

# Effect of different composition and emulsifier types on the stability of red palm oil emulsified powder

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## Abstract

Red palm oil (RPO) has high nutritional content such carotene and vitamin E. However, those components in RPO are unstable and sensitive when exposed to light or temperature. Thus, the sensitive and unstable of nature components of red palm oil highlights the importance of encapsulation process via emulsification in early stage. The objective of this study was to investigate the stability of  $\beta$ -carotene in RPO encapsulated powder using  $\alpha$ -cyclodextrin as wall material with the comparison of the emulsifier used between sodium caseinate (NaCAS) and lecithin in terms of production of RPO encapsulated powder. The protective effects provided by encapsulated powder process were evaluated in terms of the degradation kinetics of  $\beta$ -carotene at 60 °C for 25 days. Stability of  $\beta$ -carotene in RPO emulsified powder was fitted well using a first kinetic model. Degradation of  $\beta$ -carotene in encapsulated powder could be correlated using the first kinetic model. The stability of  $\beta$ -carotene with lecithin as emulsifier in both solid contents (25 wt.% and 35 wt.%) were more stable with degradation rate constant 0.018 day<sup>-1</sup> and 0.016 day<sup>-1</sup>, respectively. Encapsulation process protected RPO during storage, with lecithin emulsifier performing better than NaCAS emulsifier.

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## 1.0 Introduction

Red palm oil (RPO) is the olein fraction of crude palm oil (CPO) refining which has beneficial for maintaining the immune system and can prevent or delay several chronic degenerative diseases (Dauqan et al., 2011; Rao & Rao, 2007). It has also high nutritional content such carotene with a concentration of 500-700 ppm, vitamin E in the form of tocopherol, and tocotrienol with a concentration of 500-1000 ppm. However, those components in RPO are unstable and sensitive to oxidation when exposed to light or temperature (Fuchs et al., 2006). This degradation of active components causes the nutrients in RPO to be lost. In addition, RPO can become rancid when oxidative degradation occurs (Lee et al., 2018).

The sensitive and unstable of nature components of red palm oil highlights the importance of encapsulation process via emulsification in early stage. The encapsulation process is one of the alternatives that can be used to improve the handling and flow properties of RPO, as well as increase stability against heat, oxygen,

moisture, and facility easy transport (Nayana et al., 2021).

Extensive reviews of vegetable oil encapsulation and their stability have been published by several researchers (Lee et al., 2018; Lee et al., 2020; Sathasivam et al., 2018; Chew et al., 2018; Basu & Vecchio, 2001; Edris et al., 2016; Bordón et al., 2021; Kulandasamy et al., 2023; Pham et al., 2020), who explained different techniques of encapsulation such as supercritical carbon dioxide, extrusion, freeze drying, and spray drying. Among the various encapsulation techniques, spray drying is the most widely used technology in the food industry to produce powders at an economical cost (Edris et al., 2016), continuous processing, flexible, produce powders with good quality (Mohammed et al., 2020), can be used with various types of wall materials, has good volatile matter retention, and good product stability (Obón et al., 2009). Therefore, converting oil to powder can be consumer-friendly and widen their application in the culinary, health, food, and nutraceutical sectors (Nayana et al., 2021).

Furthermore, one of the factors to be considered in the encapsulation process is the coating material. This determines the degree of core protection, particle stability, and efficiency of encapsulated powder (Capelezzo et al., 2018). Several materials have been used as carrier material in encapsulation process, including carboxymethyl sago cellulose (CMSC), maltodextrin, gum arabic, and cyclodextrin. In recent years, cyclodextrins have been getting great interest in encapsulation research due to their multi-dimensional applications in various sectors (Narayanasamy et al., 2014). Cyclodextrins have been applied to encapsulate various of bioactive components in vegetable oil, including canola oil (Basu & Vecchio, 2001), essential oil (Capelezzo et al., 2018), kenaf seed oil (Chew et al., 2018), garlic oil (Piletti et al., 2019), cinnamon oil (Simionato et al., 2019), and coriander essential oil (Silva et al., 2019). Those reported that cyclodextrin successfully improve the oxidative stability of vegetable oil. Furthermore, cyclodextrin has also received much attention due to its non-toxicity, biodegradability, biocompatibility, and ability to form inclusion complexes with several compounds (Hedges, 1998). The commonly used cyclodextrins in encapsulation process are  $\alpha$ -,  $\beta$ -, and  $\gamma$ -cyclodextrin. The Joint FAO/WHO Expert Committee on Food Additives recommends the maximum use of  $\beta$ -cyclodextrin is 5 mg/kg per day in food, while for  $\alpha$ - and  $\gamma$ -cyclodextrins it has not been determined because of their innocuous profiles (Astray et al., 2009). The  $\alpha$ -Cyclodextrin ( $\alpha$ -CD) can be complex more easily than  $\beta$ - and  $\gamma$ -cyclodextrin with guest molecules having five carbons or less, because they have the smallest dimension of cyclodextrin (Hedges, 1998).

Yu et al. (2019) studied the encapsulation of vegetable oil using cyclodextrin as a wall material. They compared different types of cyclodextrin ( $\alpha$ -,  $\beta$ -, and  $\gamma$ -) to encapsulate garlic oil. One of the important objectives of encapsulation process is to control the release of core material. For instance, cyclodextrin complexes exhibited a slow-release rate with citral release being consistent (Phunpee et al., 2017). Chew et al. (2018), compared three effects of different wall materials (gum arabic,  $\beta$ -cyclodextrin, sodium caseinate) on the characteristics of the spray-dried and indicated that kenaf seed oil was well encapsulated by three wall materials.  $\alpha$ -Cyclodextrin had been successfully used to encapsulate tomato oil to slow down carotenoid degradation (Durante et al., 2020).

It is interesting to use  $\alpha$ -cyclodextrin to encapsulate RPO which has high nutritional carotene in a powder form for functional food application. Among the few published research, no other previous research was done to investigate RPO encapsulated powder using  $\alpha$ -cyclodextrin by spray drying. Therefore, the objective of this study was to investigate the stability of  $\beta$ -carotene in RPO encapsulated powder using  $\alpha$ -cyclodextrin as wall material. The comparison of the emulsifier used between sodium caseinate (NaCAS) and lecithin in terms of production of RPO encapsulated powder was also studied.

