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Green and rapidmethod for classification of the adulteratedcorianderpowderusing vis-NIR spectra and Principal Component Analysis

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## **ABSTRACT:**

Coriander powder is commonly used as food additives, flavouring agent, medicine, preservation element due to its nutritional, antioxidant properties. Its nutritional and medicinal properties increase the demand and which makes it's prone to adulteration. Coriander powder is adulterated with different type of adulterants, which are low cost and harmful to human health. Present work focuses on classification and categorization of the coriander samples adulterated with common adulterants namely - saw dust, cow dung and salt using the visible near infrared spectra. Visible Near infrared spectra was obtained for the pure coriander powder and coriander powder adulterated with the given three adulterants in different ratios. Principal Component Analysis was used for the classification of the samples. Variable correlations were checked between the spectral wavelengths and the coriander concentration of the adulterate samples. It was observed that all the wavelengths above 1100 nm had strong correlation with the coriander adulteration. This results helps in understanding the importance of the wavelengths above 1100 nm for the determination of quantity and type of adulterant in the adulterated coriander powder. Principal Component Analysis was helpful in grouping the complete data set with three types of adulterant as well as one set of adulterant. Results show that the vis-near infrared spectra is capable of classification of the coriander samples adulterated with three adulterants and nine concentration.

**Keywords**: adulteration, Coriander, Near Infrared spectra, Principal Component Analysis, quality estimation

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## **INTRODUCTION:**

India is the world's largest spice producer and exporter with production of 10.88 million tonnes in 2021-22. Out of the 109 varieties listed by International Organization for standardization, India produces 75 of them, with coriander, chilli, ginger, turmeric and cumin making up about 76% of total production of spices.[1] Spices are used by humans to impart flavour to foods and beverages to enhance taste, smell and palatability. Further spices also possess cosmetic and medicinal applications and also act as natural food preservatives and food additives to prevent microbial growth and prevent food spoilage. Spices are also responsible for the antioxidant and antimicrobial properties.[2-4] Coriander is a herbal spice which grows with branching stems, its cultivated for its seeds and leaves, which are used in variety of dishes. Brown mature seeds are grounded to powder to be used as a seasoning mixture to spice a range of food items. Coriander powder due to its antibacterial, antifungal and antioxidant properties is used as natural food preservatives.[5] Coriander seeds and powder are used to treat intestinal parasites, gastrointestinal complaints. They are also used as anti-inflammatory, antiseptic and antihypertensive.[6]

Due to high in value in terms of medicinal and culinary properties, coriander powder becomes a prime target of adulterants. Grounded coriander is adulterated with less expensive or low quality spices, corn starch, saw dust, cow dung, horse dung, salt, starch, chalk powder and sometimes it's also adulterated with carcinogenic and toxic dyes to enhance their appearance and hide the added adulterant. Adulteration in food causes risk to their health and cheats the consumer. Adulteration is done for financial profit and is common in almost all backward and developing countries.

Spectroscopic techniques namely Infrared (IR) and near-infrared (NIR) spectroscopy are non-destructive, fast, non-hazardous, easy-to-use techniques with applications for food applications such to be used for measuring fat content, protein, ash, moisture and other properties. Infrared spectroscopy used with the primary method can be used as a rapid technique for detecting adulteration in herbs and spices. The technique being non-destructive does not affect the integrity of the sample. Absorbance in the range of infrared spectroscopy is result from the overtones and combinational bands vibrations. Range differs for different materials depending upon the bonds present in the element. Multivariate analysis such as Principal components analysis (PCA) is used to classify the samples and identify their adulteration concentration.[7]

Work has been reported to find the authenticity of coriander oil using Near Infrared Spectra. It was observed that Principal component analysis could differentiate the matrices of pure and adulterated oils. Further Linear discriminant analysis and k-nearest neighbour algorithms were used to identify the pure and adulterated samples. Regression model with coefficient of determination greater than 0.9 was developed for quality control.[8] Another research was carried to compare the performance of three different NR instruments for the quantitative analysis of coriander seeds. Ninety adulterated samples and two hundred authentic coriander seed samples were analysed by all devices. Results of regression analysis shows that all the instruments were capable for quantitative analysis of coriander seeds.[9] Another study showed the quality analysis of 15 samples of coriander seeds along with other herbs and spices. Different methods were used to find flour and starch as an adulterant in the coriander samples using the traditional chemical methods. Four loose samples contained adulterant and none of the FSSAI samples were found adulterated. Thus the authors concluded that it's good to use FSSAI samples rather than loose samples.[10] In another study GC-MS was used to detect adulteration and contaminants in coriander powder and other spices. The techniques were found useful for detection of authenticity of the samples. Detection of adulterants and contaminants in black pepper, cumin, fennel, coriander and turmeric using GC-MS technique for forensic investigation.[11, 12] Literature reported the assessment of the analytical methods for detection of lead in different spices namely coriander powder, turmeric powder, dried turmeric roots, blue fenugreek powder, red chilli powder, as well as khmelisuneli and

