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RESEARCH

Classification of Edible Oils by using Time Domain NMR (TD-NMR) Technique and Microwave (MW) Dielectric Spectroscopy

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Abstract
Increasing worldwide food demand as a result of the increasing population has led to an increase in vegetable oil prices. Olive oil, one of the most popular edible oils, is regrettably commonly adulterated by the addition of a different, less expensive oil to lower its price. As a result, many processes are carried out on edible and olive oil is one of the most cheated products today. Microwave dielectric technique (MW), in parallel with Time Domain Nuclear Magnetic Resonance (TD-NMR) technique, has started to be used in food analysis because it can analyze the content of the product, which cannot be distinguished by sensory characteristics in a simple, fast and high-efficiency way.In this study, spin–lattice (T_1) and spin–spin (T_2) relaxation times of edible oils were measured. Dielectric permittivity constants $\mathcal{E}_1$ and $\mathcal{E}_2$ have also been measured from 10 MHz to 20 GHz. It is observed that spin–spin (T_2) and spin–lattice (T_1) relaxation times of pure olive oil are shorter than those of sunflower oil and that T_1 and T_2 are shorter for riviera olive oils than for extra virgin olive oils. It was found that the T_1 and T_2 values increased with the increase in temperature in the measurements based on the investigation of the impact of temperature on the relaxation times. The highest $\mathcal{E}_1$ values are found in sunflower oils, while the lowest $\mathcal{E}_1$ values are found in riviera olive oil. As is well known, the amounts of oleic and linoleic acid in edible oils also affect the T_1-T_2, viscosity, and $\mathcal{E}_1$-$\mathcal{E}_2$ values. As a result, relaxation times and dielectric parameter constants are used to categorize and distinguish edible oils, particularly extra virgin and riviera olive oils.

Keywords TD-NMR · Relaxation Time · Edible Oil · Dielectric Constants

Introduction

Olive oil is a wholesome and nutritious oil that is obtained from the olive tree's fruit in the Mediterranean region. The characteristic aroma, taste, color, and nutritive properties of olive oil set it apart from others and separate it from edible vegetable oils (Criado et al. 2004). Olive oil plays a very important role in the human body, as it has positive effects such as an excellent source of energy, growth of the body, good cholesterol, and cell regeneration while reducing the risk of getting sick cardiovascular ailments, and nervous disorders (Calder 2015; Kostik et al. 2013; Pérez-Jiménez et al. 2007). Besides being a valuable product, due to the limited production areas, its price is higher than other oils. For this reason, there is frequent adulteration with cheaper seed and fruit oils. To detect olive oil adulteration upon blending with various oils, there are a lot of techniques, such as Fourier Transform (FT) infrared (IR) (Calder 2015; Rohman et al. 2010; Zhang et al. 2013), chromatographic method (Covaciu et al. 2020; Salerno et al. 2023), dielectric spectroscopy (Cataldo et al. 2010; Lizhi et al. 2010) and low-field NMR (Ok 2017; Šmejkalová and Piccolo 2010) have been utilized. Among various detection methods, the Time Domain-NMR technique is one of the most prospective ones in terms of its cost, measurement speed, and information accuracy. In the classical ^{1}H-NMR technique, nuclear spins of the H atom inside the sample, which has been exposed to the static magnetic field (B_0) are divided into different energy levels. Then, owing to the applied variable magnetic field (B_1), transitions between these energy levels occur. After the termination of B_1, spins come back to their previous levels. In TD-MNR methods, these times, known as relaxation times, are measured. There are two types of relaxation time spin–spin

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or transversal (T_2) and spin–lattice or longitudinal (T_1). As it is known, the TD-NMR relaxation times of edible oils such as sunflower, canola, and olive oil are very close to each other (Maraşlı et al. 2023). In this case, it is considered appropriate to use an additional secondary method alongside the TD-NMR method to more accurately detect adulterated oils. Considering reliability, economic feasibility, and user-friendliness, the microwave technique is thought the most suitable method. Microwave technique (dielectric permittivity constants real part ($\mathcal{E}_1$) and dielectric loss factor ($\mathcal{E}_2$)) in parallel with TD NMR relaxometry. In the microwave technique, the mechanism that arises from the interaction of electromagnetic (EM) waves with the material and contributes to EM absorption is the movement of electric dipoles by being affected by the electric field. As a result of this mechanism, the real component of the dielectric coefficient ($\mathcal{E}$), which is expressed as a measure of how much the electric dipoles are affected by the electric field, expresses the magnitude of the electric polarization, while the imaginary component expresses the damping of the activated dipoles and thus the energy loss (Abea et al. 2021; Nelson and Trabelsi 2009). T_1 and T_2 relaxation times and also $\mathcal{E}_1$ and $\mathcal{E}_2$ have different features in each material, resulting in the chemical structure of the material, bonding (Hindman et al. 1973), density and viscosity (Bryan et al. 2005; Ribose 2000 Lizhi et al. 2010).

