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Adulteration detection of brown sugar in ground coffee using FTIR spectroscopy

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Abstract

For many years, inexpensive adulterants have tainted coffee. Fourier transform mid-infrared (FT-MIR) is employed for qualitative detection of Brown Sugar (BS) adulteration in Pure Coffee (PC) due to the effectiveness of spectroscopic technologies as quick and effective routine approaches in the domain of food adulteration. Nutritionally, there is a considerable difference between BS and PC. Even though there are some available microscopic methods to detect adulteration of different adulterants, ATR-FTIR was recognized as a beneficial tool for detecting adulteration of BS in PC. PC, BS, and different percentages of spiked coffee (10%, 20%, 30%, 40%, 50%) were subjected to proximate in parallel with FT-IR spectroscopy. The spectroscopy results showed that BS was detected from PC by the nearly disappearing specific bands at 2920 cm^{-1} , 1658 cm^{-1} , 1746 cm^{-1} , and 1658 cm^{-1} , which are present in Pure Coffee. The intensity of peaks at these specific bands kept decreasing on increasing the adulteration. As FTIR is a fast and non-destructive technique, it can be employed to find BS adulteration in PC.

Keywords: FTIR, coffee, brown sugar, adulteration

1. Introduction

The coffee beverage is popular everywhere because it represents hospitality, has stimulating characteristics, and has a delightful flavor, making it a global product (Brondi *et al.*, 2017) [3]. Due to the large consumption of coffee, it becomes a focal point for adulteration by combining lower-quality products with identical physical properties. For instance, it can include adding other ingredients like coffee husks and stems, chicory, maize, brown sugar, barley, soybean, and triticale to the coffee mix to produce cheaper products, as well as the deteriorating quality of the beans (considering dull beans) (Toci *et al.*, 2016) [15]. The two most popular coffee species are *Coffea arabica* and *coffee robusta*, with arabica having a greater economic value due to its better aroma. In addition to lowering the product's quality (taste, fragrance, and nutritional value), adulterations increase merchants' profits and put consumers at risk for contracting foodborne illnesses.

Although consumers accept this practice in some nations, such as England, it is illegal to sell coffee mixed with items like chicory or figs unless the producer discloses this mixing. As coffee adulteration affects each country's economic conditions, data on the subject are almost nonexistent on a global scale (Flores-valdez *et al.*, 2020) [6]. Brazil is the world's largest producer of coffee, and an analysis of 2400 brands by ABIC found that 583 of them, or 25% of the national brands, were contaminated with husks, corn, rye, acai seeds, or brown sugar (Čurlej, 2021) [4].

Conventional techniques like HPLC (Domingues *et al.*, 2014) [5] or NMR (Bergana *et al.*, 2019) [2] are typically used to detect adulteration or authentication. However, these techniques are not practical for large samples or routine analysis because they require expensive equipment. Near-infrared (NIR) and mid-infrared (MIR) spectroscopy methods gained popularity for examining food quality and quantity. When compared to NIR, MIR spectroscopy at $4000\text{-}400\text{ cm}^{-1}$ delivers chemical information that is crisper and more intense because it depicts fundamental vibrations rather than the overtones and combination bands that are recorded in the NIR area (Lohumi *et al.*, 2015) [11].

FTIR is a spectroscopic technique extensively used to characterize food components and detect probable food adulterants based on their unique spectral fingerprints (Valand *et al.*, 2020) [16]. It ranges in 2 different regions i.e., MIR range ($450\text{ to }4000\text{ cm}^{-1}$, FT-MIR) or NIR range ($4000\text{ cm}^{-1}\text{ to }10000\text{ cm}^{-1}$, FTNIR). Currently, there is no systematically established relationship between FTIR spectral features on coffee and brown sugar, despite the appeal of FTIR as a quick, non-destructive methodology (Arslan *et al.*, 2020; Sanadgol Nezami *et al.*, 2021) [1, 13].

