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EFFECT OF HEATING ON LARD ADULTERATION IN RBD PALM OIL USING GAS CHROMATOGRAPHY AND CHEMOMETRICS

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Abstract

Adulteration has gained much concern in the oils and fats industry due to health and religious issues. This study assesses the effect of heating on the profiling of lard (15% and 30% lard) spiked in refined bleached deodorized (RBD) palm oil at 120 oC, 180 oC and 240 oC for 1, 2 and 3 hours. The volatile compounds released were identified using (gas chromatography mass spectrometry headspace) GC-MS-HS method. Multivariate data from GC-MS-HS were mean centred prior to Principal Components Analysis (PCA) using Unscrambler software. The result obtained from the scores plot for 30% lard and 15% lard in the RBD palm oil showed the same pattern for heating temperature at 120 oC and 180 oC. At 240 oC, both sample and control were scattered in the scores plot. However, the GC-MS-HS technique did not differentiate between 0% lard with 15% or 30% lard in RBD palm oil.

Keywords: Adulteration, Heating effect, Lard profiling, GC-MS-HS, PCA

INTRODUCTION

The increasing number of Muslim population by over 23.5 percent in the last fifty years showed that Islam is the fastest growing religion in the world. As food become abundant and easily accessed, the authenticity of halal food is a major concern among the Muslim consumers throughout the world. In the food industry, food adulteration practice is a common issue which resulted in uneasiness amongst consumers belonging to certain religion group (Nurrulhidayah et al., 2015). Islam is a universal and complete religion as it covers the whole aspects of Muslims life such as law, muamalah as well as food. In Al-Quran Allah said:

"Therefore eat of what Allah has given you, lawful and good (things), and give thanks for Allah's favour if Him do you serve". (An-Nahl: 114),

Many issues were raised by the public as well as the authority regarding the authenticity of halal products. Due to the awareness as the Muslim population have grown rapidly, these issues have gained attention globally (Mansor et al., 2012). In Asean country, vegetable oil such as Refined Bleached Deodorized (RBD) palm oil is an economically important cooking product due to its excellent storage stability and storage quality. However, there was a report that claimed food products such as vegetable oil was also involve in the contamination and adulteration of lard. (Marikkar et al., 2005).

Although Aishah (2015) has proposed the use of gas chromatography flamed ionisation (GC-FID) technique for the detection of lard in oil, it needs sample preparation to convert the fatty acid to FAME which is time consuming and costly. In the analysis of volatile fractions in many food samples, headspace sampling is the technique of gas extraction that has been reviewed as a rapid and efficient technique (Lorenzo et al., 2002). Therefore, it is cheaper and faster as no prior sample preparation steps are required.

The abundance of recycle cooking oil in the market nowadays is an important factor that needs to be considered when consumer decided to buy the recycle oil. There might be contamination from the previous food that is haram or shubhah in the cooking oil. Therefore, a simple and quick method is needed to screen and detect the presence of that particular food (ie: lard). Hence, the objective of this study was to analyse the pattern of volatile compounds for different percentage of lard (0%, 15%, 30%) in RBD palm oil when heated at three different temperatures (120 ^{o}C, 180 ^{o}C, 240 ^{o}C) for 1, 2 and 3 hours. This method was a preliminary investigation and served as a basic method develop more accurate techniques. Different heating temperature would give different levels of the same volatiles compounds. Chen et al. (2002) reported that significant difference ($p< 0.05$) were found for volatile compounds in lard when roasted at 150 ^{o}C and 200 ^{o}C.

MATERIALS AND METHODS

Materials

The RBD palm oil and sample of pig's adipose tissue were purchased from a local supermarket at Nilai, Negeri Sembilan. Adipose tissue was stored at -20 ^{o}C prior to the analysis.

Sample Preparation

Lard sample was extracted according to Rohman and Che Man (2009) by rendering adipose tissue in a conventional oven (Pensonic AE-11N) at 100 ^{o}C for 2 hours. The melted fat was filtered through Whatman filter paper and dried by the addition of anhydrous Na_2SO_4 to remove the water content. The

filtered samples were stored in a tightly closed container in a refrigerator until further analysis. The RBD palm oil was adulterated with lard at various percentages (0%, 15% and 30%) in w/w.

Heating Procedure

All adulterated oils were heated at 3 different temperatures (120 ^{o}C, 180 ^{o}C and 240 ^{o}C) using a digital hotplate (Daihan, Korea) with a controlled temperature probes for 3 hours. Samples were taken at 1, 2 and 3 hours and cooled to room temperature before kept in a tightly closed and seal universal bottles in a refrigerator until further analysis.

Analysis Using Gas Chromatography Mass Spectrometer with Headspace Analyser (GC-MS-HS)

The method for determining the volatile compounds was done according to Lorenzo et al. (2002). For the analysis of volatile compounds, 4.0 ml of each oil samples was placed into 10 ml vials sealed hermetically with a cap. The experimental conditions of the head space sampler were as follows; oven temperature: 120 ^{o}C; loop temperature: 130 ^{o}C. Transfer line temperature: 135 ^{o}C and headspace generation time: 30 min. The mass range measured between in the mass spectrometer was 35-100. The carrier gas was helium, at an approximate flow rate of 20 ml/min. The mass spectra obtained were compared to the NIST Mass Spectral Search Program for compound identification. The peak areas were selected for further data analysis using chemometrics technique.

