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Demulsification of Virgin Coconut Oil by Centrifugation Method: A Feasibility Study

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Abstract: In this study, the potential of centrifugation method in demulsification (emulsion breaking) of coconut milk emulsion is investigated. The conventional methods to produce Virgin Coconut Oil (VCO) by using fermentation and cold pressing need longer time to break these emulsions. Coconut milk from the local market was used as samples for the study. The centrifuge speed was varied from 6000 to 12000 rpm and the centrifugation time was varied from 30 to 105 min. The present research found that, centrifugation method can enhance the demulsification of oil-in-water (O/W) coconut milk emulsions in a very short time compared to the conventional methods. Due to its fast and higher quality of Virgin Coconut Oil (VCO), centrifuge can be used as an alternative demulsification method for oil-in-water coconut milk emulsions. This method provides higher yields, quicker and less expensive.

Key words: Fermentation, demulsification, o/w emulsion, centrifugation, conventional, coconut milk, fermentation

INTRODUCTION

An emulsion consists of two immiscible liquids (usually oil and water), with one of the liquids dispersed as small spherical droplets in the other (a continuous phase). Emulsions are thermodynamically unstable due to the unfavorable contact between oil and water molecules (Friberg, 1997) and as a consequence their physical structures will tend to change over time by various mechanisms (e.g., creaming, flocculation and coalescence), eventually leading to complete phase separation (McClements, 2005).

Coconut milk is the natural oil-in-water emulsion extracted from the endosperm of mature coconut (*Cocos nucifera* L.) either with or without the addition of water (Seow and Gwee, 1997). It contains fat, water, carbohydrate, protein and ash with the major components being water and fat (Tansakul and Chaisawang, 2005). The emulsion is known to be naturally stabilized by coconut proteins: globulins and albumins and phospholipids (Birose *et al.*, 1963). However, the coconut milk emulsion is unstable and readily separates into two distinct phases—a heavy aqueous phase and a lighter cream phase (Cancel, 1979; Gonzales, 1990). The reason for the instability is that the protein content and quality in coconut milk is not sufficient to stabilize the fat globules (Monera and del Rosario, 1982).

Gravity is mainly associated with the slow sedimentation process of an immiscible mixture. A common way to accelerate this sedimentation is by the use of centrifugation, where the high achievable rotation frequencies permit an effective acceleration; highly superior to the simple gravitation case (Starobinets *et al.*, 1979). Sometimes, gravity separation may be too slow because of closeness of the densities of the particles and the fluid, or because of association forces holding the components together. Gravity separation takes hours, while centrifugal separation is accomplished in minutes,

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(Geankoplis, 2003). The centrifuge works using the sedimentation principle, where the centripetal acceleration is used to separate substance of greater and lesser density. By using centrifuge, it is possible to break down emulsions and to separate dispersions of fine liquid droplets, though in this case the suspended phase is in the form of liquid droplets which will coalesce following separation (Coulson and Richardson, 1991).

MATERIALS AND METHODS

Sample Preparation and Procedures

Fresh coconut milk without added water were bought from a local market and passed through cloth filters before experiments. Four bottles filled with 150 mL of coconut milk each. The four bottles placed inside a high speed centrifuge (Sorvall Evolution RC) with rotor used is SLA-1000 and centrifuged for different speeds ranging from 6000 to 12000 rpm (the angular velocities ω of the rotor ranging from 628.32 to 1256.64 rad sec⁻¹) and different times ranging from 30 to 105 min, all experiments were run at 30°C. Same coconut milk used on each speed and centrifugation time. Centrifugation produced layers of an aqueous phase (water) on the bottom, an emulsion phase (cream) in the middle and an oil phase on top. The height of the separated layers was measured and converted to relative percentage as follows: for the same emulsion:

$$\text{Layer (\%)} = \frac{\text{The height of the separated layer}}{\text{Total height of the emulsion}} \times 100\%$$

Moisture Content and Rancimat Analysis

These analysis include determine of moisture content and rancidity of VCO by referring standard procedure (AOCS ca 28-38). All analysis was done in triplicate. Moisture balance from Mettler Toledo was used to determine moisture content. Rancimat machine supplied by Metrohm, Germany was used for rancidity determination.

