

# Effect of MgO to Fatty Acid Molar Ratio on the Production of Magnesium Salt of Fatty Acid from Palm Fatty Acid Distillates (PFAD) for Food Additives

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**Abstract.** Palm Fatty Acid Distillate (PFAD), a by-product of CPO industry, contains approx. 82%-wt of free fatty acids, which can be utilized as raw material for Magnesium Salts of Fatty Acids (Mg-PFAD). The objectives of the experiment were to produce Mg-PFAD salts through saponification-fusion reaction of PFAD at low temperature and ambient pressure and investigate the effect of MgO to fatty acid molar ratio on reaction conversion and yield. Next, washing Mg-PFAD salts with ethanol was able to facilitate the recovery of vitamin E (tocopherol and tocotrienol) from PFAD. Composition of Mg-PFAD were determined by AAS and GC analysis. Based on the data, yield of Mg-PFAD was increased by the increased of MgO to PFAD molar ratio. Mg-content of the product was within the standard according to FAO reference (4-5%-wt). Reaction was completed within 5-7 min after the addition of H<sub>2</sub>O (as catalyst).

## 1 Introduction

Indonesia is one of the largest Crude Palm Oil (CPO) producers in the world, where the production was reported to reach 33.4 million tons in 2015 [1]. Palm Fatty Acid Distillate (PFAD) is by-product of the CPO refining process from the deodorization unit, with a high fatty acid content of 82% and is estimated to be about 4% -w CPO [2]. This fatty acid can be utilized to produce metal salts, such as magnesium stearate, which is abundantly used in food and pharmaceutical industries as emulsifier, lubricants, anti-caking agent. In addition, thermal treatment in deodorization unit will transfer most of vitamin E content from CPO to PFAD. Reduced mass flow and massive stacking of vitamin E will increase the vitamin E content in PFAD up to 5 times higher than in CPO, which is about 4000-5000 ppm or about 0.5%-w [3]. This will increase the feasibility of vitamin E extraction from PFAD.

Metal salts are defined as products of reaction from saturated or unsaturated fatty acid with alkali, alkaline or transition metals. There are various methods usually applied for the commercial manufacture these metallic salts, namely decomposition process, fusion process,

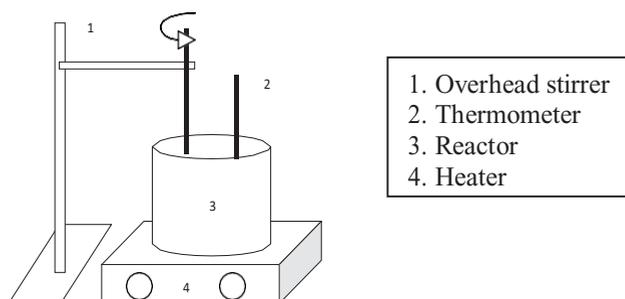
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and modified reaction process. The fusion reaction was known to have advantages such as the low temperature reaction and atmospheric pressure, fast reaction (3-5 minutes), and the possibility to utilize its exothermic reaction heat [4]. This research aims to investigate the effect of molar ratio between free fatty acid (FFA) in PFAD and MgO on reaction conversion and its magnesium salts' yield, which are substantial for process optimization at later stages.

## 2 Materials and Methods

This research adapted the method of Rogers patent (1959), US Patent number 2890232 for PFAD [5]. 100 grams of PFAD and 10.6 grams of MgO was mixed at 80 °C, then 3.2 grams of water was added while maintaining the heating process. The saponification reaction will occur at 98 °C and completed when the temperature reaches around 105 °C [6]. The experimental scheme of research is shown in Fig.1. In this experiment, we varied the molar ratio of fatty acids in PFAD to MgO, as shown in Table 1. Amount of water added as a catalyst was equally set of 3.2 grams in all variations.

