

Validation of a microscopic method and mathematical model for the detection of corn and wheat starch in adulterated ground black pepper

Matheus Aparecido de Paiva Caixeta ¹, Maria Gessi Teixeira ², Katia Alves Campos ³, Brígida Monteiro Vilas Boas ⁴

¹ Instituto Federal de Educação, Ciência e Tecnologia do Sul de Minas Gerais, IFSULDEMINAS, Campus Machado. E-mail: matheuscaixeta1997@gmail.com

² Instituto Federal de Educação, Ciência e Tecnologia do Sul de Minas Gerais, IFSULDEMINAS, Campus Machado. E-mail: maria.teixeira@ifsuldeminas.edu.br

³ Instituto Federal de Educação, Ciência e Tecnologia do Sul de Minas Gerais, IFSULDEMINAS, Campus Machado. E-mail: katia.campos@ifsuldeminas.edu.br

⁴ Instituto Federal de Educação, Ciência e Tecnologia do Sul de Minas Gerais, IFSULDEMINAS, Campus Machado. E-mail: brigida.monteiro@ifsuldeminas.edu.br

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Abstract

Adulteration of spices may negatively impact food quality and safety. The addition of starch is a fraud unlikely to be detected by consumers. Therefore, this study was developed in order to quantify, by optical microscopy, the presence of corn and wheat starch in ground black pepper and to validate equations for estimating starch percentage to identify adulteration. Black pepper grain samples were ground and intentionally adulterated with 3 %, 5 %, 10 %, 15 %, and 20 % corn starch and wheat flour, separately, and subsequently analyzed by optical microscopy. A linear regression model was adjusted to estimate the percentage of adulterant starch. A spreadsheet was developed to calculate the estimated mean starch percentage and its lower and upper limits. The model was validated by means of a blind test (samples adulterated at unknown levels to the analyst). Additionally, three commercial samples and three suppliers were evaluated. Structural differences were observed between corn and wheat starch granules, enabling their identification and quantification. The blind test confirmed the effectiveness of the method in identifying and counting starch granules, validated the adjusted mathematical model, and enabled satisfactory quantification of the adulteration percentage, reinforcing the reliability of the results. Wheat starch was detected in all three commercial samples and in two of the three supplier samples. Therefore, optical microscopy may be highlighted as a practical tool for the food industry, contributing significantly to fraud detection, quality assurance, and authentication of spices.

Keywords: Quality control. Spices. Fraud. Food microscopy. Black pepper. *Piper nigrum*.

Introduction

Food fraud consists of economically motivated adulteration, often involving the substitution or falsification of ingredients, which leads to reduced quality of finished products and, in many cases, harm to consumers (Global Food Safety Initiative – GFSI, 2014). In this context, herbs and spices are highly vulnerable to food fraud, particularly due to their extensive global trade (Velázquez *et al.*, 2023). Moreover, spices are frequently marketed in ground form, making them susceptible to the incorporation of lower-value ingredients without consumer detection (Prado *et al.*, 2021).

One of the most frequently adulterated spices worldwide is black pepper (*Piper nigrum* L.) due to its high commercial value (Tremlova,

2001; Vidal, 2020; Zaukuu *et al.*, 2022). Such adulteration practices can be sophisticated and difficult to detect. In response, several techniques have been investigated to identify adulterants in black pepper to mitigate fraudulent activities. These include near-infrared (NIR) spectroscopy (Vadivel *et al.*, 2018; Wilde *et al.*, 2019, Lima *et al.*, 2020), quantitative PCR (qPCR) (Sousa, Ferreira, Faria, 2019), optical microscopy (Vadivel *et al.*, 2018), as well as combinations of methods, such as microscopy and spectroscopy (Lohumi, Lee, Cho, 2015; Lafeuille *et al.*, 2020; Meadow *et al.*, 2021). However, some of these techniques are costly and not feasible for routine application.

For fraud involving starch-based adulterants, optical microscopy offers distinct advantages, such as direct morphological identification

of starch granules (corn and wheat) without the need for high-cost equipment, as required for chromatographic techniques or mass spectrometry. These methods often require hazardous solvents and specialized personnel training, whereas optical microscopy is easy to perform, has low operational costs, and is more sustainable, making it suitable for quality monitoring in small laboratories or spice-processing industries (Vadivel *et al.*, 2018).