In this study, T_1-T_2 and $\mathcal{E}_1$-$\mathcal{E}_2$ parameters have been investigated as well as to classify (discriminate) between various kinds of edible oils (sunflower oil, traditional oils, especially extra virgin, and riviera olive oils) by using Bruker Minispec mq20 TD-NMR (20 MHz) and Agilent Technologies E8364B PNA series network analyzer and Agilent 85070E Dielectric Slimform Probe Kit, respectively.

Material and Methods

In this study, we used four groups of samples which are called sunflower, extra virgin olive, riviera olive, and traditional olive oils. 17 selected samples were given in Table.1 and purchased from a local market in Istanbul, Turkey. The samples and devices have been conditioned at room temperature (25 °C), and the samples have been put down in an 8 mm diameter glass tube for TD-NMR and a 20 mL transparent plastic box for microwave dielectric measurements. A commercial Bruker Minispec mq20 device has been used to measure the relaxation times T_1 and T_2. Before each measurement, the samples were placed in the NMR device for at least 10 min to reach thermal equilibrium. T_2 and T_1 have been obtained using the Carr-Purcell-Meiboom-Gill (CPMG) and inversion recovery (IR) pulse sequences. In the TD-NMR technique, the signal-to-noise ratio (SNR) should be big enough to determine the relaxation time curves. to improve the SNR, each measurement has been repeated five times for each sample, and the exponential fitting has been applied to the average of measurements. The relaxation delay between these measurements was chosen to be approximately 5 times longer than the observed values of T_1 and T_2 values to be sure that samples have fully relaxed between the measurements. It should be noted that various echo-time have been tested to check the effect of self-diffusion. For minimization of the contribution of the diffusion term, the echo time was fixed to be 0.5 ms. The measurement parameters (CPMG and IR sequences) are given in Table 2. Dielectric constant $\mathcal{E}_1$ and $\mathcal{E}_2$ measurements have been performed by using Agilent Technologies E8364B PNA series network analyzer and Agilent 85070E Dielectric Slim form Probe Kit. Calibration of the liquid test fixture has been performed using 25 °C air, distilled water, and a 50 Ohm load terminal. Measurements of samples have been taken at 25 °C in a range of 10 MHz-20 GHz frequency.

Results and Discussion

NMR Properties of Edible Oils

The values of T_1 and T_2 calculated from the average of each sample's five measurement curves are shown in Fig. 1.a-b, and the types of oil can be determined. T_1 values for extra virgin olive oil and riviera olive oils vary between (109–112) ms and (107.6–110.6) ms, while T_2 values vary between (95.44–97.96) ms and (92.96–94.36) ms. T_1 and T_2 mean values for extra virgin olive oil and riviera olive oils are nearly identical (Fig. 1. c), indicating that T_1 and T_2 parameters are (110.68; 96.28) ms, and (109.12; 93.88) ms, respectively. Although Fig. 1. a and b shows that there are some overlapping in the T_1 and T_2 value of extra virgin olive oils and riviera oils. Figure 1. c shows that the distribution of the mean value of relaxation times is different for each type of oil.

