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Adulteration Control of Lavender Essential Oil by using Its Electrical Properties in the Low-Frequency Range

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Abstract: With the development of technology, analysis methods have also increased and, as with many products, the properties of essential oils can be studied using methods based on electrical properties. In this study, the electrical properties of lavender essential oil mixed with various fixed oils were to be investigated to determine the adulteration rate more quickly and easily. Corn oil, olive oil, and sunflower oil were selected as fixed oils to obtain the oil samples with different adulteration rates. In this context, the real and imaginary parts of the permittivity, the loss tangents, the electrical conductivities, the amplitudes, and the phases of the impedances of the oil samples were measured. The measurements were also used to create an equivalent Debye circuit model for the lavender essential oil in order to analyze its electrical properties. The results showed that the lavender essential oil and the oil samples mixed at different rates showed different results in terms of electrical properties at different frequencies. According to the measurements, electrical conductivity was a critical parameter for a mixture of pure lavender essential oil and corn oil. The loss tangent was investigated for the mixture of pure lavender essential oil and olive oil, and the mixture of pure lavender essential oil and sunflower oil. It was observed that the electrical conductivity in the studied frequency band increased when the adulteration rate in the mixed corn oil samples increased. In addition, the loss tangent of the olive oil mixture and the sunflower oil mixture increased as the adulteration rate of said oil samples increased. By measuring the electrical properties, it may be possible to determine whether there is an adulteration and dilution process for particularly valuable essential oils, and if so, in what proportion.

Keywords: Debye model, dielectric properties, electrical impedance spectroscopy, lavender essential oil

1. INTRODUCTION

Medicinal and aromatic plants, which are widely used in the food, medicine, and cosmetics industries, are one of the most remarkable product groups of recent years. Essential oils and aromatic extracts obtained from medicinal and aromatic plants are used by the fragrance and flavor industries in the composition of perfumes, food additives, cleaning products, cosmetics, and medicines, as a source of flavor chemicals, or as a starting material for the synthesis of nature-identical and semi-synthetic beneficial aroma chemicals [1]. Due to the antioxidant [2], antimicrobial [3], antibacterial [4], and antifungal [5] properties of essential oils, they are used in agricultural production as insecticides, fungicides, herbicides, and nematocides. It is used as a source of struggle agent [1] and as a natural preservative in the food industry. The main factor affecting the quality of the essential oil is the growing conditions of the plant. Many factors such as altitude, soil conditions, the use of chemicals such as pesticides, and precipitation affect the quality of the oil obtained. The time at which the plant is harvested affects the quantity and quality of the essential oil, and for some plants even the harvest time during the day is important. In addition, running the essential oil through standard distillation processes at the right temperature, pressure, and time depending on the location in the plant, applying hygienic conditions to the equipment during collection and processing of the plant material, chemical degradation due to exposure to heat, light or oxygen during packaging and transportation, and proper packaging and storage are also important factors that affect the quality and quantity of essential oil.

Lavender (*Lavandula sp.*) is a very valuable essential oil plant from the Lamiaceae family. Lavender, whose produc-

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tion is increasing every year in our country, is used in many areas such as perfume, cosmetics, medicine, herbal tea, and landscaping. It is known that lavender can affect the central nervous system and is used as a sedative. Lavender oil owes these properties to the high proportion of linalool it contains. The strong antiseptic (germicidal) and antibiotic (bactericidal) effect of lavender oil is also well known. That is why lavender oil is particularly important among aromatherapeutic oils [6].

Due to the increasing demand for essential oils and the low yield of essential oils extracted from raw materials, adulteration of essential oils with high commercial value has become a common problem. The most common forms of adulteration in essential oils are the addition of fixed oils to essential oils, the use of solvents for dilution (such as dipropylene glycol (DPG), propylene glycol (PG)), the addition of other essential oils with low production costs, and the addition of chemical components (such as active ingredients) to improve content analysis results. There are many studies on the determination of irritants in essential oils [7]-[9].

