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Detecting adulteration in mustard oil using low-frequency dielectric spectroscopy

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Abstract: This paper presents a dielectric spectroscopy approach for analysing the quality of food products. This study aimed to detect the adulteration in mustard oil using dielectric spectroscopy in the 1 to 10 MHz frequency range at a temperature of 30 to 50 °C. The dielectric data were used to predict the adulteration in oils at the given frequency range. The finding indicates that using data analysis techniques can further improve the capacity of dielectric sensing to detect adulterated edible oil. Using MATLAB R2021a, linear relationships between the frequency and adulteration percentage variables were obtained to predict the dielectric constant and dielectric loss factor values. A paired sample *t*-test was used to analyse the effects of the frequency and adulteration on the dielectric parameters, with a significance level of 0.05 being set for the differences. Correlation coefficients (R^2) > 0.96 were established using regression equations relating the dielectric constant, dielectric loss, and adulteration.

Keywords: dielectric constant; loss factor; regression; statistical analysis; temperature

Adulteration is inserting or mixing materials that would not be in food products for financial gain or other motives (Banti 2020). Edible oil has been found as the maximum ingredient involved in food mixing, a previous study showed that adulteration in fats and oils is economically motivated and, in some cases, intended to enhance the food flavour (Sairin et al. 2017). Adulteration in food materials is a worldwide severe issue not only because it deceives the consumers, but also it harms their health and causes severe consequences to the well-being of people. In general, the adulteration of edible oil causes two significant concerns to the consumer. First, it concerns consumers who practice a vegetarian diet and followers of religions that prohibit the consumption of pigs, pork, or any other products.

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Mustard oil is adulterated with argemone oil, olive oil and rice bran oil are mixed with sunflower oil, soybean oil, palm oil, while ghee (clarified butter) is mixed with animal fat and cold pressed oil is admixed with refined oil (Huq et al. 2022). Adulteration is rampant in many foods, but perhaps none as much as edible oils due to chronic shortages and the volatility in prices. The argemone oil present in mustard oil, as an adulterant, can cause oxidative stress and the death of red blood cells via methaemoglobin formation by altering pyridine nucleotide(s) and the glutathione redox potential in human beings, causing glaucoma, nausea, and loss of eyesight (Das and Khanna 1997).

Argemone (*Argemone mexicana*) and mustard (*Brassica juneca*) oilseeds look similar in shape and size. Due to the close similarity, argemone oilseeds are frequently mixed as an adulterant with mustard seeds at the time of the oil extraction. Such oils are remarkably health-vulnerable; these oils are toxic and should not be consumed. They contain toxic alkaloids, such as sanguinarine and dihydrosanguinarine; the consumption of adulterated oil can lead to health disorders like dropsy, neural degeneration and paralysis. Prolonged consumption of argemone oil causes epidemic dropsy disease, which causes several health issues, such as vomiting, diarrhoea, and loss of appetite (Ekop et al. 2007). In 1998, approximately 65 people died, and more than 3 000 people required hospitalisation in New Delhi, India, due to the intake of mustard oil adulterated with argemone oil (Das et al. 2005). It may also cause serious health-related issues, particularly for those who have allergies to certain types of substances or consumers who suffer from different diseases.

Until recently, dielectric spectroscopy techniques (Liangxiao et al. 2017), widely used to analyse food products, have attracted the research community's attention in studying the adulteration of fats and oil. This method is non-destructive and non-invasive and does not require the pre-treatment of the sample or reagents. Electrical parameters, such as the dielectric constant and dielectric loss, depend on the composition of the complex mixture and the food itself. In the past, studies on the dielectric properties of oils have been focused on the corrosion, and the estimation of adulterated oils, and applications of these properties to oil handling, storing, and food making have been limited. Therefore, the basic knowledge of a dielectric study is appropriate for the quality of edible oils. The earlier reported study was conducted on the interaction mechanism of oil subjected to microwave radiation at a broad range of frequencies. In general, the dielectric constant refers to the ability of a material to store electromagnetic energy, and the dielectric loss factor signifies its capability to dissipate electromagnetic energy, i.e. this technology has been extensively applied in the analysis of agricultural materials (Chen et al. 2016; Woo et al. 2019). In dielectric spectroscopy, information is provided about the dielectric response of materials to the electric field at selected frequencies (Cimbala et al. 2022). However, more literature is needed on using dielectric spectroscopy to detect adulteration in vegetable oils.