Starch has frequently been detected as an adulterant in seasonings such as garlic, ginger, onion powder, and ground black pepper. This fraudulent practice is achieved to obtain economic gain by partially replacing higher-value ingredients with lower-cost raw materials. Thus, starchy materials are used as adulterants due to their wide natural availability, ease of access, low cost, and inertness in terms of color, odor, and flavor (Lohumi *et al.*, 2014; Lima *et al.*, 2020; Macedo, 2023; Velázquez *et al.*, 2023; Sari *et al.*, 2025).

Given this context, this study was developed in order to quantify the presence of corn (*Zea mays*) and wheat (*Triticum aestivum*) starch in ground black pepper using optical microscopy, and to validate equations for estimating starch percentage based on starch granule counts. Additionally, commercial samples and samples from suppliers to the food industry were evaluated.

Material and methods

Mathematical modeling for determining the percentage of starch granules in ground black pepper samples

Black pepper grain samples (*Piper nigrum*) free of impurities, obtained from a producer in Linhares, Espírito Santo State, Brazil, were ground using an industrial processor and sieved through a 60-mesh sieve (0.250 mm) to achieve commercial granulometry (maximum of 50 %

of the material passing through the 60-mesh sieve). These samples were classified as the pure standard.

Corn starch was donated by a sauce and seasoning manufacturer, and wheat flour (Type 1) was purchased from a local business; both were located in Machado, Minas Gerais State, Brazil. To simulate adulteration, two experiments were conducted in a completely randomized design (CRD), with four replicates, by separately adding two starch sources to pure ground black pepper.

In the first experiment, black pepper was adulterated with corn starch at levels of 3 %, 5 %, 10 %, 15 %, and 20 %. In the second experiment, wheat flour was added at the same concentrations. Adulteration levels above 20 % were not tested, as they altered the characteristic color of ground black pepper, resulting in a whitish appearance.

Microscopic analysis of the intentionally adulterated samples was performed according to a methodology adapted from Prado *et al.* (2021). Briefly, 0.4 g of the sample was homogenized, clarified with 2.5 % sodium hypochlorite for ten minutes, and filtered using a glass funnel and filter paper. A portion of the filtered sample was then placed on a microscope slide, followed by the addition of three drops of Lugol's solution (1 % potassium iodide and 0.35 % sublimated iodine). Excess Lugol's solution was removed with blotting paper and one drop of glycerinated water (1:1, v/v) was added before placing the coverslip.

One slide was prepared for each replicate of each corn starch and wheat flour concentration. Three photomicrographs were taken from each slide using a Leica DM 2500 optical microscope (Leica Microsystems, Wetzlar, Germany) equipped with a Leica EC3 camera and a 40X objective lens, totaling 60 images of each starch source. Images were captured using Leica LAS EZ software, and starch granules were subsequently counted using ImageJ software. For the quantification of corn

and wheat starch granules, a minimum particle size of $5\ \mu\text{m}$ (based on the smallest granule diameter) was established to facilitate visual counting and ensure standardization.

Statistical analysis was performed using Sisvar software (Ferreira, 2011). To meet the assumptions of analysis of variance (homoscedasticity and normality of errors), data transformation was required, adopting $y = \sqrt{z+0.5}$, in which y represents the transformed value and z is the average number of granules observed according to the methodology previously described. Analysis of variance and regression analyses were performed using transformed data. Polynomial regression models were selected based on the significance of the F-test, goodness of fit, and coefficient of determination (R^2). After model selection, 95 % confidence intervals were estimated to calculate the adulteration percentage from the mean number of corn or wheat starch granules.

A spreadsheet was developed using Microsoft Office Excel to calculate the estimated mean starch percentage, as well as lower and upper limits.

Validation of the mathematical model (blind test)

To validate the fitted equations for starch granule counting, pure ground black pepper samples were intentionally adulterated with 1 % and 7 % corn starch and 4 % and 12 % wheat flour; one sample remained unadulterated (control). Seven replicates were prepared for each concentration, totaling 35 samples. The selected concentrations were randomly assigned and unknown to the analysts (blind test). Samples were randomized and arbitrarily coded from 1 to 35 prior to analysis. For each sample, three slides were prepared, and three images were captured from each slide, totaling 315 images. Image analysis was performed as previously described.

The mean number of starch granules detected in the samples was grouped according to the type of adulterant identified and the estimated percentage of adulteration calculated from the fitted equation for each starch type, including the corresponding lower and upper limits, using the developed spreadsheet. These results were subsequently compared to the actual adulteration levels, which were disclosed after analysis, aiming to validate the proposed equation.

