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Article

Formulation and Characterization of Microencapsules Containing Ethanol Extract of Sungkai Leaves (*Peronema canescens* Jack)

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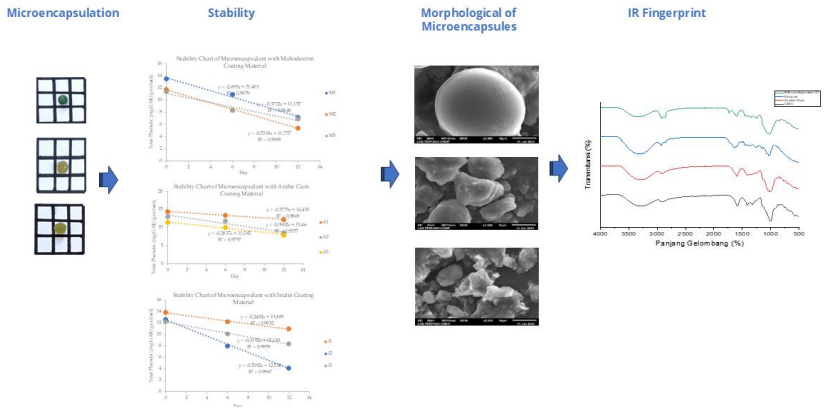
Abstract

The Sungkai plant (*Peronema canescens* Jack.) is a widely recognized as medicinal plant in Indonesia, with its leaves recently gaining attention for their potential health benefits. This study explores the microencapsulation of ethanol extract from Sungkai leaves using three different coating materials—maltodextrin, inulin, and Arabic gum—at varying concentrations. The aim of this study was to identify the optimal microencapsulation formulation using these materials. Microencapsulation was performed using the extrusion method, and the best formulation was characterized by evaluating its physicochemical properties, morphology, and infrared (IR) spectrum. Antioxidant activity was measured using the 2,2-diphenyl-1-picrylhydrazyl (DPPH) assay. The results showed that microencapsule formulation **A1** exhibited superior physicochemical properties compared to other formulations. Scanning electron microscopy (SEM) analysis of **A1** revealed a smooth surface with a slightly rounded shape and minimal wall folds or cracks, suggesting good stability. Moreover, fourier-transform infrared (FTIR) analysis confirmed effective encapsulation of the ethanol extract as well. While the crude extract demonstrated the highest antioxidant activity, microencapsulation slightly reduced this activity. Among the microencapsule samples, formulation A1 (using Arabic gum) retained the most antioxidant potential. In conclusion, formulation A1, utilizing Arabic gum as the coating material, was found to be the optimal microencapsulation formulation for the ethanol extract of Sungkai leaves.

Keywords: *Coating agent, microencapsulation, ethanol extract, Sungkai*

Graphical Abstract



Introduction

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One of the medicinal plants that grows in Indonesia is the Sungkai plant (*Peronema canescens* Jack). *Peronema canescens* Jack (*P. canescens*) in Jambi Province is ethnobotanically used for various therapies such as antifever, antioxidant, and immunostimulant [1]. This plant is traditionally used by the community in medicine or health care, such as bruising medicine, fever medicine, cold medicine, worming medicine, and mouthwash [2]. *P. canescens* contains bioactive compounds that play a role as antidiabetics [3], antihyperurisemia [4], anti-inflammatory [5], potential anticancer [6], and immunostimulant [7]. *P. canescens* also has potential bioactivity as an antibacterial and antioxidantwhich is related to the content of secondary metabolite compounds possessed by *P. canescens* plants, such as alkaloids, terpenoids, phenolics, flavonoids, tannins, and saponins [5].

However, bioactive compounds in extracts have several disadvantages, such as a high organoleptic impact due to the bitter and sour taste of some compounds, low solubility, a tendency to oxidize, and a limited shelf life, reducing the utilization of bioactive compounds [8]. Therefore, some kind of processing or delivering system as an alternative is needed that can overcome this problem, to ensure their effectiveness and target function. Encapsulation is an effective method that improves the phytochemical stability by entrapping the core material with the coating agent [9].

Albeit, the application of encapsulation technology continues to experience developments, such as nanoencapsulation and microencapsulation. Microencapsulation is one of the methods used to protect active substances, improve their physico-chemical properties, and protect them from unpleasant flavors and aromas even adverse environmental conditions [10]. The extrusion method is one of the most popular and simple methods in microencapsulation technology. The advantages of this method compared to other methods are that it is easy to perform, does not require high temperatures, has gentle formulation conditions that ensure higher cell viability, does not use harmful solvents, and can be performed under aerobic and anaerobic conditions [11].
Biopolymers are oftentimes used as coating materials for microencapsulation of various bioactive compounds because they have attractive physico-chemical properties [12]. The selection of a coating material that can avoid compositional changes due to damage to the bioactive compounds in the extract is a crucial point for the success of the microencapsulation process [13]. Coating materials such as inulin, maltodextrin, and Arabic gum are recognized as safe and have been used for the stability of bioactive compounds [14]. Maltodextrin is a polysaccharide class compound consisting of β-D-glucose units obtained by acid or enzyme hydrolysis of some starches (corn, rice, potato, starch, or wheat). Maltodextrin is highly soluble in water, has a neutral taste, and low viscosity, and is easily obtained [15]. Inulin is a polymer of fructose units linked by a terminal glucose unit at the end of the chain.

Inulin has activity as an anticytotoxic and immunomodulator. In addition, inulin also behaves as a prebiotic and stimulates the activity of beneficial microflora in the colon. Since its release only occurs in the gut, inulin can be used to protect bioactive compounds in extracts that are susceptible to degradation along the human digestive tract [15–17]. Arabic gum is a complex heteropolysaccharide composed of D-glucuronic acid, L-rhamnose, D-galactose, and L-arabinose. Arabic gum is often used as a dressing in microencapsulation technology because it has good emulsification properties, high solubility, and low viscosity in aqueous solutions. In addition, it provides good retention of volatile substances and effective protection against oxidation [15]. These three biopolymers were chosen to be used as coating materials in this microencapsulation study of an ethanol extract of *P. canescens* leaves extract due to their functional properties. The purpose of this study was to determine the best microencapsulation formulation using maltodextrin, inulin, and Arabic gum.

Materials and Methods

Chemicals

The main material used in this study was Sungkai leaves (*Peronema canescens* Jack.) obtained from Pamuatan Village, Kupitan District, Sijunjung Regency, West Sumatra Province, Indonesia. Other materials used were $FeCl_3$, 2N sulfuric acid, Dragendorff reagent, Lieberman-Burchard reagent, HCl, Mg powder, HCl, tween-80,

Aquadest, maltodextrin (Lihua Starch), carbomethyl cellulose, CMC (Sigma), Inulin (Pep'D), Arabic gum (Orlife), $CaCl_2$ (Pudak Scientific), Glutaraldehyde (Sigma-Aldrich), Gallic acid (Sigma), Folin-Ciocalteu reagent, Na_2CO_3 (Emsure®), Ascorbic acid (Emsure®), methanol p. a (Emsure®), DPPH (Sigma-Aldrich), The equipment used in this research is glassware (Pyrex®), Fouri-er-Transform infrared spectroscopy (Alpha II-Bruker), a UV-Vis spectrophotometer (Thermo-Fischer), and a SEM-EDX JEOL JSM-6510LA.

