Journal of
Food and Pharmaceutical Sciences

Original Article

Detection of Garlic Powder Adulteration Using FTIR Spectroscopy and Chemometrics: A Case Study in an Indonesia Marketplace

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Received: 16 May 2024; Revised: 06 August 2024; Accepted: 24 August 2024; Published: 31 December 2024

Abstract: Garlic is a plant with numerous benefits, including its use as a culinary ingredient and for medicinal purposes. However, the rising demand does not align with the availability of garlic in Indonesia, resulting in high garlic prices. Excessively high prices may potentially lead to garlic powder adulteration. The objective of this study is to determine whether there are garlic powder products adulterated with maltodextrin in the Marketplace. A total of 10 garlic powder samples were purchased on Marketplace. The study utilized a combination of FTIR spectroscopy with chemometrics. The chemometrics employed in this study were OPLS-DA (Orthogonal Partial Least Squares Discriminant Analysis) for sample grouping, as well as PLS (Partial Least Squares) for multivariate calibration. The findings revealed that OPLS-DA could effectively group the samples. Additionally, the best multivariate calibration model (PLS) was obtained at the wavenumber range of 679-440 cm^{-1} with a calibration R^2 value of 0.9981, RMSEC value of 1.11, validation R^2 value of 0.9926, and RMSEP value of 2.12. Based on the research results, it was found that three products were not pure garlic powder, with details indicating two products containing maltodextrin as an adulterant and one product containing a non-maltodextrin adulterant.

Keywords: Garlic powder; maltodextrin; FTIR; OPLS-DA; PLS

1. INTRODUCTION

Garlic (Allium sativum) is a horticultural plant commonly used as a primary seasoning in various forms such as fresh bulbs, dried powder, fried garlic, or garlic oil. The addition of garlic to dishes provides a unique flavor that cannot be replaced by other spices. The aroma and taste are attributed to the presence of allicin compound, which is the primary secondary metabolite in garlic. When garlic is damaged, it releases the enzyme alliinase to convert alliin into allicin. Apart from its culinary use, garlic is also utilized for medicinal purposes. Garlic is believed to have antimicrobial, antihypertensive, antioxidant, antitumor, and anticoagulant activities [1].

Garlic is known to have numerous benefits, making it an important commodity that increases consumption demand by 1.38% annually. The estimated consumption demand for garlic in 2023 reached 517.930 tons, increasing to 526.770 tons in 2024. However, the available amount of garlic in Indonesia cannot meet the projected consumption demand. From 2020 to 2024, Indonesia will

experience a significant decrease in garlic availability. In 2023, Indonesia experienced a garlic availability deficit of 405.200 tons, which increased to 411.440 tons the following year [2]. Based on this data, it can be observed that the garlic balance in Indonesia is unstable, indicating an imbalance between garlic availability and demand.

According to data from the Ministry of Trade's Market and Basic Needs Monitoring System (SP2KP), this phenomenon has led to a 6.99% increase in garlic prices from July 2023 to August 2023. Therefore, garlic is dubbed as "white diamonds" due to its easy saleability and high economic value resulting from continuous increasing demand [3]. High garlic prices can increase the likelihood of fraud, with one potential fraud being the adulteration of garlic powder. Garlic powder is obtained from garlic that has undergone drying and grinding processes. Adulteration of garlic powder is aimed at minimizing production costs with greater profits. This practice is known as adulteration, involving the addition or substitution of cheaper ingredients than the main raw materials [4]. Garlic powder can be adulterated with various substances such as maltodextrin, sodium caseinate, talc, cornstarch, corn flour (maizena), peanut powder, potato starch, and rice flour [5].

The FDA's Forensic Chemistry Center in the United States has found instances of garlic powder products being adulterated, where only one ingredient, garlic (pure garlic), is listed. However, the product actually contained 70% maltodextrin [6]. Additionally, research by Patarroyo-Leon discovered 15 samples of counterfeit garlic powder commercially available, mixed with cornstarch without labelling the addition of cornstarch as an additional ingredient [7]. Furthermore, in 2017, several food companies in the USA and Australia found peanut contamination in garlic powder [8]. Given these issues, there is a need for methods to detect food adulteration.

