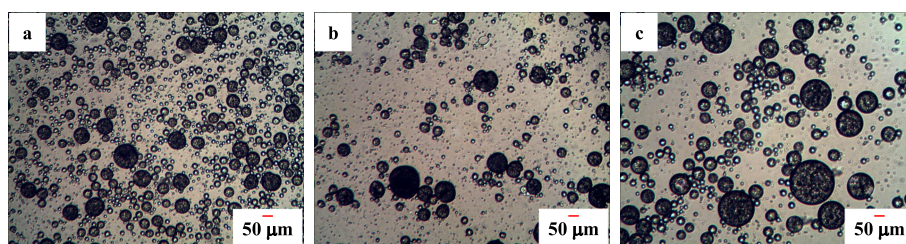


Fig. 3 Optical micrographs of PLLA (MW = 200,000 g/mol): essential oil after solvent evaporation at ratios: (a) 90:10, (b) 70:30, and (c) 60:40.

Table 3 Percentage of loading and encapsulation at different PLLA-MWs and PLLA:essential oil mass ratios.

PLLA molecular weight	Percentage of loading and encapsulation	PLLA:essential oil (% w/w)		
		90:10	70:30	60:40
77,000 g/mol	Loading (%)	5.53 ± 1.35	15.09 ± 0.98	13.98 ± 1.02
	Encapsulation (%)	55.27 ± 2.02	50.29 ± 1.89	34.94 ± 1.67
140,000 g/mol	Loading (%)	9.70 ± 1.45	25.82 ± 0.96	29.35 ± 1.30
	Encapsulation (%)	97.03 ± 2.76	86.06 ± 3.01	73.37 ± 2.80
200,000 g/mol	Loading (%)	8.43 ± 2.14	21.27 ± 2.09	24.55 ± 1.79
	Encapsulation (%)	84.31 ± 3.78	70.89 ± 3.46	61.38 ± 2.97

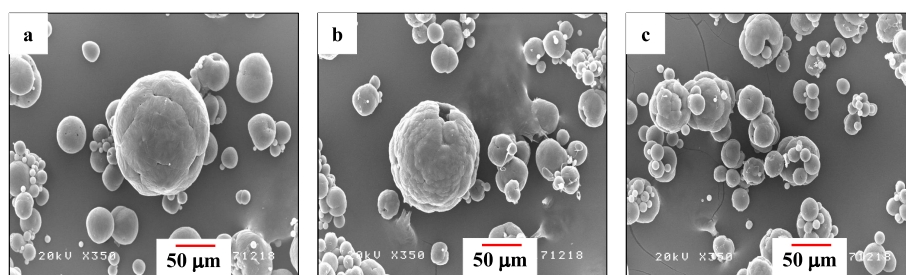


Fig. 4 SEM micrographs of PLLA microcapsule prepared at PLLA:essential oil ratio of 90:10 using PLLA with MW of (a) 200,000 g/mol, (b) 140,000 g/mol, and (c) 77,000 g/mol.

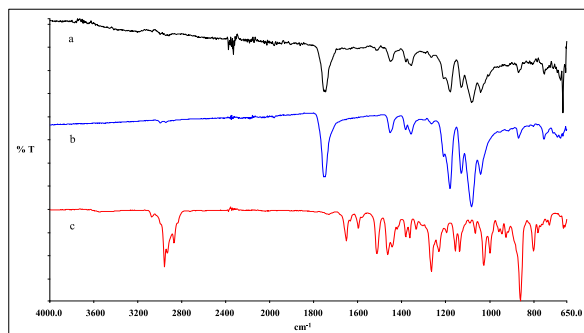


Fig. 5 FT-IR spectra of (a) essential oil polymer capsules (90:10), (b) PLLA, and (c) essential oil.

produced relatively uniform and intact capsules with a more spherical morphology than those prepared from PLLA with MW of 77,000 and 200,000 g/mol (Fig. 4). During solvent evaporation, PLLA precipitated at the droplet interface and formed a shell surrounding the essential oil. The morphology of the resulting microcapsules depended on droplet stability and shell formation during solvent removal. PLLA with lower and higher MWs resulted in less regular capsule structures, reflecting differences in solution viscosity during emulsification and solvent removal. The more uniform morphology observed for PLLA at 140,000 g/mol is consistent with its higher encapsulation efficiency.

Structural characterization of microcapsules

FT-IR was used to identify functional groups of PLLA, essential oil, and the corresponding capsules.

