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Wall Material Formulations for *Zingiberaceae family* Rhizome Extracts *(Curcuma longa, Curcuma xanthorriza* and *Zingerol officinale)* on Microencapsulation Efficiency of Spray Drying Processing

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Abstract: Research has found that *Curcuma sp.* (*Curcuma xanthorrhiza (CX), Curcuma longa* (CL) and *Zingerol officinale* (ZO)) contain bioactive compounds that possess antioxidant, antimicrobial and anti-inflammatory properties. The advancement to bring out bioactive compounds from traditional medicine into modern medicine have investigated. The process used to encapsulate the extracted bioactive compounds is spray drying. This study proposes formulating different wall materials such as gum arabic (GA), xanthan gum (XG) and maltodextrin (MD) with different combinations to determine the properties of each wall material and how it affected the microcapsules (product). The spray-dried microencapsulated product was evaluated in terms of its yield analysis, size distribution, moisture content, color, functional group and the efficiency of microencapsulation (MEE%). Results indicated in terms of product yield, the current processing method only yields between 20 - 26% of encapsulated products, with moisture contents between 83.8 - 88.4%. The size of microcapsules is distributed in the span of 1.93 - 28.4 and the microencapsulation efficiency (MEE%) between 33.3 - 96.3%. Overall results showed that the best wall material combination for each rhizome extract of *C. xanthorrhiza*, *C. longa* and *Z. officinale* is XG:MD (83% MEE), GA:MD (96% MEE) and GA:MD (65% MEE) respectively. The results indicated the possibility of producing microencapsulated extracts from plants material for incorporation into beneficial products.

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Keywords: *Zingiberaceae family,* Spray drying, gum Arabic, xanthan gum, Maltodextrin

1. Introduction

The knowledge of using plants as medicine has passed down from generation to generation particularly for treatment by traditional healers in the community. Medicinal plants are based on knowledge, approaches, health practices, diagnose and prevent illnesses or maintain well-being. However, in the last decade, medicinal plants are becoming popular worldwide due to public interest and a tremendous surge in acceptance of natural therapies for developed countries [1]. In fact, many new active compounds have been found in medicinal plants. Medicinal plants have stated to be the sources of that bioactive compounds and have been applied in modern medicines. One of the medicinal plants is *Zingiberaceae family.* A *Zingiberaceae family's* member that consists of about 120 species Curcuma have been found in South Asia, Southeast Asia and China. For example, *Curcuma Longa, Curcuma Xanthorrhiza*, *Curcuma amada* and *Zingerol officinale*. A few *Zingiberaceae family* also found in southern Pacific region and Australia [2].

Zingiberaceae family have been well studied and these species promotes beneficial pharmacological properties including anti-inflammatory, anticancer, hypotensive and antimicrobial [3]. Every medicinal plant will have their bioactive compound produce certain physiological effects. In *Zingiberaceae family*, there are two major bioactive compounds which are curcuminoids and sesquiterpenoids that able to treat several diseases. The other bioactive compounds such as demethoxycurcumin, zederone, germacrone and others. The bioactive compounds from *Zingiberaceae family* have been highlighted in pharmaceutical field as it was found with many benefits. However, derivation of bioactive compound are facing few challenges in biomedical field due to its characteristics such as limitation of solubility and stability which are affected by the temperature, pH and additives presence [4].

The application of technology of plant-derived bioactive compounds in the system of delivery has to be innovated in order to overcome the challenges. The found technology which is microencapsulation has been applied in related fields as well as in agricultural, food industries and pharmaceutical. Particularly, in few cases, microencapsulation has been applied in encapsulation of colourings, flavouring, essential oils and microorganisms. In the pharmaceutical field, microencapsulation provides necessary protection for any active substances that present in medicinal plants. At the end of the microencapsulating process, there is a small particle formed that is called microcapsules [5]. The final microcapsules can be stored by a shell, coated or surrounded with polymeric material to produce particles in the range of micrometer to millimeter for protection [6].

