

Investigating the potential of Fourier transform mid-infrared spectroscopy combined with chemometrics for detecting camel's milk adulteration

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Abstract

This study explores the effectiveness of mid-infrared (MIR) spectroscopy in combination with chemometrics as an alternative to sensory analysis for detecting camel's milk adulteration with cow's milk. A paired comparison test involving various concentrations of adulterants was initially conducted to assess consumers' ability to detect such adulteration. The analysis successfully classified samples into adulterated and authentic camel's milk using principal component (PC) analysis and hierarchical cluster analysis. Moreover, the application of partial least squares regression and PC regression calibration models demonstrated high-performance capabilities in revealing the level of adulteration. These findings highlight the potential of MIR spectroscopy combined with chemometrics for the authentication of camel's milk.

Keywords: camel's milk; cow's milk; vibrational spectroscopy; chemometrics; adulteration

Introduction

Global camel's milk production reached three million tons per year in 2020, reflecting a 42.8% increase over the previous 20 years (2000–2020) (FAOSTAT, 2022). This increase is mainly attributed to the growing interest of consumers in this product due to its superior nutritional and therapeutic features compared to cow's milk (Ait El Alia *et al.*, 2023). This includes its capacity to treat autism and allergies and prevent diabetes, autoimmune illnesses, and cardiovascular problems (Hammam, 2019). Indeed, camel's milk is a suitable alternative to cow's milk due to its low concentration of β -lactoglobulin, making it

less allergenic. Additionally, it contains five times more vitamin C and ten times more iron. Camel's milk is also known to have a high concentration of bioactive peptides, lactoferrin, zinc, and mono and polyunsaturated fatty acids (Kamal *et al.*, 2017; Kumar *et al.*, 2016; Swelum *et al.*, 2021; Redwan *et al.*, 2022).

Considering its higher price compared to cow's milk, camel's milk is often subject to adulteration. In Morocco, for example, a liter of raw camel's milk can cost between three and ten times more than a liter of raw cow's milk, depending on the location. The most common type of fraud is substitution in which a food component is

partially or completely replaced by another less expensive alternative without the knowledge of the customers. Cow's milk, which is largely available in global markets and less expensive, is the main adulterant in camel's milk. In addition to economic losses, fraudulent practices can also have adverse effects on consumer health, particularly for those with allergies to cow's milk (Azad *et al.*, 2016; Windarsih *et al.*, 2021).

Sensory evaluation analysis is a technique widely used in the food industry to identify and characterize the sensory components of food, both qualitatively and quantitatively (Varela *et al.*, 2012). The International Organization for Standardization (ISO)-5495 (2005) defines a method for determining if there is a noticeable difference or similarity in the intensity of a sensory attribute between two food samples using the 2-AFC (Alternative Forced Choice) test or the directional difference test. The paired comparison test, a two-alternative forced choice test, can determine if there is a remarkable difference in one or multiple sensory attributes and determine the direction of the difference. However, it does not measure the extent of the change and thereby the amount of added adulterants (Zine-Eddine *et al.*, 2021).

In recent years, analytical techniques have been developed to confirm the authenticity of camel's milk products and protect consumers' health. For instance, the Ultra Performance Liquid Chromatography (UPLC) approach has been developed for detecting the addition of cow's milk in camel's milk powder using Bovine B-lactoglobulin as a marker (Li *et al.*, 2021). Polymerase chain reaction (PCR) and real-time PCR have also been recently utilized to investigate the presence of sheep and cattle milk components in camel's milk powder (Wu *et al.*, 2022) especially the adulteration of milk and dairy products, is one of the important issues of food safety. The large price difference between camel milk powder, ovine, and bovine milk powder may be an incentive for the incorporation of ovine and bovine derived foods in camel milk products. This study evaluated the use of ordinary PCR and real-time PCR for the detection of camel milk powder adulteration based on the presence of ovine and bovine milk components. DNA was extracted from camel, ovine, and bovine milk powder using a deep-processed product column DNA extraction kit. The quality of the extracted DNA was detected by amplifying the target sequence from the mitochondrial Cytb gene, and the extracted DNA was used for the identification of milk powder based on PCR analysis. In addition, PCR-based methods (both ordinary PCR and real-time PCR. To detect possible adulteration of camel's milk with cow's or goat's milk, a standardized real-time PCR system based on single-copy nuclear genes, real-time PCR with melting curve analysis, and Fourier transform near-infrared (FT-NIR) spectroscopy

with chemometric methods have also been developed (Mabood *et al.*, 2017; Souhassou *et al.*, 2018; Wajahat *et al.*, 2022; Wang *et al.*, 2020).

Near-infrared (NIR) and Fourier transform mid-infrared (FT-MIR) spectroscopies have revealed interesting results for screening and identifying additional substances in dairy products. Vibrational spectroscopy combined with chemometric methods is indeed becoming a promising, nondestructive, rapid, and reliable analytical approach for the exact and accurate authentication of dairy products (Kamal *et al.*, 2015; Windarsih *et al.*, 2021). Chemometrics tools have been widely used for data treatment and valuable information extraction. In general, chemometrics can be divided into two main categories: supervised and unsupervised data analysis. The objective of unsupervised methods is to reveal any underlying trends in the dataset without any prior knowledge. Examples of unsupervised data analysis include principal component (PC) analysis, hierarchical clustering (HC) analysis, and k-means, among others (Windarsih *et al.*, 2021). Focusing our attention on supervised approaches for classification purposes, some of the most popular methods are kNN (k-nearest neighbor), PLS-DA (partial least-squares discriminant analysis), and SIMCA (soft independent modeling of class analogy). However, when developing regression models to quantify a distinctive adulterant, widely used methods include PC regression, partial least square regression (PLSR), and support vector regression (SVR) (Grassi *et al.*, 2023; Windarsih *et al.*, 2021).