Returning to Fig. 1c, we note that spin–spin and spin–lattice relaxation times of extra virgin and riviera olive oil are shorter compared to the traditional and sunflower oil. Production methods and different geographical conditions, such as climate, harvest time, maturity, humidity, altitude, etc., are fare two main factors that affect the acidity level of oils (Khdair et al. 2015). In traditional methods, the extracting period is very long, and the oils are exposed to air. Because of this reason the oxidation level increases, and consequently, the amount of acidity and peroxide increases. On the contrary, in modern systems, the acidity is less because it takes less time to produce olive oil. For these reasons, the relaxation times of traditional oils and other types of olive oils are different from each other. The gas

Table 1 List of four groups of edible oil samples and their abbreviations

Sample	Sample Name	Sample Abbreviation
Sunflower oil	Yudum Sunflower	YSO
Extra Virgin Olive Oils	Komili Extra Virgin Oil	KOVO
	Kristal Extra Virgin Oil	KRVO
	Yudum Extra Virgin Oil	YVO
	Tariş Extra Virgin Oil (North Aegean)	TVO (NA)
	Verde Extra Virgin Oil	VV0
	Güven Asa Extra Virgin Oil	GAVO
	Ahsaf Extra Virgin Oil	AVO
Riviera Olive Oils	Komili Riviera olive oil	KORO
	Kristal Riviera olive oil	KRRO
	Yudum Riviera olive oil	YRO
	Tariş Riviera olive oil	TRO
	Verde Riviera olive oil	VRO
	Ahsaf Riviera olive oil	ARO
	Kırlangıç Riviera olive oil	KRO
Traditional Olive Oils	Traditional Olive Oils 1 (Aegeon Region)	TRD-1
	Traditional Olive Oils 2 (Aegeon Region)	TRD-2

chromatography methods show that the percentage of oleic acid (C18:1) is (78.1–79.2) % for olive oils and (19.5–27.49) % for sunflower oils. Also, the percentage of linoleic acid (C18:2) is (2.7–14.4) % for olive oils and (59.55–68.5) % for sunflower oil (Řezanka and Řezanková, 1999; Santos et al. 2017; Vávra et al. 2021; Xing et al. 2019). The main reason for the difference in relaxation times between sunflower and olive oil is the different amounts of oleic and linoleic acids (Abea et al. 2021; Hindman et al. 1973; Ok. 2017; Ribose 2000). In this study, it is observed that the amount of oleic acid and relaxation times are proportional to each other. To confirm this relation, relaxation times of the pure fatty (oleic and linoleic) acids have been measured. T_1 and T_2 are obtained as (211–204.4) ms for oleic acid and (287–285) ms for linoleic acid (Maraşlı et al. 2023). Generally, it is known that olive oils are classified into two main groups riviera and extra virgin. Research on olive oil shows that the biggest difference between the two types of oil is the percentage of oleic and linoleic acids. Iverson et al. (1965) found that the average percentages of oleic and linoleic acids in extra virgin olive oil were 59.3% -19.1%, and 65.8% -14.0% in riviera olive oil, respectively. As a result, it can be observed that the studies on the fatty acids composition of olive oils and relaxation times measurements are consistent with each other. To classify the types of olive oils, the bi-exponential fitting has been applied to T_2 curves, and two components of T_2 (T_{21}, T_{22}) have been obtained. It is seen that the olive oils are localized to particular regions according to their types in drawing T_1 versus T_2 and T_{21} versus T_{22} graphs (Fig. 2a,b).

To observe the effect of temperature changes on relaxation times in edible oils, relaxation times have been measured by increasing the temperature of the device and samples from 22 °C to 38 °C in 4 °C increments. The graph of temperature versus relaxation time and its slopes are shown in Fig. 3 and Table 3.

The viscosity is related to the decrease in the slope of the graphs as temperature increases. According to this graph, the relaxation times for all edible oils increase as the temperature rises. It is well known that the relaxation times are affected by the viscosity value. When the viscosity is low, the relaxation times are long. and it is well known that the viscosity value falls with increasing temperature. Abramovič and Klofutar 1998; Rotimi 2017; Tajweed et al. 2014 investigated the viscosities of several oils at temperatures ranging from 20 °C to 100 °C. According to Abramovic and Klofutar, the corresponding mean viscosity ranged from 48.98 to 17.63 cP for refined sunflower oil, 49.14 to 17.71 cP for unrefined sunflower oil, and 63.28 to 21.03 cP for olive oil. Viscosity measurements of olive oil by Davies ranged from 27.0 to 18.0 cP, while sunflower oil measurements varied from 29.0 to 17.0 cP. The dynamic viscosity of the olive oil sample, as determined by Tajweed, was 80.5 cP at 20 °C, 60.7 cP at 25 °C, and 33.5 cP at 40 °C. Kuroda et al. (2009) demonstrated the temperature dependence of proton relaxation times in fatty acids. The obtained low viscosity resulted in a longer

