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Vitamin E Extraction from Magnesium Salts of Palm Fatty Acid Distillates

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Highlights:

- Increasing the number of extraction stages increased the yield of the extract concentrates and the vitamin E recovery rate from Mg-PFAD.
- Three-stage hexane extraction resulted in the highest yield and recovery rate of vitamin E from Mg-PFAD compared to isopropanol or ethanol extraction.
- Sequential ethanol extraction followed by hexane extraction is recommended to increase both the yield and the vitamin E concentration in the extract concentrate.

Abstract. This research studied how the type of organic solvent and the number of extraction stages affect the vitamin E cumulative extraction yield and recovery rate from Mg-PFAD. First, PFAD was saponified to produce Mg-PFAD, then vitamin E was extracted from the Mg-PFAD using ethanol, isopropanol, or hexane, followed by evaporation to produce vitamin E concentrate. Three-stage hexane extraction with a solvent to Mg-PFAD mass ratio of 3 kg solvent/kg Mg-PFAD produced the highest vitamin E recovery rate. Organic solvent with a lower polarity gave a higher extraction yield and recovery rate of vitamin E from Mg-PFAD. In general, an increase of the number of extraction stages led to an increase of the vitamin E extraction yield and recovery rate from Mg-PFAD.

Keywords: *Magnesium salts of PFAD; PFAD; saponification; solvent extraction; vitamin E.*

1 Introduction

Oil palm (*Elaies guineensis*) is a vegetable oil source with high oil content and the highest oil productivity per hectare among other vegetable sources. In 2019, Indonesia produced 48.4 million tons of crude palm oil (CPO) from 14.6 million hectares of plantation area [1]. Crude palm oil (CPO) is extracted from palm fruit, followed by a sequence of oil refining processes, such as degumming, bleaching,

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and deodorization. Palm fatty acid distillate (PFAD) is a by-product of crude palm oil deodorization, which is one of the stages in crude palm oil (CPO) refining. The yield of PFAD is approximately 6%-wt of CPO [2], or about 2.9 million tons per year in Indonesia. PFAD has a high fatty acid content of 82%-wt [3]. In addition, most of the vitamin E in CPO is carried over to the PFAD, resulting in 4000-5000 ppm or about 0.5%-w vitamin E in the PFAD, which is five times the amount found in CPO [4]. About 70% of the vitamin E found in PFAD exists in the form of tocotrienol, which is a much higher percentage than in other sources, such as olive oil, rice bran, berry, and cereals [5]. Therefore, PFAD is a highly potential raw material for vitamin E production.

Vitamin E is essential for humans, with an intake of about 15 mg per day recommended by the National Institute of Health [6]. Thus, because of the large size of the Indonesian population there is a high potential demand of vitamin E. However, Indonesia's domestic production of vitamin E is still low, thus resulting in high imports of vitamin E. In 2018, 1900 tons of vitamin E were imported according to the International Trade Centre [7]. Two methods to separate vitamin E from CPO and fatty acids are: 1) esterification of the fatty acids followed by distillation, and 2) saponification/neutralization of the fatty acids using alkali metal base (Na or K) in alcohol, followed by extraction with hexane [8]. The disadvantages of these methods are high investment costs due to the high energy requirements and large amounts of waste. Therefore, this research isolated vitamin E from PFAD through neutralization of the fatty acids (saponification) using magnesium oxide (MgO), resulting in a mixture of magnesium salts of palm fatty acid distillates (Mg-PFAD) and unsaponifiable vitamin E, followed by extraction of vitamin E using food-grade organic solvents [9,10]. Mg-PFAD salt is insoluble in water and organic solvents, and solidifies at room temperature in the form of a white precipitate, which enables separation of vitamin E by solvent extraction, resulting in an extract rich with vitamin E. This research studied how type of organic solvent and number of extraction stages affect the vitamin E cumulative extraction yield and recovery rate from Mg-PFAD.