This study aims to evaluate the consumer's ability to detect adulteration of camel's milk by cow's milk using paired comparison tests. Afterwards, the use of mid-infrared (MIR) spectroscopy in combination with chemometric tools was investigated for creating a model able to detect camel's milk adulteration by cow's milk.

Materials and Methods

Samples preparation

Samples of raw camel's milk were collected directly from nomads in the Beni Mellal region, Morocco with a guaranteed authenticity. Cow's milk was bought from local farmers near Beni Mellal, and the purity of the raw cow's milk used as adulteration was also assured. The milk samples were filtered and kept at a temperature of 6°C until analysis.

Sensory analysis

Paired comparison tests

Consumer test was conducted using participants of different ages and genders as well as officials, academic

researchers, and students of the Higher School of Technology, Sultan Moulay Slimane University, Morocco. All participants in this study were regular cow's milk drinkers who had been introduced to camel's milk during training sessions.

In accordance with the protocols described in ISO-5495 (2005), 50 mL of each milk sample was placed in separate white plastic cups and identified with distinct numbers. To eliminate any lingering flavor, mineral water was offered between samples. Participants were then instructed to compare the five samples individually, which included 100% camel's milk (CAM), 100% cow's milk, a blend of 25% cow's milk and 75% camel's milk (CAM25), a blend of 50% cow's milk and 50% camel's milk (CAM50), and a blend of 75% cow's milk and 25% camel's milk (CAM75).

By using the one-sided paired test, the standard ISO-5495 (2005) can determine if there is a difference. The paired comparison test was used to see if the tastes of the four samples were different from each other compared to the pure sample. For each test, two cups marked with numbers (CAM vs. cow's milk, CAM vs. CAM25, CAM vs. CAM50, and CAM vs. CAM75) were given to the consumers. They were asked to taste both samples and indicate whether they differ. Each test sample was categorized differently across raters, and each respondent had a separate order for the samples.

Data analysis

ISO 5495 (2005) was used to assess significant differences in consumer responses using a one-sided paired nonhe-donic test (difference case).

Equation (1): The smallest number of replies (x) equals the nearest whole number higher than:

$$x = \frac{(n+1)}{2} + z\sqrt{0.25n} \quad (1)$$

Where z changes according to the significance level: 1.28 for $\alpha = 0.10$; 1.64 for $\alpha = 0.05$; 2.33 for $\alpha = 0.01$ and 3.09 for $\alpha = 0.001$.

IBM SPSS Statistics version 25 was used to perform the chi-square tests. The interval plot was created with Minitab's statistical package, version 18 (Minitab, Inc.).

Vibrational spectroscopy and chemometrics

Samples preparation for spectral acquisition

Adulteration analysis involved the provision of various sets of adulterated samples. These samples were

created by mixing camel's milk with varying levels of cow's milk as adulterant. The samples were thoroughly mixed prior to their analysis using MIR spectroscopy. The levels of camel's milk adulteration were recorded as weight-to-weight (w/w) percentages, ranging from 1% to 99% (w/w) of cow's milk. As described in the following equation (2):

$$\% \text{ Adulteration} = \frac{\text{Mass of cow's milk in camel's milk}}{\text{Total mass of sample}} \quad (2)$$

Acquisition of the Spectrum

Spectra of the samples were obtained using a PerkinElmer spectrometer equipped with an Attenuated Total Reflectance (ATR) accessory, a DTGS detector, a Globar source, and a KBr Germanium separator. The spectra were scanned with a resolution of 4 cm^{-1} to 98 scans in the absorbance range of $4000\text{--}450 \text{ cm}^{-1}$. The analyses were carried out at room temperature. The recorded spectra of pure and adulterated camel's milk samples were entered into Unscrambler software, Version 10.1, along with the mass percentages calculated.

Spectrum analysis

In this research, we utilized a range of statistical techniques to process and analyze spectral data obtained through MIR spectroscopy. To ensure a comprehensive exploration of the dataset, we initiated the analysis by performing PC analysis and HC analysis.

PC analysis is an unsupervised model that is commonly used in exploratory data analysis to identify patterns and clusters in the collected data. This method is particularly useful when dealing with large amounts of quantitative data, as it extracts crucial information from the data table by projecting it onto a set of new orthogonal variables known as PCs. These PCs, which represent the largest variations between characteristics, are calculated to be independent of one another and provide an overview of the data structure by revealing the relationships between objects and detecting anomalous features (Roggo *et al.*, 2007; El Orche *et al.*, 2020). The outcome of HC analysis, however, is a dendrogram that illustrates the grouping of samples in a hierarchical manner and measures the similarity between them based on their different attributes (Miller *et al.*, 2000).

To determine the amount of cow's milk present in adulterated camel's milk samples, we employed the PLSR, PCR, and SVR methods. The samples were separated into calibration and validation sets only for PLSR and PCR methods, with the former containing 80 samples and the latter containing 20 samples. PLSR can analyze data from samples spectra at various frequencies and link spectral absorption changes with analyte concentration while simultaneously accounting for additional spectra that may interfere with the analyte spectra (El Orche *et al.*,

2022). PCR, however, is a type of factor analysis that integrates spectral and concentration data into the model in a single step (Hirri *et al.*, 2023). SVR is a two-step machine learning algorithm that uses a kernel function to map training data to a higher dimensional feature space and then builds an optimal separating hyperplane in that space to achieve maximum margin (Shi *et al.*, 2015). We applied the regression models to a medium infrared spectral range between 4000 cm^{-1} and 950 cm^{-1} , with and without spectral preprocessing.

