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# Microencapsulation of Xanthone extracted from *Garcinia Mangostana* pericarp using freeze drying

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**Abstract**

*Garcinia mangostana* fruit is one of the most popular fruit and known as a tropical fruit that has high nutrition value and widely cultivated in the tropical rainforest of some Southeast Asian nations. The main objectives of this research were to identify the composition of extracted xanthones and to determine the antioxidant activity, total phenolic content, moisture content and antimicrobial activity of encapsulated *Garcinia mangostana* pericarp. Encapsulation process was done by using freeze drying technique. Antioxidant activity, total phenolic content, moisture content and antimicrobial activity of encapsulated xanthones analysis were conducted. The amount of xanthones was detected at range of 515 to 4000 $cm^{-1}$ through Fourier transforms infrared spectroscopy (FTIR). The highest value of radical scavenging activity for non-encapsulated xanthones was 82.69% whereas 88.01% for encapsulated xanthones. The total phenolic content (TPC) was found 5.240 mg GAE/g and 6.359 mg GAE/g for non-encapsulated and encapsulated samples, respectively. In conclusion, encapsulated xanthones from *Garcinia mangostana* pericarp has high antioxidant activity and antibacterial properties which has potential to the manufacturing functional food, cosmeceutical and pharmaceutical industries.

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## 1.0 Introduction

The *Garcinia mangostana* fruit is a standout amongst the most famous fruit and known as a tropical natural fruit that has high nourishment value. *Garcinia mangostana* belongs to the family of *Guttiferae* and is named as “the queen of fruits” which is broadly planted in the tropical rainforest of some Southeast Asian nations like Indonesia, Malaysia, Sri Lanka, Philippines, and Thailand (Pedraza-Chaverri et al., 2008).

Xanthones is a phenolic compound that has strong antioxidant properties. It highly contains in *Garcinia mangostana* pericarps. The present of antioxidant can prevent the oxidation occurs in chemical reaction which can cause the damage of cell. Moreover, antioxidant is also known as anti-cancer agent. There is abundance of *Garcinia mangostana* pericarps thrown away as a waste by the consumer during the season of fruit. Currently, due to the most previous research done, the pericarp of *Garcinia mangostana* is not just a waste product but has much beneficial compound.

Xanthones can be widely extracted by using the conventional extraction method. Xanthones are commonly obtained by extraction with organic solvents such as ethanol, acetone, dichloromethane and hexane (Joana Gil-Chávez et al., 2013). Chromatography has been used to isolate two new highly oxygenated prenylated xanthones and twelve well-known xanthones from the pericarp of mangosteen. The content of xanthones inside the *Garcinia mangostana* (GM) pericarps have beneficial properties such as antitumoral, anti-inflammatory, antiallergy, antibacterial, antiviral, antifungal and antioxidant activities. According to Palakawong et al. (2010), antioxidant activity of *Garcinia mangostana* (GM) pericarp extracted with 50% ethanol, $IC_{50}$ had 5.94 μg/ml antioxidant activity which was the highest compared to its bark and leaves.

Soxhlet extraction method is widely used as a conventional standard extraction method compared to other extraction methods such as normal stirring extraction, ultrasonic extraction and microwave-assisted extraction (Yoswathana & Eshtiaghi, 2015). The main advantages of using soxhlet extraction method is, it can increase the temperature and help to reduce the viscosity of required solvent. Generally, Soxhlet extraction can retain the extracted nutrients from *Garcinia mangostana*

(GM) pericarp without making any changes or cause any damages.

Microencapsulation is one of the potential techniques to preserve the beneficial compound from extracted *Garcinia mangostana* pericarp as well as can preserve the taste and the time span of usability of product. Microencapsulation of xanthones also can keep its unique appearance, season and dietary substance (Zuidam & Nedović, 2010). Furthermore, freeze drying can avoid microcapsule of xanthones to recoil or toughen. The procedure of freeze drying as a rule can holds roughly 97% of the nutritional value of xanthones. In contrast, the disadvantage of other drying process particularly the dryer is utilizing high temperature during the operation which can destroy a significant amount of xanthones. The substance of xanthones does not contract or toughen through freeze drying process, however it can retain the odour, flavour, colour shape and nourishing substance of the xanthones.

