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Feasibility of a chromameter and chemometric techniques to discriminate pure and mixed organic and conventional red pepper powders: A pilot study

Muharrem Keskin[1] | Aysel Arslan[1] | Yurtsever Soysal[1] | Yunus Emre Sekerli[1] | Nafiz Celiktas[2]

[1]Department of Biosystems Engineering, Faculty of Agriculture, Hatay Mustafa Kemal University, Antakya, Hatay, Turkey

[2]Department of Field Crops, Faculty of Agriculture, Hatay Mustafa Kemal University, Antakya, Hatay, Turkey

Correspondence
Muharrem Keskin, Department of Biosystems Engineering, Faculty of Agriculture, Hatay Mustafa Kemal University, Antakya, Hatay, Turkey.
Email: keskin@mku.edu.tr; mkeskinhatay@gmail.com

Abstract

Food adulteration is a major problem causing significant economic and health risks for consumers. Non-destructive, quick, and inexpensive methods are needed for food authentication. No study has been found on the adulteration of organic pepper powders. This study examined the feasibility of a chromameter to discriminate pure and adulterated (mixed) organic red pepper powders obtained from intermittent microwave drying for the first time. PCA, SIMCA, and PLSR were used for the data analysis. The PCA exhibited a very good distinction based on product type (organic and conventional) and drying powers (150, 300, and 450 W), while the SIMCA effectively classified the samples as organic or conventional with a correct classification ratio of 94%. Also, the adulteration rate (%) of the organic pepper powders was predicted using PLSR with promising results ($R^2 = 0.90$). To summarize, the chromameter has a good potential to classify the red pepper powders as organic or conventional.

Novelty Impact Statement: Food adulteration has become a major problem on a global scale but no study has been conducted on the adulteration of organic pepper powders to the best knowledge of the authors. This work investigated the feasibility of a chromameter and chemometric techniques for the discrimination of organic red pepper powders from the ones adulterated with the same type of conventional pepper powders. The results revealed that a chromameter has a promising potential to classify the red pepper powders as organic or conventional.

1 | INTRODUCTION

Organic farming is a sustainable farming method in which production takes place in accordance with regulations that allow no synthetic chemical fertilizers and pesticides. It has gained importance since organic food products are free from synthetic chemicals that have had negative impacts on the environment and human health. Production is carried out under national and global regulations and certifications with systematic inspections by the private and state authorities (Gomiero, 2018; Guclu et al., 2021). It can offer environmental, social, and health benefits (Shennan et al., 2017). Organic foods are believed to be healthier and more nutritious but there have been some findings with no significant differences (Gomiero, 2018; Guclu et al., 2021; Shennan et al., 2017).

Drying is one of the crucial food preservation methods by removing excess unbound water from the agricultural products causing some chemical reactions and quality changes. Hence, the drying method and drying parameters (drying temperature, air velocity, material size, slice thickness, etc.) considerably affect the product quality including color, flavor, texture, nutrition, etc. (Arslan et al., 2020; Calin-Sanchez et al., 2020; Karam et al., 2016). Intermittent (pulsed) microwave drying is an effective method with energy savings, better quality products, and shorter drying time due to the effective permeation of the applied radiation energy into the material (Esturk et al., 2011; Keser et al., 2020; Soysal, 2009; Thamkaew et al., 2020).

There has been a growth in the production and market of organic foods in the world and in Turkey, but the selling price of the organically produced foods is higher than the conventionally produced ones. Mixing similar conventional food products into organic ones is common and considered as a type of food adulteration or fraud and requires food authentication. There are two food fraud types: intentional adulteration is the deliberate inclusion of substances that have similar properties with low cost into the adulterated foods to increase its amount or to enhance its quality, while unintentional adulteration is the contamination of the foods during production or storage with biological or chemical factors (arsenic, mercury, pesticides, insects, larvae, droppings of rodents, etc.) (Banti, 2020; Choudhary et al., 2020; Fikselova et al., 2020). Intentional adulteration can be in different forms including the substitution of ingredients, masking of food origin, mislabeling, contamination, and enhancement with artificial ingredients suitable or unsuitable for human consumption (Fikselova et al., 2020). The frauded foods affect the consumers in different ways as being less nutritious, unsafe for health, and causing economic loss (Banti, 2020). On a global scale, adulteration has become a major problem in food products and the rate of frauded foods rises to 70%–90% in some countries (Mirza, 2014; Mouhiddin, 2019). Along with milk, olive oil, honey, seafood, meat, and meat products, spices are one of the food groups susceptible to adulteration as they are usually marketed in powder form (Dubecke & Weiss, 2020; Hassoun et al., 2020; Hong et al., 2017). The addition of edible oils and synthetic dyes especially Sudan dyes, which is a carcinogen and banned worldwide, into spices is common (Hong et al., 2017). It is impossible to detect the adulteration in organic foods by human eyes or taste. Thus, various quick techniques were studied to be alternative to costly and time-consuming laboratory analysis such as chromatography, spectroscopy, image processing, chromametry, etc. (Hong et al., 2017). The color of fresh or dried foods has been used as an indirect measure of their quality (Soysal et al., 2005; Keskin et al., 2017; Pathare et al., 2013). Chromameter-based assessment of food and agricultural products can be an alternative to the traditional laboratory methods with the ease of sample preparation in addition to time and cost savings.

