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AUTHENTICITY ANALYSIS OF BEEF MEATBALL ADULTERATION WITH WILD BOAR USING FTIR SPECTROSCOPY COMBINED WITH CHEMOMETRICS

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ABSTRACT

The aim of this research to detect and analyze the wild boar in beef meatball using FTIR combined with chemometric. Then, the specific of FTIR region for detecting wild boar in beef meatball in the commercial product. The wild boar meatball was made with different a concentration range between 0-100%. The wild boar meatball was analyzed using FTIR. The FTIR spectrum was processed using chemometrics method like Part Least Square (PLS) and Principal Component Analysis (PCA). The PLS look for the highest of determination coefficient and smaller of root mean square error of calibration (RMSEC) or Root Mean Square Error of Cross-Validation (RMSECV). PCA analysis will distinguish the wild boar meatball and beef meatball product. The result of this research showed that the FTIR spectrum of wild boar and beef in meatball have different characteristics. The result from FTIR spectrum processing showed that specific spectrum of wild boar at wavenumber 999 - 1481 cm^{-1} combined with 1650 - 1793 cm^{-1} which is processed at 1st derivative. The PLS resulted from the calibration method has the determination coefficient (R2) value is 0.9991 and RMSEC of 1.028%. While the validation methods resulted are R2 and RMSECV which are 0.9999 and 0.300% respectively. This research showed the meatball from wild boar can be distinguished from beef and the commercial meatballs are not made from them.

Keywords: FTIR spectrum, halal authentication, wild boar, beef meatball

INTRODUCTION

Meatball is a mixture of the ingredient of food such as flour and some food additives. Meatball is known from by society as the food consists of meat mixture of not less than 20%. Therefore, the beef meatball consists the beef in the meatball as much as 20%. So, we must be sure about the meat while usually used in meatball formulation. Islam gives information about some meats is haram such as pork, dog and carrion. Fadzlillah et al., (2011) reported that lard and pork are problems in Islam. The halal meat as protection for Muslim consumers in choosing food product.

The instruction for halal food consumption encourages an analysis of halal. The analysis of halal food has some goals to give good quality, safety and noting contaminant the non-halal in the food product. Danezis et al., (2016) reported that many different methodologies for the analysis of halal food which are based on authenticity indicators. Some methods were developed to detect the non halal food such as HPLC (Rohman et al., 2012[a]; Jorfi 2012), FTIR (Rohman et al., 2012[b]; Rohman and Che Man, 2011; Pu et al. 2014), and can also use with electrophoresis (Hermanto et al., 2013). The analysis method was used in the authentication process for halal products depend on compound character and type's sample which will be analyzed.

This research focused on the analysis of beef meatball with comparing the FTIR spectrum of wild boar and beef meatball to find the difference of functional groups. FTIR instrument has been chosen for wild boar analysis in beef meatball because it has some advantages such as a quick method, green environment, and free chemical solvent. The research from Che Man, et al., (2005) showed that FTIR combined with Partial Least Square (PLS) used as an instrument for pork analysis in chocolate by quickly. Rohman et al., (2014) also reported that FTIR spectroscopy combined with PLS is able to analyze the lard content in cosmetics at wavenumber 1785-702 cm-1 and 3020-2808 cm^{-1}. Besides, FTIR also can be used for quantification and classification of lard in cream with total time analysis for 3 minutes/sample (Rohman and Che Man, 2010).

The FTIR application also can be used in halal detection in the meatball. Many researchers focused on halal detection because halal is a global commodity for Muslim in the world. Reference from Kurniawati et al., (2014) was able to the classification of lard in beef meatball commercial which it was performed at wavenumber 1200-1000 cm^{-1}. Application of FTIR spectroscopy for analysis of rat meat in beef meatball can be detected at FTIR region 750-1000 cm^{-1} (Rahmania, eta al., 2015). Ahda and Safitri also reported that optimization of wavenumber for detection of lard in Crude Palm Oil can be analyzed and discriminated at the combination of FTIR region between 1481-999 & 1793-1650 cm^{-1} at normal spectra FTIR. This research also performs and optimization of FTIR region for analysis and detection of wild boar in beef meatball formulation. The result of optimization can be made as basic for a halal authentication process of rat meat in meatball formulation.