The main objectives for this research are to identify the amount of extracted xanthone in encapsulated *Garcinia mangostana* pericarp and to determine the antioxidants activity, total phenolic content, antimicrobial activity and moisture content of encapsulated xanthone. The scope of this research was to discover and identify the amount of xanthone from *Garcinia mangostana* pericarp. The abundance of *Garcinia mangostana* pericarp was collected and the content of xanthone inside the pericarp was extracted by using the solvent extraction. The main type of solvent that has been used in this extraction process was methanol.

## 2.0 Methodology

### *2.1 Sample preparation*

The mature fruit of *Garcinia mangostana* is obtaining from the trader in the night market located at Shah Alam, Selangor. *Garcinia mangostana* pericarp was manually separated from the fruits. *Garcinia mangostana* pericarp samples was cut into small pieces and roughly ground into powder. The initial moisture content of fresh *Garcinia mangostana* pericarp was analysed using moisture analyser.

### *2.2 Sample Extraction and Quantification*

Xanthone was extracted from *Garcinia mangostana* pericarp using Soxhlet extraction with methanol as a solvent (Ramluckan et al., 2014). 30g of the *Garcinia mangostana* pericarp was weighed using electronic balance. The temperatures that required during the extraction was between 80°C and 100°C. This experiment was run for 4 hours and 6 hours before sent to the evaporation process. Determination of the functional group of the phenolic content and its composition was carried out using Fourier transform infrared spectroscopy (FTIR).

### *2.3 Free Radical Scavenging Assay*

Free radical scavenger is utilizing the technique of 2,2-diphenyl-1-picrylhydrazyl (DPPH) with some adjustment. 10 mg DPPH was weighed and then dissolved in 100 ml of 80% v/v methanol (0.1 mg/ml). Next, 1 ml of extracted sample was mixed with 4 ml of 80% v/v methanol and 1 ml of DPPH in amber bottle. The mixture was shaken vigorously and kept in dark for 30 minutes at room temperature. The colour of the mixture changed from purple to yellow due to the present of antioxidants. Then, the absorbance reading of mixture without any sample was measured as control which consist of 1 ml DPPH and 4 ml 80% v/v methanol. The control was kept in the dark for 30 minutes at room temperature. The absorbance was analysed at 517 nm against blank using UV-visible spectrophotometer. The percentage of radical scavenging (antioxidant activity) was determined by applying the formula given as shown in Eq. (1) (Evary et al., 2019):

$$\% \text{ Free radical scavenging} = [(A_{sample}-A_{control})/A_{control}] \times 100\% \quad (1)$$

where: $A_{control}$ is absorbance of the control and $A_{sample}$ is absorbance of the sample

### *2.4 Total Phenolic Content (TPC) Analysis*

Total phenolic content (TPC) was determined by using Folin–Ciocalteu method. The assay was carried out in 1 ml of extract which had been transferred to the bottle to quantify the total phenolic concentration. Next, 500 µl of Folin-Ciocalteu solution, 1 ml of the sodium carbonate solution and 10 ml distilled water were added. The absorbance was measured at 765 nm (80% v/v methanol) after standing at room temperature (30°C) for 30 min by using UV-visible spectrophotometry. Gallic acid was used as a standard with concentrations ranging from 0.0025 to 0.6 mg/ml. The total phenolic content was calculated as gallic acid equivalent (GAE) by following equation as shown in Eq. (2):

$$T = C \times V/M \quad (2)$$

where:
T = total phenolic content (TPC)
C = concentration of gallic acid from the calibration curve (mg/ml)
V = volume of extract solution (ml)
M = weight of extract (g)

### *2.5 Microencapsulation of Extracted Sample*

The microencapsulation of extracted *Garcinia mangostana* pericarp was conducted using freeze drying process. The sample of extracted xanthones from *Garcinia mangostana* pericarp was placed into the freeze drying and the duration of freeze drying process was 48 hours to get the formation of microcapsule which is physically powder or tiny particles.