2. Materials and Methods

2.1 Sample Acquisition and Preparation

Adulterant BS and ground arabica coffee were purchased for the study at the local market in Thanjavur, Tamil Nadu, India. The merchant assured that the samples were genuine. Coffee was mixed with various amounts of brown sugar, ranging from 0% to 50%, and the final concentrations were scaled using an analytical balance (Ohaus Scout Pro SPS202F). As indicated in Table 1, 7 samples were prepared, each with a

different percentage of brown sugar added to the coffee: 0%, 10%, 20%, 30%, 40%, 50%, and 100%. Although the BS and PS were acquired dry, the mixtures were dried at 70 °C in a tray dryer before collecting spectral data to remove extra water since moisture content significantly influences infrared spectroscopy. Then, using a Remi CM-101 Plus Vortex, each admixture was stirred for 5 minutes in a sealed container until homogenized.

Table 1: Various combinations of PC and BS used as samples

Adulteration	Pure Coffee		Brown Sugar	
	%	Weight (g)	%	Weight (g)
0%	100	50	0	0
10%	90	45	10	5
20%	80	40	20	10
30%	70	35	30	15
40%	60	30	40	20
50%	50	25	50	25
100%	0	0	100	50

2.2 Proximate analysis

The moisture, proteins, carbohydrates, fats, fiber, and ash content in PC and BS were determined by A.O.A.C (2016) (I.S. Akande *et al.*, 2011)^[9].

2.3 Fourier transforms infrared (FTIR) spectrometric analysis

The IR spectra of PC, BS, and spiked samples were attained at room temperature using a Thermo Scientific Nicolet iS50 spectrometer (ThermoFisher, USA). All spectra were recorded in the Attenuated Total Reflectance (ATR) mode. This technique was applied to conduct 32 scans for each sample in the spectral range of 4000-400 cm⁻¹. Samples to be analyzed are placed directly on the diamond crystal of ATR-FTIR. Moreover, the spectra were obtained using the background

spectrum of air. There is minimal sample preparation since it is ATR-FTIR. Samples were kept in a hot air oven at 70 °C until the initiation of FTIR analysis to prevent moisture absorption from the atmosphere (He *et al.*, 2021).

FTIR spectra of Pure coffee, Brown sugar, and spiked samples with different concentrations from 0% to 50% were recorded. Thermo Fisher's OMNIC Spectra software's spectral data were exported as .SPA files and entered straight into the Unscrambler X 10.4 program (Camo Analytics). Using the spectra obtained, the overlapping FTIR spectra are built in which the x-axis is wavenumber (cm⁻¹) and y-axis as % transmission values and analyzed for the detection of adulterant.

3. Results and Discussion

Table 2: Proximate composition of different spiked percentages of BS in PC

	Moisture	Protein	Fat	Crude Fibre	Carbohydrate	Ash
PC	3.04±0.15 ^f	17.39±0.17 ^a	8.02±0.27 ^a	18.75±0.19 ^a	46.50±0.09 ^g	6.26±0.08 ^a
BS	22.86±0.55 ^a	0.3±0.03 ^g	0.27±0.04 ^f	2.24±0.10 ^g	70.22±0.44 ^a	3.15±0.15 ^f
10%	5.46±0.23 ^e	15.74±0.29 ^b	6.99±0.22 ^b	17.74±0.18 ^b	48.2±0.19 ^f	5.79±0.11 ^b
20%	6.95±0.05 ^d	13.46±0.26 ^c	6.9±0.09 ^b	16.22±0.11 ^c	50.47±0.16 ^e	5.69±0.19 ^{bc}
30%	7.24±0.12 ^d	11.32±0.06 ^d	6.09±0.21 ^c	14.41±0.13 ^d	55.34±0.37 ^d	5.34±0.11 ^{cd}
40%	9.70±0.14 ^c	9.65±0.11 ^e	5.40±0.08 ^d	11.78±0.18 ^e	58.26±0.23 ^c	5.10±0.12 ^{de}
50%	10.65±0.10 ^b	7.75±0.13 ^f	4.33±0.07 ^e	7.55±0.15 ^f	65.08±0.22 ^b	4.72±0.17 ^e

Note: Significant differences exist between means that do not share a letter. ($p < 0.05$). Each value is the average of three replicates, i.e., $n=3$.