Preparation and Extraction

Sungkai leaves are selected in good condition, wet sorted, then washed to separate the test material from dirt, and dried for 7 days [18]. Furthermore, the simplisia was pulverized using a grinder and obtained 2,500g of simplicia powder, and the yield obtained was 10%. The extraction process of *P. canescens* leaves was carried out by maceration using 96% ethanol solvent at 37°C to prevent damage to compounds contained in Sungkai leaves, such as phenolics. The process of separating extracts and solvents is carried out using a vacuum rotary evaporator at temperatures below the boiling point of the solvent to minimize damage to bioactive compounds due to high temperatures [19]. The ethanol extract yielded 303 g, corresponding to an extraction yield of 12.12%. The crude extract was subsequently analyzed by LC-MS/MS. The resulting chromatographic data were presented as peak height plots, and the molecular weights of the detected compounds were identified and analyzed using the MassLynx Mass Spectrometry Software.

Phytochemical and Total Phenolics

Phytochemical screening was carried out to qualitatively test the compounds contained in the ethanol extract of Sungkai leaves, such as flavonoids, tannins, alkaloids, saponins, steroids, and phenolic compounds, following the procedures of previous studies [20]. A gallic acid standard solution was made by dissolving 10 mg of gallic acid in a 10 mL volumetric flask using methanol solvent, thus obtaining a concentration of 1000 mg/L in the mother solution. Then the 1000mg/L mother liquor was diluted into several concentrations, namely 20; 40; 60; 80; and 100 mg/L. Determination of total phenolic content by the Folin-Ciocalteu method A total of 0.5 mL of sample (standard solution and test solution) was put into a 10 mL volumetric flask, 0.5 mL of Folin-Ciocalteau reagent was added, and the flask was allowed to stand for 5 minutes. Then 1 mL of 20% sodium carbonate was added and diluted with Aquadest until the limit mark. The mixture was incubated for 2 hours. The absorbance was then measured at a wavelength of 750 nm [21]. Based on the absorbance values obtained, a calibration curve was made and a linear regression equation for the standard solution was obtained. The total phenolic content of each test solution was determined from the linear regression equation of the standard solution. Total phenolic content is expressed in mg Galic Acid Equivalent (GAE)/g sample.

Table 1. Microencapsulation formula

Microencapsulation

The microencapsulation process of the ethanol extract of Sungkai leaves was carried out using the extrusion method by following the procedure of the study [22]. The dressing mixture was put into 100 mL of 85^0C aquadest, then 1 mL of tween-80 and 1g of dried Sungkai leaf extract were added. After homogenization, the solution was dripped into a 0.2 M $CaCl_2$ solution with ethanol solvent (70%). The formed granules were allowed to stand for 15 min, after which they were filtered. Crosslinking was done with glutaraldehyde crosslinkers. The gel beads were soaked in glutaraldehyde solutions for 5 min. The gel beads were drained and dried to a constant weight (Table 1).

% Yield

The calculation of percent yield is done by calculating the overall weight of the microencapsule product and the total weight of the microencapsule material, which are then calculated using equation 1.

$$\% R = \frac{W0}{WT} \times 100\ \% \quad (1)$$

Notes: %R: Yield product; W0: Microencapsulated weight (g); WT: total weight of microencapsulated material (g)

Samples code	Extracts (g)	Aquadest (mL)	Tween-80 (mL)	Arabic gum (g)	Inulin (g)	Maltodextrin (g)	CMC (g)
A_1	1	100	1	2.7	-	-	0.3
A_2	1	100	1	2.4	-	-	0.6
A_3	1	100	1	2.1	-	-	0.9
I_1	1	100	1	-	2.7	-	0.3
I_2	1	100	1	-	2.4	-	0.6
I_3	1	100	1	-	2.1	-	0.9
M_1	1	100	1	-	-	2.7	0.3
M_2	1	100	1	-	-	2.4	0.6
M_3	1	100	1	-	-	2.1	0.9

Water solubility

One gram of the microcapsule was accurately weighed and dissolved in 20 mL of distilled water. Filtration was carried out using a pre-weighed filter paper that had been dried in an oven at 105 °C for 30 min. After filtration, the filter paper containing the residue was dried again in the oven at 105 °C for 1 hr. The filter paper was then cooled in a desiccator for 15 min before being reweighed to determine the amount of undissolved residue (equation 2).

$$\text{Solubility (\%)} = 1 - \left[\left(\frac{c-b}{\frac{a\times(100-D)}{100}}\right)\times 100\%\right] \quad (2)$$

Where, a: weight of sample used (g); b: weight of filter paper (g); c: weight of filter paper and residue (g); d: moisture content of the sample (%).

Stability and efficiency

One gram of microcapsules was placed in a sealed vial and incubated at 60 °C for a period of 12 days. The total phenolic content was determined on days 0, 6, and 12 to assess product stability as a function of storage duration and temperature. Microencapsulation efficiency was expressed as the ratio of the total phenolic content encapsulated (a) to the total phenolic content measured prior to encapsulation (b) (equation 3).

$$\%\ \text{ME} = \frac{a}{b} \times 100\ \% \quad (3)$$

Notes ME: microencapsulation efficiency; a: total phenolics successfully encapsulated; b: total phenolics before encapsulation.

Morphology analysis and IR-spectrum

Maltodextrin, inulin, Arabic gum, carboxymethylcellulose, and microencapsule had their infrared spectra recorded using Fourier-Transform infrared spectroscopy (Alpha II-Bruker) at wave numbers ranging from 500 to 4000 cm^{-1}. Using a SEM-EDX JEOL JSM-6510LA, the morphology form of the microencapsule produced by the microencapsulation method were examined.

Antioxidant activity

The antioxidant activity was assessed using the 2,2-diphenyl-1-picrylhydrazyl (DPPH) radical scavenging method. Briefly, 3 mL of a freshly prepared 0.1 mM DPPH solution in methanol was mixed with aliquots of the sample solutions prepared at various concentrations. As a negative control, 2 mL of methanol was combined with 3 mL of the DPPH solution, while ascorbic acid was employed as the positive control. All sample preparations and incubations were carried out in the dark rom. Following thorough mixing, the reaction mixtures were incubated for a specified duration (commonly 30 min) at room temperature. Subsequently, the absorbance of each mixture was measured spectrophotometrically at 515 nm using a UV-Vis spectrophotometer. The percentage of DPPH radical scavenging activity was calculated using equation 4.

$$\%\ \text{Inhibition} = \frac{\text{Abs.blank} - \text{Abs. sample}}{\text{Abs.blank}} \times 100\% \quad (4)$$

A_{blank} is the absorbance of the negative control (methanol + DPPH).

Result and Discussion

The ethanol extract of Sungkai leaves is known to be positive for flavonoids, steroids, tannins, phenolics, saponins, and alkaloids based on the findings of phytochemical screening (Table 2). The results of previous studies also reported positive results for the same group of compounds [23]. The total phenolic content was determined in this study using the Folin-Ciocalteau method with gallic acid solution as the standard. The total phenolic content can be determined from the linear regression equation obtained from the gallic acid standard calibration curve, which can be seen in Figure 1.