Table 2 CPMG and IR parameters

Parameters for CPMG		Parameters for IR	
B_1 Frequency	19.95 MHz	B1 Frequency	19.95 MHz
90^0 Pulse Amplitude	3 dB	900 Pulse Amplitude	3 dB
180^0 Pulse Amplitude	9 dB	1800 Pulse Amplitude	9 dB
Echo Time	500 us	Max Delay	1500 ms
Number of Echoes	1500	Number of Steps	50
Repetition Time	1500 ms	Repetition Time	1500 ms
90^0 Pulse Length	3.32 us	900 Pulse Length	3.32 us
180^0 Pulse Length	6.4 us	1800 Pulse Length	6.4 us
Receiver Gain	64 dB	Receiver Gain	64 dB
Number of Scans	4	Number of Scans	2

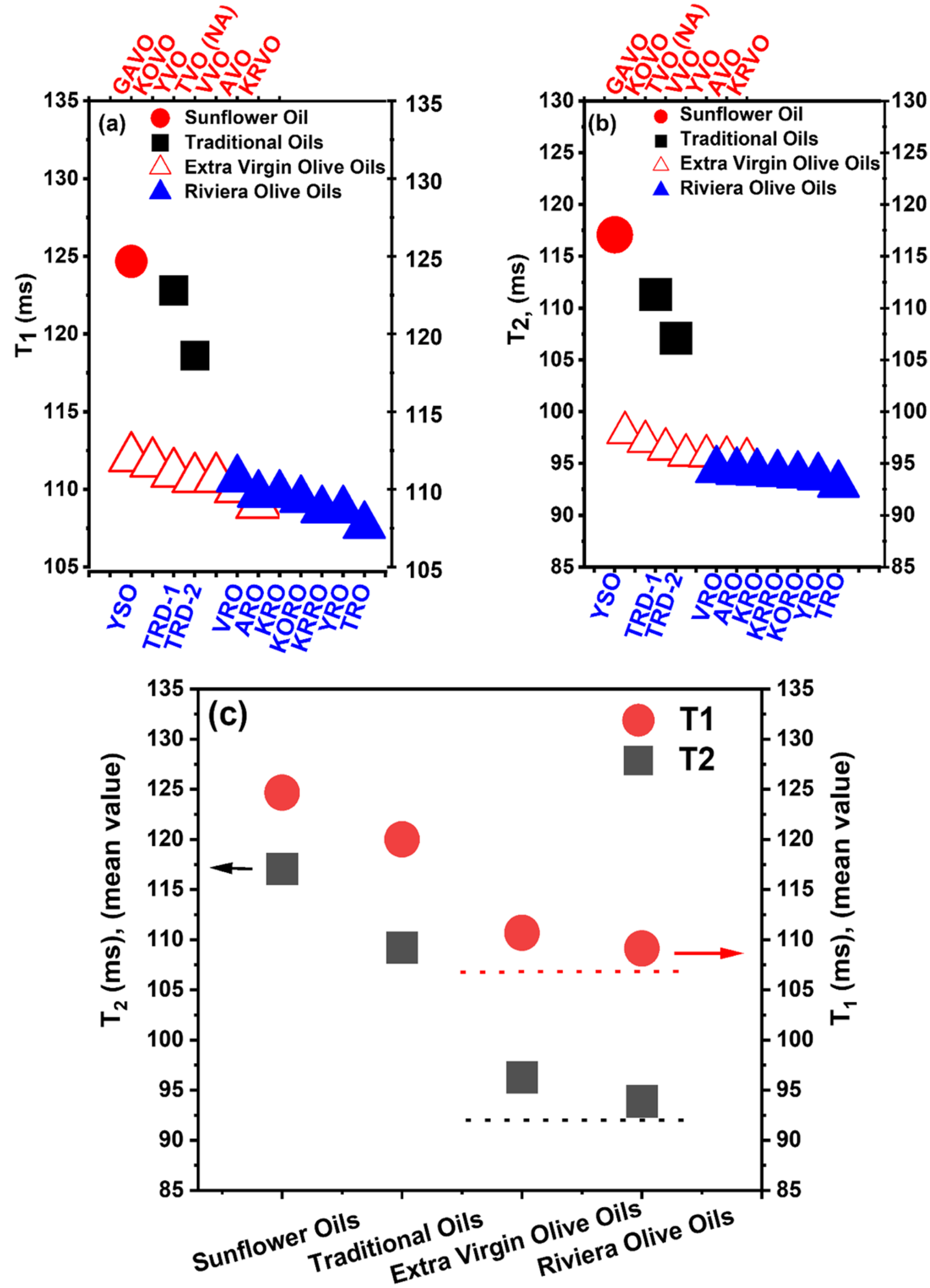
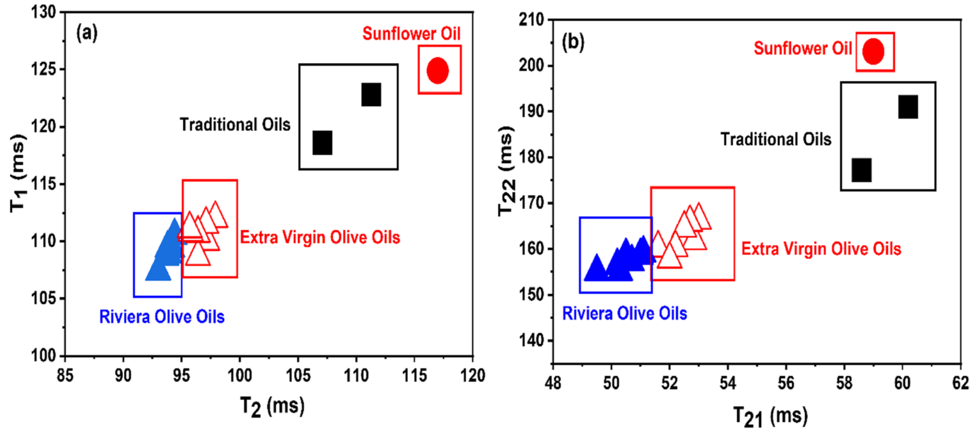