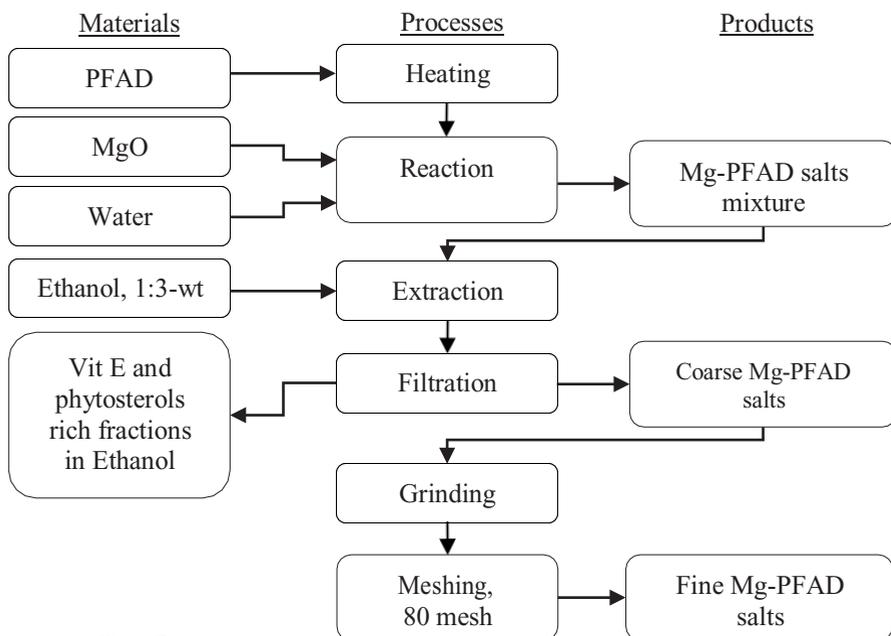


**Fig. 1.** Schematic experiment diagram

**Table 1.** Variation of molar ratio of fatty acids contained in PFAD and MgO

No.	Molar ratio of fatty acids : MgO	Temperature feeding of MgO
1	1.4 : 1.00*	80 °C [5]
2	2 : 1**	
3	1 : 1.00	
5	1 : 1.25	
6	1 : 1.50	

The flow diagram of experiments is shown in **Fig 2**. Reaction yield was calculated based on the reaction's mass balance. Atomic Absorption Spectroscopy (AAS) was used to measure Mg content of Mg-PFAD salts and Gas Chromatography – Mass Spectrometry (GC-MS) was used to observe Mg-PFAD salt formed as well as its quantity. The data from GC-MS was showed as percent area detected related to the percent composition of the sample.



**Fig. 2.** Experiment Flow Diagram

### 3 Results and Discussions

#### 3.1 Palm Fatty Acid Distillate Analysis

Table 2 presents that PFAD used in this experiment has PFAD content of 94.35%, which was obtained from acid value analysis. Later, it was required to determine the conversion of reaction, through Equation 1. Most types of FFA content of PFAD are palmitic and oleic acid by 51.38% and 39.85% consecutively.

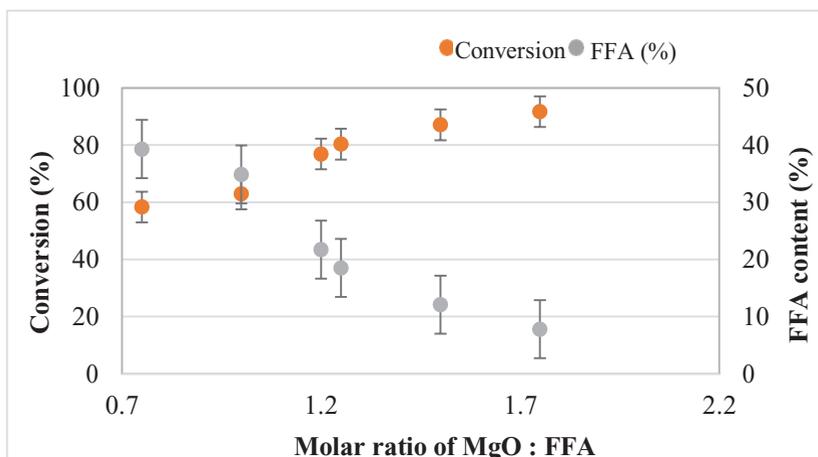
**Table 2.** Fatty Acid Composition of PFAD

	Fatty acid content (%)
PFAD	94.35 ± 0.35 (AV = 198.2 mg KOH/ g sample)
Palmitic acid	51.38
Linoleic acid	4.44
Oleic acid	39.85

The number of FFA in PFAD obtained from different by-products of CPO processing may vary. FFA composition in PFAD may range from 65.50% to 95.55% depends on the quality of purified CPO and the process condition of refining.