The proximate analysis of PC, BS, and different ratios of spiked samples were evaluated as given in table 2. The acquired results showed a significant difference in nutrient content among all the samples. There was an increasing order in moisture and carbohydrate content. In contrast, downward trend was observed in protein, fat, and ash content on

increasing the spiking percentage of BS, which is an adulterant in PC, i.e. BS > 50% > 40% > 30% > 20% > 10% > PC. Total Nutrient content was more in PC when compared to BS and it follows the decreasing order as follows; BS < 50% < 40% < 30% < 20% < 10% < PC as shown in figure 1.

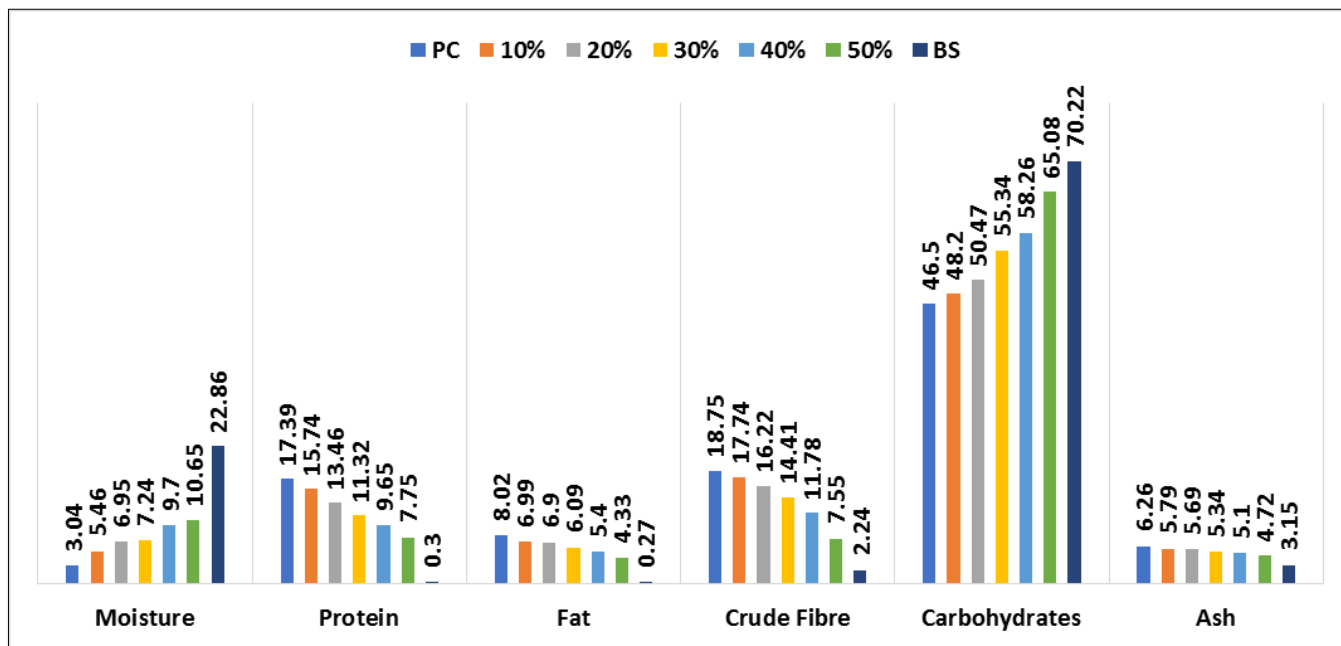


Fig 1: Proximate composition of PC+BS samples.

From FTIR analysis, the difference between the pure and spiked samples has been adopted based on molecularity. Table 2 lists the FT-IR absorption band assignments according to nutritional characteristics.