The factor that influenced the microencapsulation technique is the polymeric or wall material formulation. The choice of wall material will be discussed as it is the main aspect in this research which encapsulates the extracted bioactive compounds from different species of *Zingiberaceae family.* The characteristics such as physical, structural and chemical are analyzed from the resulted microcapsules. Besides, the wall materials must have properties like film-forming, moderate viscosity and excellent emulsifying properties [7]. In this project, the different wall materials will be used such as gum Arabic, xanthan gum and maltodextrin. The wall materials are combining with two different types by ratio that have been formulating which is 1:1. The purpose of using different types of wall materials is to meet an ideal formulation to obtain the best physically and chemically microcapsules characteristics for different extracted

rhizome of *Zingiberaceae family*.

There are chemical and physical methods for microencapsulation. For example, polymerization, solvent evaporation and air suspension coating, freeze drying and spray drying. This project chooses spray drying to encapsulate the extracted bioactive compound. Spray drying is the technology to produce dried microcapsules from any oil extracted plant mixing with wall materials. The spray drying process is stating as the best process among others due to its benefit which provides fast water evaporation and is able to maintain low temperature in the particles. In addition, spray drying is a common technique in microencapsulation because of its high cost-efficiency, flexibility and continuous production [9].

2. Materials and Methods

The methodology that is used to achieve the objectives of this study. The flowchart is illustrated in Figure 1.

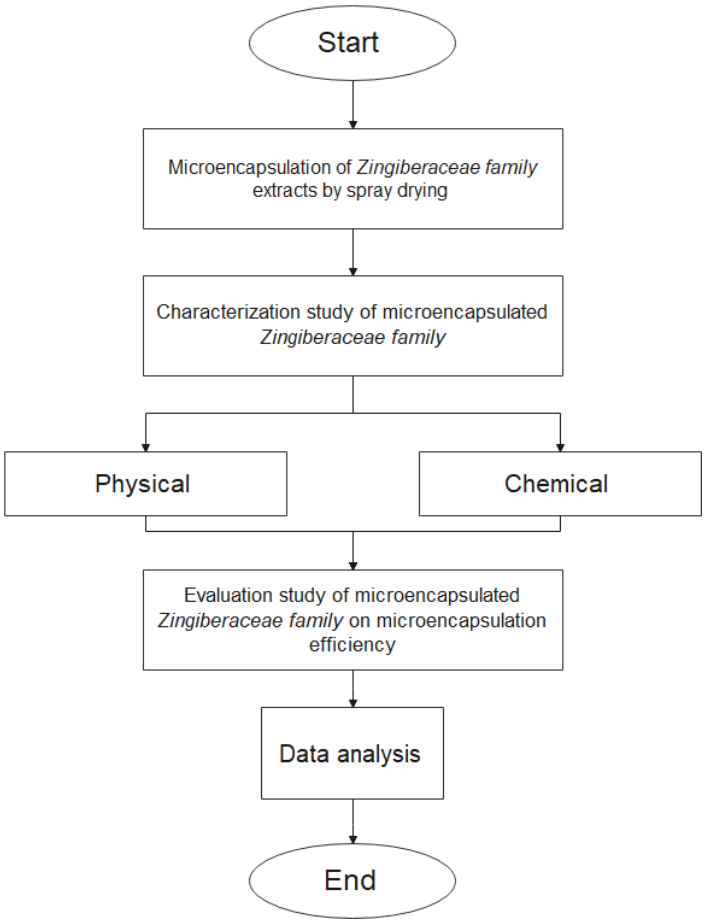

Figure 1: Flow chart of the project

2.1 Experimental procedure

There are six runnings with three types of rhizome extracts and different wall material formulations. By particular volume where the homogenized solution contained 800 ml (8 g of wall material and 8 ml of sample) and the remaining is distilled water. All running has been encapsulated by spray drying where the value of flow rate and temperature are 5 rpm and 180 °C respectively. Table below shows the different wall material formulations for different extracts.