## 2.0 Materials and methods

### 2.1 Materials

Red Palm Oil as the main ingredient was obtained from SME Bumirahayu Lampung, Indonesia. Sodium caseinate was obtained from Thong Sheng Food Technology Sdn Bhd, Malaysia. Lecithin was obtained from PT Multi Kimia Raya Nusantara Semarang, Indonesia.  $\alpha$ -Cyclodextrin ( $\alpha$ -CD) was obtained from Cyclochem Co.Ltd, Japan. Hexane and distilled water were obtained from CV. Kimia Indrasari Semarang, Indonesia.

### 2.2 Methods

#### 2.2.1 Preparation of RPO feed emulsions

A wall material solution was prepared by dissolving  $\alpha$ -CD in distilled water at 40 °C and cooling to room temperature. Two emulsifier solutions (NaCAS and lecithin) were prepared by dissolving in distilled water at 45 °C with constant shaking for approximately 30 min. The RPO feed emulsions were prepared with two different solid contents (25 wt.% and 35 w.t%). The red palm oil was blended with the wall solution and emulsifier emulsion to give each solid content composition. The solution was mixed using an Ultra Turrax T25 mechanical homogenizer (Janke & Kunkel, GmbH, Staufen, Germany) operated at 18,000 rpm for 5 min. The experiment design is shown in Table 1.

#### 2.2.2 Measurement of oil droplet size

Measurement of oil droplet size was determined using microscopic method, following the method reported by (Williams et al., 2023). RPO feed emulsions were captured visually in a high-resolution

**Table 1:** Experiment design for the emulsion

Formulation	Solid content (wt.%)	Red palm oil (wt.%)	$\alpha$ -Cyclodextrin (wt.%)	Sodium caseinate (wt.%)	Lecithin (wt.%)	Distilled water (wt.%)
A	25	10.11	12.01	2.88	-	75
B	35	10.11	12.01	4.03	-	75
C	25	14.15	16.82	-	2.88	65
D	35	14.15	16.82	-	4.03	65

optical microscope with 100x magnification. Then the picture of emulsion was further analysed using open source of ImageJ tool 1.51j8 version to obtain oil droplet size distribution.

### 2.2.3 pH of RPO feed emulsions

The pH of RPO feed emulsion was determined using seven compact Metler Toledo pH meter (Mettler-Toledo AG, Grifensee, Switzerland). Initially the electrode was calibrated then dipped into the prepared emulsion. The pH value appeared and automatically recorded.

### 2.2.4 Viscosity measurements

The apparent viscosity of RPO feed emulsion was measured at 50 °C using Brookfield DV-I rheometer (Brookfield Engineering Laboratories, Inc., MA, USA) with a speed of 30 rpm and a spindle of 62. The viscosity analysis method was conducted following the method described by Kędzia et al. (2018). A 100 mL of emulsion was put into the measuring cup of the viscosimeter then pushed the viscosity measurement button. Then the viscosity value will appear on the layer with the unit used is centipoise (cP).

### 2.2.5 Encapsulation of RPO feed emulsion

RPO feed emulsions were spray dried using a lab-scale of Buchi spray dryer B-290 (Büchi, Buchi Indonesia, Indonesia) which equipped with a centrifugal atomizer. The spray drying condition followed the method by Lee et al. (2018) with some modifications. The parameters used were as follows: inlet temperature, 165 °C; feed flow rate, 20 mL/min; aspirator rate, 100%; and compressed air flow rate, 40 m<sup>3</sup>/h. The outlet temperature was recorded around 100±3 °C, respectively. The powders were stored at 30 °C until use.

### 2.2.6 Determination of moisture content of spray-dried powder

Determination of moisture content followed the method by Chinta et al. (2009). The moisture content of each sample (1 g) was determined from its weight loss in an infrared moisture analyser (HC103, Mettler-Toledo AG, Grifensee, Switzerland) at 105 °C, until the weight was stable for 3 minutes.

### 2.2.7 Scanning electron microscopy (SEM)

The surface structure and morphology of RPO spray-dried powder was observed using SEM-EDX JEOL JSM-650LA scanning electron microscope (JEOL Co. Ltd., Akishima, Japan). The samples were placed on the SEM stubs using a double-sided adhesive tape (Nisshin EM Co., Tokyo, Japan). The specimen was subsequently coated with Pt-Pd, using a magnetron sputter coater (MSP-1S, Vacuum Device Inc., Tokyo, Japan). The coated sample was then observed at an electron accelerating voltage of 10 kV. The SEM images were taken following the procedure by (Sultana et al., 2018).

### 2.2.8 Concentration of $\beta$ -carotene in RPO spray-dried powder

Concentration of  $\beta$ -carotene in RPO spray-dried powder was analysed following the method reported by Sathasivam et al. (2018) and Nokkaew et al. (2019). In brief, 0.1 g of red palm oil powder was dissolved in 25 mL of hexane solution. Then the absorbance was measured using a visible light spectrophotometer Shimadzu UV-1780 (Shimadzu, Kyoto, Japan) at a wavelength of 446 nm with a cuvette (1 cm wide). Total  $\beta$ -carotene was calculated from Eq. (1) (Nokkaew et al., 2019).

$$\text{Carotene (ppm)} = \frac{383 \times A_{446} \times V}{W \times 100} \quad (1)$$

where  $A_{446}$  is absorbance at 446 nm; 383 is diffusion coefficient;  $V$  is volume of hexane (mL) and  $W$  is weight of RPO powder (g).

### 2.2.9 Stability of $\beta$ -carotene in RPO spray-dried powder

The stability characteristics of RPO spray-dried powder were investigated by degradation analysis of  $\beta$ -carotene during storage time at 60 °C for 25 days. The stability of  $\beta$ -carotene content during incubation time could be correlated with the first-order encapsulation equation as in Eq. (2) (Lee et al., 2020).