To quantify adulteration, a two-step calibration and validation approach was utilized (Kamal *et al.*, 2015) researchers, governments, consumers and so on due to the increase of falsification procedures inducing lost large of money as well as the confidence of consumers. The determination of the authenticity and the detection of adulteration of milk and dairy products have been determined by several analytical techniques (e.g., physico-chemical, sensory, chromatography, and so on. The model's performance was assessed through root mean square error of calibration (RMSEC), root mean square error of cross-validation (RMSECV), and R-square. Subsequently, the chosen model was employed to determine the concentration of samples from a distinct set of predictions. The effectiveness of the model's prediction was evaluated using root mean square error of prediction (RMSEP) (Mabood *et al.*, 2017). A lower RMSE and higher R-square values indicate a favorable prediction quality.

Results and Discussion

Paired comparison tests

The role of sociodemographic data analysis has been essential in comprehending eating habits (Dominici *et al.*, 2021). Table 1 contains the analysis of sociodemographic data obtained from 120 people who participated in the paired comparison test. The participants in the study completed a questionnaire, and the results indicated that most respondents were young individuals, with an almost equal gender distribution (females 51.67%; males 48.33%) and 60% of them were below 25 years of age. Furthermore, the participants evaluated 960 milk samples in the paired comparison test.

The graph in Figure 1 displays the consumers' responses interval with a 95% level of confidence (CI) for the mean (p value of 0.05) of those who were able to distinguish between cow's milk and camel's milk. As can be seen, the obtained data illustrates a notable contrast in the testers' responses.

Furthermore, the reported results in Figure 1 demonstrate that the increasing addition of cow's milk positively

Table 1. Paired comparison test's sociodemographic characteristics.

| Variables | Number of participants N (120) | Proportion (%) |
|---------------------------|-----------------------------------|----------------|
| Sex | | |
| Female | 62 | 51.67 |
| Male | 58 | 48.33 |
| Age range | | |
| Less than 25 years old | 72 | 60 |
| From 26 to 35 years old | 18 | 15 |
| From 36 to 50 years old | 18 | 15 |
| Fifty-one years and older | 12 | 10 |

affected the ability of consumers to detect the difference between pure camel's milk and pure cow's milk.

Table 2 displays the findings of the camel's milk paired comparison test. The number of assessors was 120, which is larger than the minimum number required by ISO-5495 (2005) (e.g., 18) for revealing the difference between the two milk types using a one-sided paired test.

The results indicate that around 26.67% of the evaluators were able to identify the presence of cow's milk in the camel's milk when the amount was less than 50%. However, this detection was not statistically significant. As the amount of added cow's milk exceeded 50%, the participants were able to significantly detect the adulteration, with 70% of them identifying the presence of cow's milk with a p -value of 0.001 when 50% of cow's milk was added. Moreover, when the added quantity reached 75%, a majority of 86.67% of the evaluators were able to detect the difference, with a p -value of 0.001.

It can be concluded that the inability to identify adulteration of raw camel's milk by low quantities of cow's milk may be attributed to the milk's unique organoleptic properties, which differ from those of cow's milk. Indeed, camel's milk has a normal odor, an opaque white color that can be attributed to the low carotene content, and homogenized fat facts which gives the milk a smooth appearance (Ahmed *et al.*, 2014), and a more salty taste (which can vary based on the diet of the animal) compared to that of cow's milk (Profeta *et al.*, 2022; Singh *et al.*, 2017). The intensity of these characteristics can therefore hide low amounts of added cow's milk.

To investigate the relationship between the responses of the paired comparison test and the parameters age and gender (Table 3), chi-square tests were employed. The findings indicate that the responses of the paired comparison test are not significantly associated with the

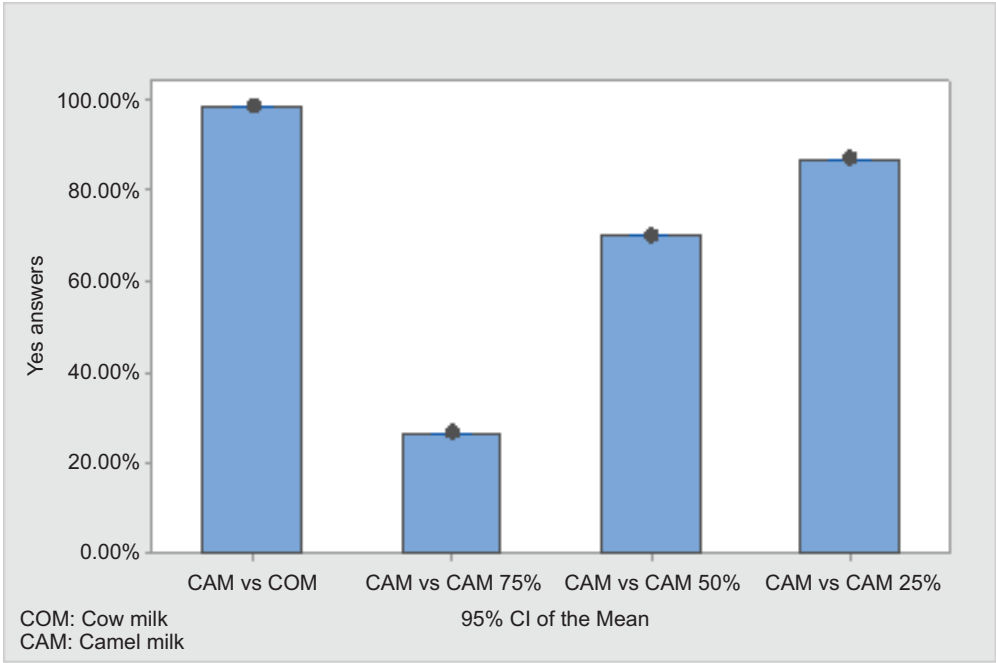


Figure 1. Plot demonstrating the range of consumer responses who could differentiate between cow's milk and camel's milk at a 95% confidence level for the mean.

