

## Detection of adulteration in olive oil using rheological and ultrasonic parameters

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### Abstract

Commercially available extra virgin oil is added with coconut oil and sunflower oil at different proportion. The variation of rheological and ultrasonic parameters in the binary mixtures of the oils are studied and reported. The oil is subjected to frying temperature 210°C repeatedly so that compositional changes take place in the fatty acids. A general decrease in the viscosity of oils is observed with increase in temperature and the difference in the variation in terms of percentage of adulteration is also studied. An empirical correlation between viscosity and temperature is also determined so as to predict viscosity and adulteration in oil at intermediate temperatures. The changes in the quality of olive oil is premeditated from the changes in the parameters such as viscosity, density, ultrasonic velocity, acoustic impedance, adiabatic compressibility and intermolecular free length, from which the adulteration in the oil can be easily perceived compared to analytical techniques.

### Keywords

Olive oil

Coconut oil

Sunflower oil

Viscosity

Ultrasonic velocity

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### Introduction

The adulteration in edible oil is a deep rooted social evil, which is a threat to human life. Adulteration of low quality, cheap, non-edible and toxic substance in the edible oil leads to different disease to human. The studies of physical and chemical properties of edible oil are an important and rigorous research topic among the researchers. Edible oil has the composition of fatty acids and triglycerides (Daniel *et al.*, 2000). Fatty acids are straight chain aliphatic compounds terminated with a –COOH group and triglycerides as esters of the propane 1, 2, 3- triol with three fatty acid residues. Among the edible oils the most healthy, important, nutritious edible oil is olive oil which has saturated fatty acids C12:0 (Lauric), C16:0 (Palmitic), C18:0 (Stearic) and the unsaturated fatty acids C18:1 (oleic), C18:2 (Linoleic), C18:3 (Linolenic) (Daniela *et al.*, 2010).

Linoleic acid consists of two conjugated double bonds which are more liable to oxidation than the single double bond in oleic acid (Rubalya *et al.*, 2009). The oil with a rich content of oleic acid is expected to have longer life time than oil with a large content of Linoleic acid. Oxidation slightly reduce the content of oleic acid in the olive oil and increases of saturated fatty acid constituents (palmitic and stearic) (Gozde and Banu, 2009). In the sunflower oil and olive oil, oxidation induced more pronounced compositional changes: contents of linoleic and linolenic acids

dropped while at the same time concentration of oleic acid sustains (Adolfo *et al.*, 2006).

High quality, healthy and high antioxidant potent olive oil has higher market prices. This high cost oil is added with low grade and cheaper substitutes oils as they look more or less identical in colour and their presence cannot be easily identified by simple visual inspection. Adulteration of olive oil may produce adverse effect which raises a serious problem to the consumer as well as to the oil agencies.

Much analytical work has been published on the chemistry of extra virgin olive oil as a basis for the detection and quantitative analysis of the type and amount of adulteration with cheaper vegetable oils and deodorized olive oils (Gozde *et al.*, 2009). The binary, ternary and quaternary mixtures of olive, sunflower, canola, cotton, corn, and soybean oils were prepared using a random design. The absorbance spectra of these synthetic samples were measured by a near infrared (NIR) spectrometer, NMR spectroscopy, Raman Spectroscopy and Chemometrics. A genetic algorithm based variable selection algorithm coupled with an inverse least squares multivariate calibration method (GILS) was used to build calibration models for possible adulterants and olive oil in the adulterated mixtures (Gozde *et al.*, 2009; Daniela *et al.*, 2010).

There has been growing public consciousness about the health benefits of olive oil all the way through the world in recent years resulting in an important increase in its consumption as part of the each day

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diet. This demand has attracted fraudulent attempts to market olive oil which has been adulterated with cheaper oils. In the present work, we propose the study of binary mixtures of olive oils with different proportions of sunflower oil and coconut oil and study their effect on physical parameters that are simple compared to analytical technique due to the compositional changes in the oil.