Table 1: Wall material formulation in each running

Running	*Zingiberaceae Rhizomes* (8 ml)	Gum arabic (GA) (g)	Xanthan gum (XG) (g)	Maltodextrin (MD) (g)
R1: CX+GA:MD	*Curcuma xanthorrhiza (CX)*	4	0	4
R2: CX+XG:MD		0	4	4
R3: CL+GA:MD	*Curcuma longa (CL)*	4	0	4
R4: CL+XG:MD		0	4	4
R5: ZO+GA:MD	*Zingerol officinale (ZO)*	4	0	4
R6: ZO+XG:MD		0	4	4

2.2 Yield

The microcapsules of every run will be analyzed in terms of yield. The yield was determined by Equation 1 below.

$$\text{Product yield} = \frac{Mass\ of\ powder\ obtained\ at\ the\ spray\ dried}{Amount\ of\ solid\ raw\ materials\ used\ in\ iniatial\ feed\ solution} \times 100 \qquad \text{Eqn. 1}$$

2.3 Size analysis

Particle size distributions have been analyzed by using particle size analyzer. The obscuration display for the analyser is in range 0.01 to 0.15. The span of a volume-based size distribution is defined as in Eqn. 2 and gives an indication of how far the 10 percent and 90 percent points are apart, normalized with the midpoint (Liang. T, 2020) as shown in Eqn. 2.

$$\text{Span} = \frac{D_{90}-D_{10}}{D_{50}} \qquad \text{Eqn. 2}$$

2.4 Color analysis

Color analysis has been observed by using Hunterlab D25 Colorimeter. The color analysis will be categorized below: [10].

Table 2: Color analysis

Category	Color	Details
*L**	Lightness	Range from 0 (black) to 100 (white)
*a**	Redness and greenness	Determine redness with "+" while for greenness "-"
*b**	Yellowness and blueness	Determine yellowness with "+" and for blueness "-"

2.5 Moisture content

The moisture content was determined based on the Association of Analytical Communities (AOAC) method. 0.5 g of microcapsules were weighed and then dried in a hot air oven at 105 °C. The drying was continued until a constant weight was obtained and moisture loss was express in terms of percent dry basis. The weight measurement has been checked for every one hour [11].

$$\text{Percent dry} = \frac{C-A}{B-A} \times 100 \qquad \text{Eqn. 3}$$

Where, A is the weight of empty aluminium foil cup (g), B is the weight of aluminium foil cup + fresh microcapsules (g) and C is the weight of aluminium foil cup + dried microcapsules (g)

2.6 Functional group

2 mg of microcapsules of the *Zingiberaceae family* was mixed with 200 mg of potassium bromide. They were mixed by using mortar and pestle until homogenized. The functional group from the sample was identified by fourier-transform infrared spectroscopy (FTIR). This step was repeated for other combinations.

2.7 Determination of microencapsulation efficiency (MEE)

The determination of microencapsulation efficiency (MEE) as the ratio of encapsulated curcumin to the amount of curcumin in the microcapsules based on the equation below.

$$\text{Encapsulation efficiency} = \frac{(Total\ of\ curcumin - Surface\ of\ curcumin}{Total\ of\ curcumin} \times 100 \quad \text{Eqn. 4}$$

3. Results and Discussion

3.1 Determination of suitable wall material formulation

Table 3 showed every running was categorized in particular physicochemical properties. For example, product yield, moisture content, color, size distribution and efficiency. In product yield, the highest value was R6 which is 26.00% followed by R1, R3, R5, R2 and R4. Combination gum arabic with maltodextrin showed higher yield percentage than xanthan gum with maltodextrin in *Curcuma xanthorrhiza* and *Curcuma longa* yet it different when ginger in combination of xanthan gum and maltodextrin. The highest yield that obtained from the species state that it has higher curcumin content. In this case, *Zingerol officinale* have possibility to have higher yield stability and higher curcumin content than *Curcuma xanthorrhiza* and *Curcuma longa* due to its growth on productive environment [12]. Secondly, moisture content. The maximum value was obtained by R5 which is 88.4% and the lowest value was R1 which is 83.8%. Based on other research, curcumin microcapsules with wall material composition have moisture content ranging in 3% to 10% show good storage stability [13]. However, in this experiment, all running showed the high moisture content which above 50%. Moisture content on microcapsules are depending on the uses of wall

material. The different wall materials have different properties like its capability to produce the better dried microcapsules.

For color analysis, the aim was about to explain the reflection in lightness and saturation degree. All curcumin microcapsules displayed medium lightness with L^*ranging from 57.55 to 69.14. The high lightness was R3 which *Curcuma longa* with gum arabic and maltodextrin formulation while the lowest was R6 which *Zingerol officinale* with xanthan gum and maltodextrin. All b^* readings showed positive values that indicated a slightly medium tendency to yellow where the highest was R2. In comparison, most of the a^* values presented positive values which indicated small tendency to redness where R2 and R4 showed greenness tendency. For color analysis, b^* value is a crucial important indicator for the quality of curcumin microcapsules. The highest b^* value is R2 which *Curcuma Xanthorrhiza* with xanthan gum and maltodextrin formulation. It has possibility in R2, there were more bioactive compound trapped in the coating materials which influenced by wall material types that able in coating curcumin into microcapsules [10].