Table 2. Results of the pair-wise comparison test for raw camel's milk.

| | Percentage of consumers who could detect the difference | | |
|---|---|-----|--------|
| Percentages of the blended cow's milk with camel's milk | 25% | 50% | 75% |
| Participants' replies, (N = 120) | 26.67 ^{ns} | 70* | 86.67* |

^{ns}Not significant; *Significant difference at 99.9% level.

Table 3. Chi-square tests between the responses of the paired comparison test and the parameters age and gender.

| Percentage of the added cow's milk | 0% | 25% | 50% | 75% |
|------------------------------------|---------------------|----------------------|----------------------|----------------------|
| Gender | 2.174 ⁱⁿ | 0.049 ⁱⁿ | 0.057 ⁱⁿ | 0.021 ⁱⁿ |
| Age | 3.051 ⁱⁿ | 59.063 ⁱⁿ | 34.683 ⁱⁿ | 24.087 ⁱⁿ |

ⁱⁿ: Independent variables.

variables of age and gender, revealing that age and gender do not influence the detection of camel's milk adulteration using sensory analysis. Based on these results, it can be concluded that age and gender are not confounding variables and can be safely excluded from the analysis of our paired comparison test.

Vibrational Spectroscopy and Chemometrics

Mid-infrared spectra of pure camel and adulterated milk

Figure 2 presents spectra of pure and adulterated camel's milk obtained using MIR spectroscopy in the spectral band 3000-950 cm^{-1} . The MIR spectra of the samples show different absorbance bands that can be linked to the main components of the milk due to the absorption of infrared light at specific wave numbers. Concerning the lipid content, three bands were observed at 2857, 1754, and 1175 cm^{-1} , which can be attributed to the presence of fats (B), fats (A), and fat (C), respectively (Mohamed *et al.*, 2021). The wide water band coincides with narrower bands that indicate amide I and amide II bands present in the proteins. These protein-specific bands appear in the 1700-1600 cm^{-1} and 1570-1500 cm^{-1} ranges, respectively. Furthermore, the 1100 cm^{-1} spectral band is commonly linked to the phosphate group (O = P-O) present in casein proteins (Etzion *et al.*, 2004).

The spectral band ranging from 1250-1050 cm^{-1} exhibits several broad absorption peaks that could be assigned to lactose vibrations. The bands at 1250 and 1157 cm^{-1} are associated with C-O-C ether stretching, while the band at 1076 cm^{-1} corresponds to C-O, C-C, and C-H stretching vibrations. Additionally, the band at 1053 cm^{-1} can be assigned to the C-O stretching vibration of alcohol functional groups (Mohamed *et al.*, 2021). The alterations in the protein, lactose, and phosphate bands of camel's milk spectra following the addition of non-camel's milk can be

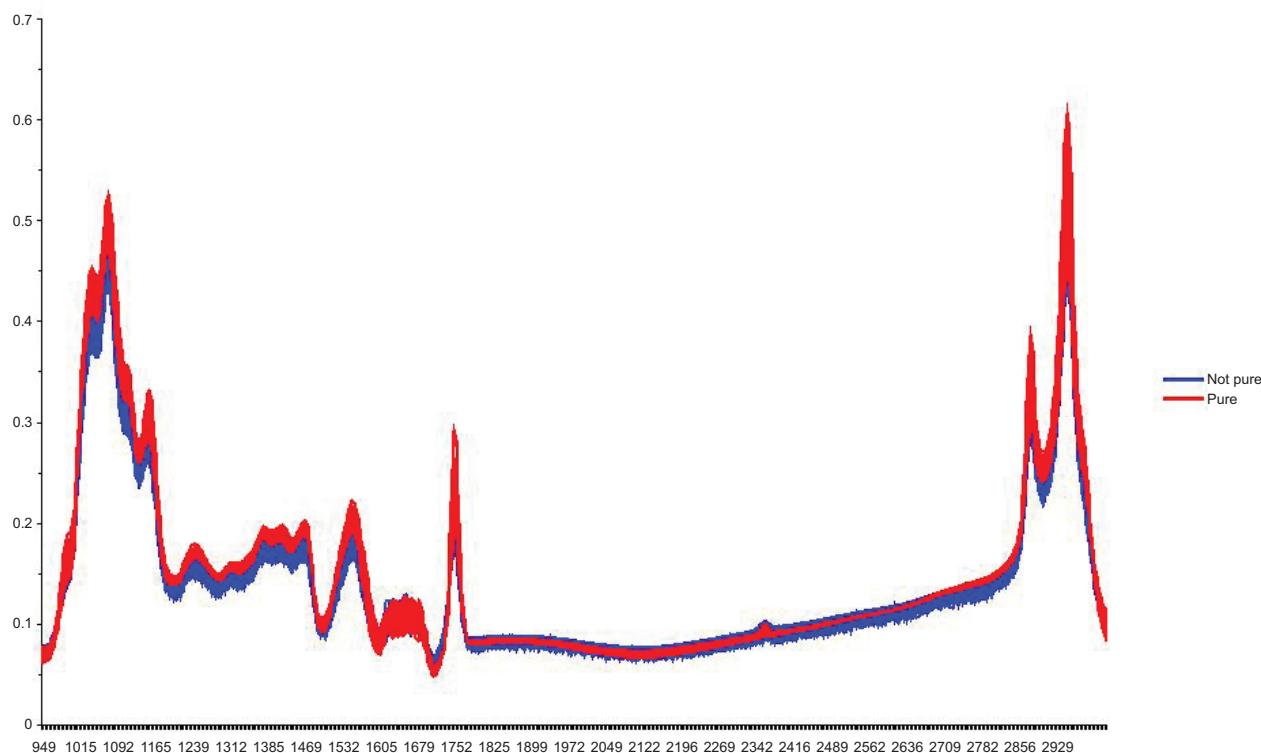


Figure 2. Mid-infrared absorption spectra of pure camel and adulterated milks in the spectral range 3000-950 cm^{-1} .

explained by the resulting change in the milk composition due to falsification.