The connection between absorbance and gallic acid standard solution concentration (mg/L) is depicted in Figure 1. For the typical solution of gallic acid, the linear regression equation is y = 0,0047x + 0,000322 with R^2 = 0.998. To calculate the total phenolic content of Sungkai leaf extract, use this equation. The total phenolic content of the ethanol extract of Sungkai leaves is calculated to be 71,828 mg GAE/g extract based on the data. The determination of total phenolic content aims to see the correlation between antioxidant activity and total phenolic content. By giving one electron from a free radical's unpaired electron so that there are fewer free radicals, polyphenolic substances are known to be able to suppress autooxidation through a radical capture process

Table 2. Phytochemical Test Results of the Ethanol Extract and microencapsule

Secondary metabolites	Extract	Microencapsule
Flavonoids	+	+
Tannins	+	+
Phenolics	+	+
Saponins	+	+
Triterpenoids	-	-
Steroids	+	+
Alkaloids	+	+

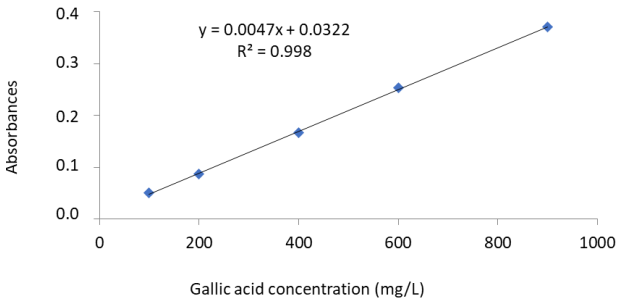
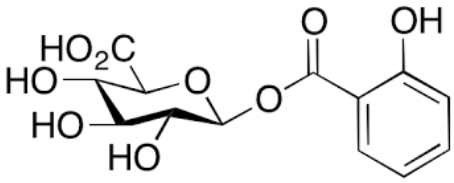


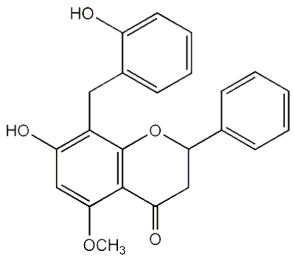
Figure 1. Gallic acid standard calibration curve

Table 3. LC-MS/MS Metabolite Profile Ethanol Extract of *P. canescens*

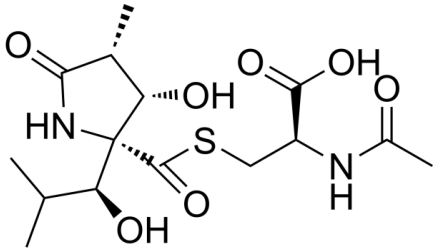
RT (min)	Measured (*m/z*)	Formulas	Proposed metabolite
13.318	375.1253	$C_{23}H_{20}O_5$	5-O-Methylchamanetin
15.473	313.0555	$C_{13}H_{14}O_9$	Salicyl Acyl Glucuronide
14.036	693.1954	$C_{34}H_{34}N_2O_{14}$	Dexylosyl Pradimicin C
13.45	399.1213	$C_{15}H_{24}N_2O_7S$	Lactacystin
13.809	383.1269	$C_{49}H_{70}N_{14}O_{11}$	Asn Asn Asn
14.694	535.2431	$C_{25}H_{40}N_2O_7S$	Lipoxin D4
16.491	699.609	$C_{49}H_{78}O_2$	22:5 Cholesteryl Ester
20.994	607.272	$C_{31}H_{42}O_{12}$	Clerodendrin A
9.857	437.2138	$C_{19}H_{28}N_6O_6$	Arg Asp Phe
3.654	317.0385	$C_{14}H_{17}Cl_3N_2$	Tetrahydro-azepinoquinolines
17.928	295.1703	$C_{13}H_{23}N_6S$	(dimethyl-[2-[(4-pyrimidin-2- ylpiperazine-1-carbothioyl)amino]ethyl]azanium)
17.568	293.1905	$C_{21}H_{25}O$	2-methylbuta-1,3-diene:styrene;hydroxide
18.839	353.2475	$C_{16}H_{37}N_2O_4S$	N,N-dimethylethanamine;dodecylazanide;sulfate
21.354	621.2868	$C_{27}H_{45}N_2O_{14}$	(hydrogen peroxide;4-(4- oxocyclohexyl) cyclohexan-1- one;5-(7-oxooxepan-4-yl) oxepan-2-one) urea
19.724	393.2782	$C_{19}H_{41}N_2O_4S$	2- [(4,4-Dimethyl-2-propan-2- ylhexanoyl) amino] ethyldimethyl-(4- sulfobutyl)azanium)



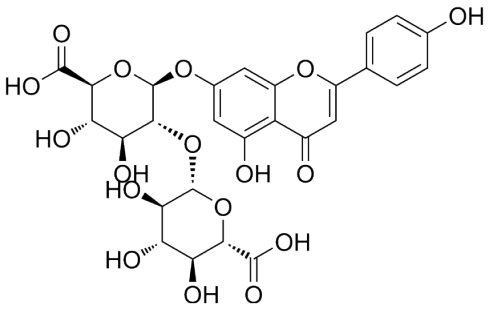
Salicyl Acyl Glucuronide



5-O-Methylchamanetin



Lactacystin



Clerodendrin A

Figure 2. Some chemical structures that play bioactivities

LC-MS/MS analysis was performed on the crude ethanol extract of *Peronema canescens* (sungkai) leaves to profile the chemical constituents. Liquid Chromatography–Tandem Mass Spectrometry (LC-MS/MS) is a powerful analytical technique that integrates the physical separation capacity of liquid chromatography with the high sensitivity and specificity of mass spectrometric detection. In this method, liquid chromatography initially separates the complex mixture into individual components based on their differential interactions with the stationary phase of the chromatographic column and their affinities for the mobile phase.

Subsequently, as each eluted compound exits the chromatographic column, it undergoes ionization and is introduced into the mass spectrometer, where the resulting charged ions are detected and measured. This process generates comprehensive data that include retention times, molecular masses, and fragmentation patterns, allowing for the elucidation of molecular structures, confirmation of compound identities, and quantitative estimation of the analytes present in the extract.

The LC-MS/MS analysis yields chromatograms displaying the retention times and relative abundances of the detected compounds in the form of peak plots. Through interpretation of the mass spectra and comparison with reference databases, the molecular weights and structural information of the detected constituents can be determined. In the present study, the analysis identified a total of 15 active compounds in the sungkai leaf extract, as summarized in Table 3. These compounds are considered to contribute to the observed biological activities of the extract (Figure 2).

Microencapsulation yield

The resulting microcapsules generally exhibited a color consistent with that of the original extract, ranging from dark green to light green, and presented as solid spherical particles (Figure 3). One of the key quantitative metrics for evaluating the efficiency and effectiveness of a microencapsulation process is the percentage yield. The yield values of *P. canescens* leaf ethanol extract microcapsules are shown in Table 4.

The highest yield was recorded for sample A1, with a value of 94.625 ± 0.625%, whereas the lowest yield was observed for sample M3, amounting to 89.325 ± 0.650%. Variations in yield are influenced, among other factors, by the water content of the raw materials. Lower water

content results in a smaller proportion of water mass within the material; consequently, upon drying, the product becomes more compact and lighter, thereby affecting the final yield [24]. Additionally, the extent to which the extract is successfully encapsulated exerts a substantial impact on yield. A higher proportion of extract effectively entrapped within the wall matrix corresponds to a higher percentage of product recovery [25].