2 Materials and Methods

2.1 Production of Mg-PFAD Salt by Saponification of PFAD

PFAD was saponified with magnesium oxide (MgO) in a 15-L jacketed reactor with a blade stirrer in the bottom part of the reactor following the methods from Listyaningrum, *et al.* [10] and Lestari, *et al.* [11, 12]. Production of Mg-PFAD salt was started by melting the 1.5 kg PFAD in the reactor at 60 °C, followed by the addition of MgO powder at a molar ratio of 1.1 mol MgO/mol PFAD. Afterwards, about 10 mL of demineralized water was added into the mixture to catalyze the saponification reaction. The reaction was completed in 5-10 minutes,

which was indicated by the formation of solid magnesium salt of PFAD (Mg-PFAD). The Mg-PFAD was then weighed and milled. The fine particle mass of Mg-PFAD was then sieved to determine the particle size distribution.

2.2 Vitamin E Extraction from Mg-PFAD and Extract Concentration Process

About 100 g of Mg-PFAD salt was mixed with organic solvent (ethanol, isopropanol, or hexane) at a solvent to Mg-PFAD mass ratio of 3 kg/kg. The extraction was conducted in a vessel with a heating mantle to adjust the extraction temperature of 60 °C following the method from Lestari, *et al.* [12]. After 45 minutes of extraction, liquid extract was separated from the solid through filtration. This process was repeated two times for two-stage extraction and three times for three-stage extraction. The 1st and 2nd stage extract were mixed to produce the two-stage vitamin E extract. Meanwhile, the 1st, 2nd, and 3rd extracts were mixed to produce the three-stage vitamin E extract. The total extract concentrates were produced after removing the solvent from the combined extracts by using a rotary evaporator.

2.3 Total Tocopherol Analysis of Vitamin E Extract Concentrate

Vitamin E concentration in the extract concentrates was expressed as total tocopherol content, which was analyzed following the procedure from Wong, *et al.* [13]. In brief, a series of standard solutions was made by diluting standard p.a. tocopherol in toluene to a certain concentration to make a standard calibration curve, while the extract concentrates were mixed with toluene, 2.2-bipyridine, $FeCl_3.6H_2O$ solution, and ethanol. The absorbance of the solution was measured using a spectrophotometer at a wavelength of 212 nm. The recovery rate of vitamin E in the total extract concentrate from the Mg-PFAD (mg tocopherol in concentrate/g tocopherol in Mg-PFAD) and the vitamin E concentration (mg tocopherol in sample/kg sample) were calculated following the formulas from Lestari [12] as stated in Eq. (1) and Eq. (2), respectively:

$$Vitamin\ E\ Recovery\ Rate = \frac{tocophero\ \ in\ concentrate\ (mg)}{tocophero\ \ in\ Mg{-}PF\ \ \ \ (g)} \quad (1)$$

$$Vitamin\ E\ Concentration = \frac{tocopherol\ in\ sample\ (mg)}{sample\ (kg)} \quad (2)$$

2.4 Acid Value Analysis

An acid value analysis was conducted following the method from Bockisch [14]. In brief, a solution consisting of chloroform, sample, and phenolphthalein was mixed thoroughly in an Erlenmeyer flask, followed by titration with an alcoholic solution of KOH until it turned into slightly pink. The volume of the titrant was

recorded. Acid values were calculated with Eq. (3), while the total amount, extraction rate, and concentration of FFA were calculated using Eqs. (4-6).

