The physical and chemical properties of VCO emulsion with citrus extract

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Abstract. Virgin Coconut Oil (VCO) is one of the oils that has many benefits. But most consumers do not like to consume it directly because of the oily taste. Therefore, one alternative to reduce its oily taste is by formulating VCO in the form of an emulsion. This study was aimed to determine the effect of VCO and citrus extract ratio on the properties of VCO emulsion using natural emulsifiers. The emulsions were prepared with a ratio of VCO and citrus extract of 9:1, 8:2, 7:3, 6:4, 5:5, 4:6, 3:7, 2:8 and 1:9, respectively. Three types of emulsifiers were used, namely Arabic gum, xanthan gum and soy lecithin with each concentration of 0.75 percent. The stability and viscosity of the emulsions were evaluated. The results of this study show that the use of the three emulsifiers resulted in different viscosities and stability. The emulsion using Arabic gum was the most stable at VCO: citrus extract ratio (9:1) with a viscosity of 230 cP. The emulsion with xanthan gum at VCO: citrus extract ratio (1:9) was stable with a viscosity of 1066 cP. However, the emulsion using soy lecithin was unstable. The contents of the peroxide number were 0.8-0.9 meq/kg sample. This stable emulsion can be used for future research to produce VCO emulsion which is good for consumption.

1. Introduction
Virgin Coconut Oil (VCO) is pure oil obtained from coconut fruit. VCO naturally contains a mixture of medium chain fatty acids (MCFA) and long chain fatty acids (LCFA) in a ratio of 3:1 [1]. The highest MCFA content is lauric acid which is ranging from 45-55%. These fatty acids are easily absorbed by the body because the molecular size is not too large as in long chain fatty acids [2]. Other studies have also reported that virgin coconut oil can help reduce total blood cholesterol, triglycerides, and phospholipids in serum and tissue [3]. Furthermore, VCO increases the body's metabolism and energy expenditure, and is directly converted into energy in the liver but not stored in adipose tissue. [1]. VCO has a higher phenolic content and antioxidant activity compared to copra-derived coconut oil. [2].

One of the derivative products of VCO that can be developed is Virgin Coconut Oil (VCO) emulsion. This emulsion product is made to reduce the oily taste in the direct consumption. Processing VCO into a more delicious and stable emulsion product will also be an advantage for VCO producers.

Thermodynamically, emulsion is an unstable system [4]. Therefore, the use of mechanical devices is needed to disperse the system and the addition of stabilizers is necessary to maintain the system in order to remain stable. Several studies on VCO emulsion have been conducted by [5] which aimed to make a VCO emulsion by studying the ratio of VCO and water using a mixed emulsifier of Tween 80 and Span 80. Then, it was followed by other studies on the addition of sucrose to VCO emulsion [6] and...
addition of honey to VCO emulsion [7]. Other research related to antioxidant activity of VCO emulsion has also been carried out by [8].

In preparation emulsions, a homogenizer is used with a certain speed and time which depends on the product being homogenized. There are several factors that influence the emulsion formation, including temperature, stirring time, and stirring speed. The addition of hydrocolloids to the aqueous phase can yield specific rheological properties to achieve emulsion stability. Some hydrocolloids act as surface active gums (e.g., Arabic gum), having the ability to form a film around the oil droplets. As a result of static stabilization, hydrocolloids aid in delaying this coalescence and prevent emulsion breakdown. Moreover, some hydrocolloids are known to stabilizes the emulsions by enhancing the viscosity of the aqueous phase (e.g., xanthan gum) [9] and [10].

The selection of emulsifier is very important in the formation of the emulsion. Several things that affect the emulsion stability are including the amount of emulsifier used, the type of emulsifier, the ratio of oil to water, and the ratio of oil to emulsifier. In addition, several factors such as pH, temperature, and processing conditions also affect the stability of the emulsion [9].

Some researchers use different concentrations of emulsifier in making emulsions. For example lecithin, xanthan gum [11], glycerin ester of fatty acids [12], Odina gum [13] and rosella extracts [14]. Likewise, in [15] conducted a study using Arabic Gum and Tween 80 emulsifiers to make VCO emulsion drinks.