FTIR has been chosen as a popular methodology for detecting food fraud [9]. FTIR is an instrument used to analyze compounds in a product [10]. The basic principle of FTIR involves the interaction between a sample (material) and electromagnetic radiation. FTIR is commonly used in the pharmaceutical, environmental, and food industries [11]. In food fraud, FTIR is often used for authentication of herbal products, agricultural products, vegetable oils, and milk [9]. The advantages of FTIR instruments include the use of small sample sizes, simple sample preparation, minimal use of solvents and chemical reagents, no special treatment required for samples, non-destructive analysis, rapid analysis, and greater sensitivity [12]. Combining FT-IR spectroscopy with chemometrics is a good method choice for food authentication [13].

Chemometrics is a branch of statistics, mathematics, and formal logic used to obtain chemical information from data. Chemometrics is often used for analysis in pharmaceuticals, forensic science, environmental science, agriculture, and food authentication [14]. In food authentication, chemometrics has been used in several studies to detect fraud in oils, honey, dairy products, and meat [5]. There are two common chemometric methods used for food analysis: partial least squares (PLS) for calibration model creation and principal component analysis (PCA) for sample classification [13].

Previous research by Galvin-King successfully detected adulteration of garlic powder samples using FTIR and NIR spectroscopy [5]. Additionally, research by Lohumi also successfully detected cornstarch adulteration in garlic powder using FTIR spectroscopy combined with partial least squares regression (PLSR) [15]. Both samples and studies were conducted abroad. In this regard, there is currently no research on the authenticity testing of garlic powder in Indonesia. Therefore, there is a need to detect garlic powder adulteration in Indonesia, especially those sold in marketplaces

like Shopee. Shopee chosen as an online shopping platform because it has a high level of visitors the highest compared to other platforms, namely 241.6 million visitors in October until December 2023 [16]. This is proven by the complete availability of goods and features on Shopee, making it easier for customers to purchase goods [17].

2. MATERIALS AND METHODS

2.1. Sample

In this study, garlic powder samples sold on the Shopee marketplace, particularly in Surabaya, were utilized. After conducting a search using the keyword "garlic powder" in the Shopee application, 19 garlic powder sellers were found in the Surabaya area, offering products under different brands. From these 19 products, samples were selected based on specific criteria.

According to the inclusion and exclusion criteria, 10 garlic powder products meeting the criteria were obtained. Therefore, this study utilized 10 samples of garlic powder that were not registered with BPOM and not certified by the Indonesian Ministry of Agriculture, purchased from Shopee in the Surabaya area. Subsequently, detection related to the presence of maltodextrin in counterfeit garlic powder will be conducted on these 10 samples.

Pure garlic is needed as a comparison to garlic powder purchased at Shopee. Pure garlic was obtained from Blimbing Market, Lowokwaru District, Malang City. Next, the merger and integration was carried out at the Batu Herbal Materia Medica Laboratory. Subsequently, identification and pulverization were carried out at the Herbal Materia Medica Laboratory, Batu.

2.2 Materials

Maltodextrin is used as an adulterant in garlic powder mixtures. Acetone (EMSURE®) is used to clean ATR crystals in FTIR instruments.

2.3 Instrumentation

The equipment used in this study included a set of glassware (PYREX®, Arizona), an analytical balance (Ohaus Pioneer®, China), FTIR spectroscopy (Shimadzu®, Japan), and Chemometrics software (TQ Analyst®, Norway and SIMCA software (Umetrics, Umeå Sweden)).

2.4 Methods

2.4.1. Sample Preparation

Garlic bulbs were sorted, peeled, and washed with running water. Then the garlic bulbs were sliced into smaller and thinner pieces. The sliced garlic was dried using an oven at 60°C for 15 hours. The dried garlic slices were ground using a blender until garlic powder was formed. The garlic powder was sieved using a 60-mesh sieve. In addition, a mixture of pure garlic powder and maltodextrin was made with a concentration of 0-50%w/w to create a calibration model for PLS.