svaneti salt. 12 samples of coriander powder were used for testing of presence of lead using pXRF and ICP-MS. It was observed that lead concentration was within 5% for the spice samples.[13] Work was reported where Aurdiuno IDE was used for spice authentication. Methods were designed and study was conducted to detect adulteration in turmeric powder, chilli powder and coriander powder. Authors concluded that use of such technology will help in avoiding the consumption of poor quality food.[14] Further a review was reported by an author from Ethiopia where different methods to identify the adulteration in herbs, spices and drugs are reviewed. Author have emphasized on the physical, chemical, biochemical and other techniques. Further the research explains it's difficult to identify the adulterant if the food and the adulterant have same physiochemical configuration.[15] Researchers from Bangladesh reported the health hazards of the adulterated spices. Authors also discussed the effects of consuming adulterated coriander powder. Authors also discussed that it's difficult to identify the adulteration by visual and sensory inputs, but still good investigations can be carried out.[16] Authors have covered the spectroscopic methods with chemometric techniques that are used for the authenticity testing of herbs and spices. The methods are using for identification of their origin and classification of the type of adulterant. Spectral methods (UV, IR, NMR) along with PCA, CA for authenticity of herbs and spices were presented. Due to their simple, non-destructive and rapid technique they are considered as best tool for characterization of the samples [17]. Literature also presents review that focuses on use of Fourier transform infrared, near infrared, Raman, hyper spectral imaging, nuclear magnetic resonance and electron spin resonance for the spice and herb authentication. The methods were used for the determination of the origin of the samples, adulterant identification and their concentration. These methods based on the principal of vibrational spectroscopy along chemometric methods had proved best method for determination of presence of the adulterants in spices and herbs. It was concluded by the authors that the spectroscopic methods can identify the adulteration in spices with great efficiency [18]. Research has been carried out in literature to show that the visible and infrared spectroscopy with the pattern recognition methods – principal component analysis (PCA), different discriminant analysis (LDA, QDA) and support vector machine methods are capable of classify different herbs, including mint, linden, nettle, sage and chamomile. It was observed that the error for the classification was below 10% for maximum methods used. The research shows that spectroscopy and pattern recognition methods are a non-destructive tool for classification of the herb and spice samples [19]. Work was reported in the literature for the authentication of coriander powder from different methods and origins. A survey in Bangladesh was done to know the consumer satisfaction towards quality of the spices and herbs. It was observed from the survey that above 90% of the consumers believed that these spices are not of good quality and are found to be adulterated with brick dust and sand. The consumers were not satisfied with the quality of the spices and requested necessary action for the same [20]. Similar survey was conducted in Dhaka city of Bangladesh to investigate the authenticity of various food and beverage products. It was observed that more than 35 of food products were adulterated [21]. Fruit and the herb of coriander are popular and having biological properties and many health benefits. Research was carried out to access the composition of nineteen commercial cilantro and coriander essential oil by GC-MS methods. Main parameters of the oil are linalool, pinene, teripene. Chiral - mass spectroscopy was capable of identification of adulteration in coriander oils by measuring the composition of essential oils [22]. Literature shows different methods for detection of food adulteration, with electronic nose as one of the upcoming and promising techniques, authors reported a review paper emphasizing the use of e-nose to perceive the aroma and distinguish among various products. E-nose technique is also used for quality monitoring with the use of gas based sensor in e-nose [23]. Detection of adulterants and contaminants in black pepper, cumin, fennel, coriander and turmeric using GC-MS technique for forensic investigation [24]. Research was conducted on different spices, dairy products, herbs and several food

products to detect adulteration using physical and chemical methods in the region of Phagwara, Punjab, India. Methods to detect the adulteration were manual, physical and chemical which were destructive in nature. Main aim of the study was to bring awareness among consumers for the food products they consume [25]. Research was carried out in Tamil Nadu, India to identify the adulterants in coriander powder along with other spices using chemical analysis and visual inspection. Test was performed on various branded and non branded samples procured from the market. Main aim of the study was to bring awareness among the consumers about the adulterated spices and herbs [26]. A detailed review was listed by the authors which covered the importance of the spices and herbs in human life, their adulteration identification and survey for the authentication of the spices. Author's covers the fundamental analytical methods, spectroscopic methods have been covered in the review [27].

