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Scientific Research

Comparison of reflectance and interactance modes of near-infrared spectroscopy for non-destructive detection of garlic powder freshness

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ABSTRACT

Garlic medicinal plant powder is significant from a commercial perspective and is widely used as an additive in the food and pharmaceutical industries. Spectroscopic techniques serve as non-destructive methods for assessing the quality of food products, medicinal plants, and related products. In the present study, the efficiency and potential of two widely used modes, reflectance and intractance, in near-infrared spectroscopy in the range of 936 to 1660 nm were compared to assess the feasibility of distinguishing fresh garlic powder. In each spectroscopy mode, 120 spectra in 2 replicates were obtained from garlic powder samples, resulting in a total of 240 spectra. Prior to modeling, 25% of the spectra were randomly selected for validation, while the remaining spectra were used to compile the models. To mitigate potential noise, the impact of common pre-processing methods on the performance of the artificial neural network (ANN), support vector machine (SVM), and k-nearest neighbors (KNN) classifiers was investigated. The principal components analysis (PCA) technique was employed to reduce the dimensionality of the spectral variables, and the first four principal components were used as classifiers inputs. In the intractance spectroscopy condition, the SVM and KNN classifiers separated spectra obtained from powders at 3 days, 3 months, and 12 months with 100% accuracy. The ANN classifier achieved 100% accuracy in distinguishing the mentioned spectra in all preprocessing conditions under investigation, except for the raw spectra (without preprocessing). Near-infrared spectroscopy in the 936-1660 nm range, combined with chemometrics, is effective for quickly detecting the freshness of garlic powder. Considering its ease of application in both industrial and laboratory settings, intractance spectroscopy mode is superior to reflectance mode.

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1-Introduction

The medicinal plant garlic (Allium sativum L., Liliaceae) is a valuable species widely used in the pharmaceutical, food, cosmetic, and healthcare industries [1, 2]. Its bulb—the primary medicinal organ—is utilized in fresh, dried, extract, oil, slice, granule, and powder forms [3]. Garlic powder, one of its main products, has considerable commercial importance and is extensively employed in both food and pharmaceutical applications [4].

This beneficial species is cultivated and distributed across many regions worldwide and ranks among the most important and widely used medicinal plants [5]. Chemically, garlic contains carbohydrates, starch, proteins, vitamins, minerals, and mucilage [1, 6], as well as various organosulfur and amino-acid compounds [7]. These constituents underlie garlic's antibacterial, antioxidant, anticancer, immunostimulatory, and other therapeutic effects, which can be influenced by factors in production and processing particularly the timing of extraction and powder storage [8–13].

In near-infrared (NIR) spectroscopy, several sampling modes are employed -reflectance, transmittance, transflectance, and interactance- each defined by the relative positions of the light source and detector [14]. In reflectance mode, the intensity of light reflected from the sample surface is measured to generate a spectrum, whereas in interactance mode the light penetrates into the sample and is modulated by its internal absorption features before being collected. The optimal mode depends on sample type: transmittance or transflectance is preferred for liquids, while reflectance is generally used for powders [15, 16]. Rio et al. (2022) identified a band at 1924 nm in powder samples corresponding to water molecular bonds [17]. To correct for particle-size effects on spectral scattering, preprocessing methods such as multiplicative scatter correction (MSC) are often applied [18].

Hemrattrakun et al. (2021) compared reflectance and interactance modes of visible/NIR spectroscopy for predicting quality indices of persimmon. Their PLS model based on interactance data successfully estimated ASC and texture firmness but failed for SSC, while the reflectance-based model did not satisfactorily predict any traits [19]. Suktanarak et al. (2014) evaluated NIR spectroscopy for non-destructive egg-freshness assessment using both reflectance and interactance modes; they found interactance to outperform reflectance and recommended it for industrial egg classification [20].

Daszykowski et al. (2023) investigated rapid, non-destructive detection of adulteration in garlic powder, corn starch, and corn flour using NIR reflectance spectra (4000–10 000 cm^{-1}). They demonstrated that PLSR models could accurately predict adulteration levels in garlic powder and outperformed PCR [21]. Lohumi et al. (2015) used FTIR spectroscopy (650–4000 cm^{-1}) and PLSR modeling to detect corn starch in garlic powder, confirming the feasibility of non-destructive detection [22]. Wang et al. (2019) assessed NIR, MIR spectroscopy, and chemometrics for distinguishing strawberry powder made from fresh versus stored fruit (2, 4, 15, 20, and 27 days) using PLSR and LS-SVM, achieving 100 % discrimination [23].