### 3.2 Effect of FFA to MgO molar ratio on fatty acid conversion and Mg-PFAD salts yield

Figure 3 shows the effect of FFA to MgO molar ratio on reaction yield. From this experiment, the reaction yield ranges from 88-92% for FFA to MgO molar ratio of 1:1 to 1:1.5.



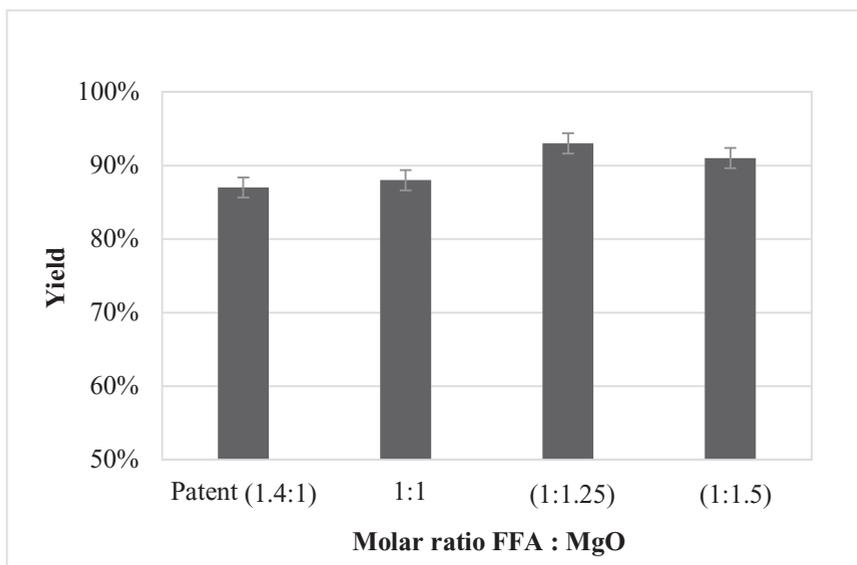
**Fig. 3** Profile of reaction conversion and FFA percentage on different MgO to FFA molar ratio

Based on the results of the experiment (**Fig.3**), it was conducted an analysis of factorial design of percent reaction conversion against the ratio of moles of MgO to PFAD. Based on the analysis of the data, the molar ratio of feed (A) greatly affects the conversion of reaction (with a P-value of 0.000), whereas the initial temperature of the reaction (B) and the interaction between the A&B has no effect against a conversion reaction (with P-value 0.931 and 0.961). The largest conversion obtained by conducting molar ratio of MgO : FFA as 1:1.75, when MgO molar ratio excess of stoichiometric requirement. Excess MgO suspected to drive the reaction to the right (product side) so that the magnesium salts products increased and thus reduced the levels of FFA content in the product. It can be seen that the more the MgO fed, then the lower the conversion reaction. However, the conversion of 91.75% did not meet the standard commercial magnesium stearate product because it still has FFA of 7.79%. Based on Ph Eur 8th Ed 2014, magnesium stearate must meet the limits of maximum FFA content by 4% or less [7]. Therefore, further processing is required to reduce the content of FFA in the final product.