The major difference between BS and PC lies in their protein, fat, and carbohydrate content. Figure 2. provides a glance at the absorbance peaks and corresponding wavenumber when the samples are subjected to FTIR analysis, with figure 2c showing a graphical representation of the distribution of nutrient components. Figure 2d shows that the moisture content of samples significantly increases as the spiking percentage increases as the intensity of peaks increases between 3500-3000⁻¹, which corresponds to the O-H stretching in the matrix (Flores-Valdez *et al.*, 2020) [6]. The peaks in the band of 3500-3000 cm⁻¹, at 3385 and 3332cm⁻¹, are attributed to the -OH stretching in polysaccharides predominantly visible in the BS spectra (Ibrahim *et al.*, 2019; Suciayati *et al.*, 2021) [14]. The absorbance values of these peaks increase as the adulteration concentration increases, which indicates the increase in polysaccharide content in the samples. The peak at 3008 cm⁻¹, which is visible in the PC

spectra, corresponds to the fat or lipid content in the sample, which decreases as the adulteration percentage increases. The C-H bond found in the methyl groups in polysaccharides corresponds to the peaks at 2920, 2940, and 2855 cm⁻¹ can be observed as strong, sharp peaks in PC whereas cannot be observed in the BS spectra and due to which the peaks intensity decreases as the adulteration increases, as the lignin concentration in the samples decreases. The stretching of the ester group OC=O also causes a peak at 1746 cm⁻¹. The peak at 1658 cm⁻¹ is visible in the PC spectra. It almost disappears on increasing the concentration of BS in sample, which can be ascribed to the C=O group present in amides, representing the protein content in the sample (Flores-Valdez *et al.*, 2020) [6], (Nguyen *et al.*, 2022). Since the protein content is decreasing in the order PC > 50% > 40% > 30% > 20% > 10% > BS, the intensity of the peak was increased in the same order. Finally, The spectroscopy results showed that BS was detected from PC by the nearly disappearing specific bands at 2920 cm⁻¹, 1658 cm⁻¹, 1746 cm⁻¹ and 1658 cm⁻¹, which are present in Pure Coffee. On increasing the adulteration, the intensity of peaks at these specific bands kept decreasing.