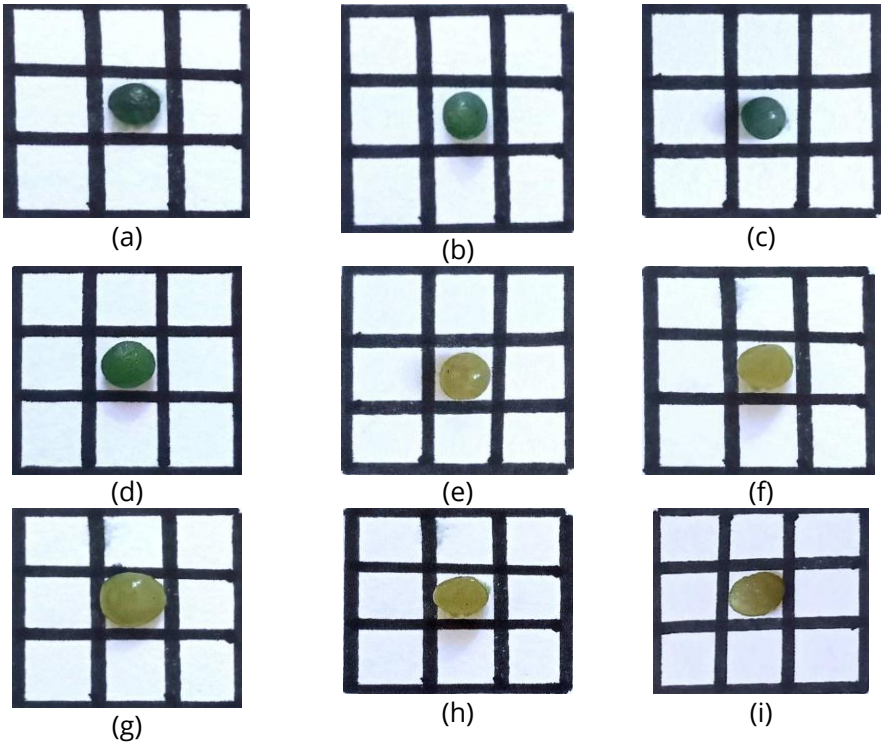


Figure 3. The photograp of microencapsules; A1(a), I1 (b), M1 (c), A2 (d), I2 (e), M2 (f), A3 (g), I3 (h), M3 (i).

Table 4. Percent yield of microencapsule

Samples	% Yield ± SEM
A_1	94.625 ± 0.625
A_2	92.825 ± 0.650
A_3	91.612 ± 0.637
I_1	93.887 ± 0.612
I_2	91.637 ± 0.621
I_3	89.700 ± 0.725
M_1	93.162 ± 0.662
M_2	89.875 ± 0.675
M_3	89.325 ± 0.650

Water solubity of microencapsule

The product's water solubility impacts the release of active compounds after applying microencapsule. A good microencapsule is expected to have a high aqueous solubility value. The data for the microencapsule of an ethanol extract of sungkai leaves in water are shown in Table 5 as a per cent solubility. According to Table 3, sample A1 with Arabic gum dressing material had a maximum microencapsulate solubility per cent in water of 98.95%, whereas sample M1 with maltodextrin dressing material had a maximum of 99.38%. This outcome aligns with previous research findings: micro encapsulants with maltodextrin as a dressing material are more soluble in water compared to microencapsulated prepared using Arabic gum and inulin as dressing materials [16]. The solubility of the microencapsulated product was observed to decrease progressively with increasing concentrations of carboxymethylcellulose (CMC).

This trend can be attributed to the rise in viscosity associated with higher CMC content, which in turn slows the drying process and results in elevated residual moisture within the microcapsules. The high moisture content adversely affects dispersibility, as the microcapsules tend to aggregate and form

cohesive clumps upon contact with water, thereby hindering the development of porous structures necessary for effective dissolution. Consequently, the microencapsulated material exhibits a reduced capacity to absorb and incorporate water [26]..

Table 5. Water solubility of Microencapsule

Samples	% Solubility ± SEM
A_1	98.845 ± 0.105
A_2	97.565 ± 0.105
A_3	97.130 ± 0.340
I_1	96.300 ± 0.210
I_2	95.470 ± 0.160
I_3	94.925 ± 0.105
M_1	99.475 ± 0.105
M_2	98.060 ± 0.050
M_3	97.62 ± 0.260

Stability of microencapsules

The stability test was conducted on extracts that had not been encapsulated and those that had been encapsulated with various coatings. Figure 4 displays the outcomes of the stability test on P. canescens leaf extract. From Figure 4 , it can be seen that the ethanol extract that has not been microencapsulated has a percentage of total phenolic content reduction of 2.695% every unit of time. The percentage of decrease in total phenolic content can be seen from the slope of the regression equation, which is 2.695. The negative sign indicates a decrease in phenolic content. These results also show that each increase in the number of heating days will cause a decrease in total phenolics of 2.695% in *P. canescens* extract. These results indicate that phenolic compounds in the ethanol extract that are not microencapsulated have poor stability when heated. Storage of samples in an oven at 60^0C can reduce the levels of phenolic compounds in the material because bioactive compounds such as phenolics can be damaged at temperatures above 50^0C.

Figure 4 demonstrates that the ethanol extract of *P. canescens* leaves that has been encapsulated has higher phenolic stability than the extract that has not been encapsulated. The findings of this study are consistent with those of previous studies, who found that microencapsulating extract can both increase its shelf life and safeguard its active ingredients [27–29].

Microencapsulation Efficiency

The encapsulation efficiency data are presented in Table 6. In general, a higher microencapsulation efficiency indicates reduced loss of bioactive compounds during processing and storage, thereby enhancing the stability and functional properties of the encapsulated extract. The efficiency of microencapsulation is influenced by multiple factors, including the concentration and physicochemical characteristics of the wall polymers, their solubility, and the solvent evaporation dynamics that occur during the encapsulation process [15,29].

In this study, the highest microencapsulation efficiency for the ethanol extract of *Peronema canescens* leaves was observed in the A1 microcapsule formulation, reaching 80.09 ± 0.105%. This suggests that the specific combination and proportion of encapsulating agents in this formulation provided more effective entrapment and protection of the extract's active constituents. Conversely, the lowest efficiency was recorded in the A3 and M3 microcapsule samples, with values of 63.50%, indicating a relatively higher proportion of compound loss or incomplete encapsulation.

Table 6. Microencapsulation efficiency

Samples	Microencapsulation Efficiency (%)
A_1	80.09 ± 0.105
A_2	72.98 ±.0.105
A_3	63.50 ± 0.260
I_1	77.71 ± 0.050
I_2	78.24 ± 0.340
I_3	70.61 ± 0.160
M_1	75.34 ± 0.210
M_2	65.86 ± 0.340
M_3	63.50 ± 0.050

Furthermore, the data demonstrated a clear trend whereby increasing concentrations of carboxymethyl cellulose (CMC) in the wall material composition resulted in decreased microencapsulation efficiency. This phenomenon may be attributed to the increased viscosity and potential phase separation during solvent evaporation, which can hinder uniform droplet formation and reduce the encapsulation yield. Overall, these findings highlight the

importance of optimizing polymer selection and concentration to achieve desirable encapsulation performance and ensure the stability of bioactive compounds during storage.

The type of dressing material greatly affects the encapsulation efficiency. Gum arabic is a dressing material because it has good emulsifying properties and can form a coating due to its protein content [30]. Gum arabic is a better dressing material than maltodextrin in the encapsulation process of Cocoa pod extract (*Theobroma cacao* L.). This is because maltodextrin has a very low emulsifier and layer-forming ability, which can lead to a lack of microencapsulation efficiency [28].