Along with the numerous sensing techniques, some enhanced qualitative and quantitative chemometric data analysis methods are employed for the easier, inexpensive, and non-destructive detection of adulterated foods. They provide an opportunity to determine the separation, classification, and correlation of the samples. Some mostly used methods include principal component analysis (PCA), classification methods such as soft independent modeling of class analogy (SIMCA), and partial least squares regression (PLSR). PCA is a pattern recognition technique in which similar samples closer to each other are grouped together and is useful to identify sample clusters, but it is not a classification method (Esbensen, 2009). SIMCA is a qualitative analysis method for the classification of the samples based on PCA (Davies & Fearn, 2008; Suhandy & Yulia, 2017). On the other hand, PLSR depends on the causal relationships to conduct estimations of unknown values of a dependent variable from a set of independent variables.

Pepper (*Capsicum annuum* L.) is an inexpensive vegetable with high nutritional value. It is one of the vegetables with the most pesticide residues (EWG, 2021). It is grown in open field conditions or in greenhouses in almost every region of the world. The global production of peppers was about 36.8 million tons and Turkey ranked third after China and Mexico with a production of about 2.6 million tons in 2018 (FAOStat, 2021). Organic pepper production is also significant in Turkey with 5,558 tons in 2018 (TurkStat, 2021). Peppers are marketed as fresh or dried and utilized to produce spices, sauces, pastes, soups, oleoresin, and natural coloring agents (Arslan et al., 2020). Pepper powders or flakes are an important spice in many countries including Turkey.

Most of the food adulteration studies have concentrated on the addition of a food ingredient or colorant into another food product (Banti, 2020; Choudhary et al., 2020; Fikselova et al., 2020). Studies on mixing similar type of conventionally produced plant-based products into organic products have been very limited. Besides, there has been no study on the adulteration of organic pepper powders with conventional pepper powders to the best knowledge of the authors. Thus, the aim of this study was to investigate the feasibility of detecting conventional red pepper powders in the organic pepper powders produced from intermittent microwave drying using chromameter and chemometric methods.

2 | MATERIAL AND METHOD

2.1 | Organic and conventional peppers

Organic (certified) and same variety conventional sweet red peppers (Capia type, Diyar F1 variety) were bought (10 kg each) from greenhouse farmers in Erdemli city of Mersin province, Turkey in the late spring season of 2019. This pepper variety is produced and consumed in Turkey in significant amounts. Peppers were grown under similar greenhouse conditions by using organic and conventional farming practices. In the production of organic peppers, manures and natural grass on the soil surface were mixed to the soil, while a mixture of lime, sulfur and laurel leaf, and azadirachtin being an organic extract were utilized as pesticides. For the conventional peppers, synthetic fertilizers including macro and micronutrient elements and pesticides (insecticides, fungicides, herbicides, etc.) were utilized. The size of the organic peppers was about 12–15 cm, while the conventional peppers had a length of about 13–16 cm. All the peppers were in the full mature (red) stage. Peppers were stored at +4°C until the drying experiments. The wet-based moisture contents of the samples were quantified based on the oven method (103°C; 24 hr). The average wet-based moisture contents of the organic and conventional peppers were determined as 91.6 ± 0.30% and 91.9 ± 0.20%, respectively.

2.2 | Drying system and drying experiments

A modified household microwave oven (Beko MD 1605, Istanbul, Turkey) having 900 W power and a microwave frequency of 2.45 GHz was employed in the drying tests. The actual power of the microwave oven was calculated as 736 W by utilizing the IMPI-2L method (Soysal, Arslan et al., 2009). The microwave oven was modified to be operated with different magnetron on and off times using an electronic controller. Materials were dehydrated using three different power rates of 150, 300, and 450 W by modifying the microwave on and off times (Table 1).

Organic and conventional red sweet peppers were dehydrated by applying three different intermittent microwave power levels (150, 300, 450 W). These power rates were selected based on the results of the preliminary experiments and the previous experience of the authors. Pepper samples were selected with similar size, color, and maturity level. After the samples were washed and excess water was removed, they were sliced (slice thickness: 1.43 ± 0.07 mm). Sliced peppers of 150 g (about three peppers) were evenly placed on the surface of drying glassware microwave oven tray (diameter: 31 cm, mass: 1,150 g) with a thickness of 7.6 ± 0.07 mm. Then the sample was dehydrated. The mass of the material on the tray was continuously measured on a 0.01 g accuracy digital scale (Sartorius TE3102S, Germany). Each drying process was completed when the moisture level of the sample reached a level of 0.10 kg [H_2O] kg^{-1} [DM] (10% wet based). The drying process was carried out with seven replications for each power rate for both pepper types giving a total of 42 drying trials (2 product types (organic, conventional) × 3 power levels × 7 replications) (Table 1). The dried products were then powdered with an electric grinder (BlueHouse, BH258CG) (Figure 1).

2.3 | Preparation of the adulterated samples

This study was carried out on both pure and adulterated (mixed) organic and conventional sweet red pepper powders. Organic pepper powder materials were adulterated with the same variety of conventional pepper powders which were dried by applying the same power in five different proportions (0%, 25%, 50%, 75%, and 100%). Each adulterated sample was prepared with a total of 10 g of mixed material sample (Table 2). Adulterated samples were prepared for each of the three drying power rates (150, 300, and 450 W) giving a total number of 15 adulterated samples (5 mixture proportions x 3 power rates).