Fig. 1 The relaxation time values of edible oils **(a)** spin–lattice, **(b)** spin–spin, **(c)** mean values of spin–spin and spin–lattice relaxation times of all the measurements

relaxation time of the olive oil, and all relaxation time values in both vegetable oils were linearly dependent on temperature. These viscosity values also depend on the levels of oleic and linoleic acid in the edible oils. As mentioned earlier, sunflower oil, which has the lowest amount of oleic acid and the lowest viscosity, is the least affected by temperature rise, next to sunflower oil, traditional oil, extra virgin olive oil, and riviera olive oil. Similarly, riviera olive oil, with the lowest linoleic acid, is most affected by increasing temperature, followed by extra virgin olive oils, traditional oils, and sunflower oils. Furthermore, research on vegetable oils and fatty acids indicates that as the percentage of oleic acid in the oil increases, so does the viscosity (Bryan et al. 2005; Khdair et al. 2015; Lizhi et al. 2010). Other studies on vegetable oils and fatty acids have found that olive oil has a higher percentage of oleic acid and viscosity than sunflower oil (Řezanka and Řezanková, 1999; Santos et al. 2017). The amount of oleic acid in fatty acids increases viscosity, while the amount of linoleic acid in fatty acids decreases dynamic viscosity, according to Gila et al. 2015. The assessment of fatty acids and oil's high-pressure viscosity was studied by Schaschke et al. (2007). They found that oleic acid has a higher viscosity than linoleic acid,

Fig. 2 T_1 and T_2 correlations (**a**), T_{21}- T_{22} correlations (**b**) of edible oils