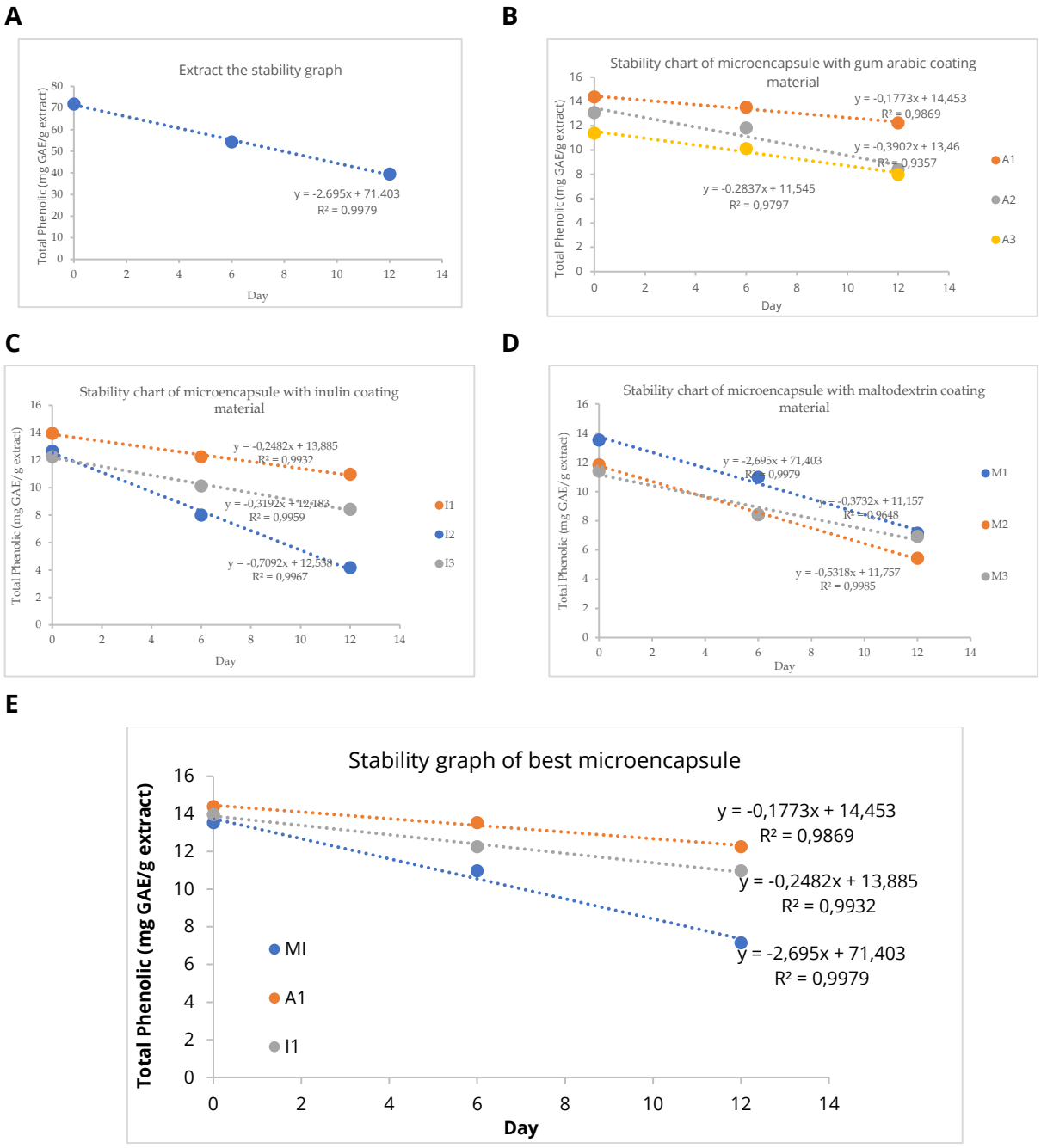


Figure 4. Stability test results of extracts and microencapsules (A) graph of extract stability test results; (B) graph of microencapsule stability test results using Arabic gum dressing; (C) graph of microencapsule stability test results using inulin dressing (D) graph of microencapsule stability test results using maltodextrin dressing; (E) graph of best microencapsule stability test results A1, I1, and M1

Morphology of microencapsules

Using SEM (Scanning Electron Microscopy), the morphological structure of the best microencapsule products from each Arabic gum, inulin, and maltodextrin dressing material can be observed at 2500 and 5000 magnification variations. Figure 5 displays a picture of the microencapsule's morphological structure.

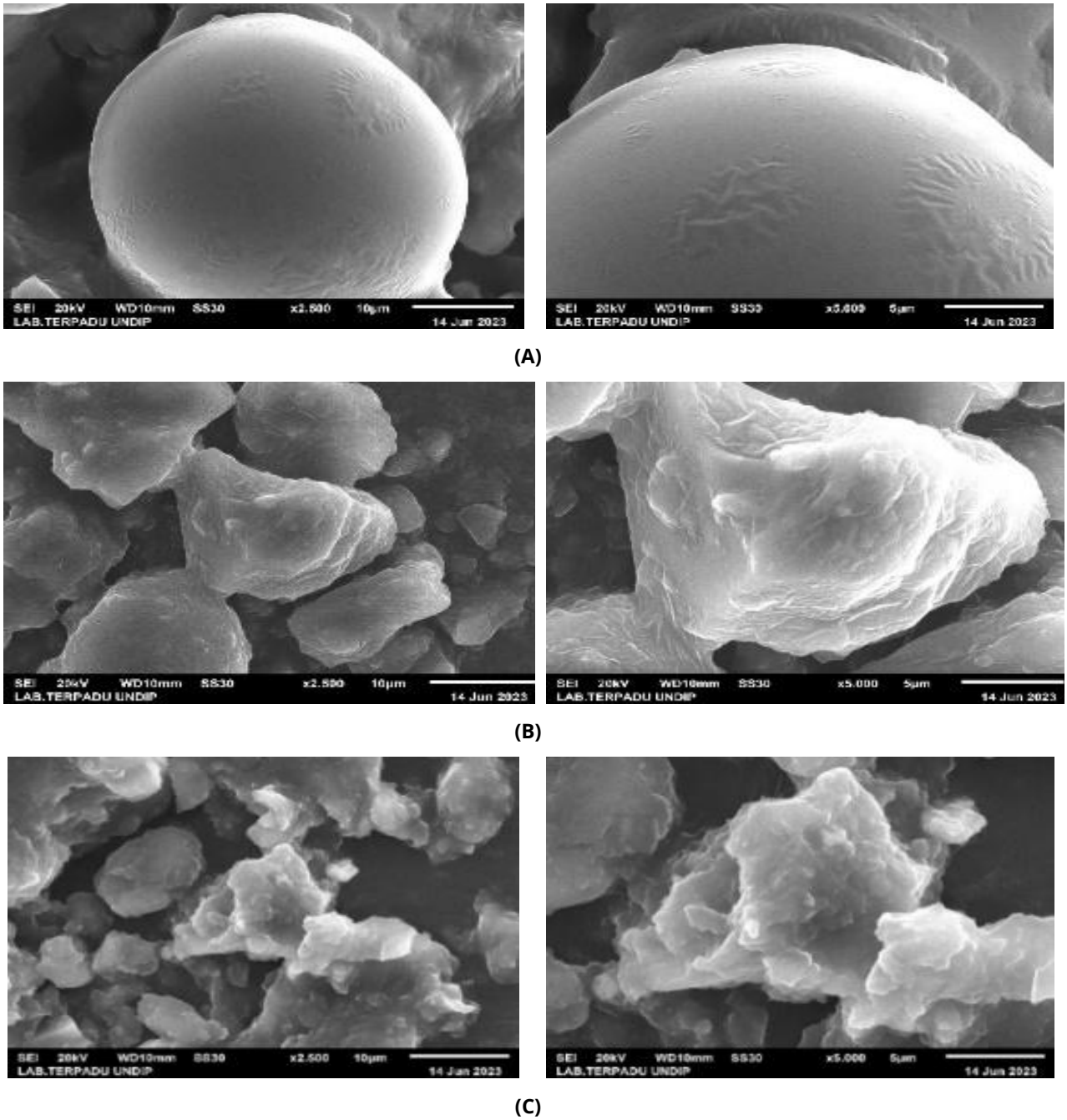


Figure 5. SEM analysis results (A) Morphological structure of the microencapsule A_1 (B): Morphological structure of microencapsule I_1 (C) Morphological structure of microencapsule M_1

Using maltodextrin (**M1**) and inulin (**I1**) dressing materials during the microencapsulation process resulted in particles of various sizes, an irregular surface structure, and deep depressions in the walls. A better microencapsules surface structure was obtained using Arabic gum, **A1**, as a dressing material. This result allows the microencapsulated with Arabic gum dressing material to have better stability. Microencapsule should have a homogeneous and smooth surface with a slightly rounded shape with minimal folds and wall cracks [17]. Microencapsules with rough

surfaces are more sensitive to oxidation reactions than those with smooth surfaces [17]. This result is supported by the data from the microencapsule stability test results, which show that **A1** microencapsule has better stability compared to **I1** and **M1** microencapsule.