One of the efforts to diversify VCO emulsion products with different tastes can be done by using various types of fruits. One of the fruits that can be used is citrus. Citrus contain vitamin C which is high enough to increase the nutritional value of VCO emulsion. In South Sulawesi, Malangke citrus are less popular because of their sour taste. during the harvest season, a lot of these citrus are wasted. This is because citrus that are not consumed then become rotten fruit. One of the alternatives to reduce orange waste is to use Malangke citrus as the water phase in making emulsions. Thus, the amount of waste is reduced so the environment can be maintained. In addition, the addition of citrus extract will produce various VCO emulsions. Based on these studies, this study will try to optimize the ratio between VCO and citrus extract on the stability and viscosity of the emulsions using 3 types of emulsifiers, namely Arabic gum, xanthan gum and soy lecithin. It is expected that the data from this research can provide useful information for the use of VCO and the development of VCO-based industries.

2. Materials and Methods
This research took place at the Chemical Laboratory of the Faculty of Industrial Technology and the Pharmaceutics Laboratory of the Faculty of Pharmacy, Universitas Muslim Indonesia. Makassar, Indonesia.

2.1. Material and equipment
The main research material is VCO obtained from CV. Avcol, Makassar. Malangke citrus were obtained from Sumber Harum Village, Sukamaju sub-district, North Luwu Regency, South Sulawesi. Arabic gum, xanthan gum, and soy lecithin emulsifiers with food grade qualification were obtained from the local chemical shop and other ingredients for analysis. The equipment used are the Ultra Turrax homogenizer, a Brookfield viscometer, a magnetic stirrer, a scale, and some glasses for analysis.

2.2. Procedure
There are two stages preparation to make VCO emulsion namely preparation of citrus extract and preparation of the emulsion.

2.2.1. Preparation of citrus extract. A total of 10 kg of citrus, each is split into 2 and then squeezed using an citrus press. The juice obtained is used for the making of VCO emulsion.

2.2.2. Preparation of VCO emulsion. Preparation of VCO emulsion is based on [5] research, with several modifications. A total of 90 ml of VCO mixed with 10 ml of citrus extract was added with 0.75 ml of Arabic gum emulsifier. Then the mixture was homogenized using Ultra Turrax at a speed of 15,000 rpm for 4 minutes to form an emulsion. Preparation of the emulsion was repeated using soy lecithin emulsifier and xanthan gum emulsifier. Then the procedure was repeated with VCO-citrin extract ratios
8:2, 7:3, 6:4, 5:5, 4:6, 3:7, 2:8, 1:9, respectively. The emulsions formed were tested for viscosity, stability, and peroxide number.

2.3. Analysis

2.3.1. Viscosity test. The viscosity test was performed using the Brookfield Viscometer. A total of 60 ml of emulsion was put in the erlenmeyer flask and its viscosity was tested using a spindle 6. The rotation speed was adjusted to 50 rpm for 30 seconds. The viscosity value will be read immediately on the tool in cP units.

2.3.2. Emulsion stability test [16]. Emulsion stability measurements were determined by the cycle method. A total of 80 ml of emulsion was put in a bottle and stored at 5°C for 12 hours. Then the emulsion was transferred again to the oven at 35°C for 12 hours. The treatment was repeated until 5 cycles at 5°C and 5 cycles at 35°C were obtained. Emulsion stability is calculated by measuring the volume of the emulsion that is still stable with the following formula: [volume of stable emulsion/volume of total emulsion] x 100 %

2.3.3. Peroxide number test [17]. The sample was weighed at 5 grams into 300 ml Erlenmeyer flask. 10 ml of chloroform and 15 ml of glacial acetic acid were added and the Erlenmeyer flask was shaken to mix the samples. Then 1 ml of saturated potassium iodide (KI) was added and the Erlenmeyer flask was immediately closed while shaking it for about 5 minutes in a dark place at a temperature of 15-25°C. Then 75 ml of distilled water was added and the Erlenmeyer flask was shaken intensely. The solution was titrated with a sodium thiosulfate standard solution of 0.2 N with starch solution as an indicator. Peroxide numbers are expressed in meq/kg of sample.

3. Results and Discussion

In Figure 1. The relationship between emulsion stability at various ratios of VCO and citrus extract using 3 types of emulsifiers, namely xanthan gum, soy lecithin and Arabic gum is presented.