The purpose of this study was to develop a system based on the dielectric spectroscopy technique for detecting adulteration in vegetable oils and to use statistical methods with dielectric spectroscopy to improve the accuracy and capability of this method to obtain more reliable results for detecting the adulteration in vegetable oils. The statistical and regression analyses for the experimental data were performed for the first time for these oils in MATLAB (version R2021a) to create a multivariate linear regression model, which is a method to obtain more reliable results in finding adulteration in vegetable oils; a similar statistical analysis was also followed by other researchers (Rubalya et al. 2018). Zhou et al. (2023) developed a simulation model to explain the approximately 10-fold stronger electric field in oil than in water, resulting in a similar amount of microwave power being absorbed by the oil and water samples. Elmosalami et al. (2022), in their studies, reported on a dielectric study and Fourier-transform infrared spectroscopy (FTIR) analysis of six oils, olive oil A, olive oil B, sesame oil, *Nigella sativa*, sunflower oil, and corn oil over the frequency range of 0.01 Hz–100 kHz. The natures of all the oils with respect to the dielectric constant, dielectric loss, and FTIR analysis were almost similar, as they contain similar fatty acids. They also used COMSOL Multiphysics (version 5.6) to simulate various results, and found that the model was in good agreement with the experimental data. Luna et al. (2020) used a dielectric analysis method coupled with neural networks (NNs) and the Internet of Things (IoT) to assess the quality of olives. Zhang et al. (2019) developed a new way to investigate the adulteration of flaxseed oil using a linear discriminant analysis. They developed a partial least squares model, which showed a good prediction capability for the measurement of adulteration

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in flaxseed oil. SPSS (version 20.0), Unscrambler (version 9.7), and Origin Pro (version 7.5) have all been used for data processing and statistical analysis. Paired sample t-tests were performed to study the effects of the frequency and concentration on the dielectric parameters. Sudhakar et al. (2023) developed a sensor for measuring the dielectric properties of oils. This sensor was used to relate the dielectric properties of oil samples to a statistically significant and robust multiple linear regression model.

MATERIAL AND METHODS

Sample preparation. To ensure the purity of the samples, the vegetable oil and argemone oil (AO) were collected from different production areas of Uttar Pradesh, India and used to prepare pure oils and their mixture with argemone oil. This research used AO as the adulteration substance. AO is usually adulterated in expensive edible oils, such as mustard oil (MO) (Wang et al. 2019), owing to its low price. To examine the ability of the dielectric spectroscopy to detect and quantify the adulteration in MO, four types of adulterated samples were prepared for investigation by mixing AO in MO-based purities of 2, 4, 6 and 8 mL·100 mL^{-1}). The prepared samples were kept in the laboratory at an ambient temperature (27 ± 1 °C) before the experiment.

Instrumentation system. The dielectric study-based system developed for the dielectric measurement is presented in Figure 1. The central unit is known as an NF Techno ZM2376 LCR Meter that was connected to a personal computer for control and data logging. The dielectric parameters were measured in frequencies between 1 kHz to 10 MHz. The procedure for the dielectric measurement is as follows:

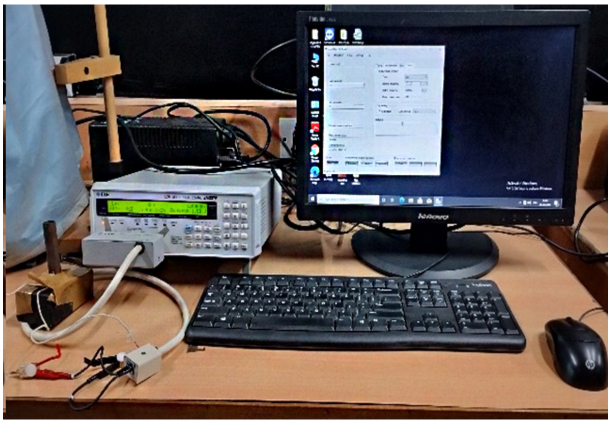

Figure 1. LCR meter set up for the dielectric measurement

LCR – inductance, capacitance, resistance

Figure 2. Temperature controller-sensor device for the temperature measurement

after the measurement system was calibrated, the air capacitance C_0 of the text fixture was tested in a temperature control system. The dielectric data of the samples were measured automatically at an interval of 7 min at the designated temperature. The measured data were recorded and plotted as a Bode diagram with ORIGIN and MATLAB softwares. Dielectric measurements of the pure and adulterated oils were taken at 30 ± 0.5 °C. The dielectric constant and dielectric loss factor were evaluated at a temperature interval of 5 °C between 30 to 50 ± 0.5 °C. A similar method was used to calculate the dielectric parameters in a previous study and also by other researchers (Lizhi et al. 2008; Mishra et al. 2023).

A temperature controller maintains the oil sample's required temperature, as shown in Figure 2. The statistical and regression analyses for the experimental data were performed in MATLAB software to create a multivariate linear regression model to obtain more reliable results in finding the adulteration in the vegetable oil.

RESULTS AND DISCUSSION

Effect of the frequency on the dielectric properties. Understanding the dielectric properties of the binary mustard and AO combinations is crucial, especially with India's weather, where temperatures frequently average about 45 °C. The frequency is a significant factor that affects the dielectric behaviour of oil samples. Figure 3 shows the variation in the dielectric constant values with the frequency at the indicated percentage of AO in the MO sample and at a constant temperature of 45 °C. The dielectric constant value for each sample substantially

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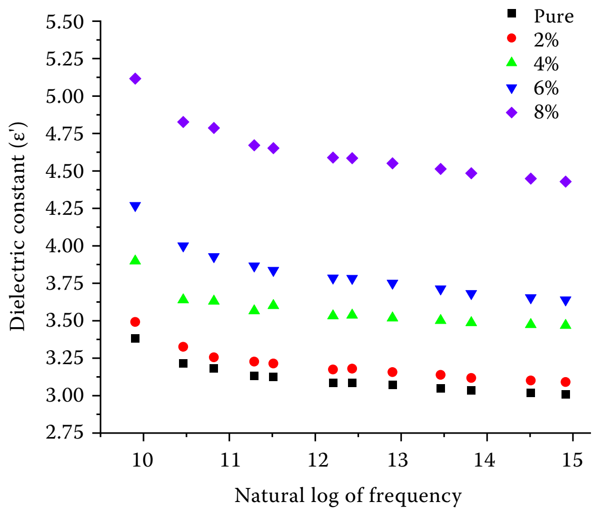

Figure 3. Frequency dependence of the dielectric constant of the oils at the indicated impurity (%) and constant temperature (45 °C)

decreases with the increasing frequency, and a decreasing trend of dielectric constant was found, ranging from 5.12 to 3.11. It was also found that the value of the dielectric constant increases by increasing the percentage amount of AO in the MO sample.

A similar decreasing trend was found for the dielectric loss. Figure 4 shows that the dielectric loss decreased from 2.21 to 0.13 with the frequency. The dielectric loss value rises with an increasing percentage of AO in the MO sample. This decreasing behaviour is due to the interactions between their molecular structures. Similar decreasing trends were reported for other edible oils (Lizhi et al. 2008).