Optical-fiber probes—especially Y-shaped interactance fibers—have become common in portable spectrometers, where interactance is often employed. However, no studies have directly compared powder scanning in reflectance (surface-sensitive) versus separate or interactance (internal-structure-sensitive) fiber modes. Therefore, the present research aims to compare reflectance and interactance sampling modes for the non-destructive detection of garlic powder freshness in the short-wave NIR range (936–1660 nm).

2- Materials and methods

2.1. Sample preparation

For this study, garlic samples were sourced from crops grown under the climatic conditions of Markazi Province (the local Arak white garlic landrace). Uniform, healthy bulbs free of any disease or pest damage were selected. At each sampling point, cloves were peeled and separated, then cut into 2–3 mm-thick slices. The

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slices were air-dried in the shade, away from direct sunlight, and then ground into powder using a laboratory mill. The resulting powders were evaluated at three storage intervals: after 3 days, 3 months, and 12 months.

2.2. Spectroscopy

Reflectance spectra were acquired by illuminating the sample at a 45° angle [24]. An Ocean Optics Flame–NIR spectrometer covering the 936–1660 nm range was used (Table 1). Prior to measurements, the light source was turned on for at least 30 minutes to ensure thermal stabilization and eliminate non-uniformity due to a "cold" lamp. Following this warm-up, absorbance spectra were calculated from the reflectance data using the relation, A=log(1/R), within the spectrometer software.

Table 1. Specifications of the spectrometer used

Spectrometer	Specifications
Ocean optics	Manufacturer
NIR- Flame	Model
88.9×63.5×31.9	Dimension (mm)
265	Weight (gr)
Hamamatsu G8160-03	Detector
128	Pixel number
936-1660	Range (nm)
6000 t0 1	S/N
10	Resolution (nm)

Two QP400-2-VIS-NIR optical fibers were used in reflectance mode, and a combined R400-7-VIS-NIR fiber was used in interactance mode to deliver and collect light (all from Ocean Optics). In reflectance mode, the two fibers were mounted in a fixed holder at a 45° angle to each other; in interactance mode, the send-and-receive fiber was likewise positioned at 45°. Each spectrum represents the average of 4 scans, and a total of 240 spectra were recorded. Prior to the first measurement and after every 10 spectra, both a background and a white-reference (barium sulfate) spectrum were acquired and saved. To maintain a constant angle and distance between the fibers and the powder surface during spectral acquisition, a custom cylindrical aluminum probe was fabricated and employed.

2.3. Chemometrics

Raw NIR spectra contain substantial noise due to particle-size variability, light-source and detector factors, and other unwanted influences. Therefore, to extract chemical information from the spectral data, various preprocessing methods are applied [25]. However, there are no definitive guidelines for choosing among these methods [21]. In the present study, several common preprocessing techniques were employed [18]. Standard normal variate (SNV) and multiplicative scatter correction (MSC) were used to remove scatter effects arising from differences in particle size and variations in the distance between the light source and the sample surface [26, 27]. Derivative processing (D) enhances peak resolution but also amplifies noise; hence smoothing is typically performed after derivatization [28]. The Savitzky–Golay (SG) smoothing method is a widely used

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post-derivative preprocessing. In this study, both first and second derivatives were evaluated, and SG smoothing was applied using a 4-point window and a second-order polynomial.

2.4. Principal Component Analysis (PCA)

In PCA, new variables are created, each of which is a linear combination of the original independent variables. These reduced new variables are the principal components: the first principal component captures the greatest variance, the second principal component covers the variance not explained by the first, and this process continues for subsequent components [29]. In this study, the first through fourth principal components were used as inputs to the classifiers.

2.5. Classifiers

Classification models have been employed in a variety of studies [30–33]. In this experiment, the capabilities of artificial neural network (ANN), support vector machine (SVM), and k-nearest neighbors (KNN) classifiers for discriminating among garlic powder samples were evaluated. Model validation is essential; hence approximately 25 % of the spectra were randomly selected by the software for validation prior to model building, and model calibration was performed using the remaining spectra. The percent correct classification of the validation samples was used to assess classifier performance (Equation 1):

$$\text{Accuracy } (\%) = \frac{TC}{N} \times 100 \quad (1)$$

where TC is the number of correctly classified samples and N is the total number of samples in the validation-set confusion matrix. Ocean View 1.6.3 was used for spectral acquisition, Unscrambler X 10.4 for preprocessing, and principal component analysis (PCA) and model building were carried out in IBM Modeler 18.