![Figure 1. The relationship between Stability and Ratio of VCO-Citrus extract in Various Types of Emulsifiers.](image)

The stability of the VCO-citrus extract emulsion using 3 types of emulsifiers is presented in Figure 1. The use of soy lecithin emulsifier resulted in unstable emulsions from the VCO: citrus extract ratio, 9: 1 to 1: 9. The emulsion stability (using this emulsifier) only reached 60 percent. In emulsions using Arabic gum emulsifier, the emulsion was stable at a ratio of 9: 1. This is different from the case with emulsions using xanthan gum, in which the emulsion stability was in the ratio of 4:6, 3:7, 2:8, and 1: 9. The emulsion destabilization process begins with droplet movement. In large emulsions, droplet movement is dominated by gravity [18]. The main factor affecting the creaming of the sample is the difference in density between the dispersing medium and the dispersed medium. The greater the
difference in density between the dispersing medium and the dispersed medium, the faster the separation due to gravity. The presence of this separation emulsifier can be reduced or prevented. Emulsifier has a property that can bind oil (lipophilic) and a property that can bind water (hydrophilic). According to [19], emulsion damage occurs through 3 main mechanisms, namely creaming, flocculation and coalescence. Creaming is a separation process that occurs as a result of up/down movements due to the gravitational force on the phases with different densities. Flocculation is the aggregation of droplets where the number and size of the globules are fixed. However, flocculation will accelerate the occurrence of creaming. Coalescence is the incorporation/combination of globules into larger globules.

**Figure 2.** The viscosity of the emulsion stabilized by xanthan gum was higher than the viscosity of the emulsion stabilized by Arabic gum and soy lecithin.

The effect of using the type of emulsifier on the viscosity of the VCO-citrus extract emulsion is presented in Figure 2. The viscosity of the emulsion stabilized by xanthan gum was higher than the viscosity of the emulsion stabilized by Arabic gum and soy lecithin. In an emulsion that used xanthan gum, the viscosity could reach 5420 cP, namely at the ratio of VCO and citrus extract of 6:4 which showed a very thick product. Emulsion using soy lecithin produced the highest viscosity of 130 cP at a VCO-citrus extract ratio of 8:2, while the emulsion using Arabic gum produced the highest viscosity of 230 cP at a VCO-citrus extract ratio of 9:1.

This difference is thought to be due to differences in the molecular structure of the three emulsifiers. Lecithin is the commercial name for phosphatidylcholine. The hydrophobic part of lecithin is 2 fatty acid chains which type depends on the source of lecithin. The fatty acids in soybeans are dominated by long chain fatty acids such as linoleic [20]. In the chemical structure of xanthan gum, it has many hydroxyl groups that are able to bind water. Besides, it has many branches that make the trapped water is able to stabilize the resulting emulsion compared to Arabic gum and soy lecithin.

In Table 1 shows the peroxide number of emulsions prepared using xanthan gum, soy lecithin and Arabic gum emulsifiers.
Table 1. Peroxide number of VCO-Citrus extract Emulsions.

| Type of emulsifier | Peroxide number meq / kg |
|--------------------|--------------------------|
| Xanthan gum        | 0.90                     |
| Soy lecithin       | 0.78                     |
| Arabic gum         | 0.81                     |
| APCC Standard for VCO | 3                       |

In Table 1., the peroxide values of the emulsion with 3 types of emulsifier are listed. The peroxide values produced from the three types of emulsion has a lower value than the standard value issued by the Asian and Pacific Coconut Community (APCC), namely the maximum peroxide value in VCO is 3 meq/kg of sample [21]. The low peroxide value is due to the fact that VCO contains around 90 percent saturated fatty acids which are more resistant to rancidity due to oxidation than unsaturated fatty acids. This low peroxide value indicates that the emulsion produced is not rancid so that it can be further developed to produce an emulsion product that is more delicious to be consumed. For example, with the addition of sugar, acid, or flavor that can enhance the taste.

4. Conclusion

Emulsion that has high stability is an emulsion that uses xanthan gum emulsifier; it produces a stable emulsion at a VCO-citrus extract ratio of 1: 9 with a viscosity of 1066 cP. Emulsion using Arabic gum produced the highest stability at 9:1 VCO-citrus extract ratio with a viscosity of 230 cP. Meanwhile, emulsion using soy lecithin is unstable. Further research is needed on the shelf life and nutritional value of VCO-citrus extract emulsion so that it can be applied to the home industry / small industry scale.

Acknowledgements

Our biggest gratitude goes to the DRPM of the Directorate General of Higher Education, Ministry of Research and Technology / National Research and Innovation Agency of the Republic of Indonesia for the funds provided through the Higher Education Leading Applied Research Grant for the 2019-2020 fiscal year.

References

[1] Liau, K.M; Lee, Y.Y.; Chen, C.K.; Rasool, A.H.G. An open-label pilot study to assess the efficacy and safety of virgin coconut oil in reducing visceral adiposity. ISRN 2011, 949686, 1-7.

[2] Marina, A.M., Che Man, Y.B., Nazimah, S.A.H. & Amin, I. 2009. Antioxidant Capacity and Phenolic Acids of Virgin Coconut Oil. Int. J. Food Sci. Nutr, 60, pp 114-123.

