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Dynamic viscosity of olive oil as a function of composition and temperature: A first approach

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Knowledge of the viscosity of virgin olive oils (VOOs) is of great importance for the design of pilot plants, to determine the time required for the settling of particles at the end of the production chain and from a sensory view point. The dynamic viscosities of French VOOs from four different cultivars ('Aglandau', 'Bouteillan', 'Salonenque' and 'Tanche') were studied as a function of their fatty acid and TAG compositions and of the temperature [10–50°C]. These four VOOs had different TAG and fatty acid compositions representative of the range of compositional variations in the main French oils. Their viscosities were similar, although small but measurable differences that depended on their compositions were apparent. All the VOO samples exhibited the same dynamic viscosity pattern over temperature. For a given temperature, the viscosity difference was the greatest between Aglandau and Salonenque oils, Aglandau being the oil with the highest viscosity. The correlation between temperature and viscosity was highlighted by an Arrhenius model for this Newtonian fluid. The Arrhenius activation energy was correlated ($R^2 = 0.993$) with the percentage of triolein, the main TAG in olive oil.

1 Introduction

Virgin olive oil (VOO) is obtained from the fruit of the olive tree by mechanical or physical means – such as washing, crushing, pressing, centrifugation, decantation and filtration – under thermal conditions that do not lead to alterations in the oil [1]. The compounds such as antioxidants and vitamins contained in the olive fruit can still be found in the VOOs, which have a better nutritional value than refined oils.

VOO viscosities are an important consideration in the design of the process pilot units (pumps, decantation and filtration units, etc.).

Whatever the process used (traditional or continuous), the settling stage is essential to remove the solid and liquid particles suspended in the oil. Those particles are often mentioned as one of the main causes of sensorial defects. Their size and the difference between their density and the oil density are key factors that influence the settling velocity. The fluid dynamic viscosity also influences the settling velocity, the latter being inversely proportional to the viscosity. Therefore, a precise knowledge of the viscosities of a monovarietal oils, or even of a multivarietal oils, is a key parameter to optimise the time required for the settling step at the end of the production chain.

Oil filtration (using hydrophilic filters materials, new ceramic membranes, etc. [2–4]) is often required at the end of the production step to ensure that residual muddy sediments have been removed, thereby preventing fermentation. Therefore, the viscosity (and its variations versus

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Abbreviations: OOO, triolein; SFA, saturated fatty acid; VOO, virgin olive oil

temperature) is a very sensitive parameter of the VOO filtration step.

Moreover, olive oil producers and official olive oil tasters in charge of the evaluation the sensory properties of VOOs frequently evoke viscosity differences that derive from differences in the varietal origins. Of course, these remarks are based only on sensory aspects such as the touch or taste in the mouth, and not on actual measurements being made.

Finally, it appears very interesting, from a sensory viewpoint, to find out whether the taster's perception of the 'viscosity' of an oil is truly representative of significant variations of the physical parameters of this oil.

Many studies have been led on the temperature dependence of the viscosity of edible vegetable oils [5, 6]. It is a function of the type of oil (sunflower, olive, corn, rapeseed, etc.), and several models have been proposed to describe its variations. Even if it is not the most accurate, it is commonly admitted that the Arrhenius temperature model, which describes the exponential decrease in the viscosity upon heating, is sufficient for process applications. The parameters of the Arrhenius model depend on the type of vegetable oil.

Valuable relations [5] have been developed between the ratios of MUFA/PUFA and the viscosities in the range 5–95°C for oils from different plants (almond, corn, hazelnut, olive, soybean, etc.). Other original studies such as the one led by Santos et al. [7] provide information on the viscosities of vegetable oils by evaluating the effect of the temperature on the viscosities of un-used and used vegetable cooking oils.

In general, 'olive' oil is one of the vegetable oils tested, but the cultivar of this oil (on which the oil composition depends) is not taken into account to comment on the study results. In fact, there is not 'one' VOO, but 'many' VOOs depending on the cultivar, the soil, the extraction process, etc.