2.4 | Color measurement of the pure and adulterated samples

Color data of the pure and adulterated red pepper powders were quantified by employing a hand-held chromameter (Model CR-400, Minolta, Osaka, Japan). The color model of CIE $L^*a^*b^*$ was utilized in the color data analysis. In this color model, L* is the color brightness (0: black or dark color, 100: white or light color), a^* denotes the red or green color (negative values up to −60 are green and positive values up to +60 are red), while b^* parameter represents the yellow or blue color (negative values up to −60: green, positive values up to +60: red) (Keskin et al., 2017). The color data were obtained from the 42 pure and 15 adulterated powder samples. The color difference (ΔE) data were computed using the formula given below (Guclu et al., 2021):

$$\Delta E = \left[\Delta L^{*2} + \Delta a^{*2} + \Delta b^{*2}\right]^{0.5}$$

where ΔL^*, Δa^*, and Δb^* denote the differences in the color data of the fresh and powdered samples.

2.5 | Data analysis

Three different chemometric data analysis methods were utilized for the classification of the pure and adulterated pepper powders by utilizing the $L^*a^*b^*$ color data. These methods include principal component analysis (PCA), soft independent modeling class analogy (SIMCA), and partial least squares regression (PLSR). Data analyses were conducted in the Unscrambler multivariate statistics program (v.10.4, Camo, Norway).

TABLE 1 Details of the drying experiments using intermittent microwave (IMW) drying system

Product	IMW power rates	Specific power rates[a]	Microwave on-off time	Drying trials	Total number of samples ($N = 42$)
Organic peppers	150 W	1.0 W/g	15 s; 59 s	7	
	300 W	2.0 W/g	15 s; 22 s	7	$N_{org} = 21$
	450 W	3.0 W/g	15 s; 10 s	7	
Conventional peppers	150 W	1.0 W/g	15 s; 59 s	7	
	300 W	2.0 W/g	15 s; 22 s	7	$N_{conv} = 21$
	450 W	3.0 W/g	15 s; 10 s	7	

[a]150 g of fresh pepper sample was dried in each drying experiment.

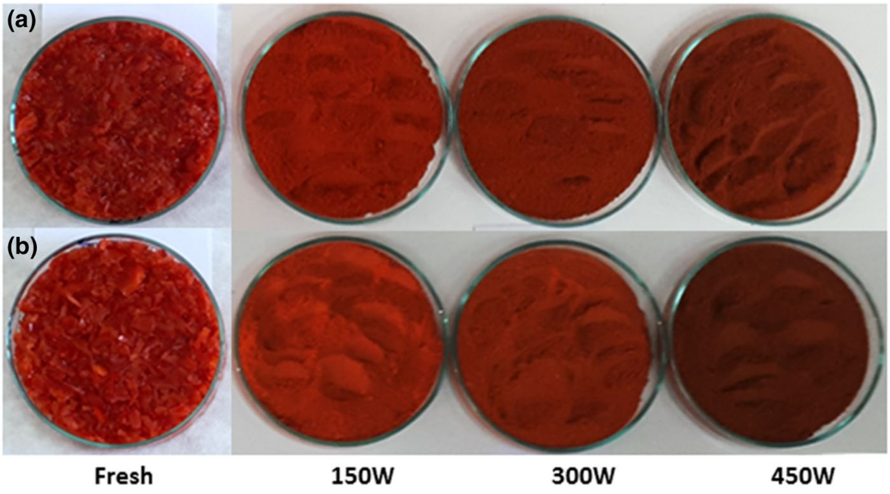

FIGURE 1 Fresh and dried organic (a) and conventional (b) sweet red pepper samples dried with three intermittent microwave power rates (150, 300, 450 W) in glass petri dishes (d = 9 cm)

TABLE 2 Proportions of the frauded (mixed) samples from organic and conventional pepper powders

Adulterated sample level	Organic pepper amount (g)	Conventional pepper amount (g)	Total amount (g)	Adulterated material code
1	10.0	0.0	10.0	0%
2	7.5	2.5	10.0	25%
3	5.0	5.0	10.0	50%
4	2.5	7.5	10.0	75%
5	0.0	10.0	10.0	100%

2.5.1 | Principal Component Analysis (PCA)

PCA is a pattern recognition technique in which samples with similarities are grouped closer to each other (Esbensen, 2009). It is not a classification method but is useful to identify sample clusters. The score plot is used to study the similarities and differences among the samples.

The color data of the pepper powders were analyzed with the PCA according to the product type (organic and conventional) and the applied microwave power levels (150, 300, and 450W). The pure pepper powder data (N = 42) were divided into training (calibration) (N_{cal}=24) and validation (test) sets (N_{val}=18). Mean centering and variable scaling of 1/StDev were used for the color variables ($L^*a^*b^*$) in the data analyses. Similarly, adulterated pepper powder data (N = 15) were analyzed using the PCA with full-cross validation since the number of total data is limited.

2.5.2 | SIMCA analysis

SIMCA (soft independent modeling class analogy) is a qualitative data analysis method for the classification of samples based on PCA (Davies & Fearn, 2008).

The color data of the pepper powders were analyzed for the classification of the samples into two groups (organic or conventional) by using the SIMCA technique. The pure pepper powder data (N = 42) were divided into training (calibration) (N_{cal}=24) and validation (test) sets (N_{val}=18). SIMCA classification was applied with two stages with 5% significance level. In the first stage, two different PCA models (organic or conventional) were created and saved by using the training data (N_{cal}=24). In the second stage, the data in the test set (N_{val}=18) were classified according to the previously created PCA models. It was examined whether organic and conventional pepper powders could be classified as organic or conventional. The classification results were evaluated based on Cooman's plot that was created after the SIMCA analysis. This plot has four regions as organic, conventional, organic or conventional (overlap), and non-classified. Classification ratios were calculated and tabulated based on Cooman's plot.