Although there are differences in the determination of essential oils and fixed oils, such as chromatographic and analytical methods, the separation and chemical components of these oils can be determined in the laboratory using gas chromatography (GC-MS) [10], high performance liquid chromatography (HPLC) [11], and nuclear magnetic resonance (NMR) spectroscopy [12]. There is no direct and rapid evaluation method for analyzing the adulteration rate in essential oils in the laboratory environment. On the other hand, the conventional methods are costly and laborious. The aim of this study was to make the determination of adulteration rate based on the electrical properties of lavender essential oil faster and easier. Therefore, the variation in the electrical characterization of lavender oil and fixed oils was investigated. The following parts of the study, including the extraction of the lavender oil and its electrical properties, are explained. Then the equivalent circuit model is established based on the electrical measurement results. The other part includes the evaluation of the dielectric properties of different oil mixtures at various ratios.

2. Material and method

In this study, the essential oil extracted in a laboratory environment from the species *Lavandula intermedia* was investigated. The fixed oils used in the study were obtained from the market as packaged shelf products.

A. *Essential oil extraction*

The dry plant material was distilled for 120 minutes using a Clevenger apparatus to extract the essential oil. The essential oil components were extracted following Özek et al. [13], using an essential oil component analysis device (GC/-MS-FID; gas chromatography (Agilent 7890A) – mass detector (Agilent 5975C)), and a capillary column device (HP Innowax Capillary; 60.0 m × 0.25 mm × 0.25 µm). The samples were diluted 1:50 with hexane for analysis. Helium gas was used as a carrier gas in the analysis at a flow rate of 0.8 mL/min, and samples were injected into the device at a split ratio of 40:1 as 1 µL. The injector temperature was set to 250 °C, the column temperature program was set to 60 °C (10 minutes), 4 °C/min from 60 °C to 220 °C and 220 °C (10 minutes).

The total analysis time with this temperature program is 60 minutes. A scanning range (m/z) of 35-450 atomic mass units and an electron bombardment ionization of 70 eV were used for the mass detector. The diagnosis of essential oil components was based on information from commercial WILEY and OIL ADAMS libraries. The percentages of the components were determined with the flame ionization detector (FID) and the diagnosis of the components was carried out with the mass spectrometry (MS) detector.

The content of the lavender oil used in the study is given in Table 1. The main component of the oil is linalool with a content of 43.90 %. linalyl acetate (19.99 %), camphor (9.10 %) and 1,8-cineole (7.36 %) can be reported. These four components make up 80.35 % of the total.

Table 1. The content of pure lavender oil used in research.

Peak	RI	RT	Component	Pct total
1	1028	10.804	α-pinene	0.41
2	1115	14.056	β-pinene	0.45
3	1169	16.418	β-myrcene	0.72
4	1207	18.047	Limonene	1.08
5	1218	18.524	1,8-cineole	7.36
6	1241	19.508	cis-beta-ocimene	0.57
7	1258	20.253	trans-β-ocimene	1.30
8	1266	20.554	3-octanone	0.83
9	1425	26.804	Hexyl butanoate	0.60
10	1538	30.627	Camphor	9.10
11	1554	31.160	Linalool	43.90
12	1567	31.549	Linalyl acetate	19.99
13	1617	33.116	Neryl propanoate	3.23
14	1686	35.138	Lavandulol	0.56
15	1714	35.946	α-terpineol	2.50
16	1717	36.044	Borneol	2.56
17	1739	36.644	Neryl acetate	0.51
18	1769	37.484	Geranyl acetate	0.90
19	1811	38.637	Nerol	0.55
20	1857	39.846	Geraniol	1.18
21	2242	49.083	α-bisabolol	1.64

RI: retention indices; RT: retention time

B. *Electrical properties of materials*

Dielectric properties allow material to be studied together with circuit theory and electromagnetic theory [14]. The complex impedance expression in the equivalent circuit model of any material is defined as follows.

$$Z = |Z| \langle \phi = Z' + Z'' \tag{1}$$

where $|Z|$ is the amplitude of the impedance (Ω) and ϕ is the phase angle of the impedance. Z' and Z'' are the real and imaginary parts of the complex impedance, respectively.