Literature shows that though a lot of work has been carried out to check the authenticity of the spices, coriander seeds and coriander oil using chemical and destructive methods, very few research have been reported for the classification of the adulteration of coriander powder using saw dust, salt and cow dung as an adulterant using the visible and near infrared spectra. The present work focuses on authentication of coriander powder sample. The research results in the classification of the pure and the adulterated samples of the coriander powder using correlation matrix and principal component analysis (PCA) using the visible near infrared spectra in the range of 400-2500nm. The work shows that the samples with different concentrations of coriander powder in the adulterated samples could be classified easily with vis NIR spectra.

## **MATERIALS AND METHODS:**

## **Sample Preparation**

Two Coriander seeds were purchased from local market, cleaned and grounded using mini grinder. For obtaining the same size of the powder samples, grounded sample was sieved through the sieve of mesh size of 0.2 mm. Particle size have a significant effect on the infrared spectra, thus all the powder samples were kept of the same size.[28-30] Four set of coriander samples were prepared. First set was of pure coriander powder samples containing only the coriander powder. Rests of the three sets were prepared by adulterating the pure coriander powder samples with powdered saw dust, salt and cow dung adulterants. Cow dung powder was prepared by collecting and drying the cow manure in sunlight for 5 days, it was then grounded and sieved with sieve of mesh size 0.2 mm. Saw dust powder was prepared by gathering the saw dust from carpenters, drying, grinding. Sample was also sieved from the same sieve. Salt was purchased from the market and sieved using the same sieve. Six branded samples were also collected from the market for the prediction purpose. Net weight of all the prepared pure and adulterated samples was 100g. Pure samples were adulterated by adding 5, 10, 15, 20, 25, 30, 50 and 100g of the powdered adulterant of the same particle size as of the pure coriander powder. Classification of the adulterated samples using the PCA method was applied on the four set of samples. The branded samples were also sieved for the required size. In all 62 samples were prepared including the pure coriander powder, adulterated and the branded samples.

## **Spectra Acquisition**

Spectra were obtained in the vis-NIR region using the NIR DS2500 Analyzer (Metrohm) available at CSIR – Central Scientific Instruments Organisation (CSIO) research center at Chandigarh, India. DS2500 Analyzer measures the specific interaction between light and the sample in a nondestructive manner. Spectra was obtained from 400-2500nm with a gap of 0.5

nm. Silicon and InGaAs detectors were used in the spectrometr to collect the data. All the measurements were taken in triplicate and average of the same was calculated using the inbuilt software and absorbance of the samples for the complete wavelength range was obtained.

## **Principal Component Analysis**

The NIR spectra obtained was complex and have unwanted wavelengths. To obtain a smooth spectra, preprocessing is required. The spectra were preprocessed using the baseline effect, linear baseline offset to smoothen the spectra and remove the unwanted noise and to provide a baseline to the complete spectra. Further 1st derivate preprocessing was applied to the spectra using the savitzkygolay algorithm to have a clear picture of the wavelengths important for the identification of the adulterants effect on the coriander sample. Preprocessing and principal component analysis was employed on the data set using the licensed software CAMO Unscramble X 10.5 version available at Lovely Professional University, Punjab, India. Correlation matrix was obtained for the preprocessed spectra to check the correlations of different wavelengths with the composition of coriander in the adulterated samples. Further principal component analysis was employed on the data set to obtain score plot, loading plot to know the relation between different samples and variables respectively.

## **RESULTS AND DISCUSSION:**

Samples for the analysis were prepared by adding three adulterants in the coriander powder sample in the proportion as given in Table 1. Three sets of adulterated samples were prepared according to same concentration – for salt, saw dust and cow dung.

Table 1 Composition of the adulterated samples for all the three types of adulterant – saw		
dust, salt and saw dust powder.		