The span value of size analysis is ranging from 1.93 to 28.43. The maximum value was 28.43 which come from R3 whereas the minimum value was 1.93 which is R1. There is a relationship in size distribution for microcapsules which when the span value is closer to 0, the size consistency is better and the granularity is more uniform [14]. Based from the table 3 below, the smallest span is R1 followed by R6, R5, R4, R3 and R2. This is showing that R1 which *Curcuma xanthorrhiza* with gum arabic and maltodextrin formulation have better size consistency.

Other than that, there are several factors that affected size distribution on microcapsules including matrix type, ratio, concentration and core ingredient types [15]. In this research, the wall material formulation was studied. However, the result showed that the wall material formulation is totally influence the size distribution. Particle size distribution concerned about the molecular weight of wall material used. Difference molecular weight will contribute in different particle size which increasing molecular weight will increase the particle size distribution according to the D(90) value from the table. The molecular weight of gum arabic is 240 to 580 kDa [16] while xanthan gum is more than 2000 kDa [17]. Thus, the D(90) value of xanthan gum used in R2, R4 and R6 are bigger than gum arabic value. Therefore, gum arabic is good in contributing small particle size for curcumin microcapsules.

Table 3 below showed the percentage of encapsulation efficiency of every curcumin microcapsules that belong to each running respectively. The efficiency is ranging from 33.33% to 96.30%. The highest value was reached 96.30% which R3 (*Curcuma longa* with gum arabic and maltodextrin) and the lowest value was 33.33% which R1 (*Curcuma xanthorrhiza* with gum arabic and maltodextrin). The efficiency percentage was depending on curcumin total and surface curcumin in microcapsules itself.

The measurement of encapsulation efficiency is to observe and analyse the ability of wall material formulation in trapping curcumin into microcapsules. The different value of efficiency percentage explained that type of wall materials used need to be considered first before undergo microencapsulation process. In this experiment, the wall material used that reach the highest percentage is gum arabic. It can be stated that gum arabic able to capsulate the bioactive compound in particularly *Curcuma longa*. According to the other research, gum arabic give high efficiency that range from 93.8% to 97.6% [18] due its property which is low viscosity. The low viscosity will contribute in efficiency. This can be concluded the properties of gum arabic able to be good in encapsulating agent.

Table 3: The summary of physicochemical properties of spray-dried for every wall material combinations

Running Combination	Product yield (%)	Moisture content (%)	Color			Size distribution Span	Microencapsulation Efficiency (MEE%)
			*L**	*b**	*a**		
R1: CX+GA:MD	25.96	83.8	58.67	1.78	0.74	1.93	33.33
R2: CX+XG:MD	20.99	85.8	68.59	7.82	-0.14	95.5	83.33
R3: CL+GA:MD	24.88	86.0	69.14	4.16	0.58	28.43	96.30
R4: CL+XG:MD	20.00	87.4	60.83	6.81	-0.56	4.36	36.62
R5: ZO+GA:MD	23.42	88.4	61.43	1.44	0.42	3.42	65.22
R6: ZO+XG:MD	26.00	87.6	57.55	2.63	0.34	2.40	48.48

3.2 Identification of functional group

There are many types of functional groups in that family including phenol, ether and ketone. In this research, the functional group that be highlighted is phenol. Phenol is an organic compound that composed of phenyl group bonded with hydroxyl group. The graph shown in figure 1 below is one of the functional groups of curcumin for R1 (*Curcuma xanthorrhiza*+ GA:MD) that showed few peaks related. The graph for other wall material combinations were shown in appendices attached.