[3] Nevin, K.G. & Rajamohan, T., 2004. Beneficial Effects of Virgin Coconut Oil on Lipid Parameters and in Vitro LDL Oxidation. *Clinical BioChemistry*, 37, pp.830–835.

[4] Maskan, M.; Gogus, F. 2000. Effect of sugar on the rheological properties of sunflower oil-water emulsion. J. Food Eng. 43, 173-177.

[5] Wiyani, L., Aladin, A., Yani, S. dan Rahmawati. 2016b. Stability of virgin coconut oil emulsion with mixed emulsifiers tween 80 and span 80. ARPN Journal of Engg. App. Scie. Vol. 11, No. 8.: 5198-5202

[6] Wiyani, L., Aladin, A., Yani, S., Mutmainnah S.H.N. and Mandang, H.D. 2018. Effect of Sucruse and citric acid addition in the Virgin Coconut Oil Emulsion. IOP Conferences Series: Earth Environ. Sci. 175 012024. https://doi.org/10.1088/1755-1315/175/1/012024

[7] Wiyani, L., Aladin, A., Mustafiah., Rahmawati & Abriana, A. 2020. Characteristics of virgin coconut oil emulsion with honey and citric acid. Proceedings of the 5th International
[8] Wiyani, L., Rahmawati; Aminah; Aladin, A.; Mustafiah., Juniar, M.E. 2020. Antioxidant activity of virgin coconut oil and virgin coconut emulsion. SRP. 2020; 11 (12): 973-976. DOI: 10.31838/srp.2020.5.139. http://www.sysrevpharm.org/index.php?mno=137586

[9] Khor, Y. P., Koh, S. P., Long and K., Long, S. 2014. A Comparative Study of the Physicochemical Properties of a Virgin Coconut Oil Emulsion and Commercial Food Supplement Emulsions. Molecules.19.9187-9202;doi:10.3390/molecules19079187. www.mdpi.com/journal/molecules

[10] Taherian, A.R.; Fustier, P.; Ramaswamy, H.S. 2006 Effect of added weight agent and xanthan gum on stability and rheological properties of beverage cloud emulsions formulated using modified stach. J. Food Process Eng. 30. pp. 204-224.

[11] Traynor M. P., Burke R., Frias, J.M., Gaston, E. & Barry-Ryan, C. 2013. Formation and Stability of an Oil in Water Emulsion Containing Lecithin, Xanthan Gum and Sunflower Oil. International Food Research Journal 20(5): 2173-2181.

[12] Bao Chengwei, An Shulin, Wang Wenli., Wang Fei, Y. Shuangchun & Pan Yi. 2013. Research Progress in Glycerin Fatty Acid Ester Emulsifier. International Journal of Scientific & Engineering Research. Volume 4: 924-925.

[13] Samanta, A., Ojha D., Mukherjee B., 2010. Stability Analysis of Primary Emulsion Using a New Emulsifying Agent Gun Odina. Journal Natural Science. Vol 2, No: 495-505.

[14] Ibrahim N. H., Lee T. S., Rozaini M. Z. H. 2013. Potential Application of Rosella Extract in Functional Food Emulsions. J. Tech and Food Industry. Vol 24, No. 1: 22-26.

[15] Mandei, J. 2019. Formulation of VCO emulsion drink using emulsifier variations (Arabic gum, Tween 80) and water. Journal of Plantation Product Industry, 14 (1), pp. 11–20.

[16] Tabibi dan Rhodes. 1996. Disperse Systems. In: Modern Pharmeceutics, Revised and Expanded. Banker G. S. and Rhodes C. R. (Eds.). CRC Press, USA.

[17] Indonesian National Standard. 1994. How to Test Oils and Lipid. (in Bahasa Indonesia). (SNI 01-3555-1994). Jakarta, Indonesia.

[18] McClements, D.J. 2011. Edible nanoemulsions: fabrication, properties and functional performance. Soft Matter 7: 2297-2316. DOI: 10.1039/COSM00549E.

[19] McClements, D.J. 2015. Food Emulsion : Principles, Practices, And Techniques Second. F. M. Clydesdale, ed., Florida: The CRC Press

[20] Wang, G. and Wang, T. 2008. Oxidative stability of egg and soy lecithin as affected by transition metal ions and pH in emulsion. J. Agric Food Chem 56: 11424-11431. DOI: 10.1021/jf8022832.

[21] APCC. 2009. APCC Standards For Virgin Coconut Oil Asian and Pacific Coconut Community. http://www.apccsec.org/document/VCO standard.pdf