About 200 French varieties of olive trees have been recorded in France. Only 15 of them have an economic importance in terms of olive oil production [8]. Oils from those cultivars have different compositions (in fatty acids, in TAGs, etc.) and different sensory characteristics. In this study, the viscosities of four mono-varietal French VOO cultivars ('Aglandau', 'Bouteillan', 'Salonenque', 'Tanche') were experimentally determined and compared within a temperature range 10–50°C. These cultivars were selected to cover the whole range of possible olive oil compositions in fatty acids and TAGs representative of the French production market [9, 10].

In this study, we will determined specific correlations for VOOs from different cultivars between viscosity and fatty acid and TAG compositions and between viscosity and temperature at atmospheric pressure.

2 Materials and methods

The monovarietal oils came from four major French cultivars [8, 10]. 'Salonenque' and 'Tanche' VOO samples (crop 2007/2008) were obtained from the French Inter-

Professional Olive Oil Association (AFIDOL), Aix-en-Provence, France. 'Aglandau' and 'Bouteillan' VOO samples (crop 2008/2009) were obtained from 'Moulin des Terroirs de Saint Laurent', Beaumont de Pertuis, France.

2.1 Chemical characterisation

2.1.1 Fatty acid determination

VOO in *n*-heptane (0.05 g/mL) was transmethylated with a cold solution of KOH (2 M) according to the European Standard NF EN ISO 5508 Norm (1995) [9, 10]. FAME were analysed according to the European Standard NF EN ISO 5508 (1995). Analyses were performed on a Perkin-Elmer Autosystem 9000 gas chromatograph (GC) equipped with a split/splitless injector ($T = 250^{\circ}\text{C}$) and an FID ($T = 250^{\circ}\text{C}$). A silica capillary column (60 m \times 0.25 mm id, 0.25 μm film thickness) coated with DB WAX (polyethylene glycol, JW) was used. The inlet pressure of hydrogen as carrier gas was 154 kPa with a ratio 1:70. The oven temperature programme was as follows: 13 min at 200°C, from 200 to 230°C at 6°C/min, 17 min at 230°C.

2.1.2 Triacylglycerol determination

The TAGs were analysed using an HPLC system composed of a LaChrom type Merck liquid chromatograph equipped with a Merck Supersphere RP-18 100 column (250 mm \times 4 mm id, 4 μm particle size, temperature 28°C) coupled with a Merck refractometric L-7490 detector [9, 10]. A sample loop of 100 μL capacity was used in which 10 μL were injected. Propionitrile was the mobile phase, with a linear flow gradient of 0.5–1 mL/min for 47 min.

The TAG were designated by letters corresponding to the abbreviated names of the fatty acid carbon chains fixed on the glycerol. The abbreviations of fatty acid names are L for linoleoyl, O for oleoyl, P for palmitoyl and S for stearoyl.

2.2 Viscosity characterisation

The dynamic viscosity was measured using a controlled stress, rotational rheometer coupled with control and data analysis software (Advanced Rheometer 550 from TA Instruments, New Castle, Delaware, USA).

The classical concentric cylinder (rotor radius: 14 mm, stator radius: 15 mm, measuring gap: 1 mm, immersed height: 42 mm) was used. Each measurement on a 20 mL oil sample was done for a continuous ramp from 0 up to 60 Pa. In order to ensure precise and stable control of temperature during measurement ($\pm 0.1^{\circ}\text{C}$), a programmable refrigerating and heating circulator (Julabo FS18 HD, Julabo Labortechnik, Seelbach, Germany) was used. Measurements were carried out between 10 and 50°C at 5°C intervals. All measurements were carried out twice.

Although the solidification temperature of VOO is about 12°C, it is possible to make viscosity measurements at 10°C because the solid liquid phase change phenomenon is very slow at those temperatures (several days). Furthermore, from a regulatory standpoint, the VOO temperature must not exceed 27°C during the production process so that the label carrying the ‘cold pressure’ or ‘cold extraction’ certifier can be put on the bottle [11]. Therefore, for both aspects, pilot design as well as sensory attributes (taste at RT), the measures of interest for VOO producers and users in general are almost exclusively those at temperatures between 10 and 27°C. For these reasons, we chose to analyse data on a slightly wider temperature range, that is, between 10 and 30°C.

