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Transesterification of triglycerides from chicken fat and lard using base catalysts and its discriminant analysis

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ABSTRAK

Keamanan makanan merupakan suatu yang dibutuhkan oleh orang muslim termasuk kehalalannya. Salah satu metode yang digunakan untuk nalisis halal adalah kromatografi gas spektroskopi masa (KG-KS). Penggunaan KG-KS diaplikasikan pada senyawa yang memiliki karakteristik sifat mudah menguap. Oleh karena itu, trigliserida harus diubah menjadi metil ester melalui proses transesterifikasi. Pada penelitian, kita menggunakan katalis basa dalam proses tersebut karena katalis tersebut dapat bereaksi lebih cepat dibandingkan katalis asam. Katalis basa yang digunakan adana K_2CO_3 dan NaOH sebagai katalis heterogen dan homogen. Konsentrasi kedua katalis (K_2CO_3 dan NaOH) yang digunakan dalam proses transesterifikasi berturut-turut sebesar 2 mg/mL and 1 mg/mL. Selanjutnya, asam lemak yang didapatkan dianalisis menggunakan kemometrika untuk membedakan lemak ayam dam lemak babi. Hasil penelitian menunjukkan bahwa kedua katalis mampu menghasilkan kesamaan asam lemak dominant dari lemak ayam dam lemak babi akan tetapi konsentrasi asam lemaknya berbeda. Katalis K_2CO_3 telah menghasikan beberapa produk metil ester dari lemak babi seperti asam oleat, asam palmitat, asam linoleat dan asam stearat dengan kadar relatifnya berturut-turut 48,62%, 21,38%, 9,08%, dan 11,18% sedangkan katalis NaOH dapat menghasilkan kadar asam lemak berturut-turut sebesar 49,60%, 21,83%, 9,12%, dan 11,18%. Selanjutnya, metil ester dominan dalam lemak ayam dari katalis K_2CO_3 dan NaOH adalah asam oleat dan asam palmitat, dimana katalis K_2CO_3 menghasilkan kadar relatif sebesar 43.73% dan 22.82%, sedangkan katalis NaOH sekitar 53.52% dan 32.00%. Analisis diskriminan telah menunjukkan bahwa lemak babi dan lemak ayam dapat dikelompokkan berdasarkan kandungan asam lemaknya yang dikatalisis menggunakan K_2CO_3 dan NaOH. Karenanya katalis K_2CO_3 dan NaOH dapat digunakan sebagai katalis basa dalam proses transesterifikasi untuk analisis kehalalan. Berdasarkan hasil yang diperoleh maka dapat disimpulkan bahwa katalis NaOH lebih baik dibandingkan katalis K_2CO_3 dalam proses transesterifikasi pada trigliserida dalam lemak ayam dan lemak babi.

Kata Kunci: asam lemak, transesterifikasi katalis basa, analisis diskriminan

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ABSTRACT

The food security is urgently needed by Muslims, including halal security. One of the methods performed for halal analysis is Gas Chromatography-Mass Spectroscopy (GC-MS). The volatile compound is the specific characteristic that supported halal analysis using GC-MS. Therefore, the triglycerides in lard or oils must be converted to become fatty acid methyl ester (FAME) through the transesterification process. In this research, we used base catalysts in the transesterification process because it can react faster than acid catalysts. The base catalysts chosen are K_2CO_3 and NaOH that depict heterogeneous and homogenous catalysts. Both K_2CO_3 and NaOH levels used in the transesterification process are 2 mg/mL and 1 mg/mL, respectively. Furthermore, the fatty acids obtained were analyzed using chemometrics to distinguish chicken fat and lard. The result showed that both catalysts can produce the same dominant fatty acid from lard and chicken but the levels of the resulted fatty acids are different. The K_2CO_3 catalyst resulted methyl ester products from lard such as oleic acid, palmitate acid, linoleic acid, and stearic acid which are 48.62%, 21.38%, 9.08%, and 11.18%, respectively for the, while the NaOH catalyst resulted in about 49.60%, 21.83%, 9.12%, and 11.18%, respectively. Furthermore, The dominant FAMEs in chicken fat using K_2CO_3 and NaOH catalysts were oleic acid and palmitic acid, where K_2CO_3 catalysts produced 43.73% and 22.82%, respectively, while NaOH is about 53.52% and 32.00%, respectively. The discriminant analysis showed that lard and chicken can be grouped based on the compositions of fatty acid catalyzed using both K_2CO_3 and NaOH catalysts. Hence, K_2CO_3 and NaOH can be used as base catalysts in the transesterification process for halal analysis. Based on the results were also concluded that NaOH catalyst is better than K_2CO_3 catalysts in the transesterification process of triglycerides from lard and chicken fat.