2.4.2. FT-IR Spectral Acquisition

The FTIR instrument used is the Qatar-S Single Bounce Diamond ATR (Shimadzu, Japan) with attenuated total reflectance (ATR) sample handling, the sample area is cleaned using acetone, the basic spectrum (background) is scanned before taking measurements on the sample, the sample to be scanned is prepared, the sample is placed under the crystal ATR, the sample was measured in 32 scans and at a resolving power (resolution) of 16 cm^{-1}, scans were carried out at a wavelength of 4000-650 cm^{-1} and replicated 2 times after being recorded in the form of absorbance, after scanning, the ATR was cleaned using acetone, then dry with a tissue.

2.4.3. Data Processing

The data was processed using SIMCA software (Umetrics, Umeå Sweden) for OPLS-DA, and then the data was processed using TQ Analyst software (ThermoScientific, USA) for PLS. Data is entered into the software (numerical and nominal data), optimization is carried out, and a score plot is obtained which describes grouping OPLS-DA and R^2 in PLS.

2.4.4 Validation Process

OPLS-DA validation required 15 samples of maltodextrin and 15 samples of control garlic powder. OPLS-DA validation was carried out by taking 1/3 samples (5 samples) for validation and 2/3 samples (10 samples) for calibration. Validation of the PLS calibration model was carried out using the Leave One Out cross validation technique. Leave One Out cross validation is carried out by discarding one sample and using the remaining samples to form a new model. Optimization is carried out to discard a number of samples until the best model is found. This selection is based on the R2 value which is closest to 1; RMSEC <2.4; and RMSEP <2.79.

3. RESULTS AND DISCUSSION

3.1. Spectral Interpretation

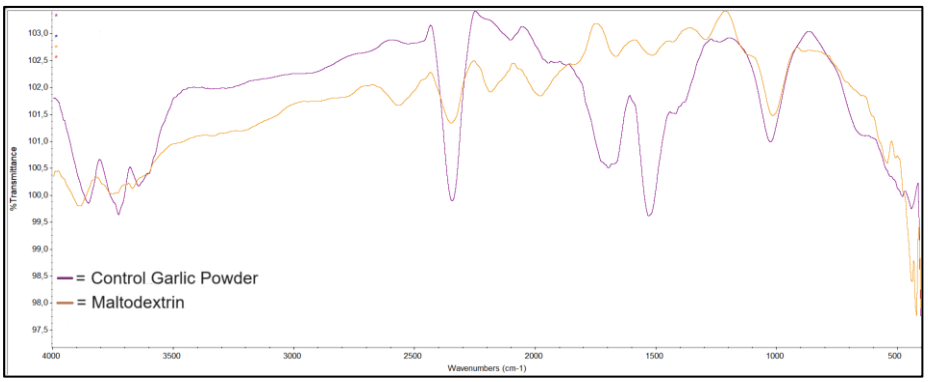

Figure 1. FTIR Spectra of Control Garlic Powder and Maltodextrin

Table 1. FTIR Interpretation Results of Control Garlic Powder

Functional Groups	Wavenumber (cm^{-1})	Control Garlic Powder		References
		Wavenumber (cm^{-1})	Functional Group Description	
O-H	3650-3600	3616.71	Stretching vibration	[18]
C=O	1830-1650	1687.80	Stretching vibration	[19]
C=C	1680-1650	1687.80	Stretching vibration	[18]
N-H	1640-1550	1521.32	Bending vibration of primary amine	[18]
CH_2 (alkane)	1465	1521.32	Cutout bending vibration	[18]
C-N	1350-1000	1021.87	Bending vibration of primary amine	[18]
$=CH_2$	900-600	665.93	Bending vibration out of plane	[18]
C-C	Around 620.72	665.93	Stretching vibration	[20]
C-S	700-600	665.93	Stretching vibration	[19]

The control garlic powder and maltodextrin as adulterants were tested using FTIR spectroscopy. The scanning process was replicated three times in the wavenumber range of 4000-650 cm^{-1}. The spectrum of the measurement results depicts characteristic functional groups in each compound. The FTIR spectra were interpreted by observing and comparing the position, intensity, and shape of the absorptions with reference tables of characteristic functional groups.