The encapsulation is known as one of the most widely used techniques to protect bioactive compounds from various environmental factors and even helps to increase the efficiency of drug delivery systems effectively [31], moisture, and light; hence, it can extend the shelf life of the product and avoid damage [29,31]. The process of forming encapsulation methods on the scale of microparticles proved to be more effective and efficient, especially in drug doses and the reaction speed in reaching the target cell. One of the factors that influence encapsulation technology is the type and ratio of coating material, concentration and structure of the active substances, emulsion properties, and drying process variables, which are important factors to be considered in the encapsulated powder's physical and chemical properties [32].
Arabic gum belongs to a protein coating material that has good layer-forming, binding, and emulsifying properties that boost yield [33]. Maltodextrin has a low surface activity, which leads to a subpar microencapsulation procedure [34]. Treatment of variations in a carboxymethylcellulose (CMC) concentration showed a decrease in per cent yield with each increase in CMC concentration. The increase in CMC content causes the diffusivity of water (the ability of water to move) to decrease so that the efficiency of extract coating decreases.

Treatment of the type of coating material showed that the encapsulation efficiency with gum arabic coating material was greater than that of maltodextrin, even inulin coating material. The type of coating material greatly influences the efficiency of the encapsulation process. Gum arabic is a coating material because it has good emulsifying properties and can form a layer/film [35]. The good emulsifying properties of gum arabic are due to its protein content. Gum arabic is a better coating material than maltodextrin in the microencapsulation process of cardamom oleoresin [36].

A_1 with gum arabic dressing material had a maximum microencapsule solubility percent in water of 98.95%, whereas sample M_1 with maltodextrin dressing material had a maximum of 99.38%. According to previous studies, the higher the maltodextrin concentration, the higher the solubility value [37]. Maltodextrin, a water-soluble polysaccharide, can bind hydrophobic compounds and disperse them uniformly in solution. Its hydroxyl groups interact with water, helping retain moisture in the material. Increasing maltodextrin concentrations improve yield, reflecting its enhanced capacity to coat extracts through emulsion formation, film development, and coating flexibility [38–40].

A1 microencapsule has the highest stability and a percentage decrease in total phenolic content ranging from 0.1773% to 0.1773% per time unit, is known from this investigation. When compared to other microencapsule, A1 is the most effective at protecting phenolic chemicals. This is because microencapsule with good oxidation protection are created when 90% gum arabic and 10% CMC are combined. Previous studies claimed that by raising viscosity, thickening agents can improve the stability of the coating substance. However, if thickening agents are added in excess, the extract will not be adequately covered, allowing for fast destruction due to inadequate protection [28].

IR – Microencapsule Spectrum

Fourier-Transform Infra Red (FTIR) analysis was used to identify the functional groups in secondary metabolite compounds from ethanol extracts, coatings, and microencapsule products. The wavelengths used in this study ranged from 4000 to 500 cm^{-1}. The results of the FTIR characteristics of sungkai leaf ethanol extract, coatings, and microencapsule products can be seen in Figure 6. The results of the interpretation of the functional groups of the microencapsule, the ethanol extract, and the of the coating material are shown in Table 5.

The results of the FTIR spectra analysis in Table 5 indicate that the peaks appearing at wavelengths 2850–2970 and 1340–1470 cm^{-1} are likely to indicate the presence of C-H Alkane functional groups. At wavelengths of 675–995 cm^{-1}, a peak appears, which is likely to indicate the presence of the C-H Alkene functional group. Peaks at wavelengths of 690–900 cm^{-1} are likely to indicate the presence of C-H functional groups in

aromatic rings, which usually appear at wavelengths of 3010–3100 and 690–900 cm^{-1}. Peaks at wavelengths of 3200–3600 cm^{-1} indicate the presence of hydrogen-bonded O-H alcohol/phenol functional groups, which usually appear at these wavelengths.

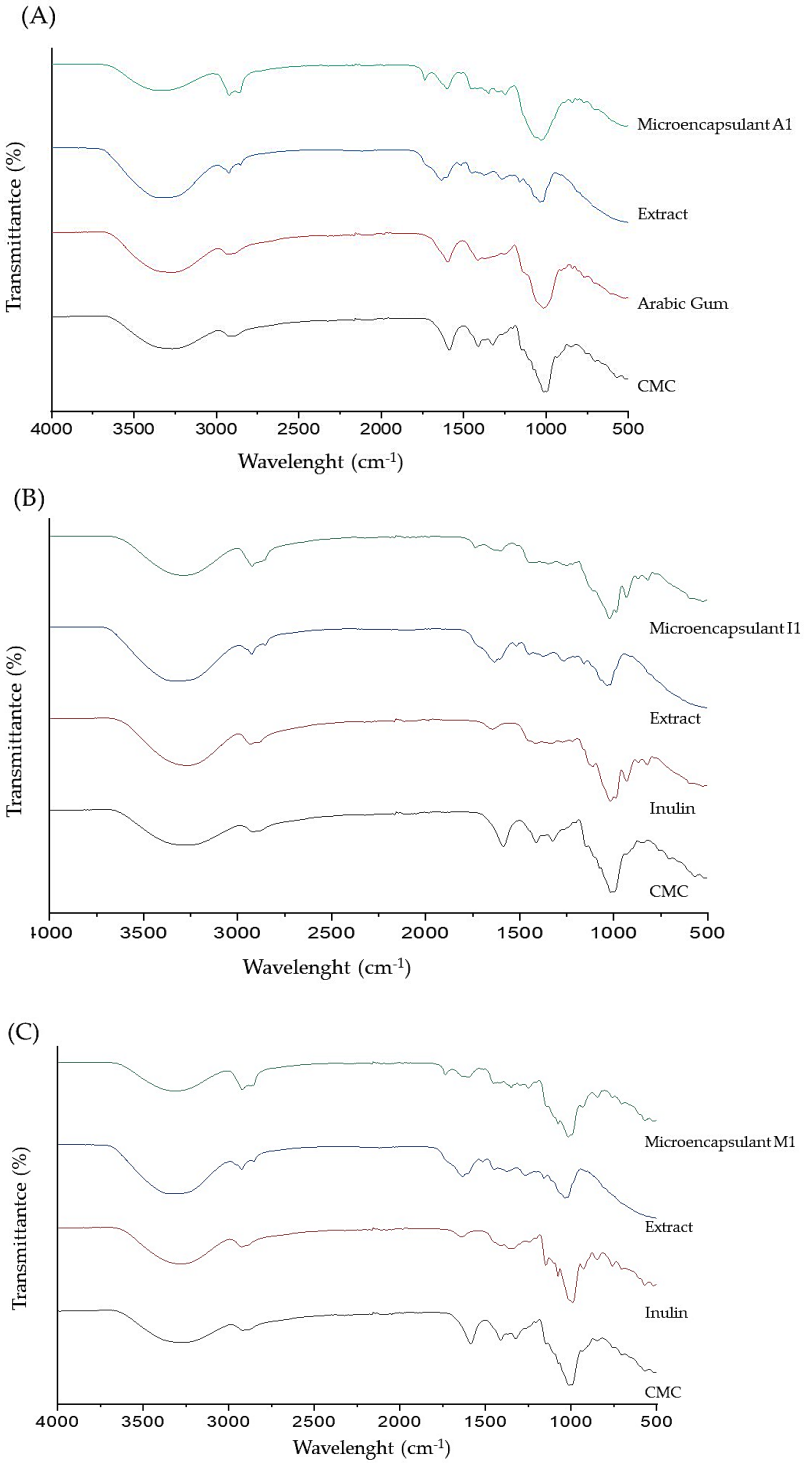


Figure 6. Comparative IR spectra of the microencapsules (A1, I1, M1), the extracts, and all component materials incorporated in the formulations (gum arabic, inulin, maltodextrin, and CMC). The spectra highlight characteristic functional groups and potential interactions, indicating successful microencapsulation through cross-linking reactions.