The impedance model of any material is based on its dielectric properties. Permittivity is one of the fundamental parameters that provides information about the electrical properties of the material [15], [16]. The complex permittivity ε is defined as in (2) for sinusoidal and continuously applied fields.

$$\varepsilon = \frac{1}{j\omega\, C_0 Z} = \varepsilon' - j\varepsilon'' \tag{2}$$

In the above equation, the capacitance of the free space is $C_0 = \varepsilon_0 A/d$, where ε_0 is the dielectric constant of the free space with the value of 8.854×10^{-12} F/m, A is the surface of the electrodes, d is the distance between the electrodes, ε' is the real part of the complex permittivity. It denotes the energy storage in the material under an electric field [17]. ε'' is the imaginary part of the complex permittivity and corresponds to the energy dissipation. The expressions ε' and ε'' of a material are defined as in (3) [18].

$$\begin{aligned}\varepsilon' &= \frac{-Z''}{|Z|^2\omega\, C_0}\\ \varepsilon'' &= \frac{Z'}{|Z|^2\omega\, C_0}\end{aligned} \tag{3}$$

The electrical conductivity of materials under a time-varying electric field is obtained by the imaginary part of the complex permittivity with the unit S/m [19]. In (4), the electrical conductivity is presented.

$$\sigma = \omega\varepsilon_0\varepsilon'' \tag{4}$$

where ω is the angular frequency ($\omega = 2\pi f$, rad/m), f is the operating frequency.

The ratio between the imaginary part and the real part of the permittivity is defined as the loss tangent and is used to determine how lossy the material is [20]. The loss tangent is given in (5).

$$\tan\delta = \frac{\varepsilon''}{\varepsilon'} = -\frac{Z'}{Z''} \tag{5}$$

Using the parameters given in the equations above, the dielectric properties of any material can be evaluated.

C. Determination of electrical properties

The electrical characterization of the oil samples was investigated using the measurement system shown in Fig. 1 [21]. The measurements were carried out at 27 different points in the frequency range of 0.1 Hz – 2000 Hz. A sine wave with an amplitude of 10 mV was used as the source signal. The measurements were carried out under laboratory conditions and at room temperature (24 °C).

The measurements were carried out with an electrochemical impedance spectroscopy device, Vertex.One (Ivium Technologies, Eindhoven, The Netherlands), which is based on a two-electrode system. In the two-electrode system, the counter electrode and the reference electrodes are short-circuited. The working electrode and the short-circuited electrodes are therefore used. According to the measurement scenario shown in Fig. 1, the R resistances were 10 Ω.

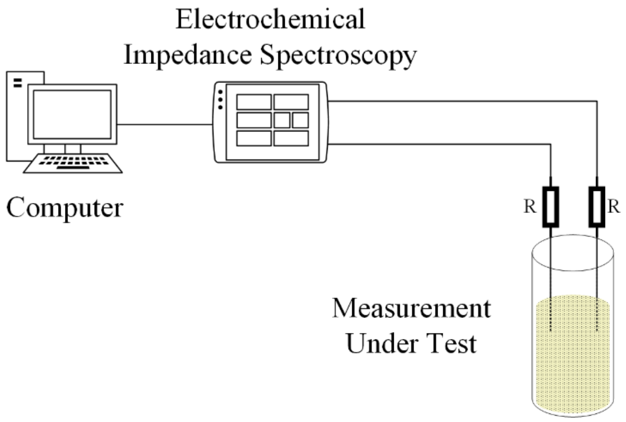


Fig. 1. Experimental measurement system.

In this study, the dielectric properties of pure lavender essential oil were discussed. In this context, the equivalent circuit model was analyzed and its dielectric properties were investigated. As a result of the measurements, the equivalent resistance R_{eq} and capacitor value C_{eq} in the circuit model of lavender oil were evaluated. In addition, the real and imaginary parts of the permittivity in the specified frequency band, the loss tangent tanδ, the electrical conductivity σ, the amplitude $|Z|$, and the phase ϕ of the equivalent impedance were determined. In addition, the dielectric properties of samples obtained by mixing the fixed oils such as corn oil, olive oil, and sunflower oil in different proportions with lavender oil were discussed. Thus, the dielectric properties of the mixture samples and lavender essential oil were compared. The oil mixtures and abbreviations investigated in the study are listed in Table 2.