2.5.3 | PLSR analysis

In chemometrics, the three mostly used regression procedures are multiple linear regression (MLR), principal component regression (PCR), and partial least squares regression (PLSR). MLR is not a suitable method when the X variables ($L^*a^*b^*$ color variables in this study) are not orthogonal; hence, PCR or PLSR are more appropriate methods (Esbensen, 2009; Keskin et al., 2013). PLSR provides better results than PCR in most applications and has been used in various food adulteration studies (Genis et al., 2021; Sezer et al., 2019; Temizkan et al., 2020).

In the present work, the color data of the adulterated red pepper powders were also analyzed by using the PLSR to predict the adulteration rate (%) of the samples. The adulteration rates of the samples were from 0% to 100% with an increment of 25%. Full cross-validation was employed as the model validation technique since the number of adulterated data was limited ($N = 15$). The accuracy of the prediction model was assessed according to the coefficient of determination (R^2) and the root mean standard error of cross-validation (RMSECV) which was considered as similar to the root mean standard error of prediction (RMSEP).

3 | RESULTS AND DISCUSSION

3.1 | Color data analysis results

Color data of the fresh and dried-powdered conventional and organic sweet red pepper materials are displayed in Table 3 and Figure 2. The mean L^*, a^*, and b^* color values were 30.93, 24.10, and 22.34 and 30.18, 25.29, and 21.81 for the fresh conventional and organic pepper samples, respectively. The mean L^* and b^* value differences between the fresh conventional and fresh organic samples were not significant ($p > .05$), while the difference in mean a^* values was only slightly significant ($p < .05$) (Table 3).

The drying process greatly increased the color brightness (L^*) making the powder samples in lighter color as compared to the fresh samples (Table 3 and Figure 2). Similar results were reported for red pepper (Moraes et al., 2013; Soysal, Ayhan et al., 2009) and carrot (Keskin et al., 2019). Similarly, Korkmaz et al. (2021) found lower L^* values in the fresh red pepper samples compared to the spices, probably due to the losses in carotenoid content or a rise in the bright pigment content during drying, resulting in a lighter color. On the contrary, Deng et al. (2018) reported lower L^*, a^*, and b^* values and lower red pigment contents in dried red peppers (pulsed-vacuum, hot-air, and combined infrared-hot-air drying) as compared to the fresh ones. Similarly, Cao et al., (2016) found lower L, a, and b values in the dried red peppers (sun, infrared, and hot-air drying) as compared to the fresh ones. Also, Ning et al., (2012) and Pinar et al. (2021) reported a reduction in the L value in red pepper after drying. These variations could be tied to the drying method and drying parameters.

Red color intensity is an indication of color quality for red peppers and is provided by carotenoid pigments which are very vulnerable to degradation and oxidation during drying causing a quality loss in color and nutrition (Yang et al., 2018). The ASTA (American Spice Trade Association) color parameter varying from 40 (orange color) to 180 (dark red color) is a global color quality standard for red peppers. The higher ASTA values imply stronger red colors and a value from 70 to 160 is generally acceptable (Hwang et al., 2017; Pripravka, 2021). It was seen in this study that when the applied drying power increased, all color parameters ($L^*a^*b^*$) were decreased (Table 3; Figure 2). Among the powder samples, the ones dried with higher microwave power (450W) had a noticeably darker color than the ones dried with lower powers (150W and 300W) (Table 2; Figure 1; Figure 2). The darker red color can be tied to the degradation of the red pigments and the darker browning pigments formed by Maillard reactions (Deng et al., 2018; Korkmaz et al., 2021). Deng et al. (2018) reported lower amounts of red pigments in dried red peppers as compared to the fresh ones, showing the degradation of these pigments. In addition, it was observed in this study that the higher applied power rate (450W) produced a lower total color difference (ΔE) as compared to the lower power rates (150W and 300W) (Table 3). Zahoor and Khan (2021) found a total color change of 10.3–38.6 in red bell peppers dehydrated by microwave-assisted fluidized bed drying similar to the findings of the present study.

3.2 | PCA and SIMCA analyses results of the pure pepper powders

In the PCA of the 42 pure red pepper powders, 24 were used in the training set, while the remaining 18 were utilized in the test set. It

TABLE 3 Colour data of the fresh and dried-powdered conventional and organic red peppers

		Color parameters[1]			
Product type	Drying power	L^*	a^*	b^*	Total color difference (ΔE)
Organic pepper	Fresh	30.93 ± 0.02^a	24.10 ± 0.03^{cd}	22.34 ± 0.06^a	
	150 W	55.66 ± 0.86^e	27.54 ± 0.97^f	37.58 ± 0.91^f	29.27 ± 1.10^d
	300 W	53.51 ± 0.91^d	24.86 ± 0.98^{de}	35.22 ± 0.94^d	26.03 ± 1.20^c
	450 W	50.81 ± 1.16^b	19.73 ± 1.48^b	31.71 ± 1.34^c	22.48 ± 1.24^a
Conventional pepper	Fresh	30.18 ± 0.02^a	25.29 ± 0.04^e	21.81 ± 0.06^a	
	150 W	59.18 ± 0.97^f	27.62 ± 0.94^f	37.06 ± 0.85^{ef}	32.86 ± 1.19^e
	300 W	58.53 ± 0.87^f	23.73 ± 0.75^c	36.25 ± 0.79^e	31.86 ± 1.00^e
	450 W	51.81 ± 1.17^c	18.23 ± 1.38^a	29.51 ± 1.47^b	24.10 ± 1.17^b

[1]The data are the means and standard deviations of the three measurements. The different letters in the same column mean significant differences ($p < .05$).