Evaluation of commercial and supplier samples

Commercial and supplier samples of ground black pepper were evaluated by optical microscopy using the same methodology previously described. Three different commercial brands (A, B, and C), packaged in 15 g units, were purchased from local retailers in Machado, Minas Gerais State, Brazil. Additionally, samples from three different suppliers (X, Y, and Z) were provided by a sauce and seasoning manufacturer located in the same municipality.

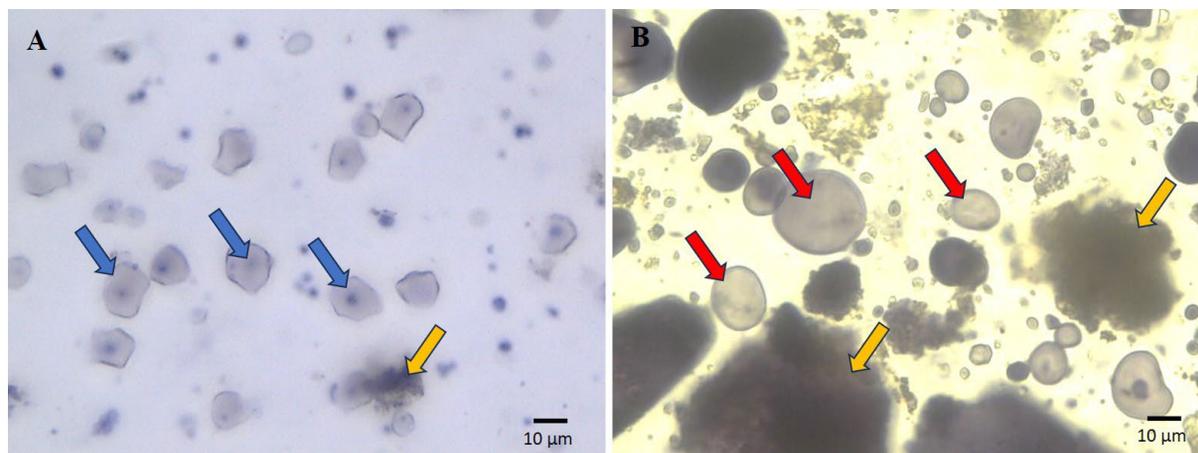
Results and discussion

Detection of starch by optical microscopy

Microscopic analysis of the intentionally adulterated black pepper samples enabled the visualization of corn and wheat starch granules, making it possible to identify their characteristic morphology. Clear structural differences were observed between natural plant tissues of black pepper and starch granules of corn (Figure 1A) and wheat (Figure 1B).

Corn starch granules exhibit a predominantly polyhedral shape, with some slightly rounded forms, and a central hilum (Figure 1A), consistent with the descriptions reported by Soares *et al.* (2017) and Pérez, Baldwin, and Gallant (2009). This morphology reflects their characteristic

Figure 1. Identification of starch granules by optical microscopy in ground black pepper (*Piper nigrum*) samples adulterated with corn starch (A) and wheat flour (B). Yellow arrows: black pepper plant structures; blue arrows: corn starch granules; red arrows: wheat starch granules. Machado, Minas Gerais State, Brazil, 2024.



Source: authors (2024).

crystalline organization (Pérez, Baldwin, Gallant, 2009). In contrast, starch granules derived from wheat display a bimodal size distribution. Larger granules, approximately $22 \mu\text{m}$ in diameter, are lenticular in shape, whereas smaller granules have an average diameter of $6 \mu\text{m}$ and a rounded shape (Figure 1B). These features are consistent with observations reported by Yonemoto, Calori-Domingues, and Franco (2007), Patel and Seetharaman (2006), and Pérez, Baldwin, and Gallant (2009).

The natural plant tissues of black pepper exhibit irregular shapes and heterogeneous cellular composition (Figure 1). Conversely, black pepper starch granules were not clearly visualized due to their significantly smaller size compared to those of corn and wheat. According to Zhu, Mojel, and Li (2017), black pepper starch granules range from 3.44 and $4.51 \mu\text{m}$ in diameter and present a polygonal shape, smooth surface, and monomodal size distribution.

Vadivel *et al.* (2018) highlight optical microscopy as an effective tool for detecting adulterants in spices, especially when morphological differences are evident, as observed between the added starch granules and the native plant matrix of black pepper. This

approach supports the findings of this study, in which the distinctive structural features of corn and wheat starch granules enabled their clear differentiation from the intrinsic pepper structures.