$$Acid\ Value = \frac{56{,}1 \times Volume\ of\ KOH\ Solution\ (mL) \times KOH\ Normality}{mass\ of\ sample\ (g)} \quad (3)$$

$$FFA\ in\ Sample\ (g) = \frac{Mr\ of\ PFAD \times Acid\ Value}{(1000 \times Mr\ KOH)} \times \text{Sample mass}\ (g) \quad (4)$$

$$FFA\ Extraction\ Rate = \frac{FFA\ in\ concentrate\ (mg)}{FFA\ in\ Mg{-}PFAD\ (g)} \quad (5)$$

$$FFA\ Concentration = \frac{FFA\ in\ sample\ (g)}{sample\ (kg)} \quad (6)$$

3 Results and Discussion

3.1 Production of Mg-PFAD Salt

Mg-PFAD was produced through two batches of saponification of PFAD with MgO, followed by milling and sieving. The fine Mg-PFAD was analyzed for the particle size distribution (Figure 1). The Mg-PFAD was dominated by larger particle sizes of 20-40 mesh. The larger particle size reduced the contact area between the solvent and the substrate, but may ease the separation of the extract and the solid soap.

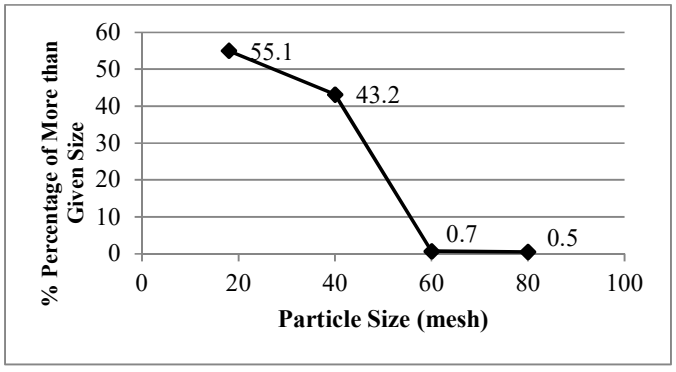

Figure 1 Particle size distribution of Mg-PFAD produced by saponification of PFAD with MgO at 80 °C and a MgO to PFAD molar ratio of 1.1 mol MgO/mol FFA.

Table 1 shows the vitamin E and the free fatty acid (FFA) concentrations of the PFAD saponification product. From the two batches of saponification, approximately 1.2 kg of Mg-PFAD was produced per kg PFAD. The Mg-PFAD product mixture still contained about 39%-wt of unsaponified FFA, which

corresponds well with the result of Listianingrum, *et al.* [10] at the same MgO to PFAD molar ratio. This high amount of unsaponified FFA may be due to the fast formation of a lumpy soap mass, which hinders the completion of the saponification reaction. In addition, the saponification product mixture may still contain acylglycerols, sterols, and squalene. Each component has a specific solubility in both polar and non-polar solvents.

Table 1 Characterization of Mg-PFAD produced by saponification of PFAD with MgO at 80 °C with MgO to PFAD ratio of 1.1 mol MgO/mol FFA.

Flow	PFAD (g)	MgO (g)	Mg-PFAD (g)
Batch 1	1500	260	1730
Batch 2	3000	520	3480
Composition in %-wt			
Vitamin E	0.034	0	0.029
FFA	95.45	0	38.95

Quek, *et al.* [8] state that the formation of a lumpy soap mass is quite common in saponification reactions. This lumpy mass may complicate the vitamin E extraction due to poor contact between the soap particle surfaces and the solvent. Therefore, appropriate solvent, solvent to soap ratio, and number of stages are required to obtain a high vitamin E recovery with low content of impurities, especially unsaponified FFA, tryglyceride, or glycerol.

3.2 Effects of Organic Solvent Type and Number of Extraction Stages on Vitamin E and Free Fatty Acid (FFA) Concentration of the Total Extract Concentrate

The effects of organic solvent type and number of extraction stages on the vitamin E concentration (mg tocopherol/kg concentrates) at various numbers of extraction stages are shown in Figure 2. The total extract concentrate with the highest cumulative vitamin E concentration was obtained after extraction using isopropanol (467-671 ppm). Extraction using hexane resulted in concentrate with a lower vitamin E concentration (308-364 ppm), while extraction using ethanol resulted in the concentrate with the lowest vitamin E concentration (92-137 ppm). The extract concentrates that were obtained after Mg-PFAD extraction using hexane or isopropanol had higher vitamin E concentrations than those obtained from Mg-PFAD (290 ppm).