Classification of the pure and adulterated camel's milk

In order to thoroughly investigate the spectral dataset, we used unsupervised chemometrics techniques, including PC analysis and HC analysis. The initial step involved applying PC analysis to the spectral data and obtained results are shown in Figure 3.

After applying PC analysis to all the samples, including pure camel's milk and those adulterated with cow's milk, we observed an overlap between the spectra. This overlap can be attributed to the similar composition of the pure milk and adulterant compared to samples with adulteration concentrations close to 50%. Then, we applied PC analysis to the 0–50% spectra (Figure 3B) and the 50–99% spectra of the adulterant and pure camel's milk (Figure 3C). This allowed us to divide the spectra into two categories: adulterated and pure camel's milk. By this approach, we were able to represent the data set in a two-dimensional space.

PC analysis indicated that the initial two components explain 95% of the overall variability in the data. Furthermore, the graph illustrates the existence of dispersion within the pure camel's milk group, which may

be related to multiple factors affecting the chemical composition, such as camel breed, age, stage of lactation, herd management techniques, and environmental conditions (Al Haj *et al.*, 2010).

HC analysis is a clustering technique that aims to create hierarchical groups of variables based on their similarity, with the closest variables being merged first to form higher-level clusters. The resulting hierarchical structure is typically represented as a dendrogram or tree (Panero *et al.*, 2018). Figure 4 shows the dendrogram produced by applying HC analysis to classify samples of camel's milk into two groups: adulterated and nonadulterated. The analysis was successful in distinguishing between the two groups using the mean-centered data and the single linkage algorithm to define the proximity between samples (Yim *et al.*, 2015). The similarity of the milk samples was assessed based on their distance from each other, and the results showed that the two types of milk were successfully differentiated from each other. Furthermore, the study employed a spectral-based version of HC analysis that assumes samples with similar spectral profiles are chemically linked and should be assigned to the same group (Chanana *et al.*, 2020).

By using HC analysis and PC analysis techniques on the MIR spectra of camel's milk, it was feasible to