Statistical Analysis

Unsupervised multivariate analysis, principal component analysis (PCA) was performed by mean centred on data of GC-MS-HS using Unscrambler Software (X10.3) version.

RESULTS AND DISCUSSION

GC-MS-HS was used to analyse the volatile compounds released in sample a (0% lard in RBD palm oil), b (15% lard in RBD palm oil) and c (30% lard in RBD palm oil) after heating at 120 ^{o}C, 180 ^{o}C and 240 ^{o}C for 1, 2 and 3 hours. The results obtained between different percentages of lard sample and control were then compared.

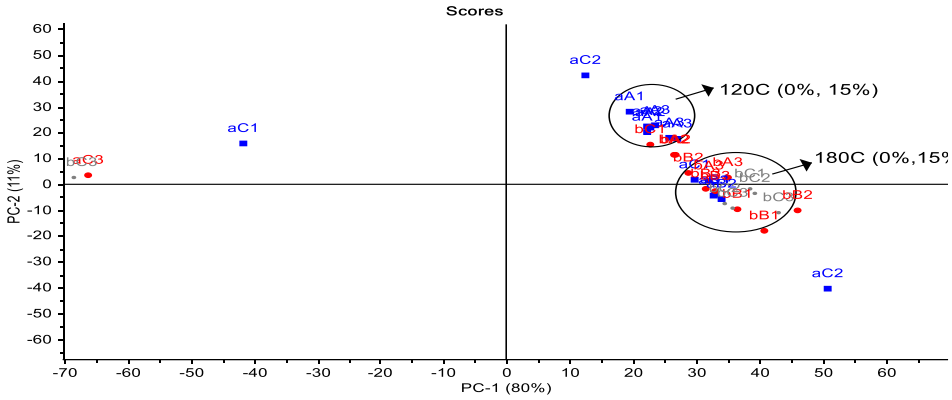

Figure 1. The Score Plot for 0% and 15% Lard Spiked in RBD Palm Oil Heated at 120 °C and 180 °C

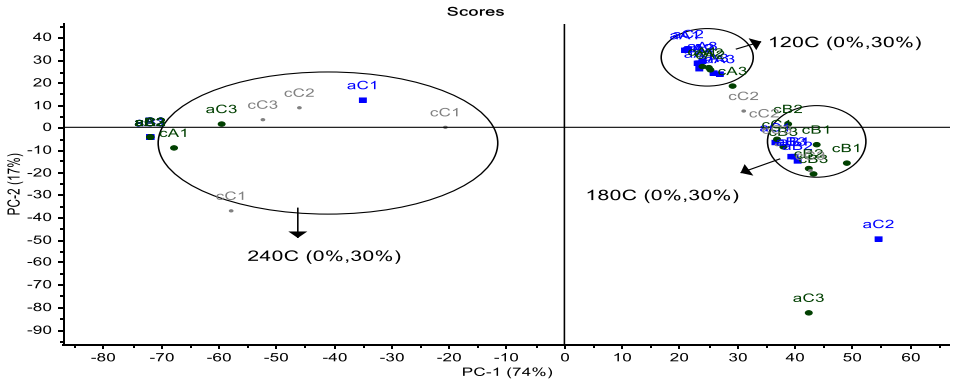

Figure 2. The Score Plot for 0% and 30% Lard Spiked in RBD Palm Oil Heated at 120 °C, 180 °C and 240 °C.

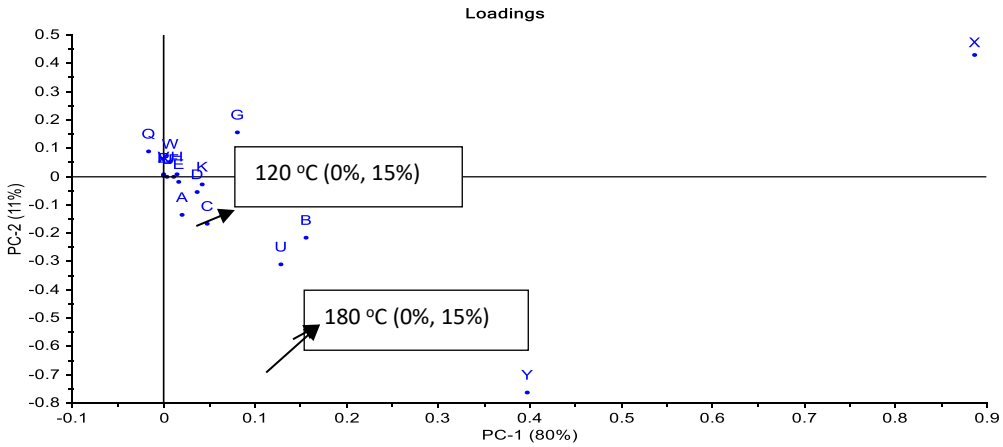

Figure 3. The loadings plot for 0% and 15 % lard spiked in RBD palm oil heated at 120 °C and 180 °C.