The vitamin E concentration in the extract concentrates was presumed to be correlated to the affinity of the solvent to other unsaponifiable components in Mg-PFAD, such as phytosterols and squalene, which have a non-polar tendency, as shown in the research of Sahu, *et al.*[15]. The highest vitamin E concentration was obtained with one-stage extraction. Increasing the number of extraction stages to two and three stages slightly reduced the vitamin E concentration of the

total extract concentrates and increased the extractability of impurities, such as unsaponified FFA, which corresponds well with the result from Lestari, *et al.* [12]. The vitamin E concentration was correlated to the amount of unsaponified FFA in the Mg-PFAD. A high FFA concentration led to a lower vitamin E concentration in the extract concentrates. The experimental data in Figure 2 show that the concentration of FFA in the hexane extract concentrates were similar to that in the ethanol extract concentrates.

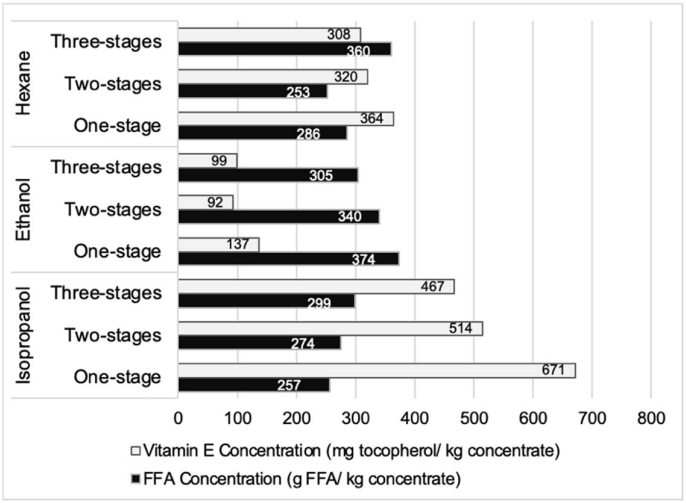

Figure 2 The effects of organic solvent type and number of extraction stages on the vitamin E and FFA concentrations of the concentrates after extraction with a solvent to Mg-PFAD mass ratio of 3 kg/kg.

Melwita, *et al.*[16] state that PFAD consists of approximately 80% of free fatty acids, dominated by palmitic and oleic acids, while Calvo, *et al.* [17] state that palmitic acid has a higher solubility in hexane than ethanol. Hoerr & Harwood [18] state that the solubility of oleic acid in ethanol is higher than in isopropanol. The isopropanol extract concentrate had the highest vitamin E concentration with the lowest FFA concentration, indicating a higher selectivity than hexane and ethanol. However, the FFA concentration in the isopropanol extract concentrate was still rather high (257-299 ppm).

3.3 Effects of Organic Solvent Type and Number of Stages on the Recovery Rate and the Yield of Vitamin E in the Total Extract Concentrate

The total extract concentrate yield and the recovery rate of vitamin E from Mg-PFAD were correlated with the ability of the solvent to extract components from

the soap mixture, consisting of unsaponified FFA, tryglyceride, and unsaponifiable components such as vitamin E, sterols, and squalene [4,8]. The effect of the number of extraction stages on the vitamin E recovery rate and the FFA extraction rate was found to be very significant for ethanol and hexane, whereas the effect was less pronounced for isopropanol (Figure 3). Figure 3 shows that the recovery rate of vitamin E increased with a decrease in polarity of the solvent. The highest vitamin E cumulative recovery rate was obtained after Mg-PFAD extraction using hexane (221-372 mg of tocopherol/g of tocopherol in Mg-PFAD). This indicates that vitamin E in Mg-PFAD (consisting of tocopherol and tocotrienol) has higher extractability in a nonpolar solvent (hexane).