Analysis of Fatty Acids

The Fatty Acid Methyl Esters (FAME) of the oil were produced by weighing 30 mg of oil in screw cap tubes to which 4 mL of methanolic HCl was added and mixed. The mixture was incubated at 50°C for 10 h and cooled to room temperature. The FAME then was extracted using hexane three times. The hexane extracts were combined and passed through anhydrous Na₂SO₄ for drying.

Gas Chromatography (GC) Analysis of Fatty Acids

The fatty acid compositions of total lipids from VCO were analyzed by GC. GC analysis was performed on a HP5890 Series II GC machine, under the following conditions: column, glass column (length 1.83 m and 2 mm ID); injector temperature, 250°C; detector temperature (FID), 250°C. Separation was done on a 100/120 Chromosorb-WAW column containing 10% SP2330. Oven temperature increased programmed from 80 to 180°C at a ramp rate of 8°C min⁻¹ for 7.5 min and nitrogen at a flow rate of 20 mL min⁻¹. The FAME was identified and quantified through comparison with standard FAME.

RESULTS AND DISCUSSION

These yields, based on the volume of the coconut milk used, which can produce VCO from 10.75 to 29.50% (Table 1). The highest yield of VCO was obtained at 12000 rpm and 105 min, where

Table 1: Experimental results of VCO yield under centrifugal force at different time and speed

Speed (rpm)							
6000		8000		10000		12000	
Time (min)	Yield (mL) (%)	Time (min)	Yield (mL) (%)	Time (min)	Yield (mL) (%)	Time (min)	Yield (mL) (%)
30	10.75	30	13.67	30	22.67	30	21.00
45	11.50	45	12.25	45	20.67	45	22.67
60	11.58	60	15.17	60	20.00	60	23.67
75	11.83	75	15.33	75	20.17	75	24.83
90	12.08	90	15.83	90	20.67	90	20.83
105	15.58	105	18.33	105	26.17	105	29.50

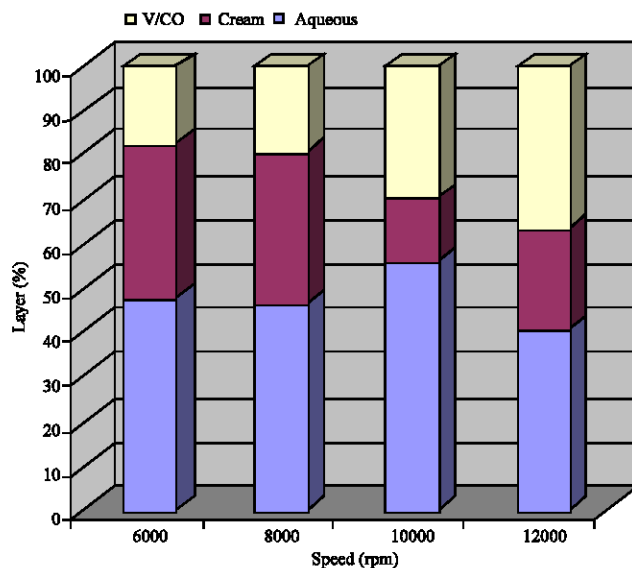


Fig. 1: Phase separation of coconut milk emulsion under centrifugation force at different speed and 105 min. Data are presented in average of four replications.

the VCO yield is 29.50%. Figure 1 shows the phase separation of coconut milk emulsion by using centrifugation force at 105 min, comprising a top layer (VCO), cream layer and a bottom layer of an aqueous phase.