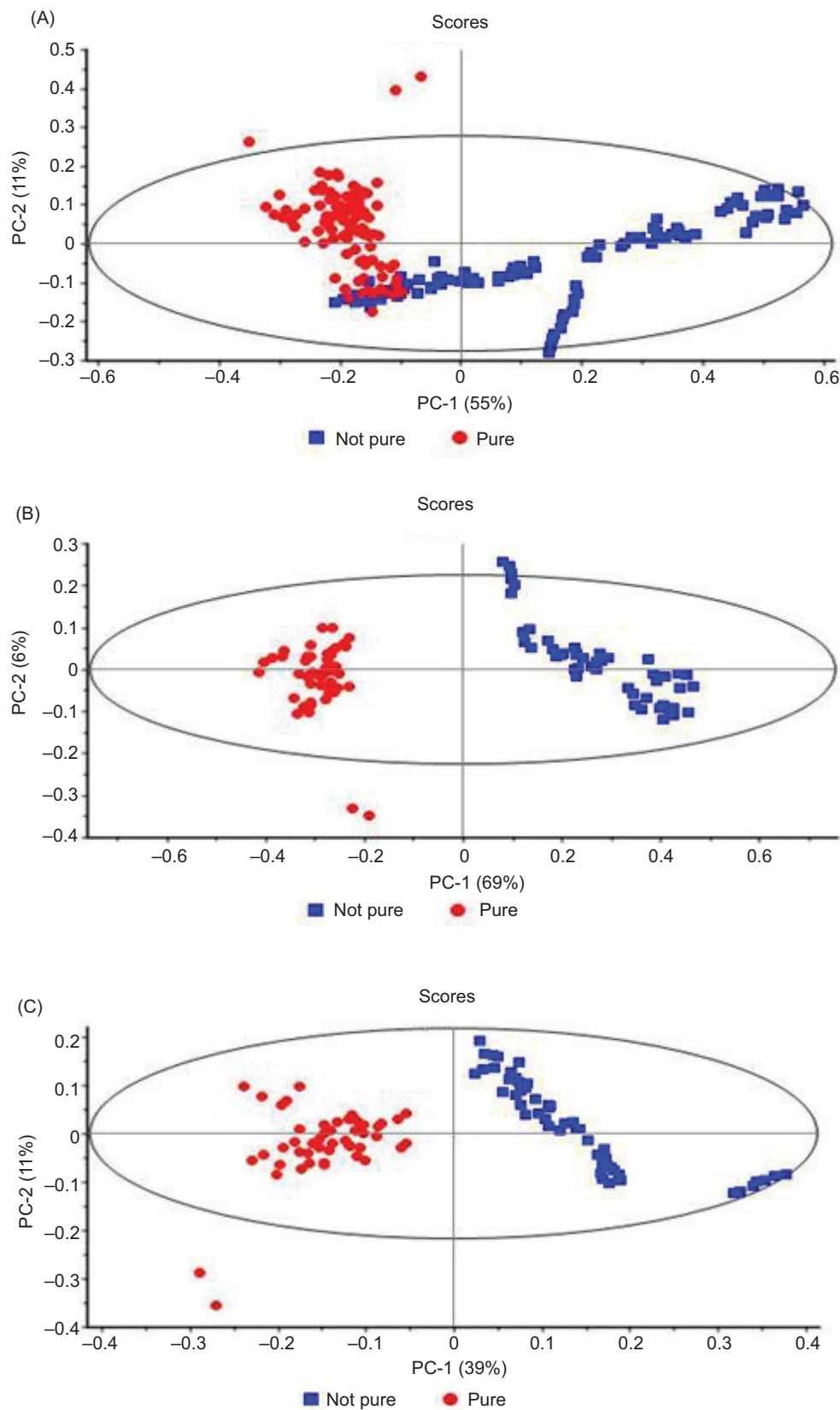


Figure 3. Scores graphs of PC analysis for all the studied samples of pure and adulterated camel's milk (A), pure camel's milk with 1–50% adulteration (B), pure camel's milk and 50–99% adulteration (C).

differentiate between pure and adulterated samples. This strong distinction lies in the spectral difference between the two studied types of milk, as revealed in Figure 2, which shows that there are noticeable differences in the spectral intensity of multiple bands.

These findings demonstrate that the used vibrational spectroscopy in the present contribution coupled with chemometric tools, such as PC analysis and HC analysis, could offer a great advantage in distinguishing between adulterated and pure camel's milk with high accuracy.

Quantification of camel's milk adulteration

To establish a linear correlation between the adulterant mass concentrations and the spectra obtained by MIR, a chemometric analysis was conducted using PLSR, PCR, and SVR techniques. The calibration and validation factors (RMSE and R-square) were presented in blue and red colors, respectively. A strong model is distinguished by a high R-square value and a low RMSE value. The processing of MIR spectra for camel's milk falsified with cow's milk by using PLSR, PCR, and SVR techniques *via* the Unscrambler software are presented in Figure 5. A total of 80 calibration samples and cross-validation samples were used. The calibration and cross-validation parameters, including R-square and RMSE, are listed in Table 4.

Based on the spectral differences obtained by the peaks, an R-square close to 0.999 was obtained in the regions of 3000-950 cm^{-1} using PLSR and PCR, and at 2500-950 cm^{-1} using SVR. Yet, the RMSE for calibration and validation was only close to 1 in the regions of 3000-950 cm^{-1} using PLSR and PCR (Table 4). The spectral range of 3000-950 cm^{-1} was chosen for processing the chemometric models of camel's milk adulteration to acquire a high value of R-square and low values of RMSEC and RMSECV of the PLSR and PCR constructed models.

According to the results presented in Figure 5, the MIR spectra of camel's milk show a strong correlation with its adulteration rate, as revealed by both the PLSR and PCR models. To develop highly accurate chemometric models that can predict the adulteration level of camel's milk, various mathematical models have been constructed using different spectral preprocessing techniques.

As seen in Table 5, the PLSR and PCR regression models were tested with and without preprocessing, using several techniques such as Savitzky-Golay smoothing, Moving Average Smoothing, Gaussian Filter Smoothing, and Median Filter Smoothing. The developed PLSR and PCR regression models induce a good performance, with a correlation coefficient fluctuating between 99.84% and 99.89% and an error ranging from 0.9456 to 1.1529 for the calibration. Cross-validation results also exhibited a high

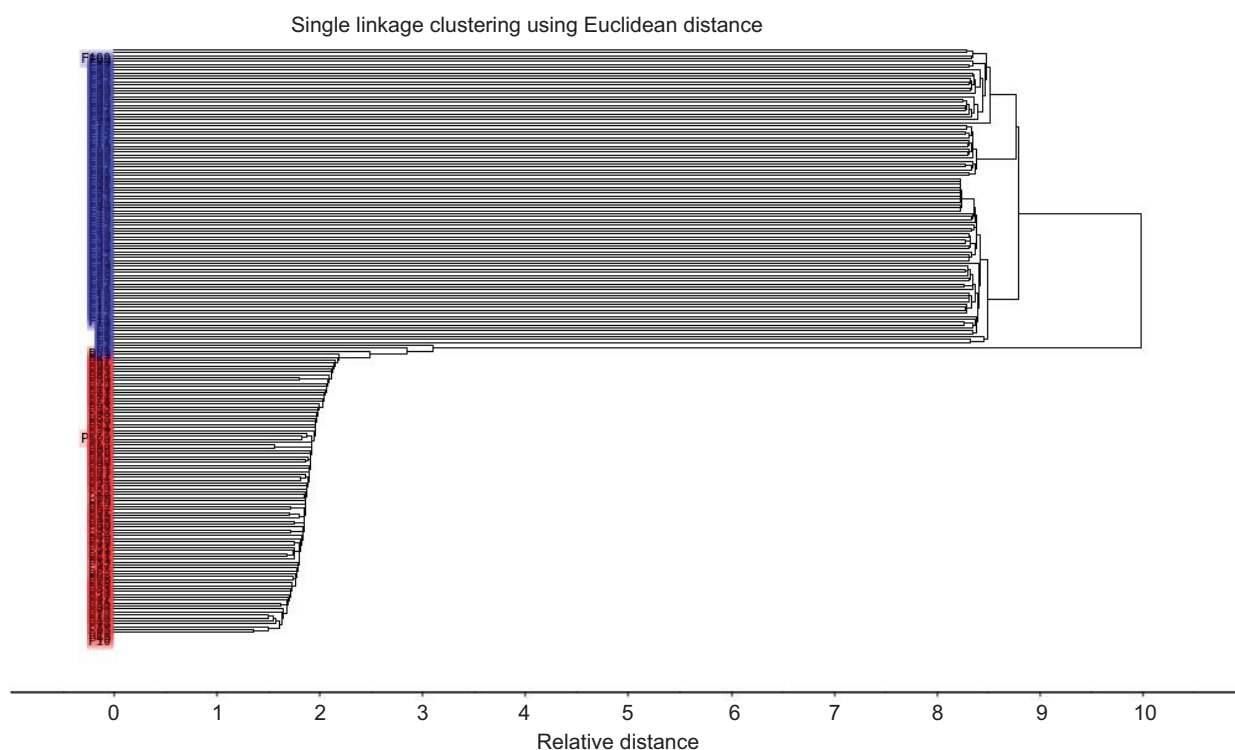


Figure 4. Dendrogram created using HC analysis with single linkage and the squared Euclidean distance to compare adulterated and nonadulterated camel's milk samples.