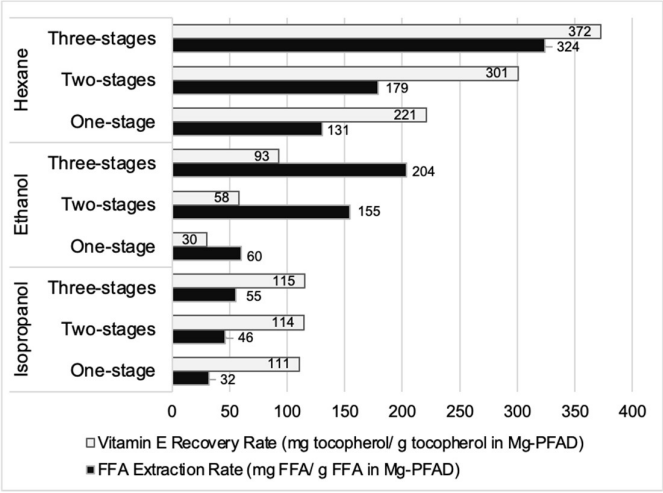

Figure 3 The effect of organic solvent and number of stages on the vitamin E recovery rate and the FFA extraction rate from Mg-PFAD at a solvent to Mg-PFAD mass ratio of 3 kg/kg.

The low polarity of hexane facilitates the formation of homogeneous dispersion between Mg-PFAD and solvent, increasing the contact area between Mg-PFAD and hexane and thus improving the extractability of vitamin E [11]. On the other hand, the high polarity of ethanol and isopropanol reduces the wettability on the surfaces of the Mg-PFAD particles and hinders ethanol diffusion into the Mg-PFAD pores, reducing the vitamin E extractability [19]. In contrast with the low recovery rate of vitamin E, the FFA extraction rate was rather high in ethanol. While the extraction rates of vitamin E and FFA were similarly high in hexane, the different extraction rates of FFA and vitamin E in ethanol may be useful to separate unsaponified FFA from vitamin E. Figure 4 shows that the highest cumulative yield of total extract concentrates was obtained after three-stage

hexane extraction of 3 kg/kg (331 g concentrates/kg Mg-PFAD), followed by three-stage ethanol extraction (258 g concentrates/kg Mg-PFAD). Isopropanol extraction resulted in the lowest cumulative yield compared to the other organic solvents. Due to the low yield of vitamin E, the extracted FFA and other unsaponifiable components may significantly contribute to an increase of the cumulative yield of extract concentrate. Increases in cumulative yield of vitamin E by 1.9-fold and 3-fold were obtained after two-stage and three-stage ethanol extraction, respectively. Meanwhile, the increases in the cumulative yield of vitamin E for two-stage and three-stage hexane extraction were 1.4-fold and 1.7-fold, respectively. On the other hand, there was no significant improvement of the recovery of vitamin E after two-stage and three-stage isopropanol extraction.

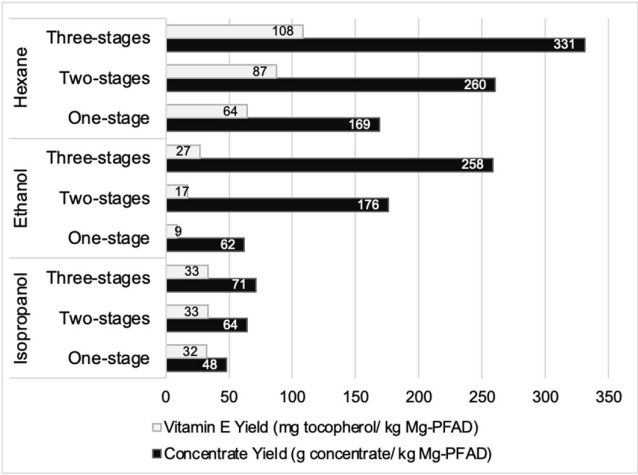

Figure 4 The effect of organic solvent and number of stages on the total concentrate yield and the yield of vitamin E from Mg-PFAD at a solvent to Mg-PFAD mass ratio of 3 kg/kg.