The FT-IR spectra of PLLA, essential oil, and essential-oil polymer capsules are shown in Fig. 5. The capsules showed bands at 2995–2939 cm^{-1} , 1747 cm^{-1} , 1451–1357 cm^{-1} , 1178–1083 cm^{-1} , 861 cm^{-1} , and 708 cm^{-1} , assigned to CH_3 stretching (alkane), $\text{C}=\text{O}$ stretching (ester), CH_3 bending (alkane), $\text{C}-\text{O}$ stretching, $\text{C}-\text{H}$ bending (aromatic), and phenyl deformation, respectively [17,21–23]. These assignments are consistent with the major constituents identified by GC-MS, including methyl 3-phenyl-2-pentenoate ($\text{C}_{12}\text{H}_{14}\text{O}_2$), 3-cyclohexane-1-ol, 4-methyl-1-(1-methylethyl) ($\text{C}_{10}\text{H}_{18}\text{O}$) [24], and sabinene ($\text{C}_{10}\text{H}_{16}$) [25].

The FT-IR spectrum of the microcapsules contained characteristic bands of both PLLA and the essential oil, confirming successful encapsulation. The absence of new absorption bands or significant peak shifts suggests that the essential oil was physically entrapped within the PLLA matrix.

Release rate of microencapsulated essential oil

Consistent with the results shown in Table 3, the highest encapsulation efficiency was obtained with PLLA at 140,000 g/mol. Release studies showed an initial

burst release at the 60:40 polymer:oil mass ratio, attributable to essential oil located near the capsule surface and a looser shell structure that permitted faster diffusion. The rate then decreased and approached a sustained regime by 120 min (Fig. S1) as diffusion proceeded through denser polymer domains [26]. This release behavior suggests a diffusion-controlled mechanism, in which essential oil located at or near the capsule surface was released rapidly during the initial stage, followed by gradual diffusion of the remaining oil through the PLLA matrix. Similar release profiles have been reported for polymeric microcapsules prepared by solvent evaporation, where the polymer shell acts as a barrier that slows the transport of encapsulated compounds [26,27]. The maximum cumulative release amount was 0.54 mg at the 60:40 polymer:oil mass ratio. These results indicate that the prepared capsules can effectively control release of essential oil, suggesting their application in exfoliating products, spa formulations, and related uses [27].

CONCLUSION

This study demonstrated that PLLA microcapsules encapsulating *Z. cassumunar* Roxb. essential oil can be successfully prepared by the solvent evaporation method. The optimal formulation used PLLA with a MW of 140,000 g/mol at a PLLA:essential oil mass ratio of 90:10, achieving an encapsulation efficiency of 97.03%. The highest cumulative release amount was 0.54 mg at 120 min for PLLA (140,000 g/mol) at a 60:40 polymer:oil mass ratio. The results show that PLLA molecular weight influences capsule morphology, encapsulation efficiency, and release behaviour. The high encapsulation efficiency and controlled-release behaviour indicate that PLLA microcapsules can preserve volatile essential oil components and provide sustained release. These findings provide useful guidance for selecting appropriate PLLA molecular weights in the design of controlled-release microcapsule systems. Based on the present results, cosmetic and spa products appear to be the most promising applications.

Appendix A. Supplementary data

Supplementary data associated with this article can be found at <https://dx.doi.org/10.2306/scienceasia1513-1874.2026.s003>.

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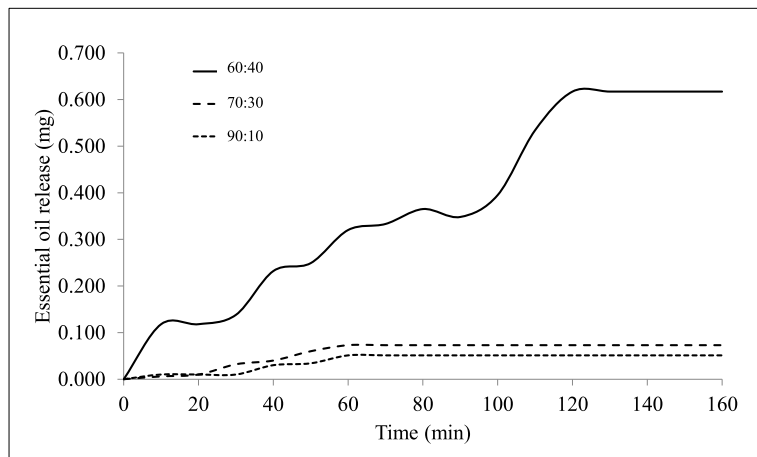
Appendix A. Supplementary data

Fig. S1 Release of essential oil from PLLA capsules of MW 140,000 g/mol.