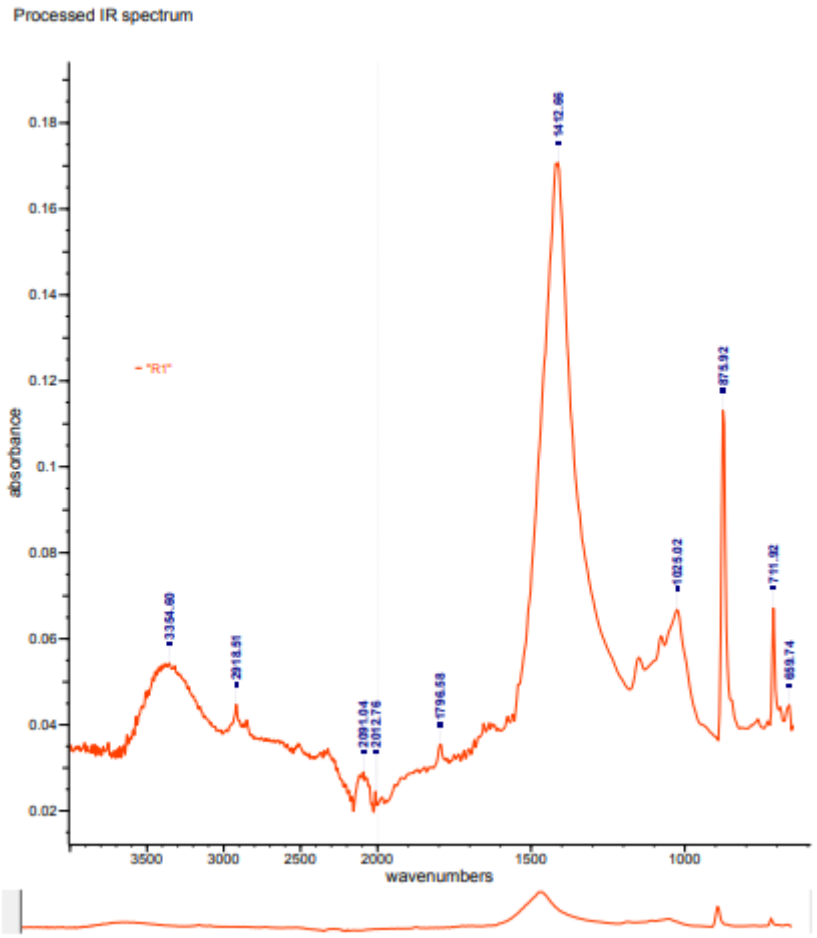

Figure 1: Peak analysis of functional group in R1

Table 4: Functional group and range value in each wall material combinations

Wall material combinations	Classification	Functional group	Range
R1: CX+GA:MD	Alcohol	(R)2CH-OH	3200-3400
R2: CX+XG:MD	Alcohol	(R)2CH-OH	3200-3400
R3: CL+GA:MD	Alcohol	R-CH2-OH	3200-3400
R4: CL+XG:MD	Alcohol	(R)2CH-OH	3200-3400
R5: ZO+GA:MD	Alcohol	R-CH2-OH	3200-3400
R6: ZO+XG:MD	Alcohol	(R)2CH-OH	3200-3400

Figure 1 stand for few peaks of functional group in R1 (CX+GA:MD. The highest peak was known as alcohol classification where - OH group has been identified. The range value was 3200 to 3400. Table 4 represented the classification, functional group and range value for each wall material combinations of microcapsules. From all the graphs that attach on appendices, most classification of functional group in every wall material combinations was alcohol which -OH group. It was same with R1, the ranging value was from 3200 to 3400. This value is highest than other functional group range.

4. Conclusion

In conclusion, microencapsulation of spray-dried extracts from *Curcuma xanthorrhiza (CX), Curcuma longa* (CL) and *Zingerol officinale* (ZO) using two different wall materials formulations has been successfully conducted. Results indicated in terms of product yield, the current processing method only yields between 20 - 26% of encapsulated products, with moisture contents between 83.8 - 88.4%. The size of microcapsules are distributed in the span of 1.93 - 28.4 and the microencapsulation efficiency (MEE%) between 33.3 - 96.3%. Besides, all samples are having same functional group of alcohol. Overall results showed that the best wall material combination for each rhizome extract of *C. xanthorrhiza*, *C. longa* and *Z. officinale* is XD:MD (83% MEE), GA:MD (96% MEE) and GA:MD (65% MEE) respectively. The results indicated the possibility of producing microencapsulated extracts from rhizome plant materials for incorporation into beneficial products.

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