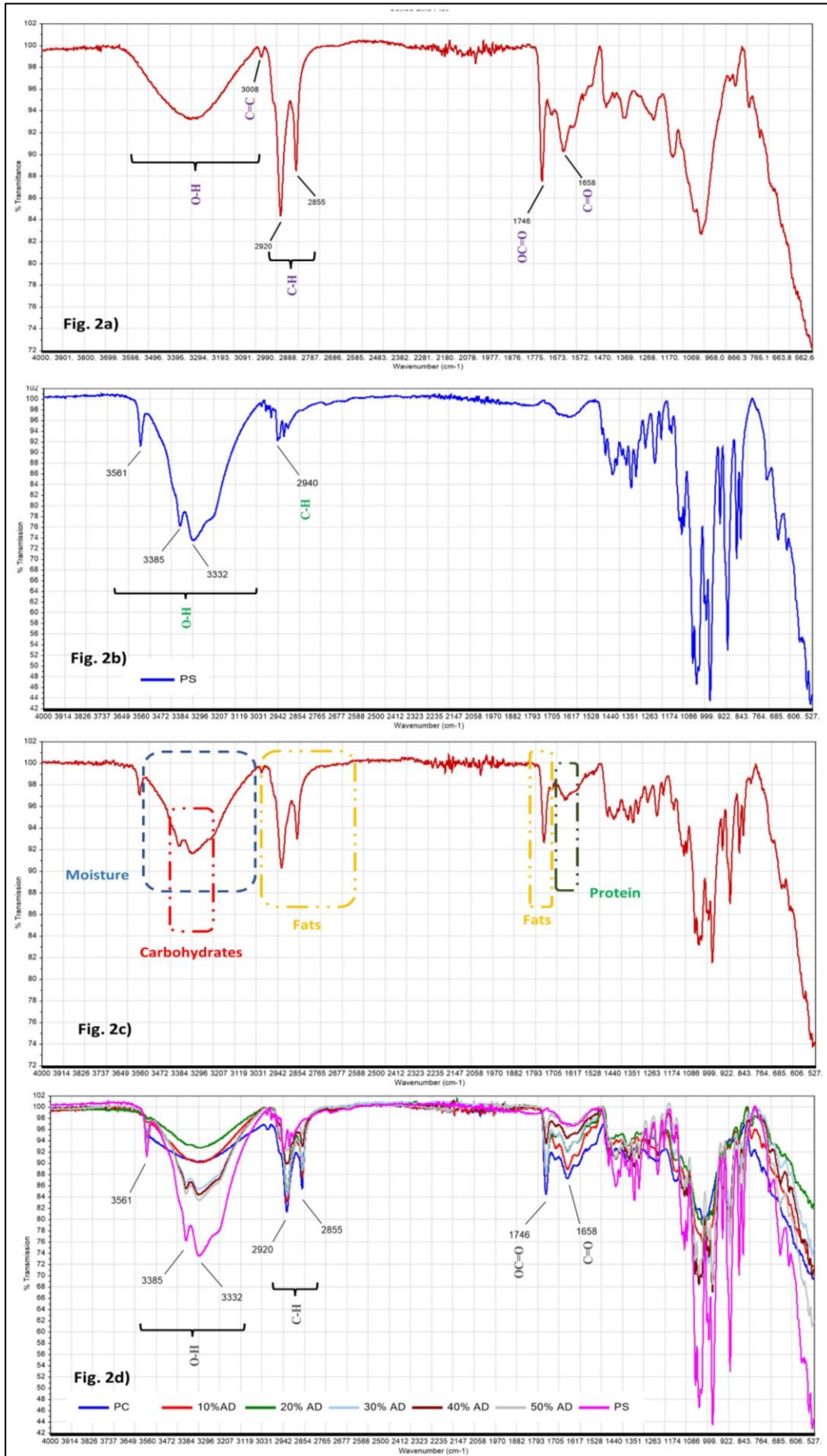


Fig 2a: Coffee FT-MIR spectra. **b)** BS FT-MIR spectra. **c)** Graphical representation of the distribution of nutrient components. **d)** Spiked Samples overlay FT-MIR spectra. Comparing the results found in FTIR and Proximate analysis, it can be observed that the results found in FTIR are substantiated by the results found in Proximate analysis. Thus, this technique can be useful in detecting BS in PC. As a result, the proposed technique has a lot of potential for detecting different percentages of BS in PC. The processing time for new samples will be less than 2 minutes.

Table 2: The Assignments of FT-IR absorption bands based on nutritional property

Nutritional Factor	Wavenumber(cm ⁻¹)	Functional Group and Vibrational mode
Moisture Content	3000-3500	-H & -OH
Protein	1600-1700	-C=O (-Amide I)
Carbohydrate	3385, 3332, 3210-3380, 1120-1280	-OH in polysaccharides, C-O
Fat	3008, 2500-3000 1700-1800	C=C in lipids, C=C, C=O stretching
Ash	835-2520	-

4. Conclusion

The present investigation unequivocally demonstrates substantial differences in the nutritional qualities of pure coffee, brown sugar, and spiked samples. Based on the findings, it is stated that FTIR offers a superior method for visualizing the chemical components of PC, BS, and spiked samples. It is a highly rapid, trustworthy, and affordable analytical approach that effectively detects BS (adulterant) in PC by locating distinctive spectral figure prints. It is a non-destructive technology with little sample preparation, exact measurement, and no external calibration requirements. It also speeds up data collecting with a scan per second. The amount of adulterant being added may be determined by using a developed approach that can apply chemometrics to those discovered spectral signatures.

5. Declaration of Conflict of Interest

The authors state that they have no known conflicting financial interests or personal relationships that may have influenced the work presented in this study.

6. Acknowledgment

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