Increasing the number of stages improved the yield of vitamin E gradually after extraction with hexane and isopropanol. In contrast, increasing the number of stages of ethanol extraction did not result in an improvement of the vitamin E extractability. This indicates that the amount of extracted vitamin E in the 2nd and the 3rd stage of isopropanol extraction was not significant. Figure 5 shows the increased vitamin E yield in the concentrate as a function of total solvent required during one-stage, two-stage, and three-stage extraction. The improvement of the vitamin E yield rate due to the increased solvent requirement during multi-stage extraction is shown in Figure 5.

The rate of the vitamin E yield improvement was the highest in hexane extraction (6.7 mg tocopherol/kg hexane), followed by ethanol extraction (2.8 mg tocopherol/kg ethanol). The lowest rate of vitamin E yield improvement occurred with isopropanol extraction (0.2 mg tocopherol/kg isopropanol). Based on this trend, increasing the number of extraction stages or the amount of solvent in hexane extraction may be the most potential strategy to increase the yield of vitamin E in the extract concentrate.

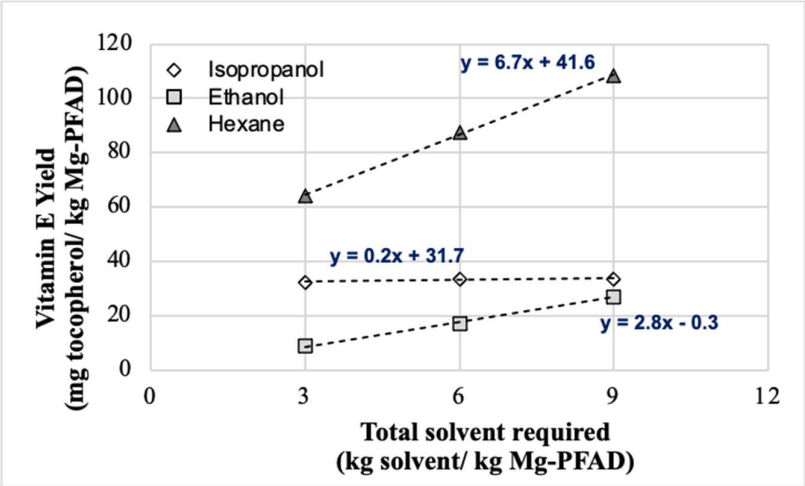

Figure 5 The yield of vitamin E in the extract concentrate as a function of total solvent required during one-stage extraction (3 kg/kg), two-stage extraction (6 kg/kg), and three-stage extraction (9 kg/kg).

The results in Figures 4 and 5 indicate that ethanol may not be a good option as a primary solvent to directly extract vitamin E from Mg-PFAD salts. However, ethanol may be a good option to pre-extract unsaponified FFA from Mg-PFAD prior to vitamin E extraction using hexane. Thus, both the yield and the vitamin E concentration of the total extract concentrate can be improved. Afterwards, further purification steps may still be required to improve the vitamin E concentration by removal of the remaining FFA and other unsaponifiable components using several alternative methods, such as molecular distillation [20], crystallization [21], or adsorption [22-26].

4 Conclusion

This study showed that the increase of the number of extraction stages proved to increase the recovery rate of vitamin E while lowering the vitamin E

concentration. The solvent polarity was inversely proportional to the vitamin E recovery rate from Mg-PFAD.

The cumulative recovery rate of vitamin E in the extract concentrate from the highest to the lowest was obtained after Mg-PFAD extraction with hexane, isopropanol, and ethanol, respectively. It is recommended to further investigate sequential extraction of Mg-PFAD by ethanol extraction followed by hexane extraction to increase both the yield and the concentration of vitamin E of the extract concentrate.

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