Table 2. Abbreviations and oil samples mixed in different proportions.

Oil sample (with the mixture of different proportions)	Abbreviation
% 90 lavender oil + % 10 corn oil	LV10C
% 50 lavender oil + % 50 corn oil	LV50C
% 10 lavender oil + % 90 corn oil	LV90C
% 90 lavender oil + % 10 olive oil	LV10O
% 50 lavender oil + % 50 olive oil	LV50O
% 10 lavender oil + % 90 olive oil	LV90O
% 90 lavender oil + % 10 sunflower oil	LV10S
% 50 lavender oil + % 50 sunflower oil	LV50S
% 10 lavender oil + % 90 sunflower oil	LV90S

D. Equivalent circuit model analysis

In order to find the most suitable circuit model of a material, approaches based on its electrical properties should be carried out. In this way, the behavior of this material can be studied as a function of frequency and it can be used in different applications according to its performance. However, it should be noted that the equivalent circuit model and the parameters corresponding to the impedance value of the material may not be unique [22]. As shown in Fig. 2, the equivalent circuit of the obtained samples can be considered as a resistor R_{eq} and a capacitor component C_{eq} connected in parallel to obtain the Debye model [23]. In this circuit system, the internal resistances of the connecting cables and conductors are neglected.

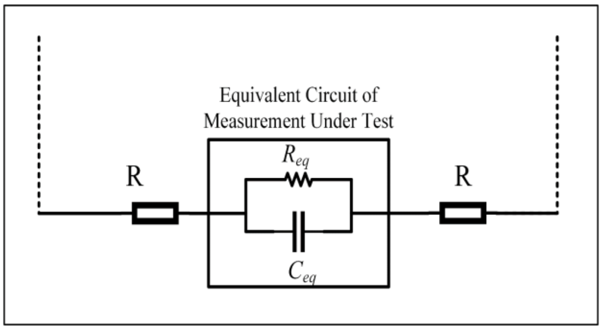


Fig. 2. Equivalent circuit model of the material under test.

Equation (1) is rearranged using the mentioned equivalent circuit components, resulting in (6).

$$Z = R_{eq}||X_c = R_{eq}||\left(\frac{1}{j\omega C_{eq}}\right) = \frac{R_{eq}}{1+\omega^2\tau^2} - j\frac{\omega\tau R_{eq}}{1+\omega^2\tau^2} \qquad (6)$$

where X_c is the capacitive reactance (Ω). τ denotes the relaxation time and is analyzed as $\tau = R_{eq} C_{eq}$. The capacitive reactance is frequency-dependent. Therefore, the electrical equivalent circuit of the oil sample under measurement also depends on the frequency. This results in different values at different frequencies.

The equivalent circuit parameters of the pure lavender oil and fixed oils used for adulteration in the study are shown in Table 3 according to the experimental measurements. As a result of the measurements, the equivalent resistance R_{eq} and the equivalent capacitor C_{eq} were determined to fit the measured impedances in the frequency range. The equivalent resistance of lavender oil was approximately 2.26 MΩ, while the equivalent capacitor value was approximately 0.99 nF.

Table 3. The values of capacitors and resistances of pure oil samples in the equivalent circuit model.

Oil sample	$R_{eq} \times 10^6$ [Ω]	$C_{eq} \times 10^{-9}$ [F]
Lavender oil	2.259	99.45
Corn oil	1.650	69.83
Olive oil	9.162	2.026
Sunflower oil	2.219	955.7