S. No.	Coriander Powder Sample (g)	Adulterated Powder sample (g)
1.	100	0
2.	95	5
3.	90	10
4.	85	15
5.	80	20
6.	75	25
7.	70	30
8.	50	50
9.	0	100

Visible near Infrared Spectra of all the 62 samples was obtained using the NIRDS 2500 Spectrometer. Raw vis-NIR and the spectra pre-processed with baseline and 1st derivative with SavitzkyGolay algorithm are shown in fig 1 (a-c). Fig 1(a) presents the raw vis-NIR spectra from 400 to 2500 nm. Fig 1(b) represents the spectra after the linear baseline correction and linear baseline offset to the raw spectra. To obtain this spectra, minimum reading of the absorbance is subtracted from absorbance values of all the wavelengths. This helps in making the graph clear and distinct. Fig 1(c) shows the 1<sup>st</sup> derivative of the obtained spectra. The 1<sup>st</sup> derivative spectra highlights the wavelength bands with highest absorbance. The spectra of all the samples showed that absorbance bands for all the adulterated samples were observed at similar bands for all the adulterant, though the absorbance units varied with respect to the concentration of the adulterant in the sample. Absorbance bands were observed around 400nm and 650-700 nm in visible region and after 1150 nm in the near infrared region. Band around 1100 nm represents the presence of R-NH<sub>2</sub> and Ar-CH bonds in third overtone region. Least absorbance were observed for the wavelengths from 993 to 1400 nm.

Least absorbance was due to absence of the CH, CH<sub>2</sub>, CH<sub>3</sub>, CONHR bonds. Absorbance at different wavelengths bands were observed higher than 1400nm. It means there is absorbance observed due to first overtone region of CH, CH<sub>2</sub>, CH<sub>3</sub>, CONH<sub>2</sub>. Absorbance band was also observed around 1930 nm, showing the presence of H<sub>2</sub>O and RCO<sub>2</sub>H bands. The results shows that the wavelengths above 1100 nm are important for the determination of the type and concentration of adulterant in coriander powder except some wavelength regions as clearly visible in the 1st derivative spectra.

To confirm the above results correlation matrix were obtained by applying the descriptive statistics option in CAMO Unscrambler software for the data set and the result is shown in fig 2. The graph shows the correlation of absorbance at different wavelengths with the concentration of coriander in the adulterated samples. The correlation matrix is observed on the basis of the vis-NIR spectra of all the prepared samples. The results shows that wavelength from 700 to 1150 nm have no or minimal correlation with the concentration of coriander in the adulterated coriander samples with value of coefficient of correlation 0.2 or less. It can be observed that wavelengths from 1150-1250 nm had correlation of 0.3 and for wavelengths above 1450 nm had correlation above 0.4 with the coriander concentration. The results of NIR spectra and variable correlation confirm that all the wavelengths above 1100 nm are important for the determination of the type and concentration of the adulterant in the samples. Highest correlation between the coriander concentration in the prepared samples and the wavelength's absorbance is observed from 1700-1850 nm and around 2100 nm. These wavelength bands are due to absorbance by the presence of first overtone region and combination band region of CH, CH<sub>2</sub>, CONH<sub>2</sub>, RNH<sub>2</sub> and ROH bonds.

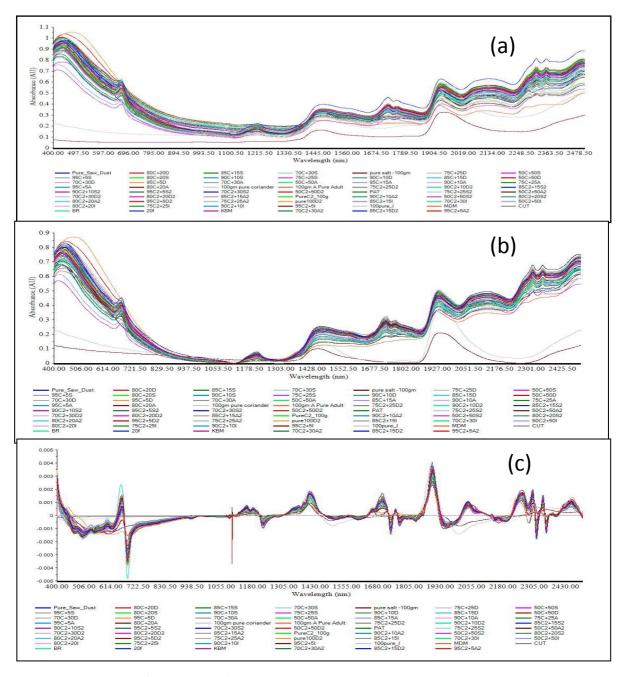


Figure 1 vis-Near Infrared Spectra from 400-2500 nm. (a) raw vis-NIR spectra (b) vis-NIR spectra pre-processed with baseline effect (c) vis-NIR spectra pre-processed with 1st derivative with savitzkygolay algorithm