MATERIAL AND METHODS

Meatball preparation

Meatball formulation was prepared with mixer the 90% meat and 10% ingredient (wheat flour, salt, onion). The standard meatballs were made in the concentration range of wild boar 0-100%. The commercial of meatball samples were obtained from the market in Yogyakarta.

Extraction process of beef meatball and wild boar meatball

The extraction process of the meatball was prepared with a Soxhlet apparatus with method: 100 mg meatball was wrapped and inserted to the Soxhlet apparatus and time extraction process until 6 hours. After extraction, the lard in n-hexane solvent was evaporated with evaporator and filtered with Na_2SO_4 anhydrate. The result of fat was collected and stored in the icebox.

Analysis of wild boar in beef meatball using FTIR spectroscopy

Identification of beef meatball contaminated with wild boar using FTIR to detect their functional groups. The wild boar detection in beef meatball use FTIR spectroscopy at wavenumber 650-4000 cm^{-1}. The several of wild boar concentrations in beef meatball were 0 % -100 % (b/b).

Data processing of FTIR spectrum

The data analysis calculated using Horizon MB QA software (Canada). FTIR spectrum combined with the PLS method in some areas the wavenumber of FTIR spectrum that is characteristic of triacylglycerol The results of this research evaluation of data processing using PLS include the R^2 value band RMSEC, RMSECV. After that, we use Principal Component Analysis (PCA) to classify the meatball product contain wild boar.

RESULT AND DISCUSSION

The discrimination process between wild boar and beef in meatball formulation was analyzed using FTIR spectroscopy combined with chemometrics. The basic discrimination process because wild boar and beef have different chemical composition. Fat is one of the components which has a contribution to the discriminant process between wild boar and beef. The research from Rohman et al., (2012[a]) reported that the triglycerides composition of lard, chicken fat, beef fat, mutton fat have different and was used for discrimination analysis. The different of triglycerides composition resulted in any different functional groups analyzed with FTIR spectroscopy. The discriminant analysis using the FTIR technique is based on fat characteristics (Rohman and Che Man, 2011).

Figure 1. showed that FTIR spectrum from wild boar and beef fat has some differences in kind of vibration and vibration intensity. Alkanes groups from wild boar and beef fat have vibration at wavenumber 2800-3000 cm^{-1}, methyl group at wavenumber 1350 cm^{-1}, Methylene groups at wavenumber 1450 cm^{-1} (Rohman et al., 2012[a]) and they have cis olefinic C-H group at wavenumber 3006 cm^{-1} Rohman et al., (2012[b]) with similar intensity. The different FTIR spectrum was showed at wavenumber 1750 cm^{-1} and 600-1300 cm^{-1}.

The data processing of FTIR spectrum results in the discrimination of wild boar and beef fat in the meatball formulation. Table 1 showed that the discriminant method of this research results different from linearity and RMSEC (Root Mean Square Error of Calibration). The result of the calibration curve has an R^2 value of 0.9991 with a limit of error of 1.028% at wavenumbers of FTIR spectrum 999-1481 cm^{-1} combined with 1650-17.93 cm^{-1} in 1st derivate spectrum. Rohman et al., (2012[b]) reported that modeling of discrimination resulted from determination coefficient value is higher and the RMSEC value is smaller. That's reason showed that the FTIR wavenumber has a linear correlation of the changes in lard concentration and its intensity. The validation curve of the PLS (Part Least Square) model also has a good model with a determination coefficient (R2) is higher and RMSECV (Root Mean Square Error of Cross-Validation) values is smaller compared with other wavenumbers (Table 1).