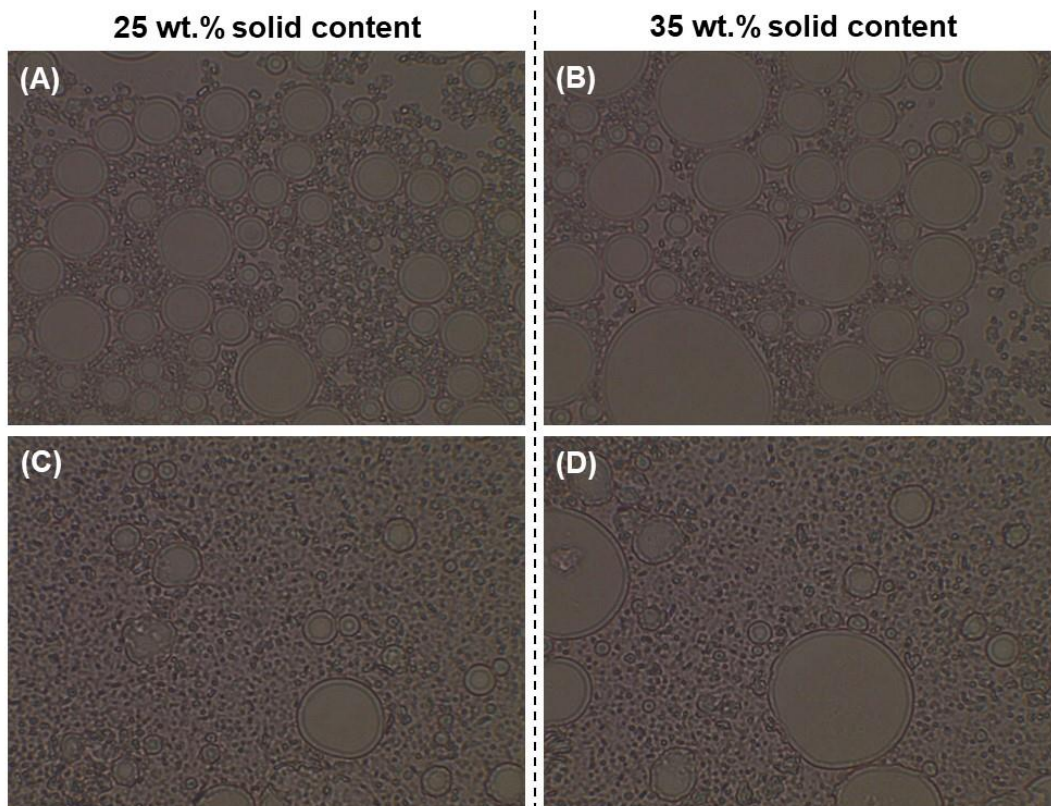
$$Y = Y_0 \exp(-kt) \quad (2)$$

where  $Y$  is concentration of  $\beta$ -carotene at incubation time,  $t$  (day);  $Y_0$  is initial concentration of  $\beta$ -carotene in the sample at day 0;  $k$  is degradation rate constant of  $\beta$ -carotene ( $\text{day}^{-1}$ ).

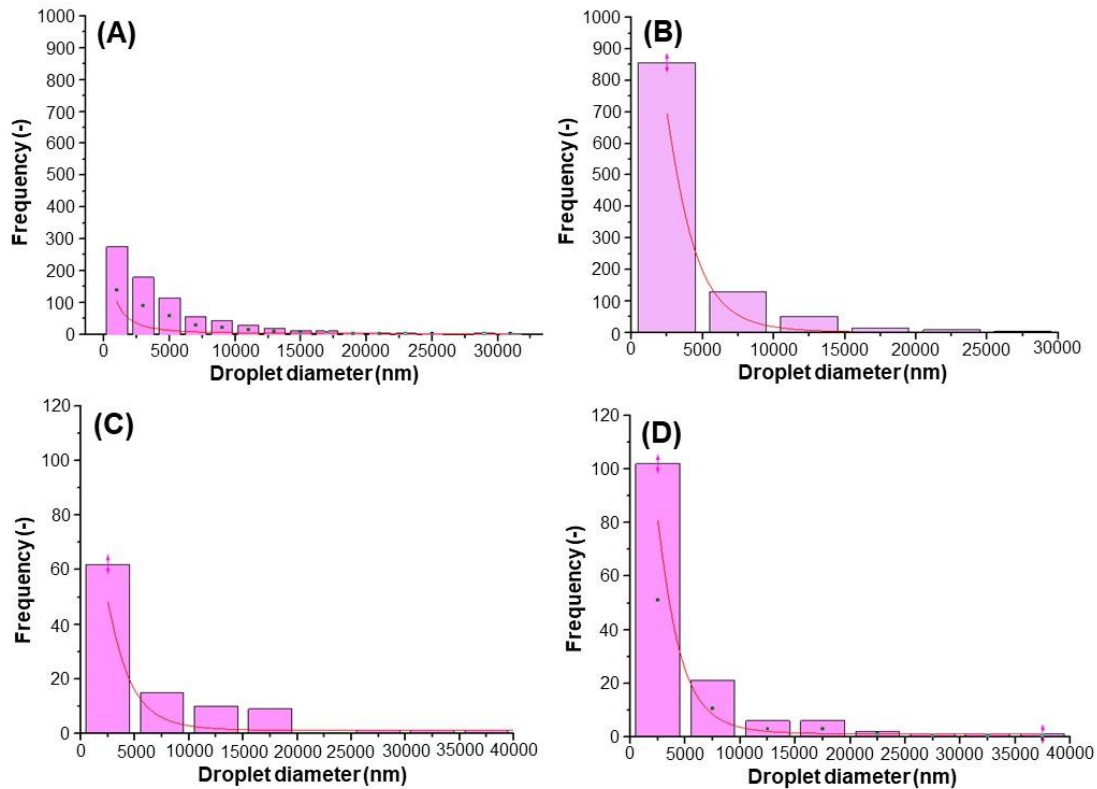
## 3.0 Results and discussion

### 3.1 Effect of solid content composition and emulsifier type on the characteristic of emulsion

Composition of solid content significantly affects the properties of the RPO emulsion. Fig. 1 shows the results of optical microscopic of oil-in-water (O/W) RPO emulsion with different solid contents of 25 wt.% and 35 wt.%. The 35 wt.% solid content produces a greater droplet diameter density compared to the 25 wt.% solid content. This result shows the amount of water added during the production of the RPO emulsification. The lower of solid content means more water ratio added to the emulsion system which affects the dispersion mechanism in the O/W emulsion. In addition, the different types of emulsifiers also affected the properties of the RPO emulsion. In this study, NaCAS and lecithin were used as emulsifiers. In the case of NaCAS, it produced more droplet diameter density than lecithin. NaCAS worked better than lecithin in forming a stable RPO emulsion.