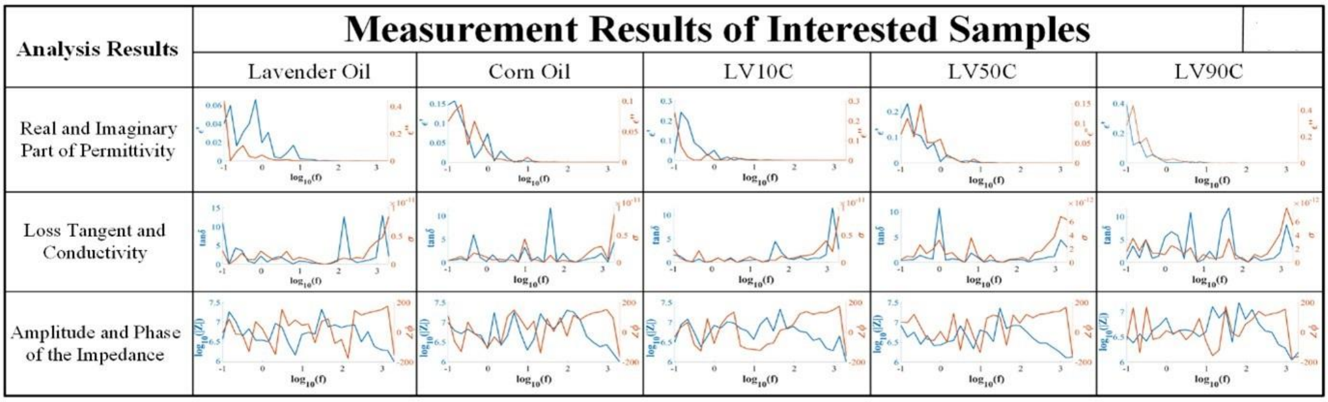
E. Measurements of dielectric properties

The dielectric properties of pure lavender essential oil in the frequency range of 0.1 Hz – 2 kHz and its mixtures with different proportions are shown in Fig. 3. The phase angle of the impedances of the pure lavender oil was about 150º in the frequency range of 200 Hz – 1.3 kHz, while about 130º was measured for the corn oil, olive oil, and sunflower oil.

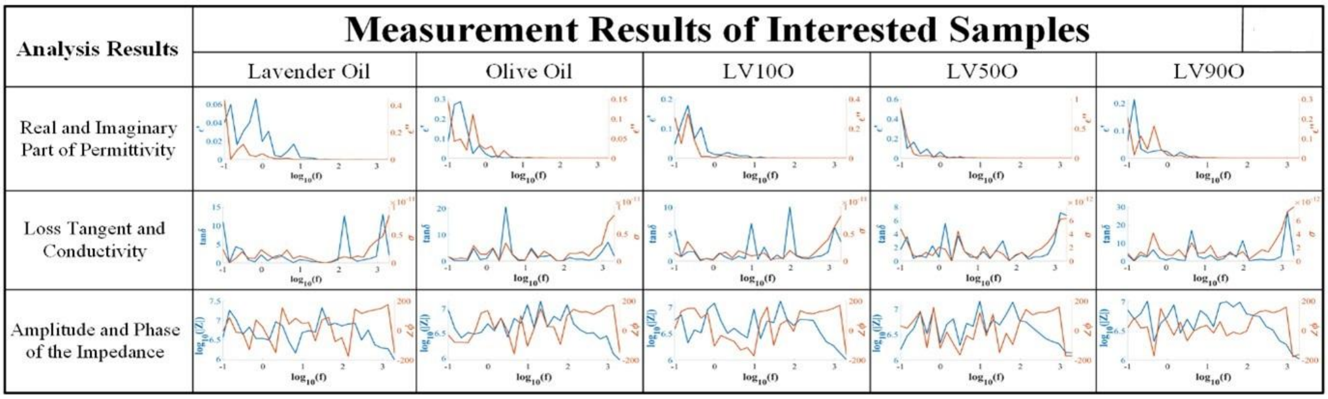
Fig. 3(a) shows the dielectric properties of the pure lavender, the corn oil, the LV10C sample, the LV50C sample, and the LV90C sample. The electrical conductivity of the LV10C sample was 8.46×10^{-12} S/m at 2 kHz. The LV50C and LV90C samples were 6.726×10^{-12} S/m and 7.994×10^{-12} S/m at 1364 Hz, respectively.

Fig. 3(b) shows the results of the pure lavender and olive oil, the LV10O sample, the LV50O sample, and the LV90O sample. The loss tangent of the LV10O sample was 10.07 at 95.06 Hz. The loss tangent of the LV50O sample and the LV90O sample was 7.14 and 26.98 at 1364 Hz, respectively.