Table 4. Calibration and cross-validation parameters for camel's milk.

| Model | Spectral Range (cm ⁻¹) | Calibration | | Cross-validation | |
|-------|------------------------------------|-------------|----------|------------------|----------|
| | | RMSEC | R-Square | RMSECV | R-Square |
| PLSR | 950-2000 | 2.0930 | 0.9947 | 2.5839 | 0.9921 |
| PCR | | 2.7952 | 0.9906 | 3.1205 | 0.9886 |
| SVR | | 3.1389 | 0.9957 | 3.3513 | 0.9946 |
| PLSR | 950-2500 | 1.2191 | 0.9982 | 1.4765 | 0.9974 |
| PCR | | 1.5324 | 0.9971 | 1.7064 | 0.9965 |
| SVR | | 2.7171 | 0.9963 | 2.8559 | 0.9960 |
| PLSR | 950-3000 | 1.0128 | 0.9987 | 1.2890 | 0.9980 |
| PCR | | 1.1529 | 0.9984 | 1.3844 | 0.9977 |
| SVR | | 2.8417 | 0.9950 | 3.0228 | 0.9942 |
| PLSR | 950-3500 | 1.7790 | 0.9961 | 2.6510 | 0.9918 |
| PCR | | 4.5938 | 0.9746 | 5.3738 | 0.9663 |
| SVR | | 3.0262 | 0.9966 | 3.4870 | 0.9951 |
| PLSR | 950-4000 | 3.1427 | 0.9881 | 3.2884 | 0.9872 |
| PCR | | 3.2498 | 0.9873 | 3.3124 | 0.9871 |
| SVR | | 2.8396 | 0.9957 | 3.2560 | 0.9941 |

efficiency as correlation coefficients ranged from 99.77% to 99.87% and error ranged from 1.0405 to 1.3844.

Furthermore, the constructed PLSR models for calibration and cross-validation were slightly better than the PCR models in terms of regression parameters. It was also demonstrated that the use of PLSR or PCR regression with Moving Average Smoothing preprocessing technique leads to establishing more efficient models with correlation coefficients that approach 99.9% and a calibration error of less than 1. The cross-validation results also indicate a correlation coefficient near 99.9%, with minimum errors of 1.04 for PLSR and 1.12 for PCR.

The effectiveness of the PLSR method for processing infrared spectral results has been revealed in various studies for detecting food adulteration. For instance, El Mouftari *et al.* (2021) investigated the adulteration of oleaster oil by olive oil using ATR-FTIR and chemometrics, and the best results were obtained by using PLSR with R-square of 0.995. Likewise, for the quantification of honey adulteration using MIR and chemometrics, including PLSR and SVM, the PLSR have induced higher efficiency compared to the SVM regression (Elhamdaoui *et al.*, 2020).

Twenty samples were used for external validation to validate these models' ability to quantify camel's milk adulteration. The test samples were examined and the results were anticipated using the PLSR and PCR models. Table 6 summarizes the findings of all the constructed models.

As seen, the R-square values for PLSR and PCR were greater than 99.69%, with a mean square error of less than 1.5 for PLSR and 1.56 for PCR. The anticipated values are quite close to the actual values. Based on these findings, it was demonstrated that the adopted approach in this study is effective to detect and measure the presence of cow's milk in pure camel's milk. Therefore, this method can help overcome the underdetection of camel's milk adulteration by using sensory analysis, especially when low amounts of cow's milk are added.

Conclusions

This work aims at investigating the potential use of MIR spectroscopy combined with chemometrics for camel's milk adulteration. The use of the paired comparison test as a sensory analysis to investigate the ability of consumers to detect adulteration of camel's milk by cow's milk shows that not all consumers are able to detect the adulteration, even in the case of high amounts of cow's milk in camel's milk, and that nondetection rises with decreasing levels of the adulterant in camel's milk. The latter MIR spectroscopic method, combined with chemometric tools, was found to be an effective method for the detection and quantification of adulteration of camel's milk by cow's milk. It was also found that the PC analysis and HC analysis models can be used as classification tools for pure and impure camel's milk. Meanwhile, the PLSR and PCR calibration models could offer great advantages for the quantification of adulterated camel's milk. This approach is simple, does not require extensive