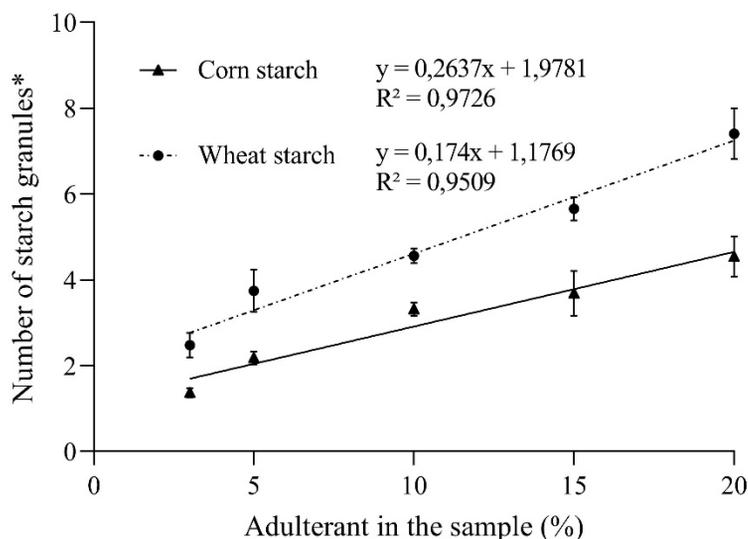
Mathematical modeling to determine the percentage of starch granules in ground black pepper samples

The number of starch granules in ground black pepper differed significantly among samples containing different percentages of corn (*Zea mays*) and wheat (*Triticum aestivum*) starch. A linear relationship was observed between starch granule counts and the estimated percentage of adulteration, enabling the adjustment of a specific mathematical model for each type of adulterant. A trend line was fitted for each model (Figure 2). Both linear equations exhibited coefficients of determination (R^2) above 0.95.

These equations enable the estimation of adulteration percentages in unknown black pepper samples based on the number of starch granules identified by optical microscopy.

Confidence intervals for the mean granule counts were estimated at ± 3.30 for corn starch and ± 3.48 for wheat starch. These values were

Figure 2. Starch granule counts (transformed data*) in ground black pepper (*Piper nigrum*) samples adulterated with 3 %, 5 %, 10 %, 15 %, and 20 % corn starch (*Zea mays*) and wheat flour (*Triticum aestivum*), analyzed by optical microscopy. Mean values \pm standard error, regression equation, and coefficient of determination (R^2). Machado, Minas Gerais State, Brazil, 2024.



*Transformed data ($y = \sqrt{z+0.5}$), in which z is the number of starch granules.

Source: authors (2024).

used to validate the starch granule counting method for estimating adulteration percentage.

The spreadsheet developed to calculate the estimated starch percentage, as well as the corresponding lower and upper limits, is available at https://docs.google.com/spreadsheets/d/1HG S8fo_5h5osMTQuXVmh3T09NzkoU3qfVgHTp_Je_T4/edit?usp=sharing. To use the spreadsheet, the user must enter the mean number of starch granules observed and select the starch source (corn or wheat). The file should be accessed using a Google account; if this is not possible, it can be downloaded and a copy locally created for use.

Validation of the starch granule counting method in ground black pepper samples (blind test)

The results obtained from the blind test were grouped according to the type of adulterant identified, the estimated adulteration percentage, and the corresponding lower and upper limits

(Table 1). These results were compared to the actual adulteration levels, which were disclosed after analysis, aiming to validate the fitted equations (Figure 2).

Among the 35 samples evaluated by optical microscopy, correct adulteration levels were estimated for 25 samples, corresponding to 71.43 % of the total, thereby supporting validation of the starch granule counting model. An additional 10 samples (11.43 %) had discrepancies only at the first decimal place, while 17.14 % fell outside the confidence interval, although the estimated values remained close to the actual adulteration levels. It is noteworthy that lower starch concentrations made detection and estimation more challenging, leading to greater uncertainty in identifying fraud in samples containing starch levels below 1 %.

Based on the data obtained from the blind test, optical microscopy proved effective in identifying and counting corn and wheat starch granules, and the linear equation fitted in this

Table 1. Estimated percentages of corn starch and wheat flour in adulterated ground black pepper samples, calculated using the developed spreadsheet. Machado, Minas Gerais State, Brazil, 2024.