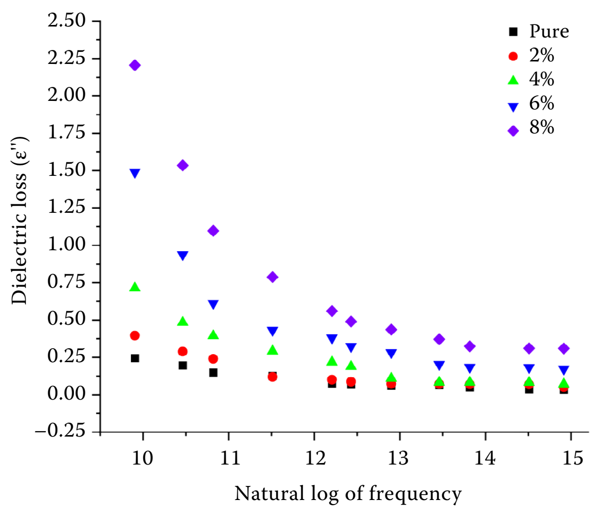

Figure 4. Frequency dependence of the dielectric loss of the oils at the indicated impurity (%) and constant temperature (45 °C)

Effect of the temperature on the dielectric properties. In Figures 5 and 6, variations in the dielectric constant and dielectric loss are shown by changing the temperature at the indicated frequency. A decreasing trend was found, and the dielectric constant and dielectric loss values decrease between 2.9 and 4.45 and 0.05 and 0.124, respectively; other researchers also reported similar results (Chandel et al. 2014). This shows a typical behaviour pattern for organic compounds, which exhibit a decrease in the relaxation time as the temperature rises due to a change in the effective dipole length; as molecules are more closely bound to-

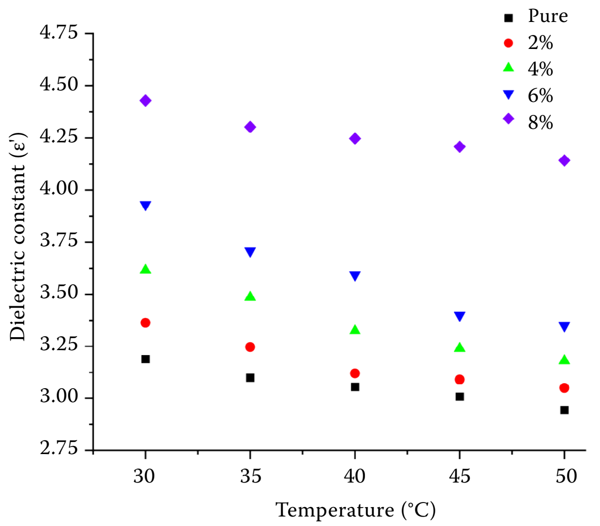

Figure 5. Temperature dependence of the dielectric constant of the oils at the indicated impurity (%) and at a constant frequency (3 MHz)

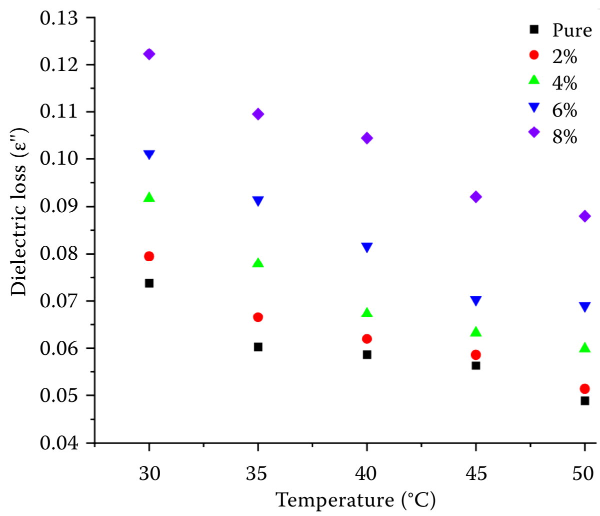

Figure 6. Temperature dependence of the dielectric loss of the oils at the indicated impurity (%) and at a constant frequency (3 MHz)

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gether at low temperatures, it takes a longer time for them to shift their orientation, resulting in long relaxation times. Similar behaviour was reported in our previous study (Mishra et al. 2023).