3- Results and discussion

In Figure 1, the absorbance spectra of the three types of garlic powder samples—each with different freshness levels—are shown in the 936–1660 nm range for both interactance and reflectance modes. The spectra within each mode, and between the two modes, appear highly similar overall, although absorbance intensities differ at certain wavelengths. Notably, the spectra collected in interactance mode exhibit greater uniformity and substantially reduced apparent noise, especially at the peaks. This improved consistency is likely due to the reduced impact of factors such as variations in the sample-to-fiber distance and diminished influence of surface irregularities in interactance sampling. The spectral profiles closely match those reported by Daszykowski et al. (2023) [21].

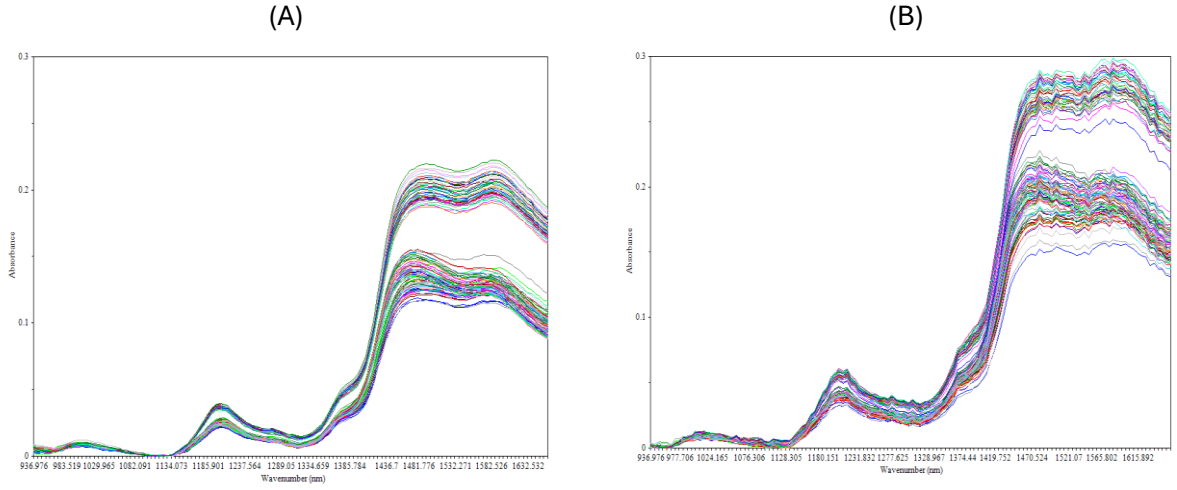

Fig 1. All acquired spectra (converted to absorption) on A: Intractance and B: Reflectance

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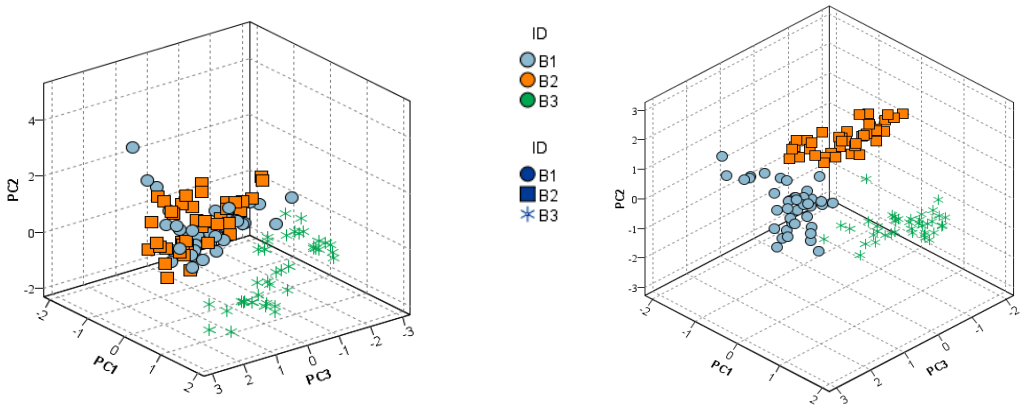
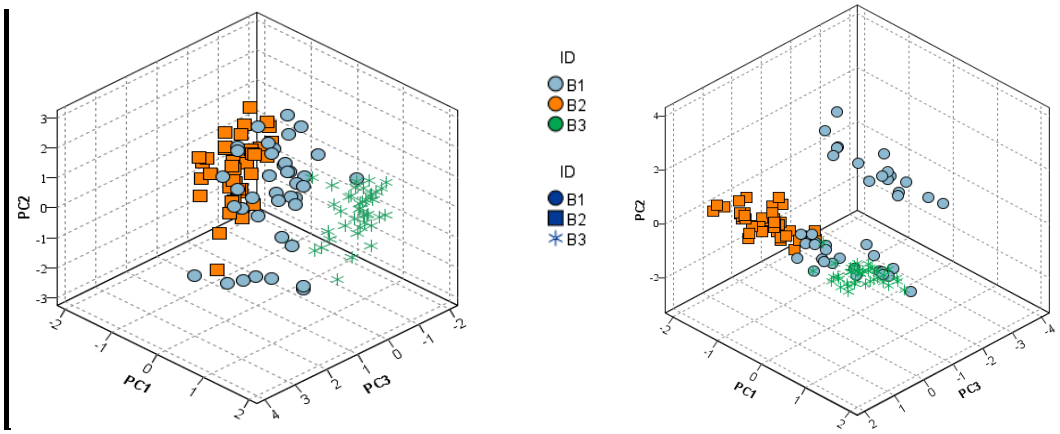
In some studies, the 1924 nm band in powder samples has been attributed to water molecular bonds [17]. In the present study, given the examined spectral range (936–1660 nm), this is viewed positively with respect to moisture effects in the samples. Garlic contains, in small amounts, sulfur-containing compounds, flavonoids, amino acids, polyphenols, water-soluble constituents, and lipids. Consequently, spectrum interpretation is challenging and complex due to potential overlap of these constituents. The key absorption bands observed in the collected spectra (Figure 1) are:

- 1120–1290 nm, corresponding to the second overtone of C–H bonds
- 1320–1520 nm, arising from the first overtones of N–H and O–H bonds [34]
- A peak at 1420 nm, reported as the first overtone of the O–H bond in water [35].

To compare how preprocessing methods affect the separability of sample spectra, the sample distributions in the space of the first three principal components are shown (Figure 2). The application of PCA and analysis of sample dispersion in principle-component coordinates has been documented in multiple studies [29, 30, 36].



No preprocessing - Raw spectra- (Right: Intractance, Left: Reflectance)



SNV preprocessing (Right: Intractance, Left: Reflectance)

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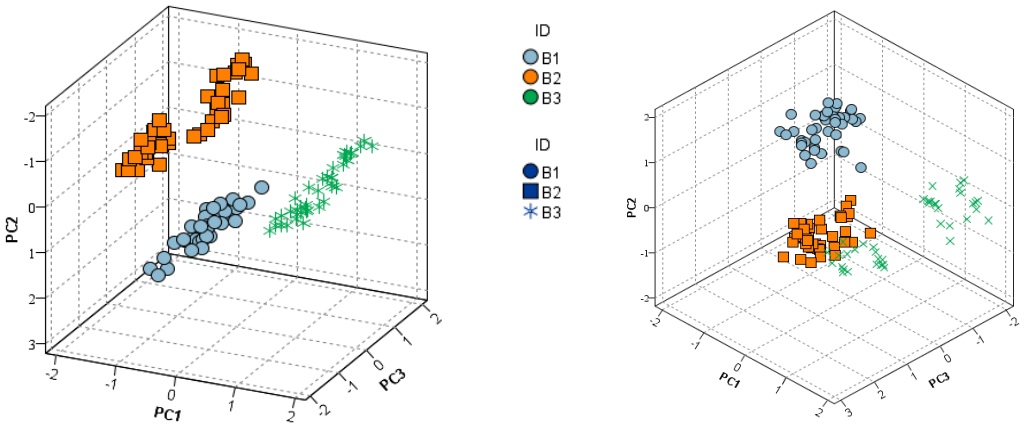

D1+SG preprocessing (Right: Intractance, Left: Reflectance)

Fig 2. The effect of the different preprocessing on the spectra scattering at PC1, PC2 and PC3 coordinate

3.1. Modeling

The results related to model calibration and validation are presented in Tables 2 and 3. The training and test datasets were the same across all classifiers. In the interactance spectral acquisition mode, the SVM and KNN classifiers achieved 100% classification accuracy across all evaluated preprocessing methods. Additionally, in the reflectance mode, the ANN classifier demonstrated 100% classification accuracy in all preprocessing methods except for the raw spectra.