Adulterant*	Estimated mean adulteration (%)	Lower limit	Upper limit	Real adulterant	Actual adulteration (%)
Corn starch	1.34	0.00	4.64	Corn starch	1
Corn starch	10.00	6.70	13.30	Corn starch	7
Wheat flour	7.76	4.28	11.24	Wheat flour	4
Wheat flour	15.13	11.65	18.62	Wheat flour	12
Not detected	0.00	–	–	None	0

*Adulterant identified by the analyst in the blind test.

Source: authors (2024).

study enabled satisfactory quantification of adulteration percentages.

In a study on the detection and quantification of impurities in roasted and ground coffee, Mendes *et al.* (2016) reported that the accuracy and precision of the microscopic method were satisfactory for determining impurity concentrations from 0.2 % onward. Method uncertainty varies according to the concentration range and tends to be inversely proportional—that is, the higher the impurity concentration, the lower the method uncertainty.

In this study, the use of optical microscopy enabled direct visual analysis of corn and wheat starch granules, allowing morphological identification and quantification of adulteration without the need for complex mathematical approaches. Conversely, spectral techniques such as NIR spectroscopy face challenges due to the overlap of absorption peaks from components with similar functional groups (e.g., carbohydrates, proteins, and lipids) (Lima *et al.*, 2020). Furthermore, validation of the fitted mathematical model via a blind test reinforces the reliability of microscopic analyses conducted in this study, whereas spectral methods may require mathematical adjustments that can reduce sensitivity, as reported by Lima *et al.* (2020).

In light of this, the fitted equations (Figure 2) are valuable tools for estimating the percentage of

corn (*Zea mays*) and wheat (*Triticum aestivum*) starch present in ground black pepper samples. These tools may assist food industry professionals who use black pepper as an ingredient.

Evaluation of commercial and supplier samples of ground black pepper

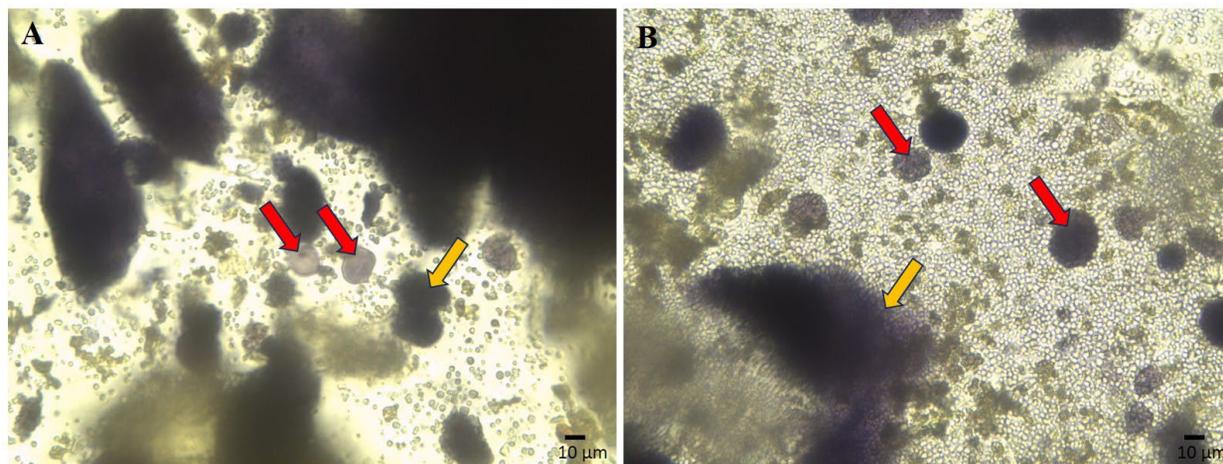
Microscopic analysis of commercial and supplier samples revealed the presence of wheat starch granules in all three commercial brands purchased locally and in two samples supplied by the sauce and seasoning manufacturer that uses ground black pepper as an ingredient (Figure 3).

According to the results obtained for commercial and supplier samples, no evidence of adulteration with corn starch was identified. However, the estimated percentage of wheat starch detected in the commercial samples may indicate possible adulteration. The highest percentage was observed in brand C (3.46 %) (Table 2).

No starch granules were observed in the black pepper sample from supplier X. For supplier Y, an average of 0.67 granules was detected, which may be considered negligible. However, the highest mean percentage of wheat starch was found in the sample from supplier Z, estimated at 4.81 %, which may indicate possible adulteration.