## Materials and Methods

### Sample preparation

Popular branded olive oil, coconut oil and sunflower oil has been collected from a local grocery shop located in Thanjavur, district of Tamilnadu, India to assess the purity of olive oil. To get sample for heated oils hundred milliliters of sample has been placed in a copper beaker and heated on an electric device, stirring manually with glass rod. A microcontroller based temperature controller has been designed and has been used to monitor and control the sample temperature. To mimic the oil oxidation process during frying, the sample has been heated up to 210°C for five times. The temperature is maintained by a temperature controller which has a very good accuracy of  $\pm 1\%$  of error.

### Methods

To find the change in viscosity with temperature a Redwood viscometer (manufactured by Associated Instrument manufacturers India Private limited, New Delhi, India) is used. It consists of a cup made of copper equipped with a pointer, which makes sure a stable head and orifice at the center of the base of inner cylinder is used. The orifice at the bottom is closed with a ball on elevation makes the oil to flow and the time is measured. The temperature of the sample is maintained by a temperature controller. The orifice is opened and the time required for collecting 50cc of oil is measured from which the viscosity of the sample is calculated.

The kinematic viscosity is calculated from the following relation:

$$(\eta) = (X^*t - Y/t) \times 10^{-4} \text{ m}^2/\text{s} \dots (1)$$

X & Y are constants, t = redwood time which measure the rate of flow in seconds.

When  $t < 34$  X = 0.0026 & Y = 1.175

And When  $t > 34$  X = 0.26 & Y = 172

The copper cup in the viscometer is washed with CCl<sub>4</sub> after each observation. Each reading is taken from the average of three trials.

A continuous wave Ultrasonic Interferometer with

a frequency of 2 MHz is used to study the changes in the ultrasonic velocity in the oils on addition with coconut and sunflower oil. The Ultrasonic Interferometer consists of an ultrasonic cell with double walled brass column with chromium plated surfaces. In the bottom of cell a piezoelectric quartz crystal of resonant frequency 2 MHz is placed with a metallic reflector attached to the shaft which moves up or down by the action of spring and a micrometer screw. The standing waves will be formed between the crystal and the reflector which is filled with the oil under study, when the crystal is excited by RF oscillator of 2 MHz. The measurement of velocity is based on the determination of wavelength in the medium. If the distance between the crystal and reflector is increased or decreased and the variation is one half the wavelengths or multiple, the current in the micro ammeter increases to maximum. The maxima and minima in the standing waves were measured using a differential amplifier which is fed to micrometer.

## Results and Discussion

Figure 1 and 2 shows a plot of the variation of viscosity of the binary mixture of olive with sunflower oil and coconut oil. The values at 30°C are quite comparable with the values given in the literature. It is observed that the viscosity decreases with increase in temperature. Molecules and functional group in saturated fatty acid are arranged in a zig-zag fashion and are piled and stacked bind with flux which regulates the viscosity of oil (Juyoung *et al.*, 2010; Rubalya *et al.*, 2012). This arrangement exerts strong inter-molecular forces (Vander waals force, London force etc) between the molecules and do not allow free flowing of the liquid hence viscosity is more. In unsaturated fatty acids a kink is produced between the C-C and decrease the rotation between the bonds. This makes the flow easier and viscosity smaller (Abramovic *et al.*, 1998; Adolfo *et al.*, 2006). The important chemical reaction that takes place in the oils on heating is hydrogenation.

From the Figure 1 it is observed that the viscosity of virgin olive oil decreases with the addition of 20% and 40% of sunflower oil. The decrease in viscosity may be due to factors like density, molecular weight and unsaturation. In this illustration this is due to the content of large amount of unsaturated fatty acid 69% of oleic acid and more than 20% of linoleic acid in sunflower oil. Figure 2 illustrate the addition of 20% of coconut oil does not make much changes in the viscosity compared to olive oil which may be due to diminutive impact of low carbon (C<sub>6</sub>, C<sub>8</sub>, C<sub>10</sub>, C<sub>12</sub>