Table 1 shows that garlic powder has typical absorption in the O-H, C=O, C-N, N-H, C=C, C-C, and C-S groups. Based on the spectral readings of the garlic powder, absorption was observed at the wavenumber of 3616.71 cm^{-1}. This absorption occurred due to stretching vibration of the O-H group

with moderate intensity, indicating the presence of carbohydrates and amino acids. The presence of hydrogen bonds caused the O-H absorption peak to appear broadened. At the wavenumber of 1687.80 cm^{-1}, stretching vibration of the C=O group was observed, indicating the presence of protein components. At the wavenumber of 1521.32 cm^{-1}, bending vibration of the N-H group with moderate intensity was observed, indicating the presence of protein components. Bending vibration can cause changes in bond angles, resulting in bent bonds. At the wavenumber of 665.93 cm^{-1}, stretching vibration of the C-S group was observed, indicating the presence of sulfur components.

Table 2. FTIR Interpretation Results of Maltodextrin

Functional Groups	Wavenumber (cm^{-1})	Control Garlic Powder		References
		Wavenumber (cm^{-1})	Functional Group Description	
O-H	3650-3600	3622.45	Stretching vibration	[18]
C-H *sp3*	1450	1518.09	Bending vibration	[18]
C-O	1300-1000	1016.12	Stretching vibration	[18]
C-C	Around 620.72	617.68	Stretching vibration	[20]

Table 2 shows that maltodextrin has typical absorption in the O-H, C-H *sp3*, C-O, and C-C groups. Based on the results of the maltodextrin spectrum readings obtained there is absorption at the wave 3622.45 cm^{-1} which is describes the stretching vibration of the O-H group. There is a bond hydrogen causes the peak to appear broadened. On numbers wave 1016.12 cm^{-1} stretching vibration occurs which describes the existence of C-O group. At a wavenumber of 1518.09 cm^{-1} bending vibration occurs from the C-H *sp3* group. Stretching vibrations can cause changes bond length. A comparison of the two spectra revealed differences in the constituent groups between garlic powder and maltodextrin.

3.2 Orthogonal Partial Least Squares-Discriminant Analysis (OPLS-DA)

3.2.1. Result of OPLS-DA Control Garlic Powder and Maltodextrin

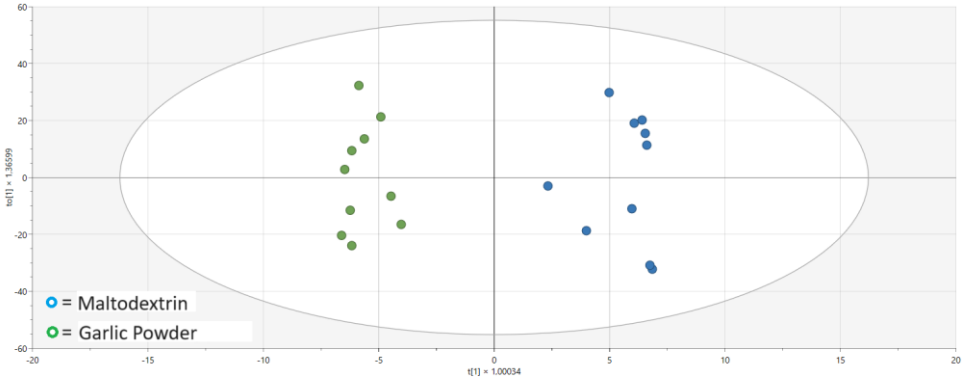

Figure 2. Score Plot Control Garlic Powder with Maltodextrin

OPLS-DA is a supervised clustering method. In this case, grouping is done by deleting the information in each group. Then, an analysis is carried out to find out whether groups whose information has been removed can gather with other groups that have similar characteristics. Based on Figure 3, it is found that OPLS-DA can group samples well. In the OPLS-DA score plot results, it was found that garlic powder and maltodextrin were well separated into two groups. The control

garlic powder group was in the left quadrant, while the maltodextrin group was in the right quadrant. In this case, both have different characteristics so they are in different groups.