## 3.0 Results and discussion

### *3.1 Microencapsulation of xanthones using freeze drying*

The mixture of xanthones and maltodextrin was having a pre-treated and glimmer solidified which was placed in a vacuum chamber inside the equipment of freeze drying. The fundamental gear that was utilized as a part of this drying procedure was a stop dryer. The moisture of xanthones was drawn off by the process of evaporating the ice in certain temperature as low as -50 ℃. The content of moisture that was removed from the extracted xanthones is around 90 percent which is exceptionally productive than the other drying process. The freeze dried xanthones in solid phase which regularly observed as powder was kept appropriately in tight container to prevent excess moisture and to guarantee the freshness of microcapsule until opened and being consumed. The extracted xanthone was dried by using freeze dryer in two days which equal to 48 hours. Freeze drying helps to remove the amount of water and excess of extraction solvent (methanol) that available in the samples in getting powder form. From the observation, the amount of extracted xanthone in powder form that obtained was about 10 g for each of samples. Physically, the colour of collected powder for each sample was yellowish as shown in Fig. 1.

**Fig. 1**: Sample in powder form after freeze drying

**Table 1**: Summary of FTIR result description of the samples before freeze drying.

| SAMPLE | Temperature (°C) Time (hour) | IR Spectrum Range ($cm^{-1}$) | Absorbance Band | Peak | Functional Group |
|---|---|---|---|---|---|
| **1** | 80 °C 4 hours | 3291.5 Alcohol, Phenols Broad Appearance | 1646.75 C=C (Both $sp^2$) Medium Appearance | 1013.60 Ordinary Bond | Fluoroalkenes |
| **2** | 80 °C 5 hours | 3275.24 Alcohol, Phenols Broad Appearance | 1645.72 C=C (Both $sp^2$) Medium Appearance | 1014.19 Ordinary Bond | Fluoroalkenes |
| **3** | 80 °C 6 hours | 3276.50 Alcohol, Phenols Broad Appearance | 1646.83 C=C (Both $sp^2$) Medium Appearance | 1013.66 Ordinary Bond | Fluoroalkenes |
| **4** | 90 °C 4 hours | 3275.73 Alcohol, Phenols Broad Appearance | 1646.96 C=C (Both $sp^2$) Medium Appearance | 1013.60 Ordinary Bond | Fluoroalkenes |
| **5** | 90 °C 5 hours | 3275.16 Alcohol, Phenols Broad Appearance | 1646.35 C=C (Both $sp^2$) Medium Appearance | 1013.48 Ordinary Bond | Fluoroalkenes |
| **6** | 90 °C 6 hours | 3275.95 Alcohol, Phenols Broad Appearance | 1646.48 C=C (Both $sp^2$) Medium Appearance | 1013.59 Ordinary Bond | Fluoroalkenes |
| **7** | 100 °C 4 hours | 33293.87 Alcohol, Phenols Broad Appearance | 1646.34 C=C (Both $sp^2$) Medium Appearance | 1014.64 Ordinary Bond | Fluoroalkenes |
| **8** | 100 °C 5 hours | 3323.53 Alcohol, Phenols Broad Appearance | 1612.30 C=C (Both $sp^2$) Medium Appearance | 1011.32 Ordinary Bond | Fluoroalkenes |
| **9** | 100 °C 6 hours | 3327.87 Alcohol, Phenols Broad Appearance | 1646.61 C=C (Both $sp^2$) Medium Appearance | 1017.59 Ordinary Bond | Fluoroalkenes |