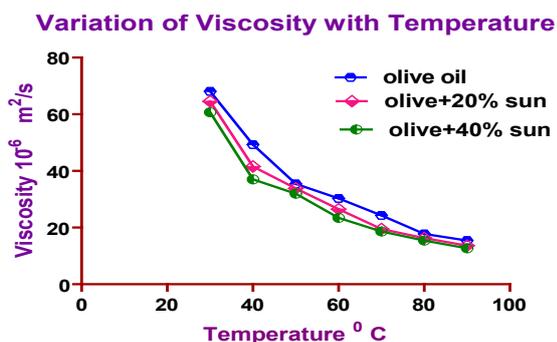


Figure 1. Change in viscosity with temperature of pure olive oil and its mixture with sunflower oil

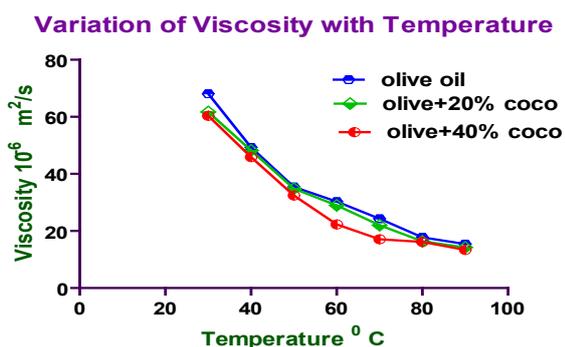


Figure 2. Change in viscosity with temperature of pure olive oil and its mixture with coconut oil

Table 1. Exponential (Arrhenius Model) function

Oil sample	R <sup>2</sup>	Significance (p)	Constants		SEE
			A (m <sup>2</sup> /s)	E <sub>a</sub> kJ/mol	
Olive oil	0.992	0.000	108.25	22.120	0.480
Olive+10%Sun	0.993	0.000	159.89	22.560	0.189
Olive+20%Sun	0.986	0.000	153.62	22.855	0.355
Olive+10%Coco	0.994	0.000	120.69	23.554	0.414
Olive +20%Coco	0.979	0.000	144.32	23.630	0.354

and C14) saturated fatty acids in coconut oil (Hui, 1999). Coconut oil consists of 92% saturated low molecular weight fatty acid and 8% of unsaturated fatty acid. Decrease in the viscosity of olive oil mixed with 40% of coconut oil exhibit more decrease in the viscosity due to large addition of short chain fatty acid.

Modeling of the temperature effect on the kinematic viscosity of oils is important and has been investigated by few researchers (Abramovic *et al.*, 1998; Anupama *et al.*, 2007; Shilpi *et al.*, 2005; Rubalya *et al.*, 2012). Two parameters and three parameters empirical equations given below were used in this study of predicting viscosity of oils in the intermediate temperature.

(1) Arrhenius equation

The following Arrhenius equation can be used to determine the activation energy of the viscous flow of oils.

$$\eta = Ae^{-Ea/RT} \dots (2)$$

where E<sub>a</sub> is the activation energy (kJ/kg), R is universal gas constant (8.314 kJ/kg mol K) and A is a constant (m<sup>2</sup>/s) (Fasina *et al.*, 2006). A plot of ln [η] against 1/T showing linear graph was drawn, from which the value of Ea is evaluated (Rubalya *et al.*, 2012). This super Arrhenius behavior of oils with temperature is illustrated in Table 1 which shows the calculated activation energy value, correlation coefficient, standard error estimate and the value of the constant A.

The temperature effect on oil viscosity has been attributed to decrease intermolecular interaction by great thermal movement. Table 1 show that the activation energy of the vegetable oils ranges from 22.12 to 23.63 kJ/mol. The highest value of activation energy was observed in olive oil added with 40% of coconut oil and 20% of coconut oil. Since activation energy refers to the sensitivity of the oil to the temperature the viscosity of this mixture of oil is more compared to other samples (Rubalya *et al.*, 2012). It was observed and suggested by the researcher (Juyoung *et al.*, 2010), that oil which has more double bond exhibit less activation energy. The composition of polyunsaturated fatty acids in olive oil is greater than 83%, sunflower oil has 89% and coconut oil comprises only 12%. The results also indicate that there is no statistically significant (p) difference in the analysis. Hence using this model we can predict viscosity at any temperature, also the degree of adulteration can be estimated.