Further principal component analysis was employed on the samples data set. The multidimensional data was projected and principal components were calculated by the CAMO Unscrambler X 10.5 version to decrease the dimensionality of the data set. Principal component analysis results were displayed as score and loadings plot to show the relations between samples and variables respectively. It was observed that loading plot result was similar to the results shown in the correlation matrix.

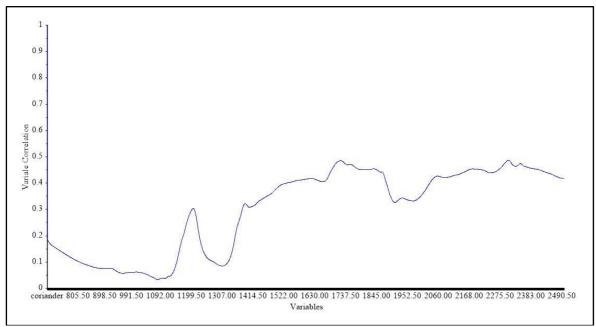


Figure 2 Correlation matrix showing the relation between the variables (wavelengths) with the adulterated coriander samples

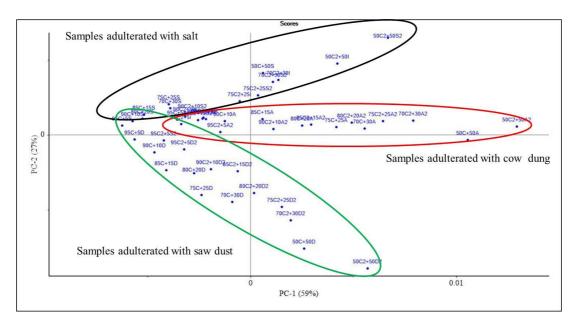


Figure 3 Principal Component Analysis of the samples adulterated with all the three adulterants.

PCA was applied first on the complete data set of 62 samples containing the entire adulterated sample. This set contained the pure coriander powder samples, samples adulterated with cow dung, saw dust and salt. Then the PCA was used on the set of data sets

with same adulterant. Thus in all four PCA analysis were performed. Score Plot of the data set is shown in fig 3 to fig 6. Figure 3 shows the PCA Score plot for all the 62 samples taken all together. It can be observed that first two PCs covered 86% of the total data set. Coverage was 86% of the data as the data has large variability due to adulteration of coriander with 3 different adulterants and seven concentrations. PCA showed that the samples with different adulterants were classified in three different groups as shown by black, red and green colour ellipse. Black ellipse shows the samples adulterated with salt as an adulterant and as the amount of adulterant increases PC 1 values of the samples increases i.e. the samples moves from left to right. Further the red ellipse shows the samples adulterated with cow dung, there is very less change in the value of PC 2 for these samples whereas PC1 changes from negative to positive with increases in concentration. Samples adulterated with saw dust are shown by the green ellipse. Variation in the PC1 and PC2 values for these samples shows large variation. With increase in adulterant concentration samples moves from top left to bottom right.

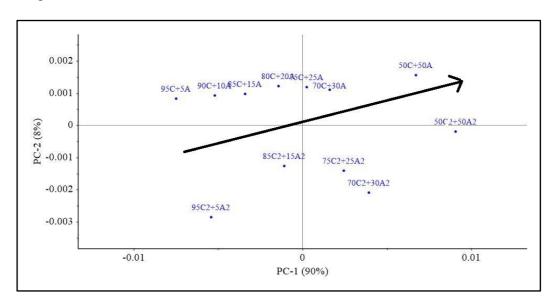


Figure 4 Principal Component Analysis of the samples adulterated with cow dung.