3 Results and discussions

The four monovarietal VOOs were chosen according to the differences in their chemical composition. Indeed, the viscosity generally increases with the TAG fatty acid chain length and decreases with the degree of unsaturation [12]. Table 1 gives for the four oils the compositions in saturated fatty acid (SFA), MUFA and PUFA and in the six main TAGs. They were all in the variation ranges published by Ollivier et al. [10, 13]. Table 1 shows that the major differences are for the SFA and MUFA compositions of ‘Salonenque’ and ‘Tanche’, i.e. SFA content: 18 and 12%, respectively, and MUFA content: 69 and 82, respectively. ‘Aglandau’ and ‘Bouteillan’ exhibit intermediate SFA composition (15%). ‘Bouteillan’ and ‘Salonenque’ have the highest PUFA content (13%) and ‘Tanche’ the lowest (6%).

TAGs make up about 98% of VOO. The TAG compositions of the four cultivars showed and confirmed large variations, as did the fatty acid compositions. For instance, the percentage in triolein (OOO) is 30.68% for ‘Salonenque’ and 54.47% for ‘Tanche’ while ‘Aglandau’ and ‘Bouteillan’ exhibit intermediate rate (Table 1).

Figure 1 illustrates shear stress versus shear rate for ‘Aglandau’ at five temperatures. The rheograms obtained for the three other cultivars were very similar. The dynamic viscosity, which is the ratio of shear stress to shear rate, is the

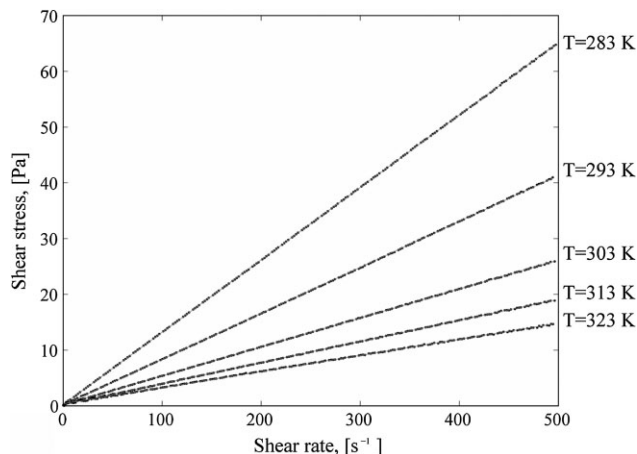


Figure 1. Rheograms for ‘Aglandau’ VOO.

slope of the linear model (Table 2) superimposed on experimental values. Its behaviour is typical of a Newtonian fluid.

Figure 2 illustrates dynamic viscosity as a function of temperature for the four cultivars tested. As expected, a decrease in dynamic viscosity as a function of temperature is observed. All the samples exhibit the same dynamic viscosity pattern over temperature. Although there are significant differences in chemical composition, dynamic viscosities are very close to each other for the four cultivars (deviation <5%).

Various empirical expressions have been proposed relating liquid dynamic viscosity to temperature. The Arrhenius model (Eq. 1) has been chosen to describe the effect of temperature on VOO viscosity:

$$\mu = A \exp\left(\frac{E_a}{RT}\right) \quad (1)$$

where μ is the dynamic viscosity (Pa s), E_a the activation energy (J/mol), R the gas constant (8.314 J/mol/K), T the absolute temperature (K) and A is the pre-exponential factor (Pa s).

Table 1. Fatty acid and main TAG compositions of the four French cultivars

Cultivars samples		Aglandau (%)	Bouteillan (%)	Salonenque (%)	Tanche (%)
Fatty acids	Saturated	15.13	14.98	17.81	11.89
	Monounsaturated	77.18	71.75	68.95	81.63
	Polyunsaturated	7.69	13.27	13.24	6.48
TAGs ^{a)}	OLO	11.57	17.83	17.04	12.03
	PLO	5.13	7.80	9.86	3.20
	OOO	53.91	34.40	30.68	54.47
	POO	22.57	18.48	21.38	17.42
	POP	3.36	3.05	4.00	2.03
	SOO	3.46	3.09	3.25	4.39

^{a)} L, linoleoyl; O, oleoyl; P, palmitoyl; S, stearoyl.