(2) Vogel Fulcher- Tammann (VFT) type of equation

VFT model is one of the empirical formula used in the prediction of viscosity of liquids for industrial applications (Arnab *et al.*, 2002). As the viscosity shows the super Arrhenius kind of behavior, we try to fit the readings to the Vogel Fulcher-Tammann equation given below

$$\eta = Ae^{-Ea/(T-Tc)} \dots (3)$$

Here T<sub>c</sub> is the critical temperature where the viscosity starts varying. Equation (3) shows that viscosity varies exponentially with the reciprocal of the temperature.

A plot of ln [η] against 1/T-T<sub>c</sub> showing linear graph was made and the value of activation energy, constant, significance of the value, correlation coefficient are calculated. Table 2 gives the value of the desired parameter and it is observed the fitting using least square is more accurate for equation (3) compared to equation (2) as the correlation coefficient is little high, there is no statistically significant (p) difference in the analysis and the value of standard error estimate (SEE) is less.

Table 2. Exponential (Vogel Fulcher-Tammann Model) function

Oil sample	R <sup>2</sup>	Significance	Constants		SEE
			A (m <sup>2</sup> /s)	E <sub>a</sub> kJ/mol	
Olive oil	0.993	0.000	58.82	18.98	0.205
Olive+10%Sun	0.992	0.000	86.01	19.62	0.282
Olive+20%Sun	0.987	0.000	83.47	19.36	0.294
Olive+10%Coco	0.993	0.000	106.11	20.26	0.296
Olive +20%Coco	0.982	0.000	113.81	20.88	0.242

Table 3. Andrade's equation 1

Oil sample	R <sup>2</sup>	Significance	Constants			SEE
			A	B x 10 <sup>3</sup>	C x 10 <sup>6</sup>	
Olive oil	0.997	0.000	1.428	-0.110	-0.453	0.027
Olive+10%Sun	0.999	0.000	0.912	-3.739	1.062	0.024
Olive+20%Sun	0.994	0.001	3.838	-5.182	1.295	0.024
Olive+10%Coco	0.996	0.000	2.791	-8.614	0.945	0.025
Olive +20%Coco	0.995	0.000	1.451	-9.483	2.016	0.023

Table 4. Andrade's equation 2

Oil sample	R <sup>2</sup>	Significance	Constants			SEE
			A	B x 10 <sup>3</sup>	C x 10 <sup>-6</sup>	
Olive oil	0.994	0.000	9.139	-0.093	100.00	0.041
Olive+10%Sun	0.995	0.000	15.430	-0.131	160.00	0.038
Olive+20%Sun	0.991	0.000	16.944	-0.149	170.00	0.027
Olive+10%Coco	0.998	0.000	12.531	-0.021	7.26	0.036
Olive +20%Coco	0.995	0.000	15.493	-0.191	248.26	0.025

### (3) Andrade's equation 1

Using the natural logarithmic format and higher-order polynomial in 1/T to give better accuracy, the modified Andrade's equation is given below. It is the equation that relates the variation of viscosity with temperature (Abramovic *et al.*, 1998).

$$\ln [\eta] = A + B/T + C/T^2 \dots (4)$$

Table 3 shows the calculated constants A, B and C. The correlation coefficient R<sup>2</sup> is found to be better fitting compared to Arrhenius and VFT equations as the value is very closer to 1. Hence the three parameter equations seem to show a better fit than the two parameter equations with a small value of standard errors and deviations. The results also indicate that significant (p) difference in the statistical analysis is p < 0.001.

### (4) Andrade's equation 2

$$\ln [\eta] = A + BT + CT^2 \dots (5)$$

The natural logarithmic value of kinematic viscosity is related to the square of temperature. To compute this equation a second order regression is used and the constants are calculated (Daniel *et al.*, 2000). Researchers had used this equation when they related viscosity and triglycerides to find the number of carbon atoms in the fatty acids.