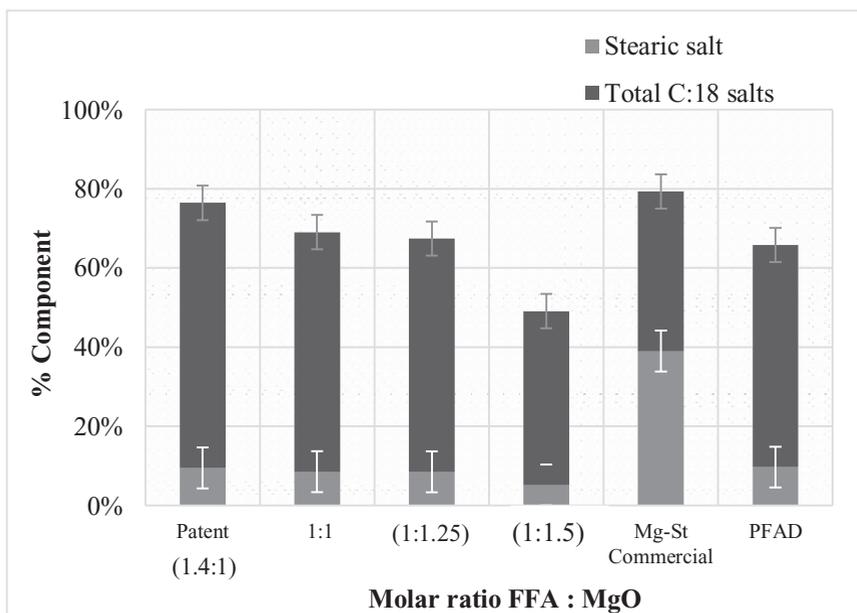


### 3.3 Effect of FFA to MgO molar ratio on Mg-PFAD salts composition

Mg-PFAD salt composition represents characteristic of the salt and determines its properties and functionality. Fatty acid composition in Mg-PFAD salts showed similar composition to fatty acid composition on PFAD (Fig. 4). Briefly, PFAD is composed by low content of stearic acid (C18:0) (4-5%) and higher content of palmitic acid (C16:0), oleic acid (C18:1), linoleic acid (C18:2) and linolenic acid (C18:3) [8]. Furthermore, the result showed that FFA to MgO molar ratio had no significant effect on Mg-PFAD salts composition.



**Fig. 4.** Reaction yield on different FFA to MgO molar ratio



**Fig. 5.** Mg-PFAD salts composition on different FFA to MgO molar ratio

### 3.4 Effect of FFA to MgO molar ratio on Mg content of Mg-PFAD salts

Fig. 6 shows magnesium content in Mg-PFAD salts on different FFA to MgO molar ratio. Based on this result, FFA to MgO molar ratio showed no significant effect on Mg content of Mg-PFAD salts.

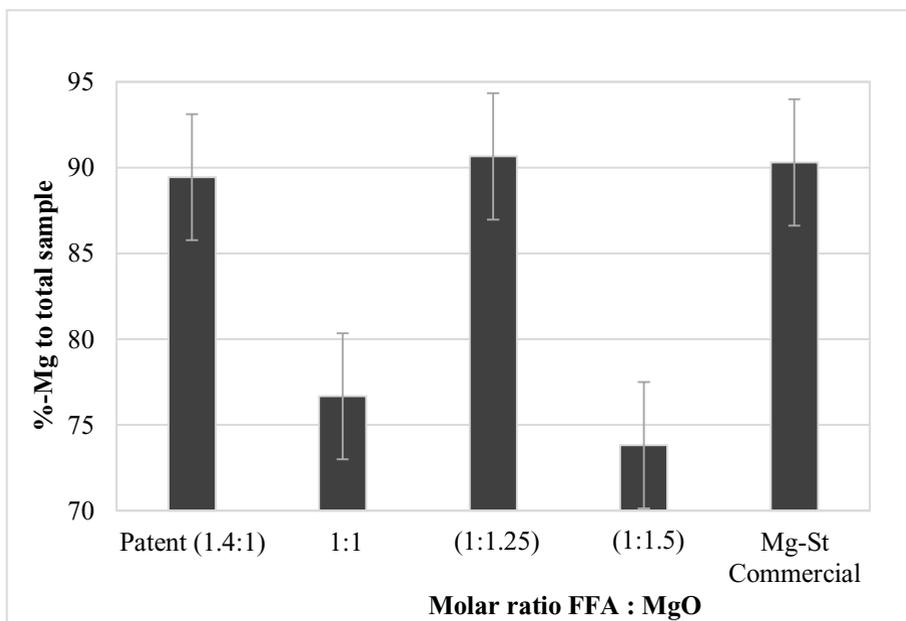


Fig. 6. Mg content in Mg-PFAD salts on different FFA to MgO molar ratio

## 4 Conclusions

Based on the experimental data, we conclude that fatty acid to MgO molar ratio affect reaction yield and composition of Mg-PFAD salts. Mg-content of the product was within the standard according to FAO reference (4-5%-wt). Reaction was completed within 5-7 min after the addition of H<sub>2</sub>O (as catalyst).

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