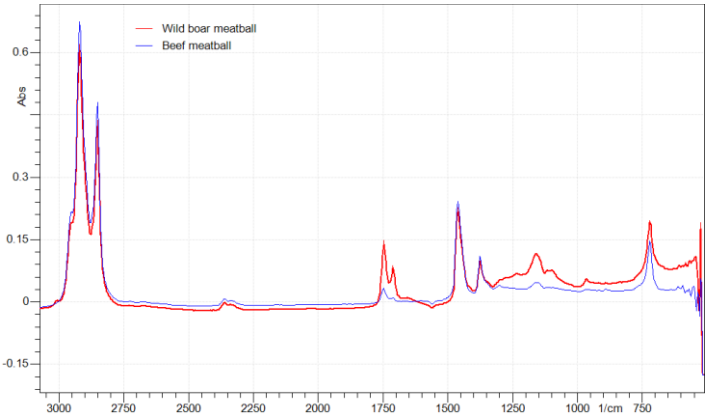


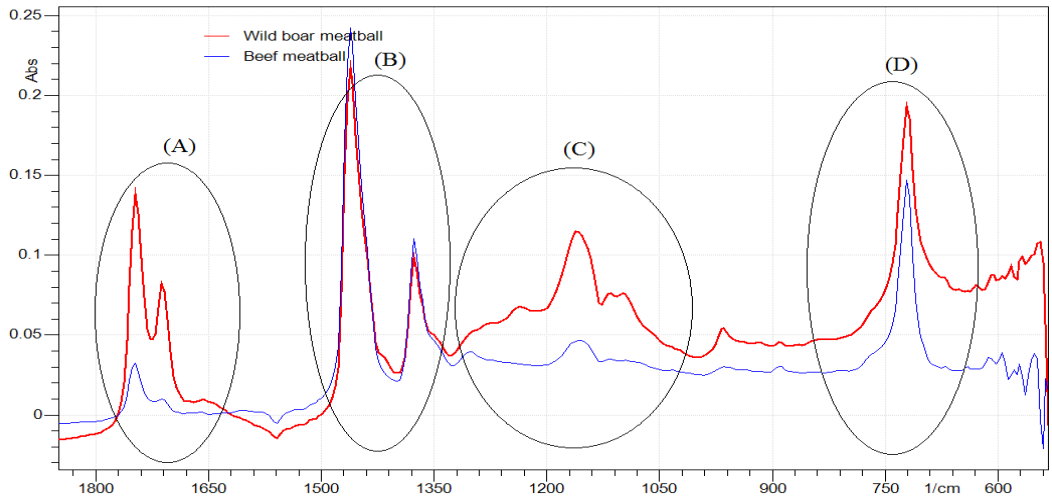
Figure 1 Normal of FTIR Spectrum from Wild boar Meatball and Beef Meatball at wavenumber 500-3100 cm^{-1}

The determination coefficient (R2) and the RMSECV values were 0.9999 and 0.300% respectively. The result of calibration and validation curve resulted from the FTIR wavenumber at 999-1481 cm^{-1} combined with 1650-1793 cm^{-1} in 1st derivative. Research from Guntarti el al., (2015) showed that the optimal wavenumbers of FTIR for identifying the wild boar in beef meatball at a wavenumber of 1000-1250 cm^{-1} with The R^2 and RMSEC values were 0.998 and 2.00%. Therefore, that's at a wavenumber of 999-1481 cm^{-1} combined with 1650-1793 cm^{-1} at 1st derivative is characteristic of FTIR spectrum to identify the wild boar in the meatball.

Table 1 Result Data of combination of wavenumber 1480-1085 cm^{-1} and other wavenumber of FTIR spectrum for wild boar detection in beef meatball

Kind of spectra	Wavenumber (cm^{-1})	Calibration curve		Validation curve	
		R^2 value	RMSEC	R^2 value	RMSECV
Normal	999-1481	0.9983	1.219	0.9998	1.342
	999-1481 & 651-763	0.9985	1.171	0.9999	1.874
	999-1481 & 1650-1793	0.9978	1.285	0.9993	1.358
	999-1481 & 2769-3020	0.9959	1.508	0.9990	1.994
1st Derivate	999-1481	0.9932	1.708	0.9983	2.187
	999-1481 & 651-763	0.9854	2.068	0.9969	3.128
	999-1481 & 1650-1793	0.9991	1.028	0.9999	0.300
	999-1481 & 2769-3020	0.9990	1.052	0.9998	0.855
2nd Derivate	999-1481	0.9940	1.660	0.9986	1.404
	999-1481 & 651-763	0.9930	1.765	0.9999	1.182
	999-1481 & 1650-1793	0.9970	1.391	0.9993	1.206
	999-1481 & 2769-3020	0.2405	5.868	0.8834	20.990