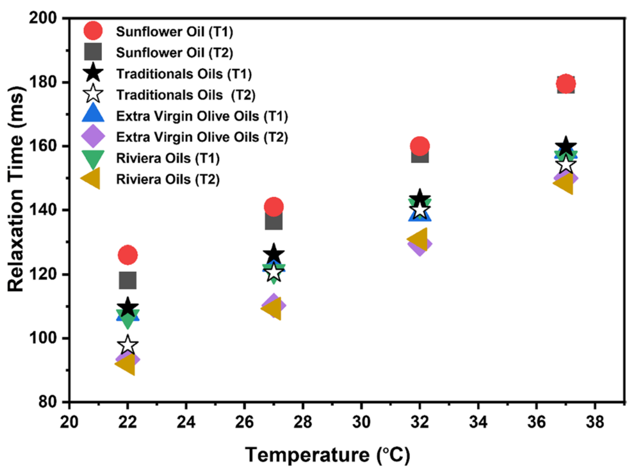


Fig. 3 Temperature-dependent graph of the relaxation times of edible oils

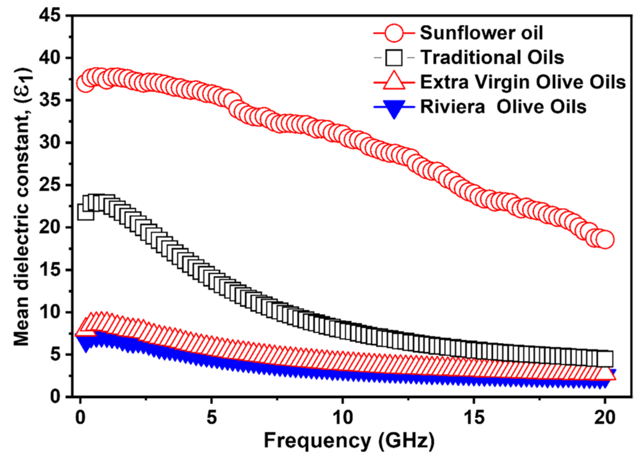


Fig. 4 Average spectrum curves of the measurements of the dilelectric spectra at 25 °C

which is 32.1 and 30.2 (MPa s), respectively. The relaxation times (T_1 and T_2) of the two pure fatty acids, oleic acid, and linoleic acid, were examined in this study. The results were (211–204.4) ms for oleic acid and (287–285) ms for linoleic acid (Maraşlı et al. 2023). These results show that the relaxation time of linoleic acid is longer than that of oleic acid, and its viscosity is lower than that of oleic acid.

Dielectric Constants of Edible Oils (ε_1—ε_2)

The mean dielectric constants (ε_1) spectra of the four types of edible oils—sunflower, traditional, extra virgin, and riviera—as a function of frequency, from 10 MHz to 20 GHz, are shown in Fig. 4. The findings demonstrated that although there was only a slight difference in the dielectric spectra of extra virgin olive oil and riviera olive oil over the studied frequency range, there were considerable discrepancies between sunflower and traditional oils. Sunflower oil had the highest mean ε_1 value across the spectrum, while Riviera olive oil had the lowest ε_1 value. The mean ε_1 of extra virgin olive oil and riviera olive oil overlapped at a high frequency; it was further observed.

Except for a few riviera and extra virgin olive oils (ARO, GAVO, KOVO, TVO(NA), and YRO), all edible oils in Fig. 5a–d (at 1 GHz and mean value) have distinct dielectric constants (ε_1 and ε_2). The very large discrepancies between the four types of edible oils are seen in

Table 3 Slope of the edible oils for relaxation time versus temperature

	Sunflower oils	Traditional Oils	Extra Virgin Olive Oils	Riviera Olive Oils
The slope of T_1 (ms/°C)	3.61	3.59	3.40	3.33
The slope of T_2 (ms/°C)	4,10	3.82	3.80	3.78