Table. 7 IR spectrums of microencapsules

No.	Functional groups	Wave number (cm^{-1})							
		a	b	c	d	e	f	g	h
1	Alkane (C-H)	2871	2924	2857	2928	2920	2930	2924	2990
2	Alkene (C-H)	887	985	933	909	920	915	913	913
3	Aromatic ring (C-H)	690-900	690-900	690-900	690-900	690-900	690-900	690-900	690-900
4	Hydrogen/ phenol bonded alcohol (O-H)	3200-3600	3200-3600	3200-3600	3200-3600	3200-3600	3200-3600	3200-3600	3200-3600
5	Alkene (C=C)	1610	1613	1631	1610	1611	1630	1632	-
6	Aromatic ring (C=C)	1600	1603	1602	-	-	-	1606	
7	Alkyne (C≡C)	2150	2112	2150	-	-	-	2120	2110
8	Amine/amide (C-N)	1349	1348	1349	1255	-	-	1263	1322
9	Alcohol/ carboxylic acids /esters (C-O)	1288	1288	1735	-	-	-	1076	1052
10	Aldehydes/ ketones/ Carboxylic acid/ester (C=O)	1734	1735	1735	-	-	-	1732	-

(a) A1 microencapsule; (b) I1 microencapsulet; (c) M1 microencapsule; (d) gum arabic; (e) inulin; (f) maltodextrin; (g) ethanol extract of sungkai leaves; (h) carboxymethylcellulose (CMC).

Peaks at wavelengths of 2500–2700 cm^{-1} may indicate the presence of O-H functional groups in carboxylic acid monomers or hydrogen bonds in carboxylic acids. A peak appears at wavelengths of 3300–3500 cm^{-1} which may indicate the presence of N-H amine or amide functional groups [38,41]. Peaks appear at wavelengths of 1610–1680 cm^{-1} which may indicate the presence of C=C alkene functional groups. Peaks appear at wavelengths of 1180–1360 cm^{-1} which may indicate the presence of C-N amine or amide functional groups that usually appear at these wavelengths. Peaks appear at wavelengths of 1050–1300 cm^{-1} indicate the presence of C-O alcohol, ether, carboxylic acid, or ester functional groups. The peak that appears at a wavelength of 1690–1760 cm-1 is likely to indicate the presence of the C=O aldehyde, ketone, carboxylic acid, or ester functional group. And the peak that appears at a wavelength of 1370 cm^{-1} is likely to indicate the presence of the NO_2 functional group of nitro compounds that usually appear at a wavelength of 1300–1370 cm^{-1}.

The C≡C alkyne functional group, which is formed from tween-80 and glutaraldehyde, is absorbed in the form of a broad peak with variable strength in the area of 2106 and 2158 cm-1 in the encapsulated ethanol extract of sungkai leaves [22]. The ethanol extract of sungkai leaves has been perfectly encapsulated or physically trapped in the tween-80-glutaraldehyde complex, according to the interpretation of the spectra shown in Table 5, so that the functional groups present in the extract are also present in the microencapsule product

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The FTIR spectral data presented in Table 7 confirm the formation of microencapsulation through cross-linking interactions between functional groups. Key absorption bands were

observed across all samples, indicating the presence of major functional groups involved in the encapsulation matrix. Broad absorption in the range of 3200–3600 cm^{-1} corresponds to O–H stretching vibrations, suggesting the presence of hydroxyl groups from polysaccharides such as gum arabic, inulin, maltodextrin, and carboxymethylcellulose (CMC), as well as plant-based bioactive compounds. The slight shifts and broadening of this peak in different samples indicate hydrogen bonding interactions, which are commonly involved in the formation of a stable encapsulation network. The presence of C=O stretching bands in the region of 1732–1735 cm^{-1} and C–O stretching around 1052–1288 cm^{-1} points to carbonyl and ester functionalities, which are typically associated with polysaccharides and organic acids. Shifts in these regions imply the occurrence of electrostatic interactions or covalent bonding, such as esterification, contributing to the cross-linking mechanism within the microcapsule structure. Absorption peaks in the 2850–2960 cm^{-1} range correspond to aliphatic C–H stretching, indicating the organic backbone of the encapsulating materials remains intact. These functional groups likely engage in non-covalent interactions, maintaining the structural integrity of the microcapsules. Moreover, signals detected around 1300–1400 cm^{-1} represent C–N stretching vibrations, commonly found in amines and amide groups, which further support the hypothesis of interaction between nitrogen-containing compounds and hydroxyl- or carboxyl-bearing wall materials [22].

These findings are consistent with previous studies. For instance, in the microencapsulation of ginger volatile oil using gelatin and sodium alginate, FTIR analysis revealed the formation of amide bonds between the amino groups of gelatin and the carboxyl groups of alginate, indicating successful cross-linking during complex coacervation. Similarly, in the preparation of cross-linked chitosan microcapsules for controlled delivery of oxytetracycline, FTIR spectra showed characteristic bands corresponding to amide and phosphate groups, confirming the formation of ionic cross-links between chitosan and hexametaphosphate. Furthermore, studies on the cross-linking of gelatin capsules with aldehydes demonstrated changes in the FTIR spectra, particularly in the amide regions, which were attributed to the formation of cross-links between amino acid chains of gelatin. These spectral changes are indicative of the chemical interactions leading to the stabilization of the microcapsule structure.

The FTIR spectrum shows a wide absorption band at wave numbers 3200-3600 cm^{-1} which indicates a loosening of the O-H (hydroxyl) and wave numbers at 2850-2970 and 1340-14700 cm^{-1} indicate stretching the C-H bond. The encapsulation formulation and the coating material have almost the same spectrum, the peak appearing at a wavelength of 675-995 cm^{-1}, which indicates the presence of the C-H Alkene functional group, which usually appears at a wavelength of 675-995 cm^{-1}. The FT-IR spectra of Tween-80 showed a major band of C-H at 675-995 cm^{-1}, and the transmittance value decreased in the encapsulation results. Meanwhile, the major band in maltodextrin at 1050-1300 cm^{-1} C-O stretching, the same band appears in all encapsulates; this is the same as previous research [42]. Theoretically, the occurrence of a cross-linking reaction between Ca^{2+} and maltodextrin affects the intensity of the asymmetry, and symmetry COO- stretching was observed at 1594 cm^{-1}, and a weak symmetric peak was presented at 1400–1500cm^{-1} Also a peak appearing at a wavelength of 1733 cm^{-1} indicates the presence of the C=O Aldehid/ketone/carboxylic acid/ester functional group, which usually appears at a wavelength of 1690-1760 cm^{-1}. The results of the comparison of the FT-IR spectra between the coating and the encapsulation results show that an encapsulation is formed, and it can be seen that all the spectra present in the encapsulation material appear on the encapsulation.

