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*Research Article*

# Halal Authentication of Toothpaste with FTIR Combine with Chemometrics

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**Abstract:** Gelatin is a raw material used in pharmaceutical and cosmetic preparations, one of which is toothpaste. Some of dental raw materials are imported from another country, and might contain porcine gelatin, Authentication of gelatin in toothpaste products is crucial due to religious and health reasons. The purpose of this study was to detect porcine gelatin in toothpaste raw materials using ATR-FTIR. Five samples of toothpaste were purchased from local market and detection was done using ATR-FTIR. The spectrum from each sample was compared against standard bovine and porcine gelatin. Experimental toothpaste containing bovine and porcine gelatin at concentrations of 5, 10, 15 and 20% were also prepared for quantification analysis. FTIR combine with chemometrics can be utilized as a reliable tool to detect gelatin in toothpaste and other pharmaceuticals.

**Keywords:** gelatin, toothpaste, FTIR

## 1. INTRODUCTION

Indonesia is a country where the majority of the population is Muslim. The awareness of halal products has increased in recent years [1]. One of the products that must be confirmed is halal for consumption cosmetics where toothpaste is included. Halal cosmetics product in Indonesia is still low compared to other countries, especially Malaysia [2]. Toothpaste ingredients can be categorized as preventive ingredients where they contain antibacterial, fluoride or therapeutic agents in dental care. Other ingredients are restorative is applied in repairing or changing tooth structure and consists of synthetic material. Most of the toothpaste ingredients in Indonesia are imported from other countries and possibly contains gelatin [3]. Irfanita et al., (2017) say that dental material product in Malaysia contains gelatin [4]. The labeling of this toothpaste product is not clear because the label does not mention the additional ingredients used.

Gelatin is a protein that is obtained from collagen from bones, hides and skin various animals such as farm animals and pigs. Based on the report it is known that gelatin from pork is present in greater quantities than gelatin from other animals [5]. Gelatin authentication is very important not only for religious reasons but including health reasons such as the presence of *Bovine Spongiform disease Encephalopathy* (BSE) or mad cow disease that can be transmitted through animals infected livestock [6]

There are many analytical methods for detection of gelatin, one of the methods used is IR spectroscopy on gummy candy [7]. The ATR-FTIR is reported to be very useful because it is reliable, accurate, and fast in delivery results in less than 2 sample minutes per analysis. This method is also known as nondestructive and requires sample preparation to obtain information

based on properties chemical and molecular of various analytes. Most of the research is done to detect gelatin or gelatin raw materials in processed foods. So the aim of this study is to detect gelatin in toothpaste using ATR-FTIR.

## 2. MATERIALS AND METHODS

### *2.1. Material*

Pure bovine and porcine gelatins were obtained from Sigma Aldrich. Commercial toothpaste were purchased from stores in Purwokerto, Indonesia. Calcium carbonat, saccharin, menthol, glycerin pharmaceutical grade from Bratachem and aquades.

### *2.2. Reference Toothpaste Samples*

Reference tooth-paste samples containing bovine and porcine gelatin at concentrations of 0%, 5, 10, 15 and 20% were prepared. The paste was prepared by blending bovine or porcine powder, calcium carbonate and menthol. The ingredients are mixed and stirred homogeneously with a mortar and stamper. Water is added and stirred until homogeneous until obtained consistency like a paste. Simulation samples were made with 5 kinds of toothpaste concentration series with or without porcine gelatin.

### *2.3. Analysis of Gelatin in Samples with ATR-FTIR*

Identification of gelatin in tooth-paste products was done qualitatively by comparing the infrared spectra data of toothpaste samples with the spectra data of pure bovine and porcine gelatin. All data were recorded within a range of 4000–400 cm–I with a 4 cm–I resolution and 32 scans [8].

## 3. RESULTS AND DISCUSSION

### *3.1. FTIR Spectrum of Pure Bovine and Porcine Gelatin*

Based on the results of measurements of 100% pork gelatin and 100% cow gelatin using FTIR, the infrared spectrum is shown in Figure 1.Based on figures 1 shows a similar pattern in the four observed areas, namely 3600-2300 cm-1 (Amide A ), 1656-1644 cm-1 (Amide I), 1560-1335 cm-1 (Amide II), and 1240-670 cm-1 (Amide III).