From Table 1, 2, 3 and 4 we can see that the

Table 5. Variation of parameter (ρ), (η), (ν), (Z), (L) and (β) of unheated olive oil and mixture of olive and coconut oil, olive and sunflower oil

Sample	Density (ρ) kg/m <sup>3</sup>	Viscosity (η) 10 <sup>-6</sup> m <sup>2</sup> /s	Ultrasonic velocity (ν) m/s	Acoustic impedance (Z) 10 <sup>6</sup> kg/m <sup>2</sup> s	Intermolecular free length (L) x 10 <sup>-10</sup> m	Adiabatic Compressibility (β) N <sup>-1</sup> m <sup>2</sup> 10 <sup>-10</sup>
Olive	830	68.13	1427	1.184	1.041	5.925
O+20sun	864	41.59	1422	1.229	1.024	5.724
O+40sun	892	60.63	1440	1.284	0.995	5.406
O+20coco	694	61.69	1417	0.983	1.146	7.176
O+40coco	682	60.37	1415	0.966	1.156	7.302

Table 6. Variation of parameter (ρ), (η), (ν), (Z), (L) and (β) of heated olive oil and mixture of olive and coconut oil, olive and sunflower oil

Sample	Density (ρ) kg/m <sup>3</sup>	Viscosity (η) 10 <sup>-6</sup> m <sup>2</sup> /s	Ultrasonic velocity (ν) m/s	Acoustic impedance (Z) 10 <sup>6</sup> kg/m <sup>2</sup> s	Intermolecular free length (L) x 10 <sup>-10</sup> m	Adiabatic Compressibility (β) N <sup>-1</sup> m <sup>2</sup> 10 <sup>-10</sup>
Olive	922	72.41	1490	1.373	0.4349	4.885
O+20sun	721	64.44	1432	1.432	0.4458	6.764
O+40sun	703	62.93	1420	0.998	0.4583	7.054
O+20coco	958	54.62	1485	1.423	0.4170	4.733
O+40coco	977	61.05	1517	1.482	0.4447	4.448

empirical relations which give the best prediction in the present study for the variation of viscosity with temperature and the adulteration in olive oil. Equations (2) and (3) are less suitable for description of the temperature dependence of oil viscosity, also the correlation coefficient value is less and the calculated errors are high. Equations (4) and (5) exhibits high correlation coefficient value ~1 and the calculated error are comparatively less.

Ultrasonic wave can be applied to solve problems found in oil related industry. In this respect, ultrasonic velocity has been measured to determine the chemical structure of different oils including chain length and degree of unsaturation. Therefore, velocity measurements can be used to access oil composition and adulteration (Aurel *et al.*, 2007). The change in ultrasonic velocity can also be correlated to viscosity of edible oils. Ultrasonic studies have enormous data in precisely understanding the molecular interactions and structural behavior of for many organic compounds. Table 5 and 6 illustrate the variation of physical parameter like density, viscosity, ultrasonic velocity, acoustic impedance, intermolecular free length and adiabatic compressibility. Increase in viscosity may be due to longer inter chain interaction and larger stiffness of macromolecules (Jose *et al.*, 2007). From the table it is observed that density and viscosity of heated oils (exposed to 210°C temperature for five times) is greater than unheated oils. Heating the oil to smoke point for a number of cycles will increase the saturated fatty acids and triglycerides in oils. This saturated and heavy molecular compound will decrease the fluid nature of the oil and hence the density and viscosity of heated oil increases (Tong *et al.*, 2002).

The ultrasonic velocity in liquid mixtures have been measured using an ultrasonic interferometer working at frequency 2MHz with an overall accuracy

of  $\pm 0.01 \text{ ms}^{-1}$ . The density and viscosity are measured using Pycknometer and Redwood viscometer with an accuracy of  $\pm 0.2 \text{ kg m}^{-3}$ . All the precautions were taken to minimize the possible error. From these data, one can readily determine the velocity of sound in a liquid with high accuracy. Formerly, the absorption of sound in the liquid and the coefficient of reflection at the reflector surface have been obtained through a complicated analysis of the electrical and equivalent-electrical circuits of the quartz crystal and the associated fluid column. The simplified analysis of the equivalent circuit given here is made possible by limiting the discussion to the conditions that exist when all parts of the system (electrical, mechanical, and acoustical) are adjusted to resonance. Under these conditions, the analysis of the complete electrical and equivalent-electrical circuit is greatly simplified.