In conclusion, the FTIR spectral analysis in this study confirms the successful formation of microencapsulation through cross-linking interactions among hydroxyl, carbonyl, ester, and amine groups. These interactions contribute to the formation of a cohesive and stable matrix capable of effectively entrapping bioactive compounds, aligning with findings from similar research endeavors.

Antioxidant activities

An antioxidant activity test was conducted on three microencapsule with the best physico-

chemical characteristics using the DPPH method spectrophotometrically to determine the antioxidant activity based on the IC_{50} value obtained from measuring the absorbance value. According to Table 6, ascorbic acid, used as a positive control, had an IC_{50} value of 5.695 (mg/L), meaning that 5.695 (mg/L) of ascorbic acid is needed to reduce the concentration of DPPH by 50%. According to this number, ascorbic acid has highly potent antioxidant activity. Strong group IC_{50} is between 50 and 100 mg/L, medium group IC^{50} is between 101 and 150 mg/L, and weak group IC_{50} is between 150 and 200 mg/L. A chemical is stated to have extremely strong antioxidant activity if the IC_{50} value is less than 50 mg/L [21].

Table 8. Antioxidant activity of microencapsule

Samples	IC_{50} (mg/L)
A_1	121.176
I_1	124.675
M_1	139.503
Extract	65.02
Ascorbic acid	5.695

The antioxidant activity assay of ethanol extracts of P. canescens leaves microencapsulated with different carriers showed IC_{50} values ranging from 121.176 to 139.503 mg/L. Among these, microencapsule A1 exhibited the highest IC_{50} value, indicating the lowest antioxidant activity compared to the others. The variations in antioxidant activity across microcapsulants are attributable to differences in the content of secondary metabolites in the encapsulated extracts. As shown in Figure 4, A1 microencapsule retained higher amounts of phenolic compounds. Antioxidant activity is closely related to total phenolic content, as these compounds can donate hydrogen atoms to neutralize DPPH radicals.

Maltodextrin is stable against oxidising agent but has poor emulsification capacity and stability and low oil retention. Maltodextrin can also reduce viscosity and has the property to prevent oxidation so that the antioxidants will be well enveloped [27,43]. The concentration of the dressing also plays an important role in the encapsulation process. Too high amount of dressing makes the emulsion dense, which makes the atomisation process difficult. The too-high dressing also causes puffing or ballooning and particle cracking. At a certain concentration of maltodextrin addition, the antioxidant quality and the ability to capture free radicals will be better [44]. The treatment of dressing type showed that gum arabic had greater antioxidant activity than maltodextrin. The use of maltodextrin dressing has high oxidation resistance properties. It can reduce the viscosity of the emulsion combined with other dressings with better emulsifying properties that cause antioxidants in the encapsulate to be enveloped and well protected [45].

Gum arabic binder has better emulsifying properties than maltodextrin. Antioxidant activity is also influenced by the properties of gum arabic binder, which can form texture, form film, bind and emulsify well so that gum arabic can maintain the core material of the product because the gum arabic binder can form a layer that can protect the core material from the process of destructive changes [12,46,47]. The antioxidant activity of the encapsulate is related to the total phenol content. A high total phenol content of the encapsulate will result in high antioxidant activity as well. Hence, the ability of the antioxidant to donate electrons in terms of suppressing the development of free radicals is also higher. Protection using the encapsulation process can prevent degradation due to light or oxygen radiation and slow evaporation, then will minimise the loss of antioxidants due to oxidation [48].

The antioxidant activity of the samples was evaluated based on IC_{50} values obtained from the DPPH radical scavenging assay, as summarized in Table 8. IC_{50} represents the concentration of sample required to inhibit 50% of DPPH radicals, with lower values indicating stronger antioxidant capacity. Among all the samples tested, the crude extract exhibited the highest antioxidant activity, with an IC_{50} value of 65.02 mg/L. This suggests a high presence of bioactive compounds—particularly phenolic and flavonoid constituents—which are known for their free radical scavenging capabilities.

In contrast, the microencapsule (**A_1, I_1,** and **M_1**) demonstated moderately reduced antioxidant activity, with IC_{50} values of 121.176 mg/L, 124.675 mg/L, and 139.503 mg/L, respectively. Among them, the gum arabic-based microcapsule (A_1)

retained the highest antioxidant activity, followed by the inulin-based (I_1) and maltodextrin-based (M_1) systems. The choice of wall material plays a significant role in determining antioxidant preservation. Maltodextrin, for example, has good oxidation resistance and can reduce emulsion viscosity, which helps protect encapsulated antioxidants. However, it also has poor emulsification capacity and low oil retention, which can limit its effectiveness as a sole encapsulating agent. Excessive concentrations of maltodextrin may further increase emulsion density, making atomization difficult and potentially causing puffing, ballooning, or particle cracking during drying. Conversely, at optimal concentrations, maltodextrin can improve the stability and antioxidant quality of the microcapsules [49].

Overall, gum arabic produced microcapsules with higher antioxidant activity compared to maltodextrin. This may be attributed to gum arabic's superior emulsifying properties, which enhance the encapsulation efficiency and protection of antioxidant compounds during processing and storage. Combining maltodextrin with other wall materials that possess better emulsifying capacity can further improve encapsulation performance and antioxidant retention [50].

Moreover, the IR-fingerprint supports these conclusions. Characteristic absorption bands observed in the spectra confirm the presence of functional groups involved in hydrogen bonding and cross-linking, such as –OH (3200–3600 cm^{-1}), C=O (1730–1735 cm^{-1}), and C–O (1050–1280 cm^{-1}). These interactions suggest that phenolic hydroxyl groups from the extract are likely bound or encapsulated within the wall matrix through hydrogen bonding or electrostatic interactions. The reduced antioxidant activity in microencapsulated forms may thus be attributed to the encapsulation of these active sites, which limits their immediate availability in the DPPH assay. Despite this decrease in apparent antioxidant activity, the encapsulation process remains beneficial for practical applications. It enhances the physicochemical stability of bioactive compounds and allows for targeted release, which is particularly valuable in functional food or pharmaceutical formulations. Furthermore, ascorbic acid was used as a positive control, displaying a remarkably low IC_{50} of 5.695 mg/L. This result validates the reliability of the assay and confirms that the tested plant extract possesses moderate antioxidant potential when compared with a well-established antioxidant standard.

Conclusions

The crude extract exhibited the highest antioxidant activity, indicating a rich presence of active compounds, while microencapsulation slightly reduced this activity, with the gum arabic-based formulation (**A_1**) retaining the most antioxidant potential among the encapsulated samples. The **A1** microencapsule had a homogenous, smooth surface and a slightly rounded shape as well as few wall folds and cracks, which suggested that the product would have higher stability, according to the results of morphological study performed using SEM. The ethanol extract of sungkai leaves was successfully encapsulated, according to the findings of functional group analysis using FTIR.

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Author Contributions

Conceptualization, ILT; ML. and F; Methodology, F; ILT; Software, RDP; ML; Validation, ILT; ML and MBS; Formal Analysis, ILT; F; and ML; Investigation, F; ILT; Resources, ML; Data Curation, ILT, F, ML, MBS; Writing – Original Draft Preparation, ILT, F.; Writing – Review & Editing, ML; MBS; Visualization, ILT; RDP; Supervision, ML.; Project Administration, ILT; Funding Acquisition, ML; ILT; RDP.

Conflict of Interest

The authors declare no conflict of interest.

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