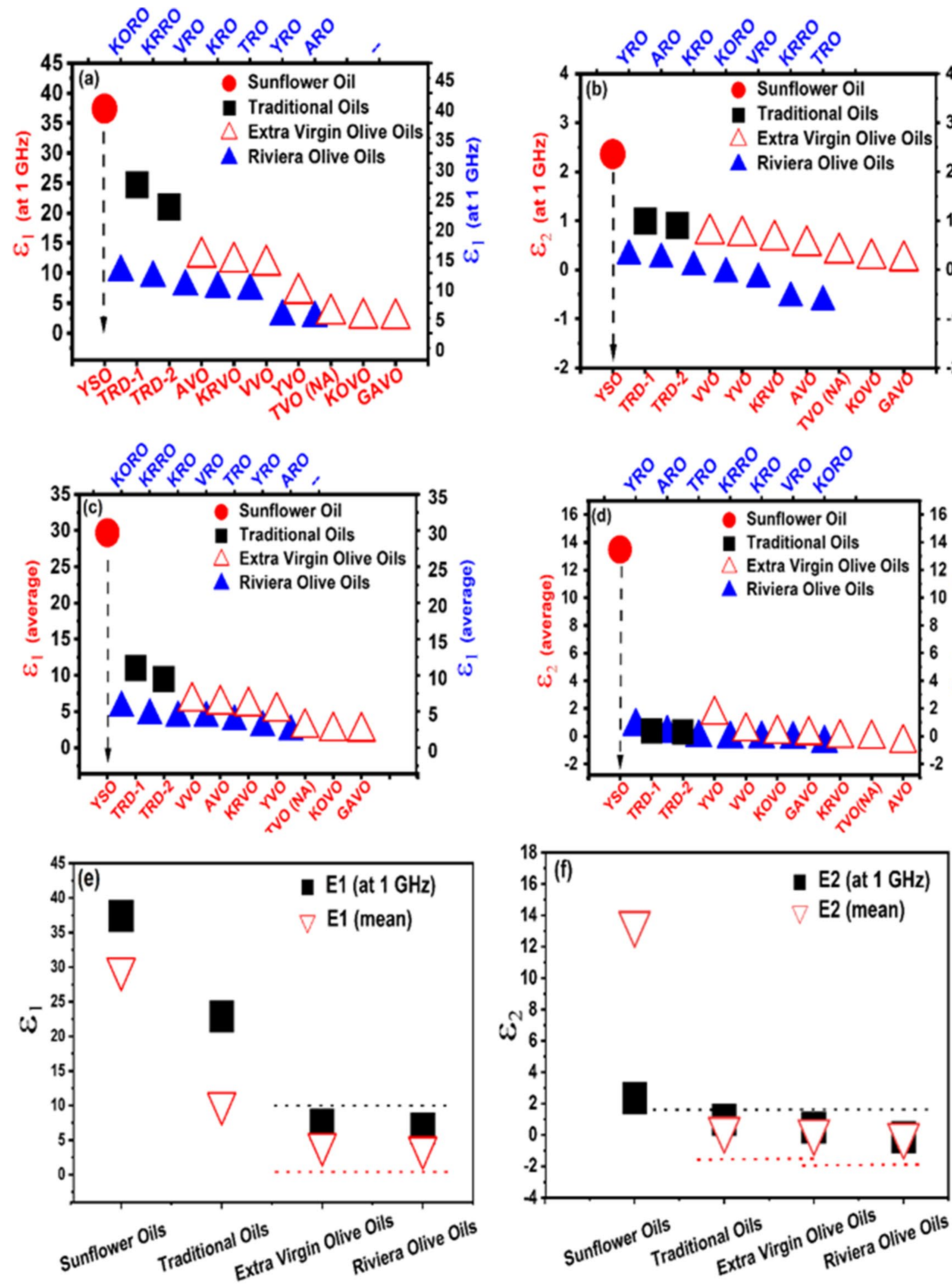


Fig. 5 Edible oil dielectric constants **(a–b)** at 1 GHz, average **(c–d)**, and oil kinds **(e–f)**

Fig. 5e–f (at 1 GHz and mean measured value). However, there is only a little difference between extra virgin and riviera olive oils. In comparison to riviera olive oil, extra virgin olive oil has been found to have higher dielectric constants ($\mathcal{E}_1$ and $\mathcal{E}_2$). It is clear from a comparison of the $\mathcal{E}_1$ data (at 1 GHz and mean value) that the dielectric constants of EVOO and RVO vary between 7.22–6.69 and 4.33–3.83. Calculations show that the differences are 7.34% and 11.5%, respectively. This research on the dielectric spectra of sunflower oils and olive oils (riviera olive oils and extra virgin olive oils) is similar to that of others (Amat Sairin et al. 2022; Deng et al. 2018; Lizhi et al. 2010, Sipahioglu and Barringer 2003). Amat Sairin et al. 2022 investigated fatty acids to determine their compositions and dielectric constants, and Sipahioglu and Barringer 2003 investigated the permittivity of vegetables and discovered both positive and negative relationships, with the permittivity and dissipation factor decreasing and then increasing with increasing frequency. Lizhi et al. 2010emonstrated that olive oil adulterated with soybean oil (linoleic and low-content linolenic acid type) and perilla oil (linolenic acid type) could be distinguished. They found that high linoleic acid contents have high dielectric constant values. Unsaturated fatty acids have a large influence on the ε_1 value of vegetable oils. Oils with high levels of oleic acid were found to have lower ε_1 values. On the other hand, oils with higher levels of unsaturation and linoleic acid showed higher ε_1 values. As previously stated, the main distinction between olive oil and sunflower oil is the percentages of oleic and linoleic acid. Olive oil has a higher percentage of oleic acid (C18:1) (78.1–79.2)% than sunflower oil (19.5–27.49)%; however, sunflower oils have a lower proportion of linoleic acid (C18:2) (2.7–14.4)% than olive oil (59.55–68.5)%. At the same meaning, riviera olive oils have a higher oleic acid (C18:1) proportion (65.84%) than extra virgin olive oils (59.36%) (Abea et al. 2021; Hindman et al. 1973; Ok 2017; Ribose 2000). I discovered through my research that materials with high oleic acid concentrations had a low dielectric constant. Also, it was found that riviera olive oils had a lower dielectric constant (ε_1 and ε_2) than extra virgin olive oils (Fig. 5. e–f).