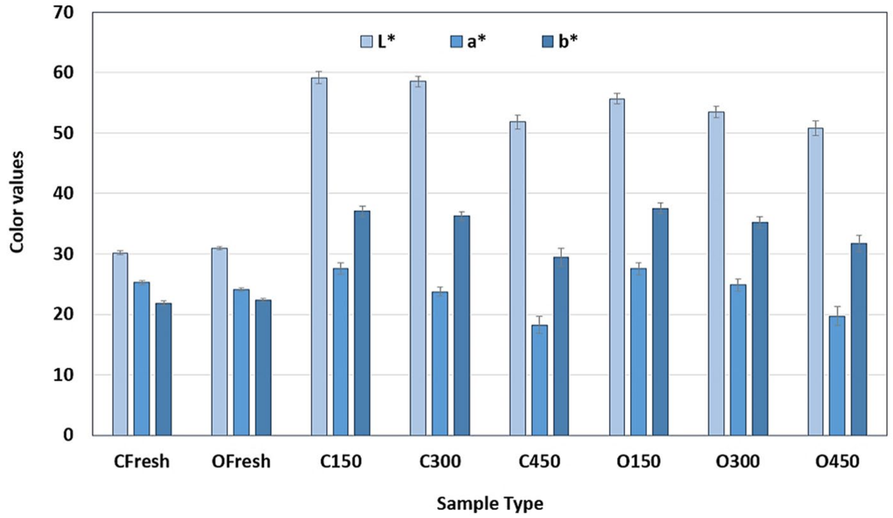

FIGURE 2 Colour data of the fresh and powdered organic (O) and conventional (C) red pepper samples (150W, 300W and 450W are the applied microwave powers)

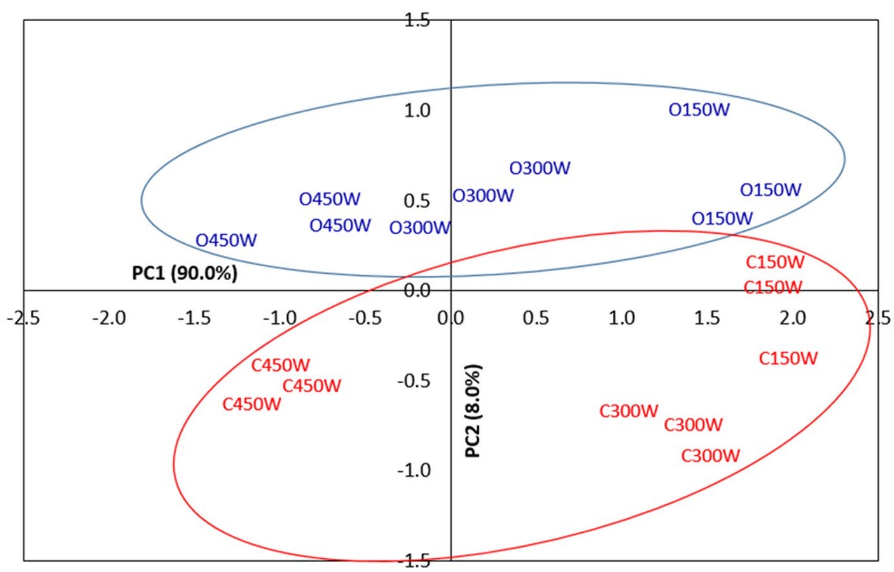

FIGURE 3 PCA score plot of the pure organic (O) and conventional (C) pepper powders using colour data (N_{cal} = 24, N_{val} = 18) (150W, 300W and 450W denote the applied microwave powers)

was seen that the first two PCA components explained a total of 98.0% of the variability (PC1: 90.0%, PC2: 8.0%) (Figure 3). The first component (PC1) represents the intermittent microwave power rate in a way that the samples dried with low power rate (150W) are located on the right side of the PC1 axis, while the ones dried with a high power rate (450W) are positioned on the left side of the same axis (Figure 3). Conversely, the second component (PC2) was important in terms of pepper type (organic or conventional) in a manner that the organic pepper samples were on the upper side, while the conventional samples are on the bottom part of the score plot. Based on these findings, it was observed that the PCA exhibited a satisfactory clustering trend for the distinction of the conventional and organic red pepper powder samples.