The results in Fig. 3(c) include the dielectric properties of the pure lavender oil, the sunflower oil, the LV10S sample, the LV50S sample, and the LV90S sample. The loss tangent of the LV10S sample was 139.3 at 0.67 Hz. For the LV50S sample, the loss tangent was 109.4 at a frequency of 6.6 Hz. The loss tangent of the LV90S sample was 9.55 at 95.06 Hz.



(a)



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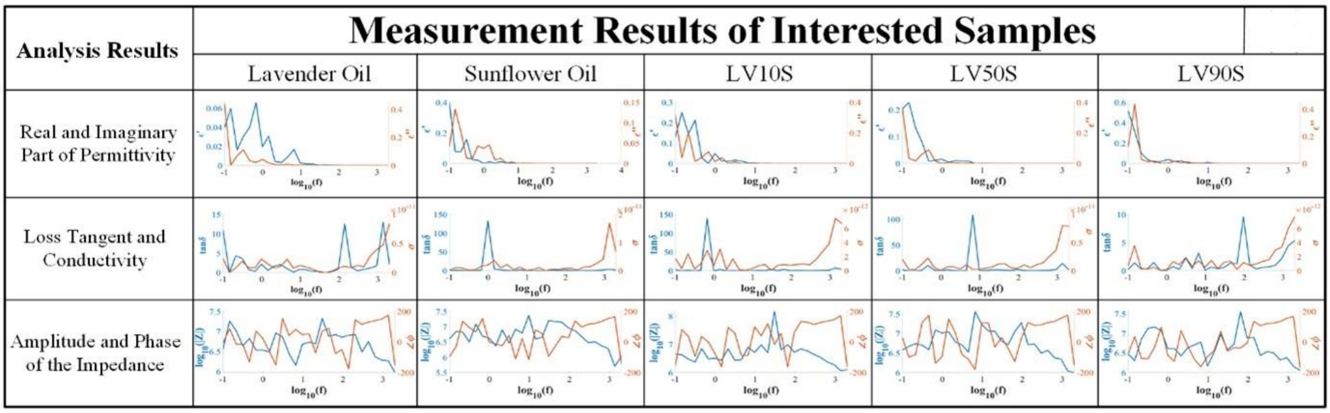

(c)

Fig. 3. The results of the measurements carried out to determine the electrical characterization of pure lavender oil and mixtures with different additives in different proportions; (a) Results of lavender oil with corn oil additives; (b) Results of lavender oil with olive oil; (c) Results of lavender oil with sunflower oil.

3. RESULTS AND DISCUSSION

In this study, the electrical properties of lavender essential oil were investigated in the frequency range of 0.1 Hz – 2 kHz to determine the adulteration rate with fixed oils. The selection of this frequency band is based on the fact that the utility frequency is defined as 50 Hz and the harmonics of this frequency lie in this spectrum [24]. In addition, common biomedical signals lie in this frequency range [25].

The experimental investigations were carried out according to the measurement setup shown in Fig. 1. As a result of the measurements, the electrical equivalent circuit models of pure lavender oil and fixed oils used for adulteration such as corn oil, sunflower oil, and olive oil were analyzed. Also, the real and imaginary parts of the permittivity, the loss tangent, the electrical conductivity, the amplitude and the phase of the impedance were experimentally determined as a result of mixing these oils in different proportions with the pure lavender oil. In this way, the electrical properties of each oil sample were evaluated.

Table 3 shows the resistor and capacitor values in the equivalent circuit model of pure lavender essential oil. The equivalent circuit is based on the Debye model, which is derived from the electrical properties of the oil. The equivalent resistance of the lavender oil was determined to be approximately 2.26 MΩ and the equivalent capacitor 0.99 nF. In addition, corn oil, sunflower oil, and olive oil were analyzed to investigate the electrical properties of the oils commonly used in the market. According to the measurement results, the equivalent resistance of corn oil was 1.65 MΩ, the equivalent resistance of olive oil was 9.162 MΩ, and the resistance of sunflower oil was 2.219 MΩ. The capacitor value of corn oil was 69.83 nF. The equivalent capacitor values of olive oil and sunflower oil were 2.026 nF and 955.7 nF, respectively. The measurements of the fixed oils were carried out on behalf of predicting the behavior of the electrical properties. Thus, the influence of fixed oils on lavender oil can be investigated. The equivalent circuit models of these oils under the electric field at the specified frequency differ from those of pure lavender oil.