Regression analysis. The dielectric constant and dielectric loss factor are two significant parameters that aid in differentiating between pure oil and adulterated oil. Dielectric properties vary with the frequency, and, as a result, dielectric spectroscopy was carried out in the frequency range of 1 Hz to 10 MHz for the pure oil and at various levels of adulteration in the oil: 2, 4, 6 and 8%. To monitor the oil quality, it is imperative to understand the relationship between the dielectric constant and the dielectric loss factor with the frequency and the adulteration percentage in the oil. Models with a robust estimate of the goodness of fit of the data, i.e. The coefficient of determination (R^2) and the standard error of estimate (SE), can explain this association (Firouz et al. 2021).

$$R^2 = 1 - \frac{\sum_{i=1}^{n}\left(\varepsilon'_{\text{actual}}(i) - \varepsilon'_{\text{predicted}}(i)\right)^2}{\sum_{i=1}^{n}\left(\varepsilon'_{\text{actual}}(i) - \overline{\varepsilon'_{\text{actual}}}\right)^2} \quad (1)$$

$$S = \sqrt{\frac{\sum_{i=1}^{n}\left(\varepsilon'_{\text{actual}}(i) - \varepsilon'_{\text{predicted}}(i)\right)^2}{n-(k+1)}} \quad (2)$$

where: n – the number of samples; $k + 1$ – the number of coefficients in the model, including the intercept; $\varepsilon'_{\text{actual}}$ – and $\varepsilon'_{\text{predicted}}$ – the actual and predicted dielectric constants, respectively; $\overline{\varepsilon'_{\text{actual}}}$ – the mean of the actual dielectric constant.

The statistical and regression analyses for the experimental data were performed in MATLAB to create such models. A paired samples t-test was used to analyse the effects of the frequency and adulteration on the dielectric parameters, with a significance level of 0.05 being set for the differences.

The multiple linear regression model also discussed by (Nelson and Trabelsi 2011) obtained below explained the relationship between the dielectric constant and adulteration percentage of the log of frequency .

$$\varepsilon' = \varepsilon'_0 + \alpha \log f + \beta A_p \quad (1\!\!\!-3)$$

where: A_p – the adulteration percentage; α, β – constants determined by the regression.

Table 1 lists the values for α and β.

Table 1. Value of the regression constants (ε', α and β) and statistics (adjusted R^2 and SE of estimate) for the dielectric constant

Oil	ε'_0	α	β	Adj. R^2	SE of estimate
AO	3.03714	–0.02320	0.28086	0.96915	0.08706

Dielectric constant is predicted by Equation (3), which is presented in Figure 7; AO – argemone oil; SE – standard error; α, β – constants determined by the regression; ε'_0 – dielectric constant

Figure 7 shows the Plane for the dielectric constant of the adulterated oil, as defined by multiple linear regression (Equation 3). It can be easily seen from Figure 7 that for the same adulteration percentage, an increase in the log of frequency leads to a decrease in the value of the dielectric constant. In contrast, at a constant frequency, an increase in the adulteration percentage leads to an increase in the value of the dielectric constant. Additionally, From figure it is evident and inferred from the values of the regression constants and that there is a steeper increase in the value of the dielectric constant with the increase in the adulteration percentage ($P < 0.05$) in comparison with the decrease in its value with the increase in the value of the logarithmic frequency.

Additionally, the linear relationship between the log of frequency and the pure oil's dielectric constant with the logarithmic frequency adequately described the fluctuation in the dielectric constant ($P < 0.05$). A similar relationship was obtained, which explained the variation in the dielectric constant with the log of frequency for the pure AO

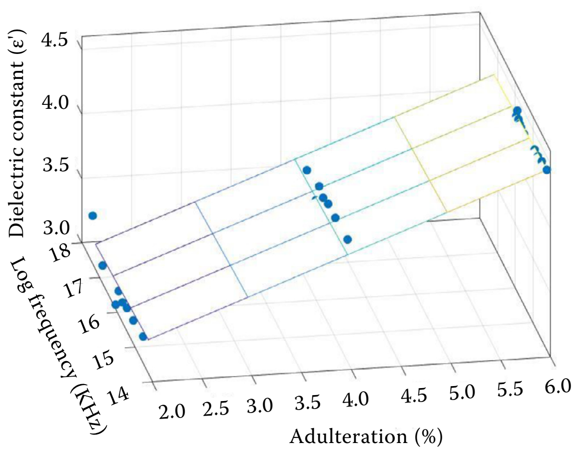

Figure 7. Plane described by the multiple linear regression model for the dielectric constant of the adulterated oil

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(P < 0.05). The obtained linear regression model is shown below:

$$\varepsilon' = \varepsilon'_0 + \alpha \log f \tag{4}$$

The values of the regression coefficient α for the pure oil and pure AO are mentioned in Table 2.