**Fig. 1:** Optical microscopy of RPO feed emulsion with different solid content and emulsifier: (A) sodium caseinate (NaCAS) with 25 wt.% solid content; (B) sodium caseinate (NaCAS) with 35 wt.% solid content; (C) lecithin with 25 wt.% solid content; (D) lecithin with 35 wt.% solid content



**Fig. 2:** Droplet size distribution of RPO feed emulsion with different solid content and emulsifier: (A) sodium caseinate (NaCAS) with 25 wt.% solid content; (B) sodium caseinate (NaCAS) with 35 wt.% solid content; (C) lecithin with 25 wt.% solid content; (D) lecithin with 35 wt.% solid content

The use of NaCAS resulted in smaller emulsion droplet sizes with better emulsion stability during storage. Furthermore, the interaction of NaCAS with  $\alpha$ -CD containing phospholipids could control the protein-stabilized emulsion system.

Fig. 2 shows the result of droplet diameter measurements using the ImageJ and Origin software with different solid contents of 25 wt.% and 35 wt.%. The droplet size was ranges from 1000 to 30000 nm (1–30  $\mu$ m). At the same time, the addition of lecithin in the emulsion resulted the droplet size ranging from 1000 to 40000 nm (1–40  $\mu$ m). With the addition of NaCAS emulsifier and lecithin, the droplet size in the emulsion was range in the 0 to 5  $\mu$ m where the distribution frequency of droplet sizes was larger at solid content 35 wt.% than at solid content 25 wt.%. Furthermore, the addition of NaCAS emulsifier has a higher frequency of droplet size distribution than the addition of lecithin, since lecithin has a higher viscosity value than NaCAS, and the higher the viscosity value, the smaller the droplet size produced. In the emulsion system, increasing the volume percentage of the dispersed phase can alter droplet size, which can enhance emulsion viscosity (Yesiltas et al., 2019). In addition, emulsifiers could influence droplet size in

case of emulsifiers with high hydrophilic-lipophilic balance (HLB) values yield small particle sizes in oil-in-water emulsions. The HLB value of lecithin was 8, while the HLB value of NaCAS was 14. As a result, the emulsion with the lecithin emulsifier was greater than the emulsion with the NaCAS emulsifier (Gasa-Falcon et al., 2021). In addition, the droplet size on emulsion system greatly affected the characteristics and stability of the emulsion.

Chung et al. (2019), measured the droplet size in MCT oil emulsions with different solid content from 0.25% to 1.5 wt.% and different use of emulsifiers. They found that droplet size of emulsion was the same which ranging from 0.03 to 30  $\mu$ m. The used of NaCAS as emulsifier in MCT oil emulsion resulted small droplet size compared to other. These results indicated that NaCAS was a more effective emulsifier than lecithin when used in the same solid content. NaCAS had more stable protein adsorption at the oil-water interface compared to the lecithin. Williams et al. (2023), reported the similar results that the droplet size of mono-dispersion emulsions using the ImageJ application ranged from 80 to 120  $\mu$ m and reached the highest peak of 100  $\mu$ m. They also found that the distribution of droplet diameters in the mono-

dispersion emulsion changed during storage time and affected to the characteristics and the stability of the emulsion.

Navarrete de Toledo et al. (2022), stated that the addition of NaCAS as emulsifier in emulsion system generated a bimodal distribution with peaks ranging from 54.3 to 301.3 nm. These results were associated with casein fragments on the different size. Meanwhile, the lecithin solution also showed a bimodal distribution, with density peaks at 125 and 670 nm. These results showed that the droplet diameter with the addition of lecithin was bigger than NaCAS and influenced the decreasing of emulsion stability. Moreover, Whitehurst (2004) and Ho et al. (2022) pointed out that droplet diameter could influenced the aggregation of droplet and changed the solubility of emulsifier in aqueous solution.

Macroscopic analysis of droplet size in emulsion was aimed to determine the droplet size and its distribution in emulsion system. The larger droplet size affects the stability of emulsion. The increasing concentration of solid content influenced the formation of droplet density with the high possibility. However, the increasing of droplet density was also followed by the decreasing droplet diameter (Williams et al., 2023). The interaction of NaCAS with  $\alpha$ -CD in the RPO emulsion showed the stable emulsion in both solid contents, 25 wt.% and 35 wt.%. The protein bond between RPO and NaCAS helps protect the core materials with addition of  $\alpha$ -CD as wall material.

### 3.2 Effect of solid content composition and emulsifier type on the emulsion stability

#### 3.2.1 Viscosity of emulsion

Table 2 shows the viscosity of the emulsion carried out on day 0 and day 25 in the analysis. Viscosity in the emulsion system is related to the characteristics and stability of the emulsion, where the greater the viscosity of an emulsion, the higher the stability of the emulsion. Viscosity analysis is carried out to determine the thickness of an emulsion that is formed. The viscosity values shows that the higher of extract content strongly influenced the viscosity (Juszczak & Fortuna, 2003).

During the storage period at 37 °C, the overall viscosity of the red palm oil emulsion increased. The viscosity changes of the emulsion during the storage period indicated the internal changes in the emulsion. The results of the RPO emulsion viscosity test using  $\alpha$ -cyclodextrin with the addition of NaCAS and lecithin

experienced an increase in the viscosity value, it was seen from day 0 to day 25 that the resulting emulsion experienced thickness. An increase in viscosity indicates a thicker emulsion stability where the dispersed phase will find it difficult to move in the outer phase so that the chances of collisions between globules will be lower and result in the globules tending to coalesce into smaller particles.