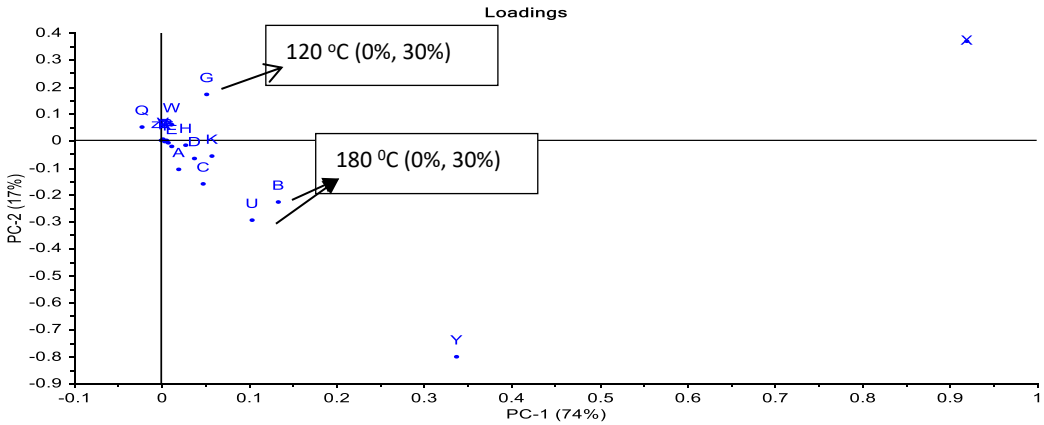

Figure 4. The loadings plot for 0% and 30% lard spiked in RBD palm oil heated at 120 °C and 180 °C.

Figure 1 shows the scores plot for 15 % lard spiked in RBD palm oil when compared with control sample (0 % lard in RBD palm oil). Samples were found to be accumulated based on the difference in their heating temperature. The same pattern in figure 2 was observed when 30% lard was spiked into RBD palm oil. The closer the samples in scores plot, the more similar they are with respect to the two components concerned. Samples and controls were closely clustered according to their heating temperatures at 120 °C and 180 °C. This cluster showed that samples contain lard (15% and 30%) could not being distinguished with control at 120 °C and 180 °C. At 240 °C, samples and controls were scattered and not clustered to each other in the scores plots. This might be due to the character of volatile compounds that were affected by high heating temperature. Since volatile compounds were influenced by fatty acids, this findings was correlated with Aishah (2015) that found that at 240 °C (maximum heating time was 3 hours), there were no difference in fatty acids between sample (contain lard) in palm oil and control. Moreover, Chen et al. (2002) also claimed that there was a significant difference between volatile compounds from pork jerky when roasted at 150 °C and 200 °C for 2 minutes.

A total of 21 compounds were tentatively identified and detected by GC-MS-HS based on matches with library mass spectral data. Based from loadings plot in figure 3 and figure 4, only 5 variables differ significantly from the other variables which were hexanal (marked as G), nonanal (marked as B), octane (marked as U), pentane (marked as X), and heptane (markes as Y) while other variables were lying closed to the centre. Variables that are close to each other in the loadings plot will have a high positive correlation if the two components explain a large portion of the variance. In figure 3 and figure 4, variables G was correlated with heating temperature 120 °C while variables B and U was correlated with heating temperature 180 °C. No variables were

correlated with heating time 240 °C. Aishah and Sukri (2015) analyzed the fatty acids in lard by gas chromatography flame ionization (GC- FID) method. They found that fatty acids linolelaidic acid methyl ester (C18:2n6t), oleic acid methyl ester (C18:1n9c) and palmitic acid (C16:0) were observed in all loading plots. However, as the heating time increased, other fatty acids were centred in the middle and located closed to each other.

Production of volatiles oxidation compound were greatly influenced by fatty acid composition of oil. According to Rohman et al. (2012), the main fatty acid compositions for lard are from palmitic (C16:1), stearic (C18:0), oleic acid (C18:1) and linoleic acids (C18:2). Meanwhile in the palm oil, the main fatty acids are myristic (C14:0), palmitic (C16:0), stearic (C18:0), oleic (C18:1) and linoleic acids (C18:2). (Koushki et al., 2015) Aldehydes compounds such as nonanal, E-2-decenal and 2-undecenal are being correlated with oleic acid. While hexanal and 2,4-decadienal are being correlated with linoleic acid. Hexanal is also a source of fatty aroma and a typical oxidation volatile from linoleic acid (Stahnke, 1994). Meanwhile hydrocarbon such as heptane and octane was from oleic acids and pentane was from linoleic acids. (Frankel, 1985).

CONCLUSION

The characteristic of volatile compounds were clustered according to the heating temperature. However, based from the result of this research, for RBD palm oil heated at 120 °C, 180 °C and 240 °C, GC-MS-HS method could not distinguished between RBD palm oil containing lard (15% and 30%) and without lard.

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