Figures 8 and 9 show that the dielectric constants of the pure oil and pure argemone oil decrease and increase, respectively, with an increase in the log of frequency ($P < 0.05$).

The obtained dielectric loss factor's association with the adulteration percentage (A_p) and frequency (f) is explained quite fruitfully by a linear relationship, which is as follows:

$$\varepsilon'' = \varepsilon''_0 + \tilde{\alpha} f + \tilde{\beta} A_p \tag{5}$$

where: $\tilde{\alpha}$, $\tilde{\beta}$ – the constants determined by the regression.

Table 2. Values of the regression constants (ε', α) and statistics for the dielectric constant (adjusted R^2 and SE of estimate)

Oil	ε'	α	Adjusted R^2	SE of estimate
Pure oil	6.6275	–0.21858	0.96245	0.03577
Pure AO	–2.6013	0.34336	0.95827	0.04164

Dielectric constant is predicted by Equation (4), which is presented in Figures 8 and 9 for the pure oil and AO, respectively; AO – argemone oil; SE – standard error; α – constant determined by the regression; ε' – dielectric constant

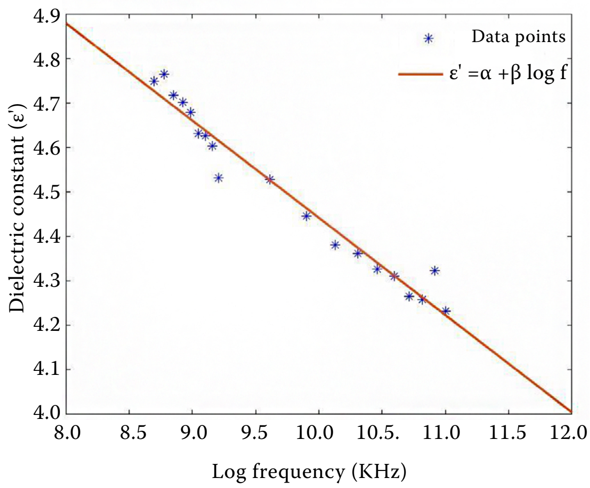

Figure 8. Frequency dependence of the dielectric constant of the pure oil given by the linear regression model
ε' – dielectric constant; α, β – constants determined by the regression; f – frequency

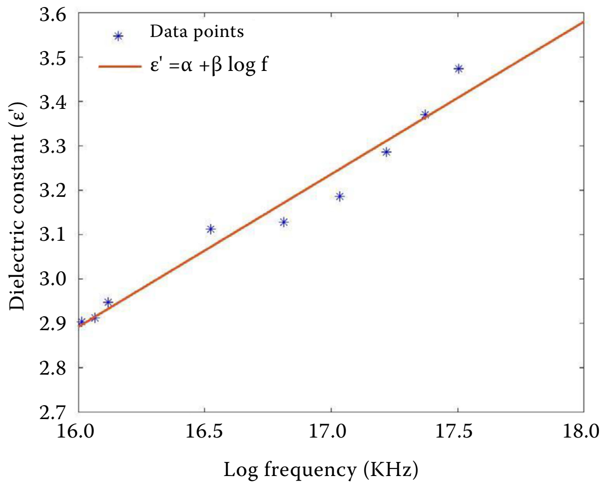

Figure 9. Frequency dependence of the dielectric constant of the pure argemone oil given by the linear regression model
ε' – dielectric constant; α, β – constants determined by the regression; f – frequency

The values of $\tilde{\alpha}$, and $\tilde{\beta}$ are given in Table 3.

A direct correlation between the loss factor parameter values with the frequency and adulteration percentage can be observed from Figure 10 and the regression constants $\tilde{\alpha}$, and $\tilde{\beta}$. In other words, an increase in the frequency and adulteration percentage values leads to an increase in the value of the dielectric loss factor ($P < 0.05$) (Equation 5).