In the SIMCA analysis, two PCA models were created, one for the organic and the other for the conventional pepper powders using the training data (N_{val} = 24) and then, SIMCA classification was applied using the test data (N_{val} = 18). The Cooman's plot obtained from the SIMCA process is displayed in Figure 4, while the classification results are presented in Table 4. According to SIMCA classification results, it was observed that the color data could be used to make a good classification of the pepper powders. All nine organic pepper powders (100%) were correctly classified as

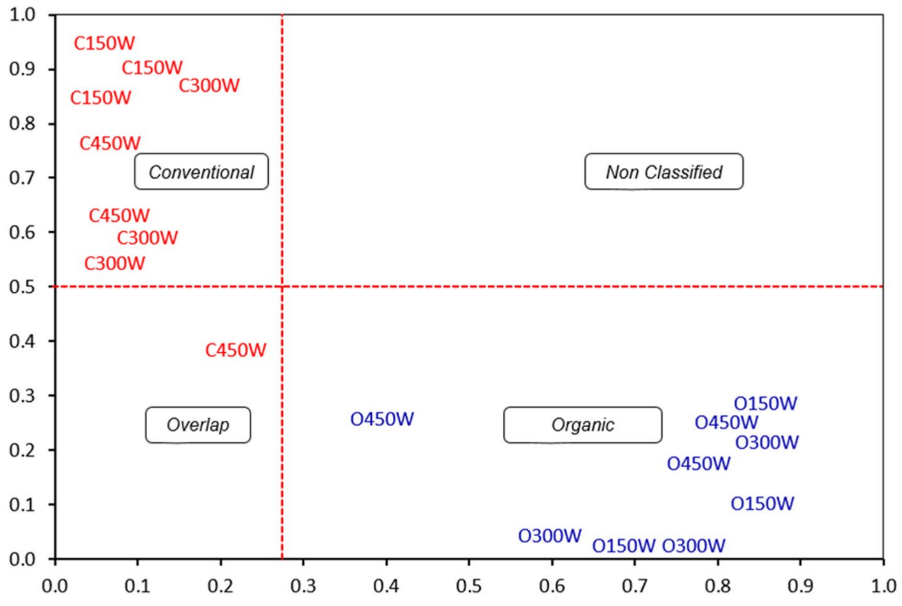

FIGURE 4 The Cooman's plot for the classification of organic (O) and conventional (C) pepper powders using colour data (N_{val} = 18) (150W, 300W and 450W denote the applied microwave powers)

TABLE 4 SIMCA classification results of the organic and conventional peppers using colour data

Samples	Classified as: Organic	Conventional	Organic or conventional (overlap)	Non-classified	Overall correct classification ratio
Organic (N_{test} = 9)	9/9 (100%)	0/9 (0%)	0/9 (0%)	0/9 (0%)	17/18 (94%)
Conventional (N_{test} = 9)	0/9 (0%)	8/9 (89%)	1/9 (11%)	0/9 (0%)	

organic, while eight of the nine conventional samples (89%) were correctly classified as conventional (Figure 4 and Table 4). One of the conventional powder sample (C450W) dried with 450W intermittent microwave power was classified as either organic or conventional (Figure 4). The overall correct classification ratio was found as 94%. It is thought that various compounds and properties in the peppers including phenolics, anthocyanins, volatiles, antioxidants, vitamins, minerals, pigments such as carotenoids, etc., may be available in different amounts in organic and conventional peppers (Guclu et al., 2021; Guilherme et al., 2020) which affect their color properties and make their classification possible.

3.3 | PCA and PLSR analyses results of the adulterated pepper powders

Organic pepper powder materials were adulterated (mixed) with the same variety conventional pepper powders which were dried by applying the same power by utilizing five different proportions (0%, 25%, 50%, 75%, 100%) (Table 2). The total number of adulterated samples was 15 (five mixtures and three drying powers). The color data ($L^*a^*b^*$) were utilized in PCA and PLSR data analyses with full cross-validation. One data were evaluated as an outlier and kept out of the calculation in the PLSR analysis.

PCA score plot of the adulterated organic pepper powders is displayed in Figure 5. It was observed that 100.0% of the variability could be modeled with the first two PCs (PC1: 92%, PC2: 8%) (Figure 5). The first component (PC1) represents the intermittent microwave power rate in a way that the samples dried with low power rate (150W) are located on the right side of the PC1 axis, while the ones dried with a high power rate (450W) are positioned on the left side of the same axis (Figure 5). On the other hand, the second component (PC2) is important in terms of the adulteration ratio (0%–100%) in a manner that the samples with lower adulteration ratio (0% and 25%) were on the upper side, while the one with higher adulteration ratio (75% and 100%) were on the bottom side of the score plot. Based on these findings, it was observed that the PCA exhibited a satisfactory cluster trend for the adulterated pepper samples.

Results of the PLSR analysis of the adulteration rate of organic pepper powders with color data are also presented in Table 5, while the reference versus predicted adulteration rate graph is shown in Figure 6. The RMSECV value was found to be 10.9% with a coefficient of determination (R^2) value of 0.90 (Table 5 and Figure 6). The outcome of the PLSR analysis exposed that the adulteration rate of the organic pepper powders can be predicted with promising results using the $L^*a^*b^*$ color data.

Non-destructive methods utilized for the evaluation of food adulteration have gradually become essential for the food quality