Keywords: fatty acids, transesterification, base catalyst, discriminant analysis

INTRODUCTION

Nowadays, the forgery cases often occur in food products by replacing or mixing the halal material with haram source. These cases have happened due to the producers want to reduce production prices and to get high profits. The case was detected in Salatiga that 13 meatball products analyzed, one product has contained pork (Fibriana *et al*., 2012). Hence, to protect the welfare and health of consumers, halal food products must be ensured. One method is commonly used to detect the pork adulteration is Gas Chromatograph (GC). The research by Suparman *et al.*, (2015) reported that the Gas Chromatograph-Mass Spectroscopy (GC-MS) can be used for the halal analysis of the imported chocolate products. The requirements of compound properties that can be analyzed using GC-MS are the volatile compounds. While the compounds do not have volatile properties, the compounds should be derived through the derivatization process. For triglycerides, the derivatization process was known as a transesterification process.

Transesterification is a derivatization process of triglycerides to produce other esters as fatty acid methyl esters (FAME) form. This derivatization process usually uses the catalyst such as H_2SO_4 and ethanol (Batti *et al*., 2008), BF_3 in methanol (Kustyawati *et al.*, 2012), or a combination of 2,4-dibromoacetophenone and triethylamine (Czauderna *et al*., 2002; Czauderna *et al*., 2008). In general, transesterification is carried out by the addition of an acid or base catalyst. The advantage of the base catalyst is more effective in the transesterification if this process is not containing the free fatty acids (ALB) above 0.5% (Shu *et al*., 2010). Whereas homogeneous acid catalysts such as H_2SO_4, HCl, and

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H_3PO_4 require a long reaction time, and the large molar ratio of alcohol to oil, and need high temperatures (Helwani *et al*., 2009).

The research from Batti *et al*., (2008) showed that the FAME conversion using 1% sulfuric acid (H_2SO_4) catalyst reached around 98.4%. While, the base catalyst is also resulting in high yields (97% or more) (Issariyakul and Dalai, 2014) and these catalysts are most commonly used in the laboratory and industrial scales (Noureddini *et al*., 2005). Hence, this study investigated the influence of K_2CO_3 and NaOH catalyst of pork and chicken fat as triglycerides from halal or haram source to find good catalyst for transesterification process. Besides that, this research also evaluated the effectivity of these catalysts to distinguish of pork and chicken fat based on the fatty acid compositions produced from the transesterification process.

MATERIALS AND METHOD

Methods

The Extraction of Fat From Chicken and Pork

Meat (pork and chicken) samples as much as 100 grams were extracted using Soxhlet for ± 6 hours using n-hexane solvent. The mixtures of oil and n-hexane were added with Na_2SO_4 to remove the water and then evaporated to get pure oil (Sari and Guntarti, 2018). Next, oil is put in a bottle and stored in the refrigerator.