Additionally, the association between the frequency and dielectric loss factor of the pure oil and pure AO is sufficiently linear and is of the form below:

$$\varepsilon'' = \varepsilon''_0 + \tilde{\alpha} f \tag{6}$$

The decreasing relationship of the dielectric loss factor with the frequency for the pure oil and pure AO is evident from Figures 11 and 12, respectively ($P < 0.05$).

Table 3. Values of the regression constants (ε'', $\tilde{\alpha}$, $\tilde{\beta}$) and statistics for the dielectric constant statistics (adjusted R^2 and SE of estimate)

Oil	ε''	$\tilde{\alpha}$	$\tilde{\beta}$	Adj. R^2	SE of estimate
AO	–0.70078	0.03666	1.0491e–07	0.98844	0.10933

Dielectric constant is predicted by Equation (5), which is presented in Figure 10; AO – argemone oil; SE – standard error; $\tilde{\alpha}$, $\tilde{\beta}$ – constant determined by the regression; ε'' – dielectric loss; e – 1.0491 × 10^{-7}

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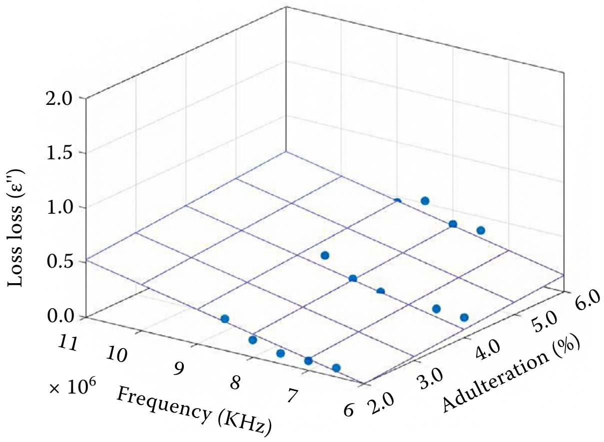

Figure 10. Plane for the dielectric loss factor of the adulterated oil, as defined by the multiple linear regression model

These linear relationships between the variables will help predict the dielectric constant values and dielectric loss factor for the given frequency and the adulteration percentage values. The standard error of the estimates is also tiny, which further authenticates the robustness of the model. Relative to the dielectric constant, the standard error of estimates is 1% to 2%; however, the percentage is slightly higher relative to the dielectric loss factor. The variation in the relationship of the dielectric constant and dielectric loss factor with the frequency is evident for the pure oil, pure argemone oil and adulterated oil in our investigation. However, it is agreed that, as the percentage of adulteration increases, the values of the dielectric constant and the dielectric loss factor increase.

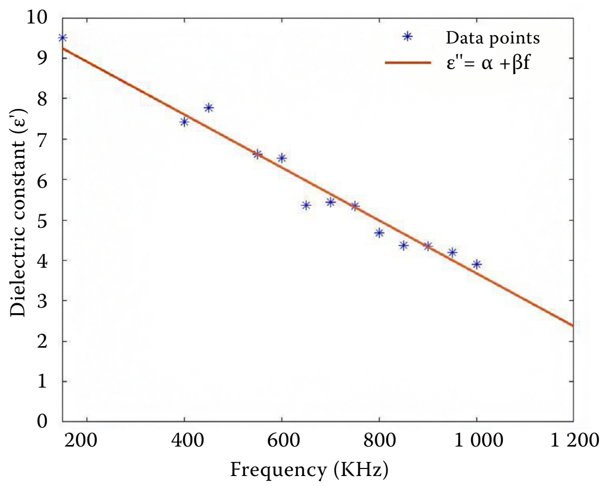

Figure 11. . Frequency dependence of the loss factor of the pure oil, given by the linear regression model l
ε'' – dielectric loss; α, β – constants determined by the regression; f – frequency