Table 2. The overall accuracy (%) by classifiers in the training and testing data sets based on different preprocessing methods (Intractance)

KNN (%)		SVM (%)		ANN (%)		Spectral Preprocessing
Train	Test	Train	Test	Train	Test	
100	100	100	100	100	96.30	No Preprocessing
100	100	100	100	100	100	MSC
100	100	100	100	98.92	100	SNV
100	100	100	100	100	100	D1+SG
100	100	100	100	100	100	D2+SG

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Table 3. The overall accuracy (%) by classifiers in the training and testing data sets based on different pre-processing methods (Reflectance)

KNN (%)		SVM (%)		ANN (%)		Spectral Preprocessing
Train	Test	Train	Test	Train	Test	
98.92	96.30	98.92	92.59	98.92	96.30	No Preprocessing
95.70	96.30	95.70	96.30	98.92	100	MSC
95.70	96.30	95.70	96.30	98.92	100	SNV
100	100	100	100	100	100	D1+SG
100	100	100	100	100	100	D2+SG

4- Conclusion

The short-wave near-infrared (NIR) spectra in the range of 936–1660 nm, collected from garlic powder samples, showed a limited number of peaks. This is likely due to the overlapping absorption of overtones and the diverse chemical bonds present in the garlic powder. Spectra acquired in the interactance mode exhibited less noise compared to those in the reflectance mode. As a result, no significant difference in the classification accuracy was observed among the preprocessing methods SNV, MSC, D1+SG, and D2+SG. The achieved classification accuracy demonstrates the potential of near-infrared spectroscopy within this spectral range for identifying the freshness of garlic powder. Considering the results and the practical ease of using a bidirectional fiber optic probe, the interactance acquisition mode is preferable. Further studies are recommended to investigate other spectral ranges, consider different garlic cultivars, particle sizes, and compare interactance and reflectance modes in assessing the quality of other food products and medicinal plant-based materials.

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چکیده

پودر گیاه دارویی سیر از حیث تجاری اهمیت زیادی دارد و به عنوان افزودنی در صنایع غذایی و دارویی کاربرد فراوانی دارد. روش‌های طیف‌سنجی به‌عنوان یک روش غیرمخرب در حوزه سنجش کیفیت محصولات غذایی، تولیدات و فرآورده‌های گیاهان دارویی مورد استفاده قرار می‌گیرد. در پژوهش حاضر کارایی و پتانسیل دو وضعیت پرکاربرد بازتابشی و تقابلی در طیف‌سنجی فروسرخ نزدیک در محدوده ۹۳۶ تا ۱۶۶۰ نانومتر برای امکان‌سنجی تشخیص تازگی پودر سیر مقایسه شدند. در هریک از وضعیت‌های طیف‌برداری، تعداد ۱۲۰ طیف در ۲ تکرار از نمونه‌های پودر سیر اخذ شد. ۲۵ درصد از طیف‌ها قبل از مدل‌سازی به‌طور تصادفی برای اعتبارسنجی در نظر گرفته شدند و تدوین مدل‌ها با طیف‌های باقیمانده به انجام رسید. برای حذف نوفه‌های احتمالی تأثیر پیش‌پردازش‌های رایج روی عملکرد طبقه‌بندهای شبکه‌های مصنوعی عصبی (ANN[1])، ماشین بردار پشتیبان (SVM[2]) و الگوریتم k-نزدیک‌ترین همسایه (KNN[3]) بررسی شد. از روش آنالیز مؤلفه‌های اصلی (PCA[4]) برای کاهش متغیرهای طیفی استفاده شد و چهار مؤلفه اصلی اول به عنوان ورودی طبقه‌بندها در نظر گرفته شدند. در وضعیت طیف‌برداری تقابلی، طبقه‌بندهای SVM و KNN با دقت تفکیک ۱۰۰ درصد، طیف‌های اخذشده از پودرها در سه بازه زمانی ۳ روز، ۳ ماه و ۱۲ ماه را جداسازی نمودند. طبقه‌بند ANN در وضعیت بازتابی به‌جز طیف‌های خام (بدون پیش‌پردازش) در همه پیش‌پردازش‌های مورد بررسی، دقت ۱۰۰ درصد در تفکیک طیف‌های ذکرشده حاصل شد. طیف‌سنجی فرو سرخ نزدیک در باند ۹۳۶-۱۶۶۰ نانومتر به کمک شیمی‌سنجی برای تشخیص سریع تازگی پودر سیر پتانسیل لازم را دارد. وضعیت طیف‌برداری تقابلی با در نظرگرفتن سهولت کاربرد در صنعت و آزمایشگاه نسبت به وضعیت بازتابشی برتری دارد.

1 . Artificial Neural Network
2 . Support Vector Machine
3 . k-Nearest Neighbors
4. Principle Components Analysis

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