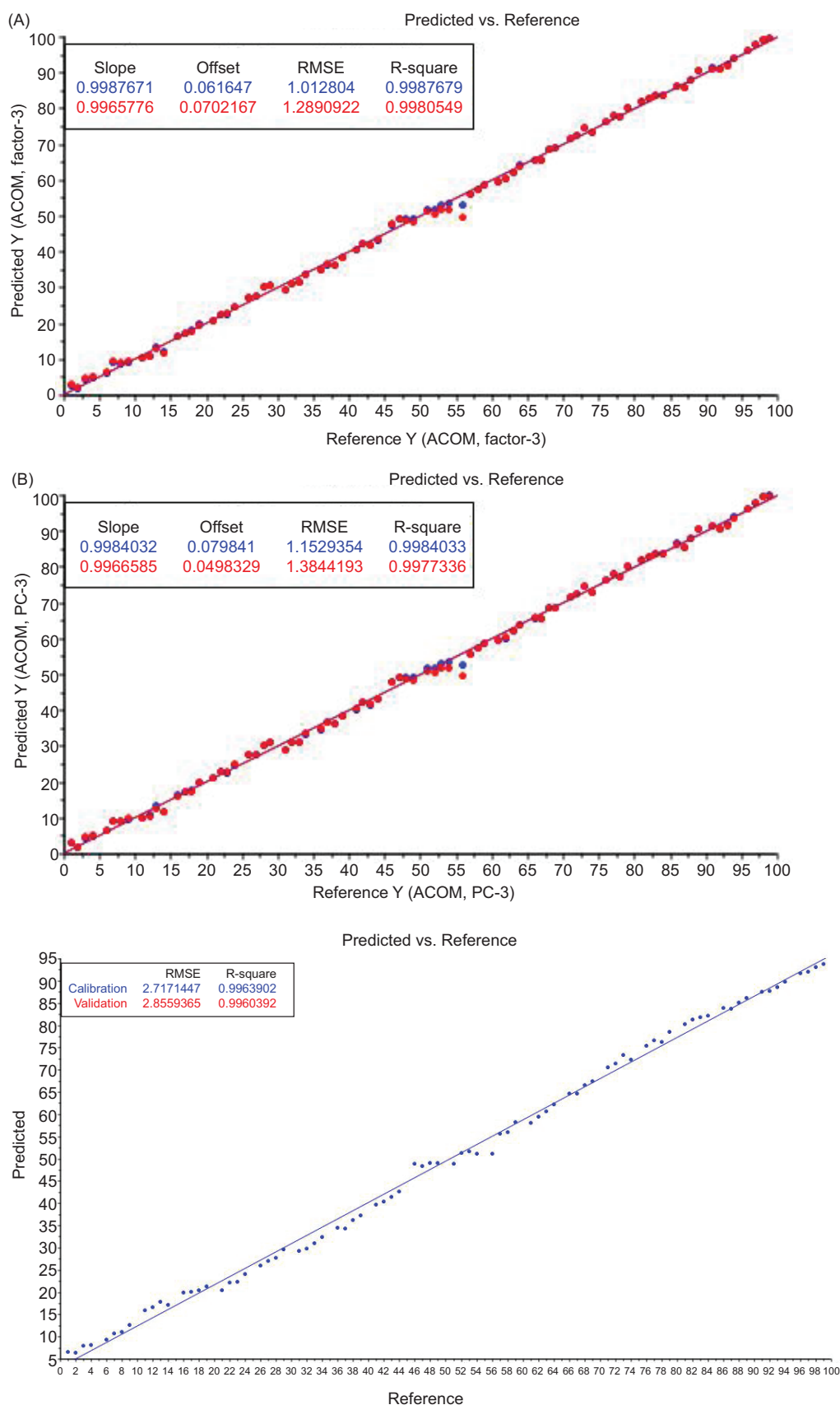


Figure 5. PLSR (A) and PCR (B) in 3000-950 cm^{-1} and SVR (C) in 2500-950 cm^{-1} of the calibration set for camel's milk adulterated with cow's milk.

Table 5. Performance parameters of PLSR and PCR.

| Model | Preprocessing | Calibration | | Cross-Validation | |
|-------|---------------------------|-------------|----------|------------------|----------|
| | | RMSEC | R-Square | RMSECV | R-Square |
| PLSR | Without Preprocessing | 1.0128 | 0.9987 | 1.2890 | 0.9980 |
| PCR | | 1.1529 | 0.9984 | 1.3844 | 0.9977 |
| PLSR | Savitzky-Golay Smoothing | 0.9684 | 0.9988 | 1.1191 | 0.9985 |
| PCR | | 1.0500 | 0.9986 | 1.1751 | 0.9983 |
| PLSR | Moving Average Smoothing | 0.9165 | 0.9989 | 1.0405 | 0.9987 |
| PCR | | 0.9850 | 0.9988 | 1.1199 | 0.9985 |
| PLSR | Gaussian Filter Smoothing | 1.0059 | 0.9987 | 1.2856 | 0.9980 |
| PCR | | 1.1237 | 0.9984 | 1.2779 | 0.9980 |
| PLSR | Median Filter Smoothing | 0.9456 | 0.9989 | 1.1648 | 0.9984 |
| PCR | | 1.0508 | 0.9986 | 1.2038 | 0.9983 |

Table 6. Performance of the PLSR and PCR models by external validation using MIR.

| Model | Preprocessing | RMSEP | R-Square |
|-------|---------------------------|--------|----------|
| PLSR | Without Preprocessing | 1.5770 | 0.9970 |
| PCR | | 1.6357 | 0.9967 |
| PLSR | Savitzky-Golay Smoothing | 1.5181 | 0.9972 |
| PCR | | 1.5686 | 0.9970 |
| PLSR | Moving Average Smoothing | 1.4937 | 0.9973 |
| PCR | | 1.5573 | 0.9970 |
| PLSR | Gaussian Filter Smoothing | 1.5502 | 0.9971 |
| PCR | | 1.6055 | 0.9968 |
| PLSR | Median Filter Smoothing | 1.5087 | 0.9972 |
| PCR | | 1.5636 | 0.9970 |

sample preparation, and has high sensitivity and repeatability. These characteristics make it a promising alternative to conventional sensory analysis.

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Conflict of Interest

The authors have no competing interests to declare that are relevant to the content of this article.

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