**Table 2**: Summary of FTIR result description of the samples after freeze drying.

| SAMPLE | Temperature (°C) Time (hour) | IR Spectrum Range ($cm^{-1}$) | Absorbance Band | Peak | Functional Group |
|---|---|---|---|---|---|
| 1 | 80 °C 4 hours | 2353.10 N–H Bond Ammonium Ions Multiple Broad Peak | 1757.03 Aldehyde Cyclic 5-Membered | 667.86 Vinyl Cis-disubstituted | Alkenes |
| 2 | 80 °C 5 hours | 2375.35 N–H Bond Ammonium Ions Multiple Broad Peak | 1757.93 Aldehyde Cyclic 5-Membered | 667.8 Vinyl Cis-disubstituted | Alkenes |
| 3 | 80 °C 6 hours | 2376.10 N–H Bond Ammonium Ions Multiple Broad Peak | 1757.00 Aldehyde Cyclic 5-Membered | 667.94 Vinyl Cis-disubstituted | Alkenes |
| 4 | 90 °C 4 hours | 2364.08 N–H Bond Ammonium Ions Multiple Broad Peak | 1540.66 Aromatic C=C Weak to Strong (usually 3 or 4) | 668.09 Vinyl Cis-disubstituted | Alkenes |
| 5 | 90 °C 5 hours | 2362.90 N-H Bond Ammonium Ions Multiple Broad Peak | 1758.18 Aldehyde Cyclic 5-Membered | 667.94 Vinyl Cis-disubstituted | Alkenes |
| 6 | 90 °C 6 hours | 2377.09 N–H Bond Ammonium Ions Multiple Broad Peak | 1758.10 Aldehyde Cyclic 5-Membered | 667.71 Vinyl Cis-disubstituted | Alkenes |
| 7 | 100 °C 4 hours | 2353.00 N–H Bond Ammonium Ions Multiple Broad Peak | 1540.97 Aromatic C=C Weak to Strong (usually 3 or 4) | 667.95 Vinyl Cis-disubstituted | Alkenes |
| 8 | 100 °C 5 hours | 2352.51 N–H Bond Ammonium Ions Multiple Broad Peak | 1756.99 Aldehyde Cyclic 5-Membered | 667.90 Vinyl Cis-disubstituted | Alkenes |
| 9 | 100 °C 6 hours | 2352.29 N–H Bond Ammonium Ions Multiple Broad Peak | 1758.27 Aldehyde Cyclic 5-Membered | 667.92 Vinyl Cis-disubstituted | Alkenes |

### *3.2 Fourier transform infrared spectroscopy (FTIR)*

The FTIR analysis is the further analysis to detect and determine the functional group of the phenolic content and its composition in extracted xanthones. All the samples were analysed and detected in the range of 515.0 to 4000 $cm^{-1}$. For samples before freeze drying, the composition detected in all samples were alcohol and phenol broad appearance. This is due to the samples still contains of methanol which not fully removes from the during evaporation stage. Furthermore, all samples were detected in C=C medium appearance and fluoroalkene ordinary bond. Most of the samples achieved the transmittance at a range between 96.1 %T to 97.6 %T which is highly corresponding to the wavelength. Table 1 and 2 show FTIR result description of the samples before and after freeze drying, respectively. For samples after freeze drying, most of the samples consist of N-H bond and ammonium ions with multiple broad peak. In additional, most of the samples were detected consist of aldehyde with cyclic 5-membered vinyl with cis-disubstituted alkenes. The samples achieved the transmittance at range between 130 %T to 150 %T which is highly corresponding to the wavelength.

### *3.3 Antioxidant Activity Analysis*

The technique of 2-Diphenyl-1-Picryhydrzyl (DPPH) had been used to identify the radical scavenging activity in each of the samples of xanthone. Physically, the purple colour of 2-Diphenyl-1- 9 (DPPH) solutions was changed to yellow colour as the reaction occurred due to the Picrylhydrazyl samples consist of antioxidant. Fig. 2 shows the radical scavenging activity (antioxidant activity) for samples before and after freeze drying.

The radical scavenging activity for sample before freeze-drying were in the range of 47.93% to 82.69% as shown in Fig. 2. The highest value of radical scavenging activity is 82.69% at temperature 90 °C and 4 hours of extraction process (sample 4). For the sample after freeze-drying, the percentage of radical scavenging activity were in the range of 77.47% to 88.01%. The highest value of radical scavenging activity was 88.01% at temperature 80 °C and 6 hours of extraction process (sample 3).

Proportionally, highest percentage of radical scavenging activity exhibited higher antioxidant which can inhibit the free radicle activities. Thus, from the results obtained, the sample after freeze drying had higher antioxidant activity as the value of radical scavenging activity was higher compared to the sample before freeze drying. The content of polyphenol in *Garcinia mangostana* pericarp known as xanthone was not degraded during the freeze-drying process and still retained the even higher compared to the content before freeze-drying.