I created a two-dimensional plot as a ε_1 versus and ε_2 graph using 1 GHz values to classify (discriminate) various types of edible olive oils (Fig. 6). It can be seen that traditional oils and sunflower oil are split into two clusters.

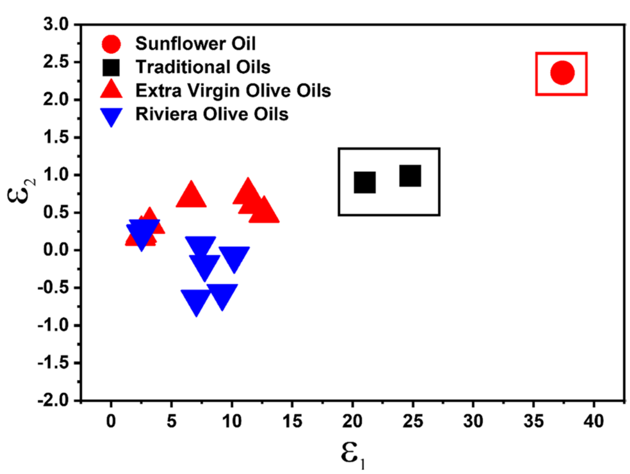


Fig. 6 ε_1 and ε_2 correlations of edible oils (at 1 GHz)

Nevertheless, differentiating between extra virgin olive oil (EVOO) and riviera olive oil (ROO) in particular is quite a struggle. Extra virgin and riviera olive oils have ε_1 values that are very similar to one another and range from (2.41–12.66) and (2.52–10.19), respectively. Some extra virgin olive oils are also mixed with some riviera olive oils. However, the majority of the extra virgin and riviera olive oil samples are divided into two distinct categories.

Conclusions

To distinguish between several types of edible olive oils, relaxation times (T_1 and T_2) and dielectric permittivity constants (ε_1 and ε_2) have been examined (sunflower oil, traditional oils, extra virgin olive oils, and riviera olive oils). Using dielectric parameter values is extremely difficult to differentiate and clarify between EVOO and ROO in particular. By utilizing the relaxation times, it will be simple to separate different edible oils from one another. Moreover, T_1 and T_2 are found to rise when the temperature rises. The temperature rise has a greater impact on riviera olive oil since it contains more oleic acid. Furthermore, I know that the viscosity of the oil rises along with the amount of oleic acid present. I also understand why the values of the edible oil's dielectric constants and relaxation times differ from one another. There were small significant differences between the riviera and extra virgin olive oils, but they were considerably different from sunflower and traditional oils. The findings revealed that oils with high oleic acid content had lower T_1, T_2, $\varepsilon_{1,}$ and ε_2, whereas oils with high linoleic acid content had larger T_1, T_2, $\varepsilon_{1,}$ and ε_2. As a consequence, I found that it is feasible to distinguish and categorize the sorts of edible olive oils, notably extra virgin and riviera olive oils. It is established that MW permittivity measurement-based detection is an excellent approach to the classification /discrimination of edible oils, which is supplementary to TD-NMR.

Authors Contributions C.O.; writing and interpretation of the manuscript, performed the measurements and all calculations.

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Data Availability The data sets generated during and/or analyzed during the current study are available from the corresponding author on reasonable request.

Declarations

Conflict of interests Cengiz Okay declares no competing interest. Cengiz Okay declares that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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