The fat transesterification using the base catalyst

Transesterification process of each sample (Fat samples from pork or chicken) following Rohman *et al.*, (2012) with slight modifications. 25 mg samples were added and mixed with n-hexane up to 25.0 ml. Each oil sample was taken as much as 3 ml, put into a test tube and added 50 mg K_2CO_3 or 3 ml $NaOCH_3$ 0.2 N. A tube containing K_2CO_3 catalyst was added with 3 ml methanol. All samples were vortexed for 4 minutes, then heated at 80ºC for 40 minutes. The mixture was added with saturated NaCl as much as 1.5 ml then centrifuged until 2 layers formed. The upper layer was taken as much as 1 µl solution and injected into the GC-MS instrument.

Data Analysis

GC-MS chromatogram data were processed with Minitab 15 English software. The relative levels of fatty acids in both fats were used for discriminant analysis between the pork and chicken meat based on the fatty acid compositions.

RESULT AND DISCUSSION

The transesterification of lard using NaOH and K_2CO_3 catalysts produced the same dominant fatty acid types. However, the resulted fatty acids from K_2CO_3 catalyst were lower concentration than NaOH catalyst. K_2CO_3 catalyst resulted oleic acid, palmitic acid, stearic acid, linoleic acid, stearic acid, and linoleic acid which are about 48.62%, 21.38%, 11.18%, 21.38%, 11.18%, and 9.08%, respectively, while NaOH catalyst produced around 49.60%, 21.83%, 11.18%, 21.83%, 11.18%, and 9.12%, respectively (Table 1).

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Table 1. Fatty acid content in lard hydrolyzed using K_2CO_3, and NaOH catalyst

RT (Min)	Molecule Formula	Fatty Acid Types	Relative Level (%)	
			K_2CO_3	NaOH
28.95	$C_{12}H_{24}O_2$	Lauric acid	1.54	1.40
33.75	$C_{14}H_{28}O_2$	Myristic acid	2.77	2.66
37.69	$C_{16}H_{30}O_2$	Palmitoleic acid	2.00	1.76
38.12	$C_{16}H_{32}O_2$	Palmitic acid	21.38	21.83
39.69	$C_{17}H_{32}O_2$	9,10-methylenehexadecanoic acid	0.31	0.22
40.11	$C_{17}H_{34}O_2$	Margaric acid	ND	0.28
41.54	$C_{18}H_{32}O_2$	Linoleic acid	9.08	9.12
41.66	$C_{18}H_{34}O_2$	Oleic acid	48.62	49.60
42.06	$C_{18}H_{36}O_2$	Stearic acid	11.18	11.18
44.65	$C_{20}H_{32}O_2$	Arachidonic acid	ND	ND
45.27	$C_{20}H_{38}O_2$	Eicosenoic acid	0.64	1.77
45.69	$C_{20}H_{40}O_2$	Arachidic acid	ND	0.51

RT= Retention time, ND = Not Detected

The transesterification process using NaOH and K_2CO_3 catalysts will produce FAME types through enolate ion formation. This ion is a reactive nucleophile that attacks the carbonyl atoms on triglycerides where this reaction will take place more quickly and produces high the FAME yields. Helwani *et al.*, (2009) reported that base catalysts are preferred over acidic catalysts because they require a long reaction time and high temperatures, and also cause corrosion in the reactor.

Table 2. Fatty acid content in chicken fat hydrolyzed using K_2CO_3, dan NaOH catalyst

RT (Min)	Molecule Formula	Fatty Acid Types	Relative Level (%)	
			K_2CO_3	NaOH
33,77	$C_{14}H_{28}O_2$	Myristic acid	1.43	1.00
37,88	$C_{16}H_{30}O_2$	Palmitoleic acid	1.28	4.01
38,10	$C_{16}H_{32}O_2$	Palmitic acid	22.82	32.00
39.69	$C_{17}H_{32}O_2$	9,10-methylenehexadecanoic acid	4.82	1.13
41.62	$C_{18}H_{34}O_2$	Oleic acid	43.73	53.52
42.06	$C_{18}H_{36}O_2$	Stearic acid	7.52	9.47
43,30	$C_{18}H_{34}O_2$	7, 10-octadecanoic acid	9.63	ND