Effect of Centrifuge Speed

The oil yield of the coconut milk emulsion has been studied for four different speeds, namely 6000, 8000, 10000 and 12000 rpm of centrifuge speed, at 30°C for different times. The highest yield of VCO was obtained at 12000 rpm. The oil droplets tend to move in the opposite direction of the centrifugal force because of their lower density. A droplet will be separated from the aqueous phase when it reaches the inner wall of the bottle. The higher centrifuge speed, gives higher yield of VCO. This observation is explained by the fact that increasing the centrifuge speed results in increasing the rate of sedimentation and consequently increases the separation of two immiscible liquid of the emulsion.

It is clear that increasing the centrifuge speed results in an increase in the VCO yield (Fig. 2). According to Stoke's law and replacing gravity acceleration by centrifugal acceleration, if water is the continuous phase, the settling velocity of oil droplets through water is given by:

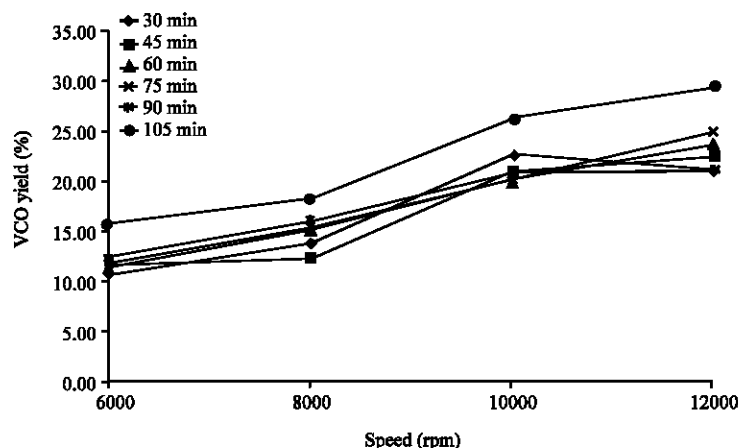


Fig. 2: VCO yield with effect of speed (rpm)

$$v_o = \frac{(\rho_w - \rho_o) \times r \omega^2 \times D^2}{18 \mu_w} \quad (1)$$

where, v_o is the settling velocity of oil, ρ_w is the density of water, ρ_o is the density of oil, r is the radius of rotation, ω is the angular velocity of centrifugation, D is the diameter of the droplets and μ_w is the viscosity of continuous phase (water).

In Eq. 1, settling velocity is proportional to the density difference, square of droplet diameter, centrifugal acceleration and reciprocal to the viscosity of water. The viscosity of oil and water are very sensitive to the temperature. Centrifuge generates heat by centrifugal rotation. When temperature increases, the viscosity will decrease much faster than density difference, $(\rho_w - \rho_o)$ does, results the increase of the droplet size. Therefore, the centrifugal acceleration increases the velocity of the oil and accelerates the separation of the emulsion.

Centrifugal force causes molecular rotations, which may reduce zeta potential of emulsion. Zeta potential is a layer of electrical charges; suspend oil droplets in oil-in-water emulsion which prevents the movement and coalescence of oil droplets. Since, water molecule is polar, it rotates at a high frequency under centrifugal forces.

The molecular rotation raises the temperature through friction and also neutralizes the zeta potential. Without the support of zeta potential, oil droplets are moved downward by gravitational force. Furthermore, when droplets collide with each other in their downward motion, coalescence takes place and droplet diameter, D increases, resulting in the acceleration of separation of emulsions.

Effect of Centrifugation Time

Six different times of centrifugation process were used, namely 30, 45, 60, 75, 90 and 105 min at 30 °C for different centrifuge speed. Figure 3 shows a plot of the VCO yield as a function of different centrifugation time. The highest yield of VCO is found at 105 min. The longer of centrifugation time, it gives higher yield of VCO. This is because the separation process will have longer times that allow the droplets of oil separate from the emulsion. The plot clearly demonstrates the fact that increasing the centrifugation time results in better separation process (VCO yield).