The ultrasonic velocity in oils depends on the fatty materials. If the molecule of the composition of the oil is not clustered the propagation of ultrasonic wave through the oil is easier hence the ultrasonic velocity in the oil is less (Rubalya *et al.*, 2010). Table 5 and 6 exemplify the change in the ultrasonic velocity through the oil on heating. The fatty acids in oil get saturated by the addition as hydrogenation is one of the chemical reactions that take place when the oil is heated. The saturated hydrogen bonding reduces the velocity of ultrasonic wave. The ultrasonic velocity also varies with addition of coconut and sunflower oil at different proportion which could be used to study the adulteration in olive oil.

Acoustic impedance  $Z = \rho v \text{ kgm}^{-2}\text{s}^{-1}$  is an important quantity which determines how much energy is transmitted and reflected by the interface separating two media of different density. Table 5 and 6 shows that heated oils illustrate more impedance than unheated oils. It is observed that addition of sunflower oil increases the impedance whereas coconut oil exhibit less impedance because coconut oil even though it is composed of high percentage of saturated fatty acids the molecular weight of the short chain fatty acids are less compared to long chain fatty acids of sunflower oil and olive oil. This study can be used not only to evaluate the adulteration but also the structure of the composition in oil (Driss *et al.*, 2009).

One of the important intermolecular properties of a liquid is the free length  $L_f$  between the surfaces of the neighboring molecules and is the distance covered by the propagating acoustic waves between surfaces of the neighboring molecules in the liquid. Jacobson has given an empirical relation between ultrasonic velocity  $v$ , the density  $\rho$  and the intermolecular free length  $L_f$  of a liquid as

$$L_f v \rho^{1/2} = K$$

where  $K$  is a constant, which depends on temperature, Jacobson has given the values of this constant at various temperatures. In terms of the adiabatic compressibility of the liquid, the equation can be written as

$$L_f^2 = K^2 \beta \quad \text{where } L_f = K \sqrt{\beta}$$

$$\text{i.e. } L_f = K \sqrt{1/(v^2 \rho)} \dots (6)$$

where  $K$  is a temperature dependent constant which is equal to  $4.28 \times 10^9$ .

The velocity deviation depends upon the increase or decrease in the free length in the solution after mixing. The reduced free length in oils is due to the mixing of different types of oil with different composition of long and short chain fatty acids, saturated and unsaturated compounds that changes the ultrasound velocity. This indicates that the intermolecular free length is a predominant factor in determining the sound velocity variation in the mixture as well as in the pure oil. In table 5 and 6 the reduction in the free length on the addition of coconut oil shows the molecular weight of the compound in the oil lowers the ultrasonic velocity in the oils.

## Conclusion

Edible oils are characterized in terms of their fatty acids and its flow behavior. The result shows that the oils with more double bonds appeared to have lower viscosity due to their loosely packed structure and the oil exhibit Newtonian behaviour. Also their flow behaviors with temperature could be well characterized by Arrhenius model, Vogel Fulcher-Tammann and Andrade's equations. From the fitting values Andrade's equation, the dependence of viscosity with temperature shows good fitting. Addition to the prediction of viscosity adulteration in the oils can also be traced. The chemical changes (polymerization) are linked to the changes in ultrasonic parameters like ultrasonic velocity, acoustic impedance, intermolecular free length and adiabatic compressibility; hence they could be used to characterize the adulteration, monitor oil quality and thermal degradation in oils. Ultrasonic velocity is related to the changes in physical and chemical properties of composition of oil that determine the quality of olive oil. The feasibility of using viscosity and ultrasonic techniques to evaluate the quality parameters of oils recommends replacing of high cost traditional analytical method with this simple

method.

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