It can be seen from day 0 that the 25 wt.% solid content with the addition of NaCAS had higher viscosity value compared to lecithin, while the 35 wt.% solid content with the addition of lecithin had higher viscosity value compared to NaCAS. At 25 days in 25 wt.% solid content with the addition of lecithin had higher viscosity value compared to NaCAS. Meanwhile, the 35 wt.% solid content added by NaCAS had higher viscosity value compared to lecithin. This should mean that the viscosity value of adding NaCAS is higher compared to lecithin even though the solid content has the same concentration because NaCAS has a larger molecular mass as an emulsifier to help solubility and dispersion (Zhao et al., 2008). Emulsion viscosity generally increases in the presence of flocculated fat lumps, which is due to the larger volume fraction (Danviriyakul et al., 2002).

For the viscosity value during storage increased according to previous research by Dabija et al. (2018), obtained viscosity values from days 1, 7, 14, 21, and 28 ranging from 210 to 900 cP where the higher concentration of solid content added will increase the viscosity of the emulsion so that it has high value of emulsion stability. In addition, research conducted by Gaygadzhev (2023), obtained viscosity values ranging from 28 to 191 cP where the higher presence of the addition of lecithin in the emulsion with the difference in the emulsion solution, which was not added with lecithin, the result was that the absence of the lecithin addition in the emulsion, the viscosity value was smaller than the addition of lecithin. This shows that the fat lumps in some emulsions as lumps of various sizes contribute to the increase in viscosity.

**Table 2:** Effect of different composition and emulsifier types on the viscosity of RPO feed emulsion

Type of emulsifier	Solid content	Viscosity of emulsion (cP)	
		0 day	25 days
Sodium caseinate	25 wt.%	12.00±0.25	149.67±123.28
	35 wt.%	14.00±1.55	5480.00±1474.45
Lecithin	25 wt.%	5.00±0.71	2626.67±363.50
	35 wt.%	28.00±7.54	4926.67±604.75

For the addition of NaCAS and lecithin emulsifiers to the emulsion, contrary to the study of Koo et al. (2019), it showed that the viscosity value of the emulsion with the addition of NaCAS was higher than emulsion with lecithin addition because the molecular mass of NaCAS was higher than lecithin and during storage the viscosity will increase. The viscosity value of the emulsion with a solid content of 25 wt.% and 35 wt.% and the addition of sodium caseinate and lecithin emulsifiers showed an increase in the viscosity value, this indicated that there had been a change in the emulsion. But it should be during storage with the same solid content and the addition of NaCAS emulsifier is higher than the addition of lecithin.

### 3.2.2 pH of emulsion

The results of pH analysis of the RPO emulsion at 25 wt.% and 35 wt.% solid content with the addition of NaCAS and lecithin, respectively, can be seen in Table 3. Based on the data in Table 2 from day 0 to day 25, each formula has a pH value not significantly different. In the emulsion with the addition of NaCAS, pH of 5.46-6.69 was obtained and with the addition of lecithin, pH of 4.16-5.52 was obtained. This result shows during the storage period the resulting emulsion is acidic over time. If the pH value of the emulsion is low, this can accelerate lipid oxidation so that the stability of the emulsion will decrease.

This is in accordance with previous studies by Yussof et al. (2023), with the same solid content but different additions of NaCAS and lecithin emulsifiers obtained the same pH value of 2-8, where during the emulsion storage period the pH value decreased and became acidic. In addition, research from Gaygadzhiev (2023), measured the pH value with different concentrations of emulsifier addition, where the obtained pH value increased the concentration of lecithin in the emulsion system with a pH of 7.0–7.5. This shows that during storage the resulting pH value is higher. The higher pH value, the higher viscosity value. The results of this study are also in accordance with the research by Koo et al. (2019), which showed that the pH value with the addition of NaCAS and lecithin resulted in an emulsion pH value ranging from

3.5 to 7.0 where during storage the pH value decreased and became acidic. Timm-Heinrich et al. (2004) stated that the speed of lipid oxidation is influenced by pH, a low pH value will accelerate the speed of lipid oxidation. The rapid oxidation of lipids is affected by temperature during emulsion storage. The difference in solid content and emulsifier resulted in decreasing pH value and became acidic during the storage period. The pH value with a solid content of 25 wt.% and 35 wt.% resulted in a pH value that was not significantly different from being acidic either with the addition of NaCAS and lecithin. This shows that the difference in solid content in the preparation of the emulsion does not affect the resulting pH value.

### 3.3 Effect of solid content composition and emulsifier type on the characteristic of RPO powder

#### 3.3.1 Moisture content of RPO powder

Table 4 shows the moisture content of RPO powder with differences in emulsifier and solid content. The emulsifier and solid content did not have a significant effect on the moisture content of RPO powder. Moisture content of RPO powder on a solid content of 25 wt.% using NaCAS and lecithin emulsifiers respectively obtained  $2.34\pm 0.36\%$  and  $2.02\pm 0.83\%$ . Meanwhile, the moisture content of RPO powder with a solid content of 35 wt.% using the NaCAS emulsifier was  $2.14\pm 0.34\%$ , which is higher than the lecithin emulsifier, which is  $1.86\pm 0.21\%$ .

The RPO powder with NaCAS as an emulsifier has a higher moisture content than lecithin as an emulsifier. This can be influenced by the difference in molecular weight of the two emulsifiers, where the molecular weight of NaCAS is greater than that of lecithin (Yussof et al., 2023). Therefore, lecithin with a low molecular weight encourages water molecules to diffuse outside the particle walls and evaporate easily during the drying process. The same results were obtained in a study conducted by (Ramakrishnan et al., 2018; Sarabandi et al., 2019), analysing the encapsulation of eggplant and tamarillo shell extracts using gum arabic and was higher than the moisture content using maltodextrin.