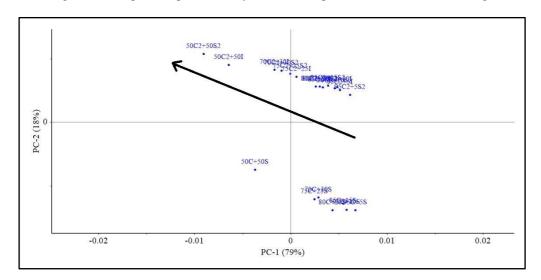


Figure 5 Principal Component Analysis of the samples adulterated with salt.

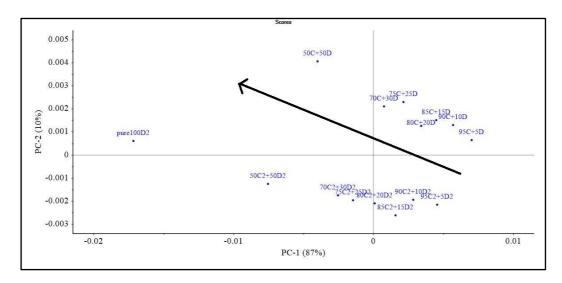


Figure 6 Principal Component Analysis of the samples adulterated with saw dust.

PCA score plot for all the samples was successful in classifying the samples on the basis of the type of adulteration. These results showed the NIR spectra is capable of differentiating the samples based upon the type of adulterant in the sample. Further it was also observed that for the samples with least adulterant present, samples were lying near to each other, but with the increase in the concentration of the adulterant in the coriander powder samples, it was easy to distinguish between the samples on the basis of the type of adulterant in the prepared sample. This result will be helpful in authentication of the coriander powder samples.

To check the variation among one type of adulterant PCA analysis were performed on set of samples with same adulterant. PCA for the samples adulterated with cow dung, salt and saw dust is shown in figure 4, 5 and 6 respectively. Each PCA from fig 4-6, around 97% data is covered by PC1 and PC2. The change can be observed as one dimension of variability is reduced by considering only one type of adulterant for one PCA score plot. It can be observed in the PCAs for one type of adulterant that with change in concentration of samples PC values shifts. This shows that small change in concentration of the adulterant effects the spectral data such that the same can be reflected in their respective PCA score plot. Further as two different coriander samples were used to prepare the adulterated samples thus two set of samples can be seen in the PCA score plot though they both are showing the similar variations with increase in concentration of the samples. The PCA graphs from 3 to 6 helps in determination of the quantity of the adulterant in the prepared adulterated samples. The use of PCA is in accordance with the results available in the literature. A research was carried out where PCA along with hierarchical cluster was used to classify the spice samples on the basis of their antioxidant activity.[31] Further a research was carried out to classify the spice samples on the basis of their proximate analysis using PCA.[32] Another study was done to classify and authenticate paprika samples using principal component analysis.[33]

The results showed that the spectral data is sufficient enough to classify the samples on the basis of the type and concentration of the adulterant for the adulterated coriander samples.

## **CONCLUSION:**

Research work carried out on the adulterated coriander samples, adulterated with three different types of adulterant for authentication of coriander samples was fruitful. Classification of the samples was done using their vis-NIR spectra in the range of 400-2500nm. Principal Component Analysis was employed on the complete data set and the data could be magnificently classified in three distinct groups with reference to the type of adulterant, thus it can be concluded that the vis-NIR spectra is capable of classification of the adulterated coriander samples with adulterants namely – saw dust, salt and cow dung. Further

PCA on one set of adulterated samples also successfully categorized the samples according to their concentration of adulterant in the samples. The study is very helpful in identifying the type and intensity of the branded and non-branded coriander samples in the market.

#### **CONFLICT OF INTEREST:**

The authors have no conflicts of interest regarding this investigation.

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#### FIGURE CAPTIONS

Figure 7 vis-Near Infrared Spectra from 400-2500 nm. (a) raw vis-NIR spectra (b) vis-NIR spectra pre-processed with baseline effect (c) vis-NIR spectra pre-processed with 1st derivative with savitzkygolay algorithm

Figure 8 Correlation matrix showing the relation between the variables (wavelengths) with the adulterated coriander samples

Figure 9 Principal Component Analysis of the samples adulterated with all the three adulterants.

Figure 10 Principal Component Analysis of the samples adulterated with cow dung.

Figure 11 Principal Component Analysis of the samples adulterated with salt.

Figure 12 Principal Component Analysis of the samples adulterated with saw dust.

## **TABLE CAPTIONS**

Table 2 Composition of the adulterated samples for all the three types of adulterant – saw dust, salt and saw dust