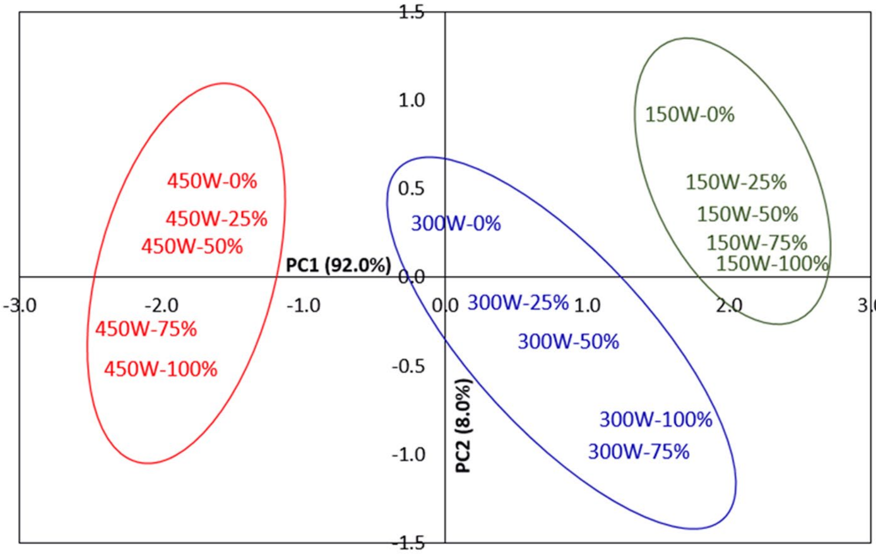

FIGURE 5 PCA score plot of the adulterated organic (O) and conventional (C) pepper powders using colour data ($N = 15$) (150W, 300W, 450W: applied microwave drying powers)

TABLE 5 Results of the PLSR of the adulteration rate of organic pepper powders with colour data

	RMSE	R^2	EV_X	EV_Y
Calibration	9.4% (RMSEC)	0.92	100	92
Validation	10.9% (RMSECV)	0.90	98	90

Note: RMSEC, root mean standard error of calibration; RMSECV, root mean standard error of cross-validation; EV_X, explained variance for X variables (L^*, a^*, b^*); EV_Y, explained variance for *Y* variable (adulteration rate).

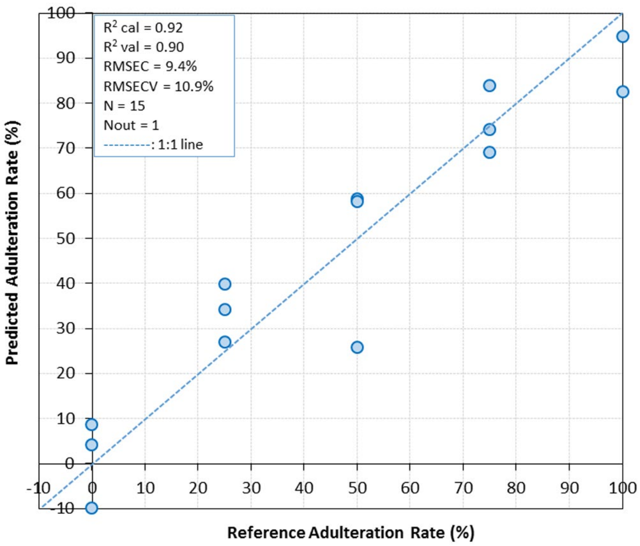

FIGURE 6 Reference versus predicted adulteration rate of the organic pepper powders using PLSR analysis with colour data

control and safety to reduce economic losses and health problems (Posudin et al., 2015). Globally, food adulteration and authentication have become a major concern in food products (Mirza, 2014; Mouhiddin, 2019). The majority of the food fraud research have dealt with the addition of a cheaper natural or synthetic ingredient or colorant into food products. Spices are one of the food categories susceptible to fraud mainly with synthetic organic dyes such as Sudan dyes (azo dyes) which are classified as carcinogens (Adjei et al., 2020; Hong et al., 2017). Red pepper, black pepper, saffron, turmeric, and onion powder are among the mostly adulterated spices (Khan et al., 2019). It is impossible to identify the frauded foods by human eyes or taste; hence, various non-destructive, rapid, and inexpensive testing methods have been studied including DNA-based methods, enzyme-linked immunosorbent assay (ic-ELISA), nuclear magnetic resonance, chromatography (GC/MS, LC/MS, etc.), vibrational spectroscopy (NIRS, MIRS), laser-induced breakdown spectroscopy (LIBS), fluorescence spectroscopy, terahertz (THz) spectroscopy, multispectral and hyperspectral image processing, electronic tongue, electronic nose, chromametry, elemental analysis including inductively coupled plasma (ICP), etc. (Hassoun et al., 2020; Hong et al., 2017; Mei et al., 2021; Posudin et al., 2015).

There have been numerous studies on the adulteration of spices including red pepper powders and flakes. A most common method of fraud has been the addition of edible oils and dyes and even brick powder into the red pepper spices to extend its shelf life, to enhance its color quality or increase its market value to obtain more profit (Banti, 2020; Gandhi & Mashru, 2019; Khan et al., 2020). In a study on the adulteration of red pepper spice, Khan et al., (2019) studied wheat flour, wheat bran, salt, and rice bran in grounded red chili peppers using hyperspectral imaging (HSI). In another work, Khan et al. (2020) detected mustard oil, olive oil, and Sudan dye in red chili pepper using a hyperspectral camera. Marquez et al. (2019) conducted a study to classify paprika samples adulterated with Sudan dyes by using UV-visible spectral reflectance. Lohumi et al. (2018) determined Sudan dye and Congo dye in paprika powder by employing Raman hyperspectral imaging ($R^2 > 0.98$). Similarly, Monago-Marana et al. (2019) used Raman spectroscopy and ASTA (American Spice Trade Association) color values to discriminate non-adulterated paprika samples from the ones adulterated with Sudan dye. Some researchers used liquid chromatography (LC) to identify adulterated red pepper spices. Karatas et al. (2019) determined eleven azo dyes in red pepper flakes by using a LC and mass spectrometry (LC-MS/MS). Ultrafast LC coupled with ultra-violet detector (UFLC-UV) was used by Adjei et al. (2020) to detect Sudan dyes in red chilli pepper powders. In addition, Sannino and Savini (2021) used ultra-high-performance LC (UHPLC) to determine synthetic dyes in chilli pepper. Near-infrared spectroscopy (NIRS) was also utilized for fraud detection in red pepper spices. Wu et al. (2017) used a NIRS system to detect adulteration in green and red peppers mixed with rice bran, wheat bran, corn flour, and rosin powder ($R^2 = 0.97$). Nuclear magnetic resonance (NMR) spectroscopy has also been used to identify adulteration in red pepper spices. Hu et al. (2017) investigated the feasibility of NMR method as a rapid and accurate determination of Sudan dye in paprika powders.