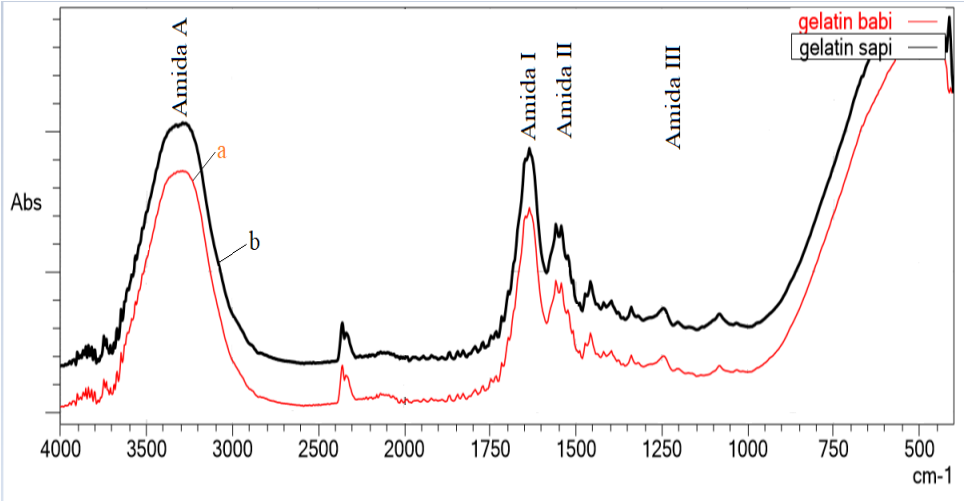


**Figure 1.** FTIR Spectra of Bovine and Porcine Gelatin

Spectrum of FTIR showed that generally bovine and porcine gelatin have similarity spectrum, but when compared more thoroughly the peaks were relatively different. Amida A at 3600-2300 $cm^{-1}$ is related to N-H stretching and intramolecular hydrogen bond of amino acids that arrange the gelatin. Parallel polarized absorption with N-H bonds showed hydrogen bond interaction with alpha helical structure of gelatin. Absorpstion peak may shift to a lower frequency when the strength of hydrogen bond rises [9]. Amide I band related to stretching vibration of C=O group (of peptide bond), with the contribution of C-N bond stretching. Amida II is generated by mixed vibration of N-H bending and C-N stretching in secondary amides [10].

*3.2. Analysis of PCA*

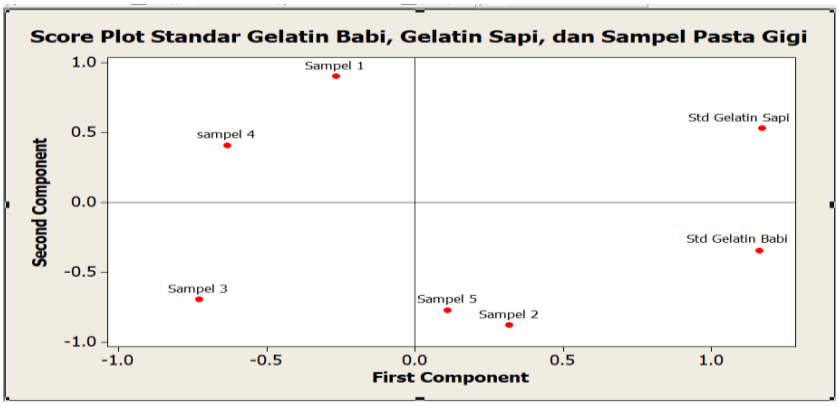


**Figure 2.** Score Plot of Pure Gelatin and Toothpaste Sample from Market

Result of PCA analysis from pure bovine and porcine gelatin and samples at 4 range wave length number ie 3600-2300 $cm^{-1}$ 1656-1644 $cm^{-1}$, 1560-1335 $cm^{-1}$ , and1240-670 $cm^{-1}$ showed that FTIR can classified pure gelatin with samples (Fig. 2).

*3.3. Analysis of PLS*

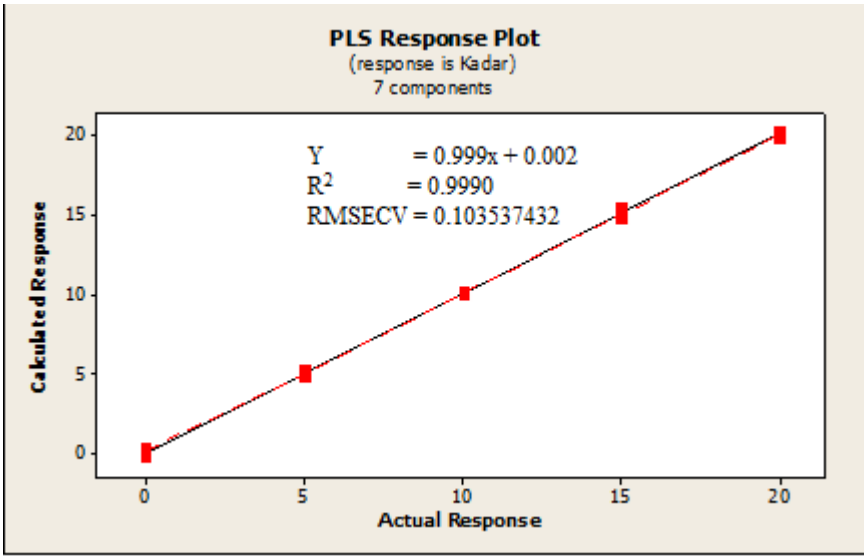


**Figure 3.** Correlation between Actual Value (x-axis) of Porcine Gelatin (%wt/wt) and FTIR Predicted Value (y-axis) of Porcine Gelatin (%wt/wt) Validation Model

FTIR spectral treatments were optimized to give the best prediction model that provides the highest value of coefficient of determination ($R^2$) and the lowest values of root mean square error of calibration variation (RMSECV). The coefficient of determination ($R^2$) obtained for correlation between actual value of porcine gelatin and FTIR predicted value was 0.999 in the calibration model. The root mean square error of cross validation (RMSECV) obtained is 0.103. Result of quantitative analysis is toothpaste sample not contain gelatin because its value is zero [11, 12].

## 4. CONCLUSION

Wave numbers region of 3600-2300 $cm^{-1}$, 1656-1644 $cm^{-1}$, 1560-1335 $cm^{-1}$ , and 1240-670 $cm^{-1}$ from FTIR spectrum showed characteristic spectrum of gelatin. FTIR spectrum combined with chemometrics can be used for rapid analysis of gelatin in toothpaste.

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