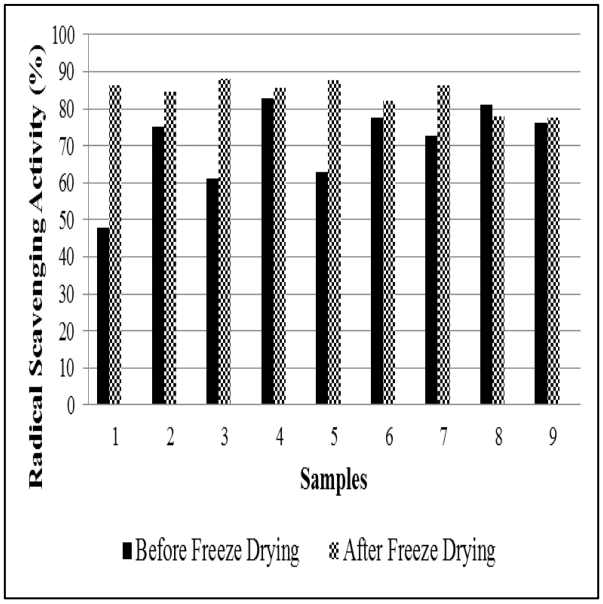


**Fig. 2**: Radical scavenging activity for samples before and after freeze drying.

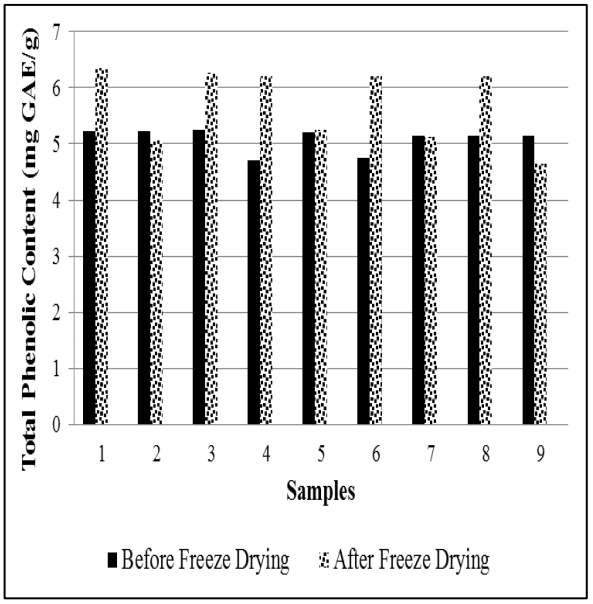


**Fig. 3**: Total phenolic content for samples before and after freeze drying.

### *3.4 Total Phenolic Content Analysis*

The samples were mixed with Folin-Ciocalteu reagent and the colour was changed from yellow colour to purple colour. The changes of colour proved that the samples consist of total phenolic content which beneficial to avoid the free radical activities. Gallic acid was used as a standard to perform the standard calibration curve. The total phenolic content is expressed as milligram gallic acid equivalents per gram of pericarp powder (mg GAE/g).

The amount of total phenolic content (TPC) for sample before freeze-drying were obtained in the range of 4.71 mg GAE/g to 5.24 mg GAE/g as shown in Figure 3. The highest amount of total phenolic content (TPC) was 5.24 mg GAE/g at temperature 80 °C and 6 hours of extraction process (sample 3). Nevertheless, the amount of total phenolic content (TPC) for sample after freeze-drying was between 4.65 mg GAE/g to 6.36 mg GAE/g. The highest amount of total phenolic content (TPC) was 6.36 mg GAE/g at temperature 80 °C in 4 hours of extraction process (sample 1). The sample after freeze-drying showed higher amount of total phenolic content compared to the sample before freeze-drying.

## 4.0 Conclusion

The extraction of xanthone from *Garcinia mangostana* pericarp proves that it consists of high antioxidant. The sample that obtained from freeze drying in form of powder has higher antioxidant activity as the value of radical scavenging activity increased. Total phenolic content (TPC) was higher in the sample after freeze drying rather than the sample obtained before freeze drying. The samples that has been analysed were detected in the range of 515.0 to 4000 $cm^{-1}$ which consists of strong C–O stretch carboxylic acid functional group that commonly recognized at the peak of 1700 to 1725 $cm^{-1}$ and 1700 to 1725 $cm^{-1}$ before and after freeze drying, respectively. Most of the samples reached the transmittance at the range between 96%T to 98%T before freeze-drying and 130%T to 150%T after freeze drying, which was highly corresponding to the wavelength. The highest value of radical scavenging activity for non-encapsulated xanthones (before freeze drying) was 82.69% whereas 88.01% for encapsulated xanthones (after freeze drying). The total phenolic content (TPC) determined were 5.240 mg GAE/g and 6.359 mg GAE/g for non-encapsulated and encapsulated samples, respectively.

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