In food industry, the color data quantified by a hand-held or desktop chromameter have been employed to assess various quality features of the food products. Numerous studies have been conducted on the appraisal of the ripeness of vegetables and fruits and the color quality of the raw and processed foodstuff such as flours, breads, doughs, molasses, juices, oils, chocolates, meats, dairy products, milks, jams, and pastas, etc. (Keskin et al., 2017). On the other hand, no study has been found on the utilization of chromameters in the detection of food adulteration.

Organic food products gain importance due to its abundant nutrition and healthy attribute. While there has been many research on the food adulteration by adding some ingredients into various foodstuff, research on the discrimination of organically produced and conventionally produced foods has been very limited. Erich et al. (2015) employed gas chromatography (GC) and nuclear magnetic resonance (NMR) to differentiate organic and conventional milk with a classification rate up to 95%. Karaman et al. (2021) assessed organic and conventional cow milk by using Fourier-transform infrared (FTIR) spectroscopy. Computer vision and hyperspectral imaging (HIS) were utilized by Xu et al. (2017) for the distinction of conventional and organic farm-raised salmon fillets. In another study, NIRS was utilized to discriminate organic and conventional rice ($R^2 = 0.84$) (Xiao et al., 2019). Organic olives and olive oils were also studied by Jurado-Campos et al. (2021) by using different analytical techniques to authenticate conventional versus organic olives and olive oils of two varieties.

Studies on adulterated organic food products have been very limited. Su and Sun (2016) evaluated organic spelt flour from three mixed compounds with rye flour, spelt flour, and organic wheat flour using a full and selected spectrum of hyperspectral imaging (HSI; 900–1,700 nm). In a similar study, the same researchers utilized the same technique to detect Irish organic wheat flour mixed with cassava flour, common wheat flour, and corn flour (Su & Sun, 2017).

Studies on mixing similar type conventionally produced plant-based product into organic product have also been very scarce. There has been no study on the adulteration of organic pepper powders with conventional pepper powders to the best knowledge of the authors. In addition, the use of chromameters on the detection of adulteration in food stuff, especially in organic food products has been very limited. Thus, this research investigated the feasibility of a chromameter and both qualitative and quantitative chemometric techniques to detect the availability of the conventional red pepper powder in organic pepper powder dried by intermittent microwave drying. To our knowledge, this is the first study dealing with the separation of organic red pepper powder from the ones adulterated with the same type of conventional pepper powder. PCA, SIMCA, and PLSR analysis showed promising results for the discrimination of organic red pepper powders from the adulterated ones with a correct classification rate of 94% and R^2 value of 0.90. More studies are needed to assess the effect of various dehydration methods and the inclusion of other food ingredients such as edible oils, dyes, etc.

4 | CONCLUSION

This study was conducted to examine the feasibility of a chromameter and three chemometric data analysis techniques (PCA, SIMCA, and PLSR) to differentiate pure and adulterated (mixed) organic sweet red pepper powders mixed with same type of conventional pepper powders produced by utilizing intermittent microwave drying with three power rates (150, 300, and 450 W).

The study results revealed that the microwave drying process greatly influenced the color of both conventional and organic pepper powders with a significant increase in color brightness values (L^*) making the dried materials in a lighter color as compared to the fresh samples. The PCA exhibited a satisfactory cluster trend with two PCs explaining about 98% of the total variability and very good distinction of the samples based on both microwave drying powers (150, 300, and 450 W) and product type (organic and conventional) using three color parameters (L^*, a^*, b^*). Based on the SIMCA classification results, the color data could be used to make a good classification of the samples as organic or conventional with a correct classification rate of 94%. In addition, the findings from the PLSR analysis exposed that the adulteration rate (%) of the organic pepper powders can be predicted with promising results (RMSECV = 10.9%; $R^2 = 0.90$). In sum, findings show that chromameter has a good potential to classify the red pepper powders as organic or conventional. More studies are needed to include the effect of various dehydration methods and the inclusion of other food ingredients such as edible oils, dyes, etc.

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CONFLICT OF INTEREST

The authors declare no conflicts of interest in this article.

AUTHOR CONTRIBUTIONS

Muharrem Keskin: Conceptualization; Investigation; Methodology; Writing-original draft. **Aysel Arslan:** Data curation; Formal analysis.

Yurtsever Soysal: Conceptualization; Formal analysis; Methodology. **Yunus Emre Şekerli:** Data curation; Investigation; Visualization. **Nafiz Celiktas:** Data curation; Formal analysis.

ORCID

Muharrem Keskin https://orcid.org/0000-0002-2649-6855
Aysel Arslan https://orcid.org/0000-0002-0060-0263
Yurtsever Soysal https://orcid.org/0000-0003-0871-8570
Yunus Emre Sekerli https://orcid.org/0000-0002-7954-8268
Nafiz Celiktas https://orcid.org/0000-0002-0467-1034

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