Figure 2 showed that the FTIR spectrum at wavenumbers at 650-1800 cm^{-1} has a different FTIR spectrum between wild boar and beef fat in meatball formulation. FTIR spectrum has different of wavenumbers 1600-1800 cm-1, 1050-1300 cm^{-1} and 650-750 cm^{-1} are very clear. While the FTIR spectrum at wavenumber 1350-1450 cm^{-1} showed similar results of the FTIR spectrum. However, the results of FTIR spectrum combined with chemometrics showed that FTIR wavenumbers at 1050-1450 cm^{-1} and 1650-1800 cm^{-1} has a good result with validation curve of linearity values of 0.9999 and small error (RMSCV) of 0.300%. This indicates that wavenumbers 1050-1300 cm^{-1}, 1350-1450 cm^{-1}, and 1600-1800 cm^{-1} can be indicated as characteristic of the FTIR spectrum for detection of wild boar fat in meatball formulation.



Fiure 2 FTIR Spectrum from Wild boar Meatball and Beef Meatball at wavenumber 500-1850 cm^{-1}

The specific of FTIR spectrum used to distinguish between beef and wild boar meatball at wavenumber range 999-1481 cm-1 and 165-1793 cm-1 in 1st derivate. Figure 3 showed that the difference of meatball products was explicit where it can be seen in the far separation. This result is a model projection used for identification of commercial samples.

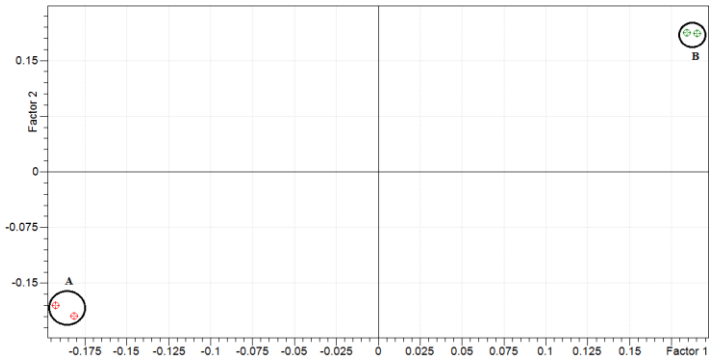


Figure 3 Classify the beef meatball (A) and wild boar meatball (B) using PCA at wave range at 999-1481 cm^{-1} and 1650-1793 cm^{-1} in 1st derivate

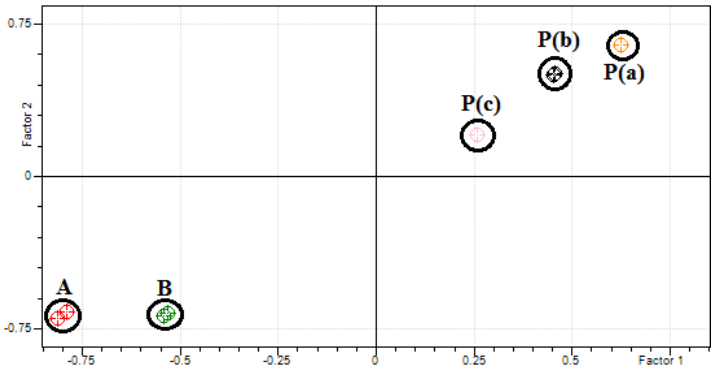


Figure 4 Detection of Meatball Sample (P(a), P(b) dan P(c)) from Commercial Product. A) Beef Meatball and B) Wild Boar Meatball

In the analysis of commercial meatballs cause the standard of beef meatball and wild boar meatball move closer to each other because all commercial meatballs are not part of both beef meatball and wild boar meatball. Based on Figure 4 showed that all commercial products that are sold, the meatball was not made from beef or wild boar. This fact was proven on PCA analysis that all commercial product far distinguished from beef and wild boar meatball. This result showed that the commercial products did not use the beef or wild boar in the meatball formulation.

CONCLUSION

FTIR spectroscopy is one of the analytical methods used for detecting wild boar in meatball where we can determine it in the specific wavenumber at 999-1481 cm^{-1} and 1650-1793 cm^{-1} in 1st derivate. The PLS optimization resulted in the calibration curve with R^2 value and limits of error which is 0.9991 and 1.028, respectively. The validation method showed a good result with an R^2 value and the RMSECV values of 0.9999 and 0.300%, respectively. The analysis qualitative of the commercial meatballs indicated that all commercial products far away from beef and wild boar meatball.

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