Prado *et al.* (2021) reported the presence of starch in 16.1 % of spice samples—including

Figure 3. Identification of starch granules by optical microscopy in a commercial sample (brand C) (A) and a supplier sample (Z) (B) of ground black pepper (*Piper nigrum*). Yellow arrows: black pepper plant structures; red arrows: wheat starch granules. Machado, Minas Gerais State, Brazil, 2024.



Source: authors (2024).

turmeric, ginger, nutmeg, paprika, and black pepper—commercialized in the state of São Paulo. According to the authors, 55.2 % contained corn starch (*Z. mays*), while 44.8 % contained both corn starch (*Z. mays*) and annatto (*Bixa orellana*) starch, suggesting potential fraud in these products. Given the relatively low economic value of corn starch compared to the evaluated spices, its incorporation may indicate intentional adulteration aimed at economic gain.

Therefore, this practice increases the economic incentive for adulteration by mixing spices with lower-cost ingredients, such as starch or flour. Moreover, the choice of adulterants is

influenced by their ready availability and their ability to avoid altering the color, aroma, and flavor of the final product (Sari *et al.*, 2025).

Fioravanti *et al.* (2024) analyzed six bulk ground black pepper samples and found unsatisfactory results in five of them due to the detection of histological elements of corn starch, characterizing adulteration. According to the authors, food products are subject to fraudulent practices that require continuous monitoring, since such adulteration may cause significant health risks to consumers and economic losses. These findings highlight the need to intensify regulatory oversight and improve good manufacturing practices within this sector.

Table 2. Estimated percentage of wheat starch in ground black pepper samples from three commercial brands and three suppliers, calculated using the developed spreadsheet. Machado, Minas Gerais State, Brazil, 2024.

Samples	Adulterant identified	Estimated mean (%)	Lower limit	Upper limit
Brand A	Wheat	0.53	0.00	4.01
Brand B	Wheat	1.91	0.00	5.39
Brand C	Wheat	3.46	0.00	6.94
Supplier X	Not detected	–	–	–
Supplier Y	Wheat	0.00	0.00	2.93
Supplier Z	Wheat	4.81	1.33	8.29

Source: authors (2024).

The adulteration of ground spices, such as black pepper, is a common practice. Lima *et al.* (2020) reported its occurrence in 62 % of black pepper samples and 79 % of cumin samples, primarily involving low-cost starches such as corn flour and cassava starch. The authors emphasized that corn flour, in particular, is a difficult adulterant to detect due to its ability to mask visual and analytical characteristics of spices. These findings are consistent with those of this study, which detected wheat starch adulteration in commercial black pepper samples and in samples supplied to the food industry, reinforcing the need for accessible and reliable monitoring methods.

Using a microscopic analysis method similar to the one in this study, Pauli-Yamada *et al.* (2021) detected foreign elements in 30 % of 43 paprika samples evaluated, with corn starch being the most frequent adulterant, present in 85 % of the adulterated samples. Similarly, Thein, Tin, and Nge (2021) identified abundant starch granules in three powdered spices (turmeric, Thai ginger, and black pepper) available on the market using microscopic analysis.

Adulterants may also be incorporated into food products as thickening agents to increase profit margins (Pantoja, 2017). Such practices may pose serious risks, particularly in terms of public health. For individuals with celiac disease—an autoimmune disorder triggered by gluten intolerance (a protein found in wheat and its derivatives)—gluten consumption impairs the body's ability to absorb nutrients, vitamins, minerals, and water (Brasil, 2020).

Food adulteration may result in the commercialization of products of inferior quality and potentially unsafe for consumption (Osman *et al.*, 2019). It also has serious implications for the food industry, given that ground black pepper is widely used as an ingredient in the formulation of various food products.

Thus, spice fraud is a relevant and emerging issue, as it may compromise food safety and product authenticity. This study, which focused on the identification of fraud in ground black pepper, contributes to quality assurance, consumer protection, and prevention of risks associated with the consumption of adulterated products.

Conclusion

Optical microscopy proved suitable for the detection and identification of corn starch and wheat flour granules in ground black pepper. The fitted and validated linear regression model enabled estimation of adulteration percentages for both corn and wheat starch, reinforcing the reliability of the results. Wheat starch was detected in commercial ground black pepper samples and in samples supplied to the food industry.

Therefore, optical microscopy can be highlighted as a practical and effective tool for the food industry, contributing significantly to fraud detection, quality assurance, and the authentication of spices.

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