3.2.2. OPLS-DA Validation

Validation is carried out to ensure that OPLS-DA can group samples well. Validation is carried out by taking 1/3 samples for validation and 2/3 samples for calibration. A total of 1/3 of the samples used for validation had their information removed (group of origin). Then, an analysis is carried out to see whether the sample without information can join according to the original group that was previously known. Based on the Table 3, it is found that the validation results show a truth level of 100%, which means that 1/3 of the samples whose information has been removed can be gathered according to the group of origin. Therefore, it can be said that OPLS-DA can group samples well.

Table 3. OPLS-DA Validation of Control Garlic Powder and Maltodextrin)

Design	Calibration		Validation		Total
	Number of samples	Correct	Number of samples	Correct	
Control Garlic Powder	10	100%	5	100%	15
Maltodextrin	10	100%	5	100%	15
Total	20	100%	10	100%	30

3.2.3. Garlic Powder Sample Prediction Using OPLS-DA

OPLS-DA which has successfully grouped samples is then used to group 10 samples purchased on Shopee. This stage was carried out to determine whether garlic powder samples 1-10 tended to group in the pure garlic powder group or the adulterant group. The large distance between samples indicates the similarity of the samples. The closer the points between samples indicate the higher the similarity, conversely, the farther the points between samples, the lower the level of similarity between the samples. In Table 4, it is found that there are three samples that do not pure, namely samples 2, 5, and 6. When grouping is carried out using OPLS-DA, the three samples converge on maltodextrin group. As for the pure sample of garlic powder are samples 1, 3, 4, 7, 8, 9, and 10. When carried out grouping using OPLS-DA, the three samples gather in the group of pure garlic powder so can be said to be pure garlic powder without mixture.

Table 4. Prediction of Garlic Powder Samples with OPLS-DA

Sample Number	Sample Total	Garlic Powder	Maltodextrin	Conclusion
1	3	3	0	Pure garlic
2	3	0	3	Impure
3	3	3	0	Pure garlic
4	3	3	0	Impure
5	3	0	3	Impure
6	3	0	3	Pure garlic
7	3	3	0	Pure garlic
8	3	3	0	Pure garlic
9	3	3	0	Pure garlic
10	3	3	0	Pure garlic

3.3 Partial Least Square (PLS)

3.3.1. PLS Wavenumber Optimization

Table 5. Wavenumber Optimization Results for PLS

Wavenumber (cm^{-1})	Calibration		Validation	
	R^2	RMSEC	R^2	RMSEP
4000-400	0.9799	3.76	0.9715	5.11
2800-1800	0.8838	8.42	0.7164	14.3
2390-2000	0.9077	7.55	0.7741	12.5
1930-1700	0.8466	9.57	0.8071	9.87
1560-1500	0.9150	7.26	0.9113	7.22
1200-600	0.9166	7.19	0.7519	11.1
1133-1000	0.9500	5.62	0.9539	5.26
679-440	**0.9981**	**1.11**	**0.9926**	**2.12**

Multivariate calibration using PLS is carried out by first selecting the most optimal wavenumber which has an R^2 value >0.99 and the smallest RMSEC (Root Mean Square Error Calibration) and RMSEP (Root Mean Square Error Prediction) values. Based on Table 5, it is found that the best wavenumbers are in the area 679-440 cm^{-1} with a calibration R^2 value of 0.9981; the RMSEC value of 1.11; the validation R^2 value of 0.9926; and the RMSEP value is 2.12. The values obtained are by the criteria so it can be said that the model used has good linearity, that is, it has a strong relationship or correlation between predicted and actual values. The optimum wave number was then used to process PLS data on control garlic powder, garlic powder samples 1-10, and a mixture of garlic powder with maltodextrin at a concentration of 0-50%w/w.