Fig. 3 shows the measurement results of the samples obtained by adding different proportions of pure lavender oil to the fixed oils investigated in the study. Based on the results in Fig. 3(a) and Fig. 3(b), it was found that the real part of the permittivity of the samples converges to zero after about 10 Hz. The same behavior was observed for the sunflower oil-added mixtures in Fig. 3(c) at frequencies below 10 Hz. It can be seen that the real part of the permittivity of the samples mentioned decreases after the specified frequencies and the withstand voltages change accordingly.

Based on the results in Fig. 3(a), it was found that the frequency with the highest electrical conductivity of the corn oil mixtures decreases as the adulteration rate increases. As the adulteration rate increased at the frequency where the highest electrical conductivity was observed, the electrical conductivity value decreased. The electrical conductivity of the LV10C sample was 8.46×10^{-12} S/m at 2 kHz. The electrical conductivities of LV50C and LV90C were measured to be 6.726×10^{-12} S/m and 7.994×10^{-12} S/m, respectively, at a frequency of 1364 Hz. Although the highest electrical conductivity values of the LV50C and LV90C samples were obtained at the same frequency, the electrical conductivity varied depending on the adulteration rate. It was observed that the electrical conductivity increases with increasing adulteration rate.

The frequency at which the largest value of the loss tangent can be observed in Fig. 3(b) and Fig. 3(c), increased with increasing adulteration rate. The loss tangent was 10.07 at 95.06 Hz for LV10O. The loss tangent of LV50O was 7.14 at 1364 Hz. The value of 26.98 was measured at 1364 Hz for the LV90O sample. Although the value of the loss tangent increased for LV50O and LV90O, the frequency at which it was observed did not vary. For the LV10S sample, the loss tangent at 0.67 Hz was measured at approximately 139.3. The loss tangent for LV50S was found to be 109.4 at 6.6 Hz. For LV90S, a value of 9.55 was measured at 95.06 Hz. It was found that the electrical losses in the frequency range were highest at these frequencies for each mixture oil. Fig. 3(c) shows that as the adulteration rate increased, the amplitude of the loss tangent decreased, and the frequency at which it was observed increased.

The measurement results showed that the change in electrical conductivity of the mixture of pure lavender oil and corn oil as a function of frequency was discussed to determine that the mixture was made with corn oil. This fact was investigated with the loss tangent for the olive oil and sunflower oil.

The amplitude and phase of the impedance of each oil sample were different. It is therefore to be expected that the equivalent circuit models differ from each other. The phase angle of the impedance of pure lavender oil at 200 Hz – 1.3 kHz was about 150º, while it was about 130º for corn oil, olive oil, and sunflower oil. The phase angles of the equivalent impedances of the mixture oils listed in Fig. 3 were also measured to about 130º. It can be said that the phase angle of the fixed oils is more dominant than that of the lavender essential oils and that the loss tangent is a significant parameter for determining adulteration.

It was observed that the amplitude of the impedances of all oil samples in Fig. 3 decreased as the frequency was increased from 100 Hz to 1 kHz. Based on the impedance responses mentioned before, it can be assumed that this is a capacitive model. A biofilter using these mixture oils can be considered based on this response. The equivalent impedance can be evaluated for the specified frequency band. For a detailed circuit model, further analysis including physical and chemical approximations should be performed.

Knowledge of the electrical properties of materials allows interpretation of their physical and chemical state and comparison by application, as indicated in the literature [26] Electrical impedance measurements for materials can be considered as an alternative before a time-consuming and costly analysis. In this study, the electrical properties of lavender essential oil were investigated for the analysis of essential oil adulteration and imitation. Future work includes investigating the electrical properties of the different essential oils in use to determine adulteration rates. Consideration will also be given to investigating the electrical properties of essential oils in the high-frequency range.

ETHICAL APPROVAL (FOR RESEARCHES INVOLVING ANIMALS OR HUMANS)

Not applicable.

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CONFLICT OF INTEREST

The authors have no conflicts of interest to declare that are relevant to the content of this article.

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