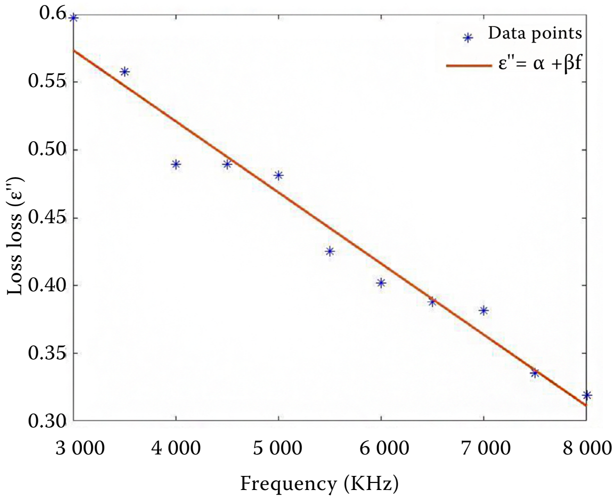

Figure 12. Frequency dependence of the loss factor of the pure argemone oil, given by the linear regression model
ε'' – dielectric loss; α, β – constants determined by the regression; f – frequency

The authors (Singh and Tarsikka 2021) studied the effect of palm oil adulteration on the dielectric properties of mustard oil. For this, a mathematical model was developed using the data of the dielectric constant, dielectric loss for different palm oil concentrations in mustard oil at different temperatures and frequencies. A liner regression model with the adulteration percentage as an independent variable and the dielectric constant and dielectric loss as the dependent variables was developed for a frequency range of 10 to 100 kHz and a temperature range of 30 to 70 °C. A R^2 greater than 0.87 was established. Their study established that the frequency had a negligible effect on the dielectric constant whereas the dielectric loss decreased with an increase in the frequency. Our study focused on detecting the adulteration in mustard oil using low frequency dielectric spectroscopy

Table 4. Regression constants (ε'', $\tilde{\alpha}$) and statistics (adjusted R^2 and SE of estimate) for the dielectric loss factor

Oil	ε''	$\tilde{\alpha}$	Adj. R^2	SE of estimate
Pure Oil	10.22764	–0.00655	0.9796	0.30348
Pure AO	0.731176	–5.3E–05	0.96092	0.01753

Dielectric loss factors are predicted by Equation (6), which is presented in Figures 11 and 12 for the pure oil and argemone oil, respectively; AO – argemone oil; SE – standard error; $\tilde{\alpha}$ – constant determined by the regression; ε'' – dielectric loss; E – -5.3×10^{-5}

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in the frequency range of 1 kHz to 10 MHz at a temperature range of 30 to 50 °C. A multiple linear regression model was established to study the relationship between the dielectric constant and dielectric loss as the dependent variables and the adulteration percentage, log of frequency as the independent variables. A R^2 greater than 0.96 was established in our case. In our study, the frequency had a negligible effect on the dielectric constant whereas the dielectric loss decreased with an increase in the frequency which is in conformity with the results obtained in (Singh and Tarsikka 2021). A similar study was conducted by (Nelson and Trabelsi 2011) in which they explored the relationship of the dielectric properties with the frequency and moisture content for selected seeds by developing a multiple liner regression model. In their study, a R^2 greater than 0.9043 was established. The linear dependence of the dielectric constant and dielectric loss on the temperature has also been established by other researchers (Lizhi et al. 2008; Bhargava et al. 2016).

CONCLUSION

According to the analysis, more regular behaviour in the dielectric constant trend than the dielectric loss factor trend was observed, possibly owing to the polarising effect. A statistical analysis was performed using MATLAB, and linear relationships between the frequency variables and the adulteration percentage were developed to detect the adulteration and predict the dielectric constant and loss factor for the pure and adulterated mustard oil for the first time. This will help detect the adulteration in edible oils. This study is helpful in determining the expected relationships between the dependent dielectric parameters and correlating them with essential features such as the adulteration. Using dielectric spectroscopy, it is easy to predict that the oil is adulterant-free. The result confirmed that the proposed system is able to detect oil-based adulteration in mustard oil. This study can be helpful for oil-identification, quality evaluation, and quality monitoring during equipment processing, storage, and design.

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