3.3.2. PLS Validation

The selected calibration model is tested to test the level of truth using cross validation. The cross-validation technique used is Leave One Out. Validation is carried out using the optimized wavenumber, which is 679-440 cm^{-1}. This validation technique is carried out by removing one of the calibration samples (for example, removing sample data with a concentration of 20%), then the remaining samples are used to create a new model [21-23] . Figure 4 that the results of the Leave One Out cross-validation on the selected model can provide a good validation R^2 value. After validation, the selected model can be used to predict maltodextrin levels in samples 2, 5, and 6.

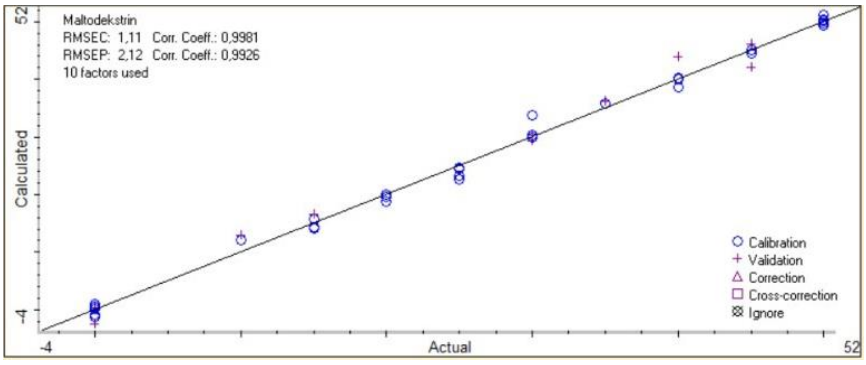

Figure 3. PLS Validation of Mixed Samples between Garlic Powder and Maltodextrin

3.3.3. Garlic Powder Sample Prediction with PLS

Garlic powder samples 1-10 were predicted to find the presence of maltodextrin as an adulterant in garlic powder sold in the Shopee. In Table 6, it can be seen that in the 5th sample there was an adulterant in the form of maltodextrin amounting to 14.96%, and in the 6th sample there was an adulterant in the form of maltodextrin amounting to 10.23%, and in the 2nd sample there was an adulterant but it did not come from maltodextrin. Therefore, it can be said that the combination of FTIR spectroscopy and chemometrics in this research has been successful and can carry out adulteration analysis in garlic powder.

Table 6. Prediction Results for Garlic Powder Samples with PLS

Sample Name	1 %(b/b)	2 %(b/b)	3 %(b/b)	Average %(w/w)	Conclusion
2nd sample	-4.03	-2.37	-1.93	-2.78 ± 1.11	The adulterant is not from maltodextrin
5th sample	4.46	22.75	17.67	14.96± 9.44	Added maltodextrin
6th sample	14.15	8.94	7.60	10.23± 3.46	Added maltodextrin

4. CONCLUSION

Based on the conducted research, the developed FTIR spectroscopy and chemometrics method can detect the adulteration of garlic powder. In sample grouping, it was found that OPLS-DA could classify samples based on their characteristic similarities, whereas PCA could not effectively classify the samples. The combination of FTIR with chemometrics can form a good model for multivariate calibration. Based on the optimization results at the optimal wavenumber range of 679-440 cm^{-1}, the calibration R^2 value obtained was 0.9981, the RMSEC value was 1.11, the validation R^2 value was 0.9926, and the RMSEP value was 2.12. There are three counterfeit garlic powder products sold on Shopee in the Surabaya area. Based on data analysis using OPLS-DA, it was found that samples 1, 3, 4, 7, 8, 9, and 10 are pure garlic powder, while samples 2, 5, and 6 are not pure garlic powder. According to the PLS data analysis, it was found that samples 5 and 6 are adulterated with maltodextrin, while sample 2 is adulterated with a substance other than maltodextrin.

Funding: This research received no external funding

Acknowledgments: -

Conflicts of interest: The authors declare no conflict of interest.

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