**Table 3:** Effect of different composition and emulsifier types on the pH of RPO feed emulsion

Type of emulsifier	Solid content	pH value						
		0 day	3 days	5 days	10 days	15 days	20 days	25 days
Sodium caseinate	25 wt.%	6.41±0.010	6.08±0.005	6.03±0.010	5.95±0.010	6.34±0.005	6.59±0.037	5.88±0.001
	35 wt.%	6.25±0.030	5.46±0.005	6.19±0.015	5.41±0.005	5.48±0.005	5.69±0.005	5.57±0.001
Lecithin	25 wt.%	5.52±0.030	5.17±0.025	5.15±0.001	4.55±0.010	4.43±0.005	4.41±0.010	4.16±0.020
	35 wt.%	5.52±0.005	5.17±0.010	5.15±0.001	4.55±0.001	4.43±0.001	4.41±0.005	4.16±0.005

This can be influenced by the greater molecular weight of gum arabic compared to maltodextrin, therefore the water molecules in the powder diffuse to the walls of the particles and evaporate easily. Apart from the emulsifier, the solid content also affects the moisture content of the RPO powder, where the RPO powder at a solid content of 25 wt.% has a higher moisture content than RPO with solid content of 35 wt.%. According to Frascareli et al. (2012), the more total solids contained in the emulsion, the higher the viscosity and the less available moisture content before the drying process.

This is in accordance with research conducted by Fernandes et al. (2008) regarding the microencapsulation of *lippia sidoides* essential oils, obtained reduced moisture content from 5% to 4% with an increase in solid content from 30 wt.% to 60 wt.%. Ng et al. (2014), regarding the microencapsulation of kenaf seed oil, the moisture content obtained at 20 wt.% solid content was 2.53 g water/100 g dry solid while at 40 wt.% solid content it was 0.92 g water/100 g dry solid.

### 3.3.2 Morphology characteristics of RPO powder

Fig. 3 shows the surface morphological structure of RPO powder with different emulsifiers and solid content. Differences in emulsifier and solid content do not have a significant effect on the morphology of the powder. In general, microcapsules have irregularly rounded, deflated and wrinkled shapes of various sizes. This size variation is a general characteristic of the powder produced through the spray drying process. There are no cracks in the microcapsules to prevent the permeability of the powder to gases and provide better protection and retention of the active ingredients (Carneiro et al., 2013). Moreover, the SEM results showed that that the differences in emulsifier and solid content produce different powder particle sizes. Powder particle size could be regulated by emulsion physical parameters such as viscosity and solids concentration (Jafari et al., 2008), as well as homogenization and spray drying conditions (Sultana et al., 2018). In this research, the operating conditions during homogenization and spray drying conditions were carried out with the same operating conditions but different in solid concentrations. These conditions influenced the increasing of emulsion viscosity. Moreover, the increasing viscosity of emulsion expanded the ability of emulsion droplets to coalesce during the atomization process in spray dryer.

**Table 4:** Effect of different composition and emulsifier types on the physical characteristics of RPO emulsified powder

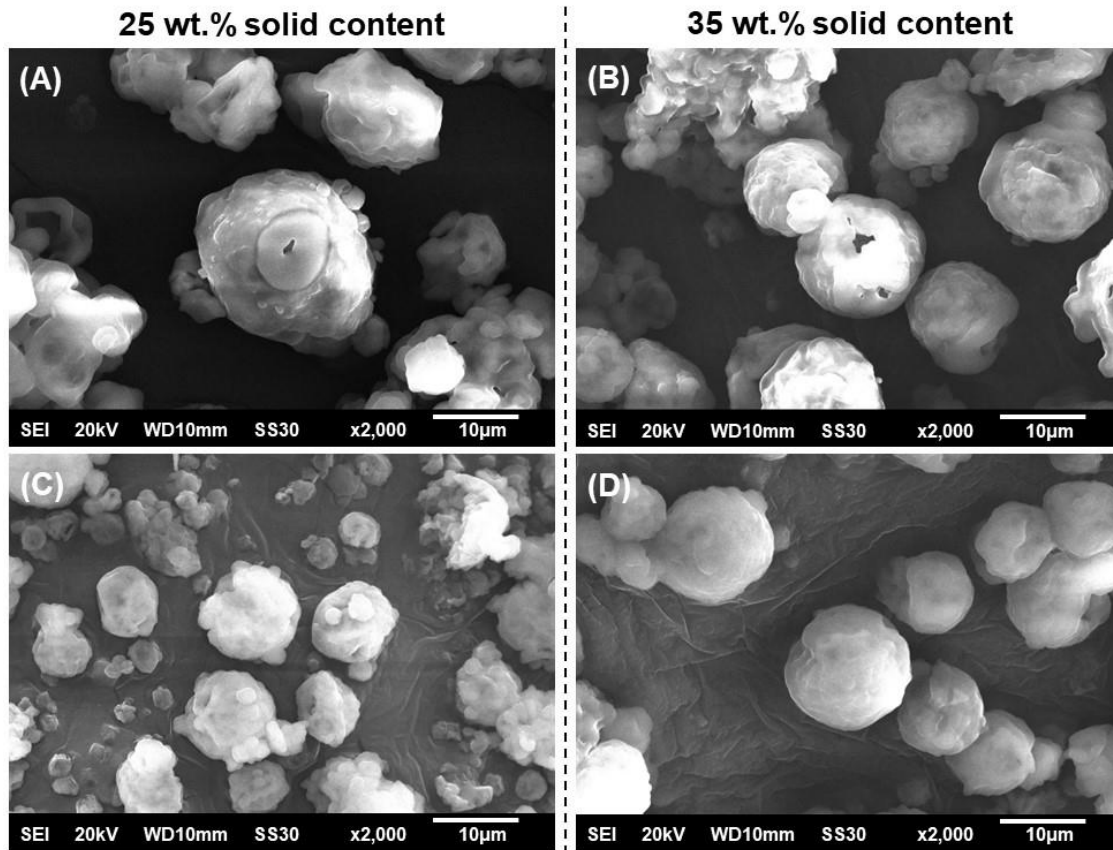
Type of emulsifier	Solid content	Moisture content (%)	$k$ ( $\text{day}^{-1}$ )	$R^2$
Sodium Caseinate	25 wt.%	2.34±0.36	0.056	0.966
Lecithin	35 wt.%	2.14±0.34	0.048	0.975
	25 wt.%	2.02±0.83	0.018	0.996
	35 wt.%	1.86±0.21	0.016	0.997

This phenomenon affected the increasing in droplet diameter of powder which due to the required of kinetic energy required to break down the size of droplet to overcome the internal forces during spray drying (Rahman et al., 2022).

