Effect of Chemical Reaction on Unsaturated Content in Palm Kernel Oil

[1]Siti Amira Hamdan, [2]Dr.Nurul Fadhilah Kamalul Aripin

Faculty of Chemical Engineering, Universiti Teknologi Mara

***Abstract*—Palm kernel oil (PKO) is edible oil extracted from kernel of palm fruit which contain mainly lauric oil. Glycosides produced from lipids in PKO by covalent bonds are recommended ingredients to produce bio-surfactant due to its membrane behavior. This research is to determine the unsaturated contents in PKO that undergoes the glycosylation process to produce glycosides. The hypothesis of the research is that the unsaturated compounds are not affected during the processes. The method to identify the component in PKO is divided into two which are the wet experiment and analytical experiment. To determine the Iodine Value (IV), wet experiment was applied by used iodine titration. While, to identify the component in PKO will used Gas Chromatography Mass Spectroscopy (GC-MS) and Nuclear Magnetic Resonance (NMR). The result shows that unsaturated content in PKO and PKO-OH is similar based on the IV, GC-MS, NMR analyses. It concluded that reduction process does not affect the unsaturated content.**

***Keywords*— Palm kernel oil, Iodide titration, GCMS, NMR**

INTRODUCTION

Palm kernel oil (PKO) is edible oil extracted from kernel of palm fruit of tropical palm tree and total yield of PKO only 10% from palm fruit produced (Norizzah, et al., 2014).The PKO has high saturated fat which contain mostly lauric oil. PKO does not contain carotene, and contains less of mono-unsaturated fats and polyunsaturated fats than palm oil (Ibegbulem & Belonwu, 2014). The characterization of PKO is it will be in solid form at ambient temperature and change into liquid at tropical temperatures (OE, HO, & AA, 2016). PKO is one of starting point of materials to get the glycoside component.

Glycoside is one of the important components in membrane behavior due to its amphiphilic structure. For example, galactosides are one of the glycosides that produced from fatty acids of PKO (Yusoff, Aripin, Aznan, & Salleh, 2018). The applications of glycosides are bio-surfactants, viscosity enhancer and detergent properties. Glycosides also used as a component for drug deliveries due to its antioxidant and antimicrobial activities, sugar-based bio-surfactants. Liposomes are one of innovative bio-surfactants formulated from glycosides (Faivre & Rosilio, 2010).

In this research, the PKO had undergoes the glycosylation process to produce glycoside. The objectives are to determine unsaturated composition of PKO that undergoes glycosylation process and to compare the effectiveness between gas chromatography mass spectroscopy (GC-MS) and nuclear magnetic resonance (NMR). The PKO will be determined the unsaturated contents by iodide titration from Tang Shin Sue (Tang Thin Sue, 2011), identification of components by GC-MS from M. et al. (M. & Thangapandian, 2012) and analyze the lipids components by NMR adopted from Aripin et al. (Aripin, Park, & Park, 2012).

METHODOLOGY

1.1. Materials

Palm kernel oil is obtained from Golden Jomaline Food Industries Sdn. Bhd. palm kernel oil alcohol is produced by reduction of $LiAlH_4$ method. 0.1M Wijs solution, potassium iodide, 0.1M sodium thiosulphate, glycial acetic acid, methanol, diethyl ether, hexane, and cyclohexane were purchasef from Merck. Methanol d-4 was purchased from Sigma Aldrich (USA).

1.2. Determination of iodine value (IV)

0.05g of sample was weighted and dissolved in 4 ml of solvent mixture of cyclohexane and glacial acetic acid then followed by 6.5ml of 0.1M Wijs solution. Later, the solution was kept in a dark place for 1 hour for the reaction to happen. After an hour, 5 ml of potassium iodide solution and 25 ml distilled water were added into the solution. Lastly, the sample was titrated with 0.1M sodium thiosulphate until it turned to a pale yellow color solution. Blank was prepared for calibration purposes. The IV is calculated by using equation below (Standard Test Method for Determination of the Iodine Value of Fats and Oils, 2006):

$$\mathrm{IV} = \frac{12.69\mathrm{N}(\mathrm{B}-\mathrm{S})}{\mathrm{W}}$$

N = Molarity of sodium thiosulphate solution used.
B = Volume in mL of sodium thiosulphate solution used for blank test.
S = Volume in mL of sodium thiosulphate used for sample test.
W = Weight in gram of sample.

1.3. GC-MS preparation

Samples were quantified by gas chromatography using the 450 GC/220 MS system (Varian. Inc, USA) and 5975C inert MSD with triple-Axis detector (Agilent Technology, USA). HP-5ms was used as column in GC/MS (5%-diphenyl, 95%-dimethylpolysioxane, 30m x 0.25mm ID x 0.25µm). The temperature programs was set up from 50°C to 250°C with 5°C/min, both the injector and detector temperatures were 280°C and He was used as carrier gas. The injection volume was 2µL. Ionization energy EI of 70 eV was used for mass spectroscopy detector.

1.4. NMR preparation

Samples were measured on JOEL NMR at 400 MHz. Samples were analyzed in deutro-chloroform and the final product were dissolved in methanol-d4 at room temperature with 400MHz.