RT= Retention time, ND = Not Detected

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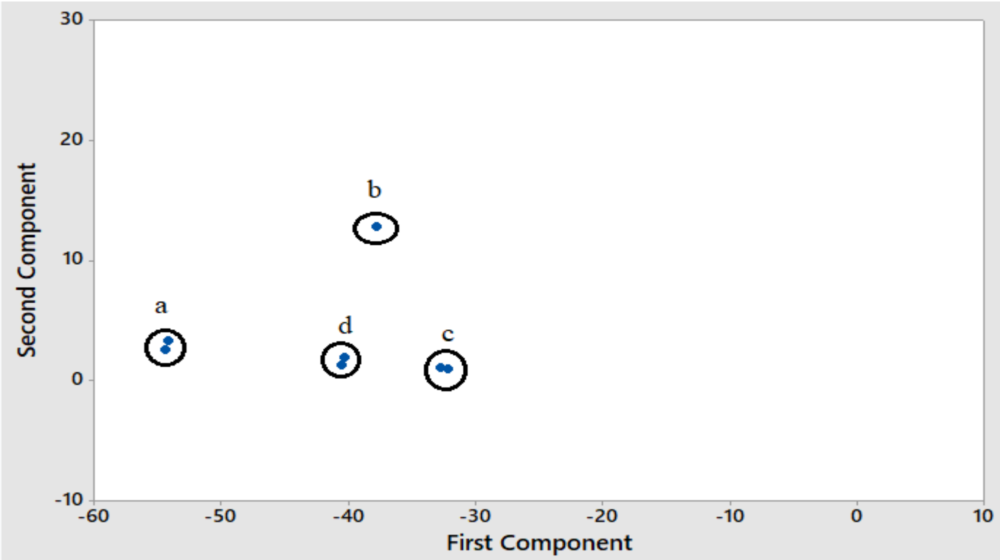

Figure 1. Score plot of Lard and Chicken Fat based on GC-MS Chromatrogram using NaOH and K_2CO_3 catalysts: a: Chicken Fat (NaOH), b: Chicken Fat (K_2CO_3), c: Lard (NaOH), and d: Lard (K_2CO_3)

Moreover, the use of the alkaline catalyst in the transesterification of chicken fat produced some fatty acids with different levels. The higher contents of fatty acid in chicken fat catalyzed using NaOH were oleic acid, acid palmitic, stearic acid, and palmitoleic acid, which are about 53.52%, of 32.00%, of 9.47%, of 4.01%, respectively. While the K_2CO_3 catalyst produced fatty acids types such as oleic acid, palmitic acid, Methyl 7, 10 oktadecanoic acid, and Stearic acid with relative concentration approximately 43.73%, 22.82%, 9,63%, and 7.52%, respectively (Table 2). Both catalysts resulted different of fatty acid types but they also produced the same dominant fatty acids such as oleic acid and palmitic acid. Hermanto *et al.*, (2008) who reported that chicken fat contains 2 dominant fatty acid including oleic acid and palmitic acid with relative concentration approximately 38.35% and 27.24%. Generally, the heterogeneous catalyst (K_2CO_3 catalyst) requires a more complex reaction due to its activity most depends on the active center catalyst (Utomo & Laksono, 2007). Therefore, the NaOH catalyst resulted is higher dominant fatty acid yields than the K_2CO_3 catalyst in the transesterification of lard or chicken fat. Besides, halal authentication between lard and chicken fat can be distinguished based on the different fatty acid compositions using both catalysts (Figures 1).

CONCLUSION

Both NaOH and K_2CO_3 catalysts can produce different fatty acids levels due to they have different interactions in the transesterification of lard and chicken fat. NaOH is a better catalyst than K_2CO_3 in this transesterification because it produced higher concentration of the dominant fatty acids than K_2CO_3 catalyst. In addition, they produced fatty acid compositions that can be performed for halal authentication because both catalysts can distinguish lard and chicken fat based on the resulted fatty acid methyl esters.

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