The same morphological structure of powder was obtained in a previous study conducted by Lee et al. (2018), who analysed the morphology of RPO powder which was encapsulated using maltodextrin and sodium caseinate (NaCAS) and lecithin emulsifiers using the spray drying method, obtained the morphology of RPO powder which had an irregular spherical shape with a deflated and wrinkled surface. However, different results were obtained in a study conducted by Lee et al. (2018), using the supercritical carbon dioxide solution-enhanced dispersion (SEDS) drying method, a finer RPO powder surface morphology with a rounded shape was obtained compared to that produced using the spray drying method. The surface morphology of powders with the spray drying method generally experiences shrinkage on the surface of the powder wall due to the influence of the cooling process from high temperatures.

In addition, powder shrinkage can be caused by the flow rate of hot air during the spray drying process (Jafari et al., 2008). The high flow rate of hot air affects the formation of water vapor during the spray drying process. Drying conditions with high temperatures and incompatibility between the encapsulation material and the spray drying process can cause the capsule wall to rupture resulting in the loss of the active ingredient content and the capsule wall to shrink (Elversson & Millqvist-Fureby, 2005). Sathasivam et al. (2018), analysing the morphology of RPO powder encapsulated in carboxymethyl sago cellulose (CMSC) with and without using plasticizers (glycerine or polyethylene glycol) through the freeze-drying method, the morphology of RPO powder using plasticizers has an irregular spherical shape with a smoother surface and has better surface characteristics compared to RPO powder which does not use a





**Fig. 3:** SEM images of RPO encapsulated powder with different solid content and emulsifier: (A), sodium caseinate (NaCAS) with 25 wt.% solid content; (B), sodium caseinate (NaCAS) with 35 wt.% solid content; (C), lecithin with 25 wt.% solid content; (D), lecithin with 35 wt.% solid content

plasticizer with an irregularly spherical shape with a deflated and wrinkled surface. The use of plasticizers can maintain the morphology and suppress shrinkage of the powder.

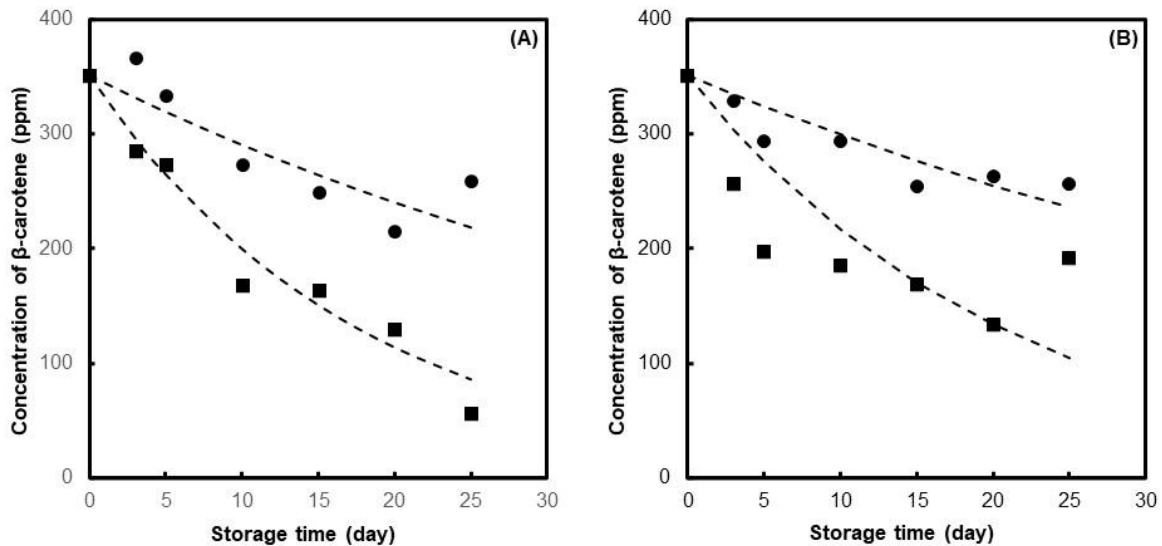
Chew et al. (2018), analysed the morphology of kenaf seed oil powder which was encapsulated with various formulas namely  $\beta$ -cyclodextrin/sodium caseinate,  $\beta$ -cyclodextrin/gum arabic and  $\beta$ -cyclodextrin/sodium caseinate/gum arabic, the results of the powder morphology analysis with the combination of ingredients used did not have a significant effect on the morphology of the powder surface, which generally has a spherical, wrinkled powder structure with various variations.

#### 3.4 Effect of solid content composition and emulsifier type on the stability of RPO powder

The degradation of RPO powder was analysed for 25 days at a storage temperature of 60 °C. Based on the data, in general the concentration of  $\beta$ -carotene significantly decreased with the length of storage time. The mechanism of carotene oxidation is the same as that of lipids (Hong et al., 2017), so that when exposed to light, heat, and oxygen carotenes will undergo

oxidation and isomerase processes which are the main causes of damage to carotenoids (Chen & Zhong, 2015). The degradation of  $\beta$ -carotene was analysed using first-order reaction kinetics. The kinetic parameter obtained from the first-order model which followed the research conducted by (Lee et al., 2020). Higher  $k$  values indicated high sensitivity to degradation.

Fig. 4 shows the degradation rate of  $\beta$ -carotene in RPO powder with the difference in emulsifier and solid content used. Based on these data, the value of  $k$  obtained at 25 wt.% solid content with NaCAS and lecithin emulsifiers was  $0.056 \text{ day}^{-1}$  and  $0.018 \text{ day}^{-1}$ . While the  $k$  values obtained at 35 wt.% solid content with NaCAS and lecithin emulsifiers were  $0.048 \text{ day}^{-1}$  and  $0.016 \text{ day}^{-1}$ . The low value of the degradation rate constant indicates that the degradation process is slow. Differences in emulsifiers have a significant influence on the degradation rate of  $\beta$ -carotene in RPO powder. The degradation rate of  $\beta$ -carotene using the lecithin emulsifier was lower than using the NaCAS emulsifier, either at a solid content of 25 wt.% or at a solid content of 35 wt.%.