Quality Characteristics

VCO produced by using the different speed and centrifugation time, had some differences in quality properties, these differences may not be large enough to significantly affect the overall quality

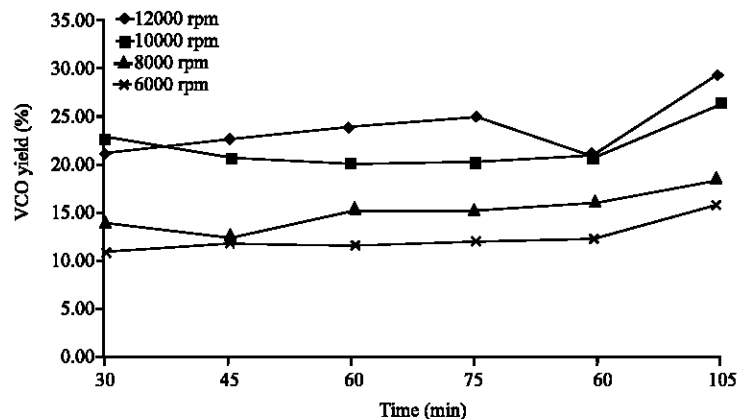


Fig. 3: VCO yield with effect of time (min)

Table 2: Quality characteristics of Virgin Coconut Oil (VCO) samples

12000 rpm (30 min)		12000 rpm (60 min)		12000 rpm (105 min)	
Fatty acid	Content (%)	Fatty acid	Content (%)	Fatty acid	Content (%)
C ₆	0.54	C ₆	0.57	C ₆	0.63
C ₈	8.77	C ₈	8.96	C ₈	9.20
C ₁₀	6.96	C ₁₀	7.05	C ₁₀	7.19
C ₁₂	47.16	C ₁₂	47.31	C ₁₂	47.64
C ₁₄	16.87	C ₁₄	16.85	C ₁₄	16.70
C ₁₆	8.58	C ₁₆	8.57	C ₁₆	8.32
C18:0	2.43	C18:0	2.40	C18:0	2.35
C18:1	6.60	C18:1	6.57	C18:1	6.39
C18:2	1.32	C18:2	1.32	C18:2	1.27
C ₂₀ +C18:3	0.21				
C ₂₀ :1	0.20				
Rancimat @ 110 = 117.64		Rancimat @ 110 = 108.87		Rancimat @ 110 = 123.73	
Moisture: 0.16%		Moisture: 0.07%		Moisture: 0.14%	

of the VCO (Table 2). Further, their levels are still within the Asian and Pacific Coconut Community (APCC) Standards for VCO. The free fatty acid content of the samples as well as their moisture content may eventually have a bearing on the qualities of the VCO during storage.

Fatty Acid Composition

The fatty acid composition of VCO sample fall within the range of Asia Pacific Coconut Community (APCC) Standards for VCO and commercial VCO. Lauric acid (C₁₂) is the major fatty acid in VCO, ranged from 47.16 to 47.64% (Table 2).

Moisture Content

Moisture Content (MC) is another parameter that will determine the quality of the VCO samples and shown in Table 2. The MC of the commercial VCO ranged from 0.10 to 0.42% with VCO sample of centrifugation for 30 min having the highest MC of 0.16% (Table 2), fall within the range of APCC standards for VCO.

The use of different speed and centrifugation time of preparing VCO did not result much differences in moisture content. This may be explained by the more efficient and effective separation of the oil from non-oil constituents for the laboratory produced samples. In oils, moisture is one of the reactants in fat hydrolysis, which can lead to the increasing of free fatty acids. Production of free fatty

acids causes hydrolytic rancidity. Moisture is one of the most important parameters for oil quality because, together with FFA, it can cause oxidation in the presence of light (Che Man *et al.*, 1992).

CONCLUSION

Based on the results of this study, it can be concluded that, centrifugal radiation can be an effective and alternative tool to break (destabilize) oil-in-water coconut milk emulsions. This method does not require chemical addition.

Centrifugation force induced molecular rotation and neutralizes the zeta potential of emulsified oil droplets. Centrifugal force provides two contribution; reduction of viscosity and neutralization of zeta potential.

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