RESULTS AND DISCUSSION

1.1 Iodine Value

The iodine value shows the amount of unsaturated compound in PKO and PKO-OH. Iodine Value (IV) is a parameter that indicates the potential of fat to be oxidized as it measure the reaction of iodine with double bonds of unsaturated fatty acids (Iodine Value, 2007). The higher the iodine value, the higher the unsaturated contents in samples. The standard range of iodine value for PKO is from 17 to 23 (Gras, 1992) .Table 1 shows the result of iodine value by titration with 2 repeated experiments.

Table 1 The IV for unsaturated contents in samples

Sample	Iodine Value	Consistency, %
Blank	12	-
PKO	19 ± 3	13
PKO-OH	19 ± 3	15

According to Table 1, the iodine value shows similar result samples PKO and PKO-OH. This confirms the fact that the unsaturated content is not affected by reduction process. The obtained iodine values are justified within the range of iodine value for PKO as reported.

1.2 GC-MS

The sample was analyzed by GC-MS to identify the component in the sample. Figure 1 show the spectrum of the component inside the reduced PKO.

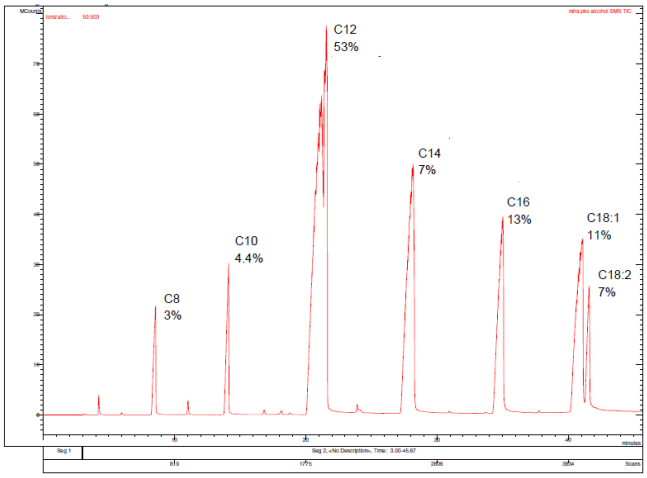

Figure 1 Chromatogram of reduced PKO by GC-MS

The chromatogram in figure 1 shows that the peak of unsaturated contents in reduced PKO is identified. According to the graph, the unsaturated compound is determined as 11% in the reduced PKO. This value is consistent with the reported amount of oleic acid in PKO (Mancini, et al., 2015). The comparison of PKO cannot be done because the unavailability of the analysis due to time constrain.

1.3 NMR

Another analysis performed to determine the unsaturated content in PKO and PKO-OH samples is NMR. Figure 2 and 3 shows the spectrum of PKO and PKO-OH respectively.

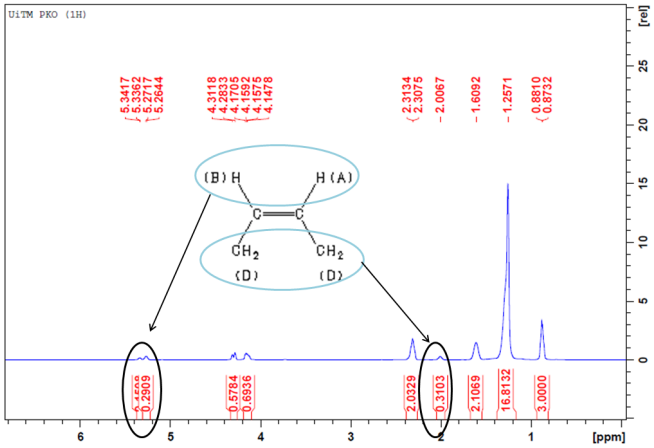

Figure 2 Spectrum of component of PKO by NMR

The unsaturated compounds are represented by peaks observed at 2 and 5.2 ppm. There is about 7.8% of unsaturated component is identified in PKO which is determined by peak integration.

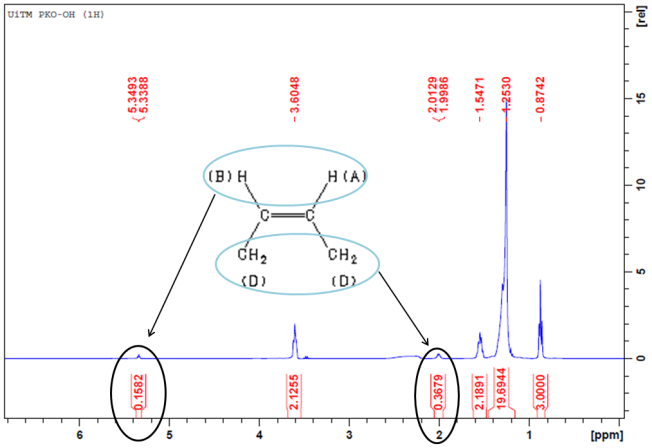

Figure 3 Spectrum of component reduced PKO by NMR

Similar to figure 2, unsaturated compounds are represent by peaks observed at 2 and 5.2 ppm. There is about 9.3% of the unsaturated compound was determined by peak integration in figure 3. The unsaturated compound in PKO and reduced PKO should be similar. The results show that unsaturated compound in PKO and reduced PKO is slightly difference. This is due to the integration is not done properly during the analysis.

CONCLUSION

The result shows that unsaturated content in PKO and reduced PKO is similar based on the IV, GC-MS and NMR analyses. We concluded that reduction process does not affect the unsaturated content.

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