**Fig. 4:** Effect of different solid content and emulsifier on the stability of  $\beta$ -carotene: (A) 25 wt.% solid content and (B) 35 wt.% solid content with (●) sodium caseinate (NaCAS), and (■) lecithin

This was due to lecithin was a phospholipid emulsifier, where this type of emulsifier could also act as an effective antioxidant agent to slow down the oxidation process and free radical permeation (Pan et al., 2013). Therefore, it can protect  $\beta$ -carotene from the degradation process. This finding was similar to research conducted by Shu et al. (2017) where compared the storage stability of ergocalciferol nano-dispersions using lecithin and NaCAS as emulsifiers. They obtained nano-dispersion with lecithin emulsifier has more stability compared to using NaCAS emulsifier.

In addition, this is in accordance with research conducted by Chen & Zhong (2015), who examined the degradation of  $\beta$ -carotene dissolved in peppermint oil using lecithin emulsifiers. The antioxidant properties of peppermint oil and the higher lecithin content in the microemulsion allows good protection against  $\beta$ -carotene. Along with emulsifiers, solid content also has a significant influence on the degradation rate of  $\beta$ -carotene in RPO powder, where RPO powder with a solid content of 35 wt.% has a lower  $\beta$ -carotene degradation rate value than that with a solid content of 25 wt.%. This was due to the solid content could affect the moisture content of microcapsules. According to Ng et al. (2014), the moisture content in microcapsules can affect the stability of microcapsules. The higher the moisture content will increase the chemical and enzymatic reactions of the encapsulated oil. The  $k$  value obtained

in this study was different with the research conducted by (Šeregelj et al., 2022), who investigated the degradation of carotenoids with a storage time of 413 days at 4, 21, 30 and 37 °C, where the  $k$  values ranged from 0.00–0.029 day<sup>-1</sup>. Song et al. (2017), observing the kinetics of carotenoid degradation during microwave-vacuum drying, obtained  $k$  values ranging from 0.0363–0.0686 min<sup>-1</sup>. Dutta et al. (2006), observed the kinetics of  $\beta$ -carotene degradation in pumpkin puree with a storage time of 0 and 2 hours and stored at a temperature of 70–100 °C, where the value of  $k$  was obtained between 0.004–0.008 min<sup>-1</sup>.

Mba et al. (2017), observing the kinetics of carotenoid degradation in palm oil and canola oil during deep-fat frying at temperature of 170–190 °C, each  $k$  value was obtained, which ranged from 0.71–1.16 × 10<sup>-6</sup>s<sup>-1</sup> dan 18–1.34 × 10<sup>-6</sup>s<sup>-1</sup>. One of the factors that can affect the difference in these values is storage temperature, an increase in the rate of degradation will increase with increasing temperature (Sierra, 2012). The degradation observed during the thermal treatment and the long storage time are a consequence of the isomerization reaction experienced by  $\beta$ -carotene at high temperatures (Chen et al., 2017), the fatty acids present in the oil also undergo oxidative processes at high temperatures, resulting in an auto-oxidation process, photo-oxidation and accelerated degradation of  $\beta$ -carotene with the formation of alkyl and peroxy radicals (Hong et al., 2017).

**Table 5:** Comparison of encapsulated carotene with different wall materials or emulsifiers

Core materials	Wall materials/ Emulsifiers	Results	References
Red palm oil	Maltodextrin DE 10, sodium caseinate, soy lecithin	Maltodextrin DE 10, sodium caseinate, and soy lecithin had been successfully used to encapsulated red palm oil to protect carotene during storage, with spray drying microencapsulated RPO.	(Lee et al., 2020)
Red palm oil	CMSC and plasticizer PEG 400.	CMSC and plasticizer PEG 400 had been successfully used to encapsulated red palm oil to improve thermal stability.	(Sathasivam et al., 2018)
Peppermint oil, $\beta$ -carotene	Lecithin	The antioxidant properties of peppermint oil and the higher lecithin content in the microemulsion allows good protection against $\beta$ -carotene	(Chen & Zhong, 2015)
Tomato oil, carotene	$\alpha$ -Cyclodextrin	$\alpha$ -Cyclodextrin had been successfully used to encapsulated tomato oil to slow down carotenoid degradation.	(Durante et al., 2020)
Sunflower oil, $\beta$ -carotene	Whey protein isolate, sodium caseinate and tween 80	The highest cellular uptake of $\beta$ -carotene was observed in the sodium caseinate emulsion, followed by tween 80 and whey protein isolate.	(Lu et al., 2017)

#### 4.0 Conclusions

The stability of  $\beta$ -carotene in RPO encapsulated powder using  $\alpha$ -cyclodextrin as wall material with different emulsifier sodium caseinate (NaCAS) and lecithin was investigated. The difference of solid content during formulation of RPO feed emulsion was also investigated. Based on the results obtained, differences in emulsifier and solid content have a significant effect on the droplet size, but do not have a significant effect on the viscosity and pH of emulsion of RPO emulsion. Differences in emulsifier and solid content do not have a significant effect on the characteristics of RPO powder, but have a significant effect on the stability of RPO powder. The stability of  $\beta$ -carotene was measured at 60 °C for 25 days. Degradation of  $\beta$ -carotene in encapsulated powder could be correlated using the first kinetic model. The stability of  $\beta$ -carotene with lecithin as emulsifier in both solid contents (25 wt.% and 35 wt.%) were more stable with degradation rate constant  $0.018 \text{ day}^{-1}$  and  $0.016 \text{ day}^{-1}$ , respectively.

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#### Contribution statement

**G. S. Putri** and **T. N. Saudia** prepared RPO feed emulsion and encapsulation process. **V. Paramita** designed the study. **G. S. Putri** and **T. N. Saudia** carried out the whole experiment and wrote the manuscript. **H. D. Ariyanto** supervised the experiment and reviewed the manuscript.

#### Conflict of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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