Table 2. Dynamic viscosity and temperature: raw data

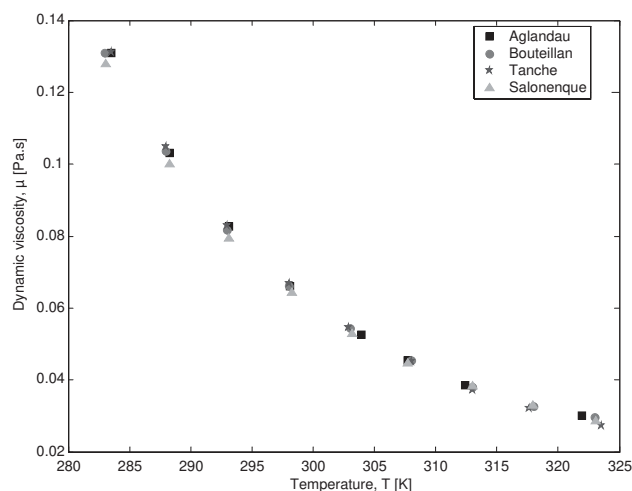
Aglандаu	T (K)	283.5	288.3	293.1	298.1	303.9	307.7	312.4	–	321.9
	μ (Pa s)	1.310E-01	1.031E-01	8.271E-02	6.607E-02	5.248E-02	4.528E-02	3.841E-02	–	3.004E-02
Bouteillan	T (K)	283.0	288.0	293.0	298.0	303.0	308.0	313.0	318.0	323.0
	μ (Pa s)	1.310E-01	1.037E-01	8.169E-02	6.615E-02	5.436E-02	4.533E-02	3.797E-02	3.270E-02	2.946E-02
Tanche	T (K)	283.5	288.0	293.0	298.0	302.9	308.0	313.0	317.6	323.5
	μ (Pa s)	1.311E-01	1.050E-01	8.292E-02	6.673E-02	5.467E-02	4.511E-02	3.731E-02	3.221E-02	2.738E-02
Salonenque	T (K)	283.1	288.3	293.1	298.3	303.2	307.7	313.0	317.9	323.1
	μ (Pa s)	1.280E-01	1.001E-01	7.928E-02	6.441E-02	5.280E-02	4.474E-02	3.817E-02	3.278E-02	2.851E-02

The logarithm of the dynamic viscosity versus the reciprocal temperature over this temperature range is plotted in Fig. 3. The linear relationship confirms that the viscosity behaviour follows the Arrhenius model (Eq. 2).

$$\ln \mu = \underbrace{\frac{E_a}{R}}_a \frac{1}{T} + \underbrace{\ln A}_b \quad (2)$$

The linear model parameters were estimated with the least square method on six different temperature ranges from $283 < T < 298$ to $283 < T < 323$ (Table 3). The activation energy and the pre-exponential factor were calculated from the slopes and intercepts of the Arrhenius plot, respectively. These values and correlation coefficients (R^2) are shown individually in Table 3 for each of the four VOOs. The high value of R^2 on each the six temperature ranges indicates that the Arrhenius model is efficient to describe the raw data even on wider temperature ranges.

The dynamic viscosities calculated (Eq. 1) using the activation energy and the pre-exponential factor estimated on the range $283 < T < 303$ K (Table 3, column 2) were plotted

**Figure 2.** Variation of the dynamic viscosity (μ) as a function of the temperature T for the four VOO samples.

versus temperature (Fig. 4). There is no intersection between the four curves of the Arrhenius temperature model. Even if at a given temperature, the deviations are small ($< 5\%$), the viscosity depends on the composition. The fatty acid and TAG composition of a VOO from a specific cultivar is weakly affected by the olive fruit ripening and by the area of origin. Moreover, the intravarietal variance of the fatty acid and TAG compositions is much smaller than the intervarietal variance [10, 14]. As a result, for a same cultivar, viscosity differences are not measurable. In any case, we will not conclude on the impact of the VOO cultivar on the viscosity but on the impact of the VOO composition.

‘Salonenque’ was the least viscous of the four cultivars. At 293 K, the viscosity of ‘Salonenque’, calculated using Eq. 1, was close to $8.11E-2$ Pa s (activation energy and pre-exponential values gathered in Table 3). In order to obtain the same viscosity for ‘Aglандаu’, the most viscous of the four VOOs, the temperature had to be increased by only 0.8 K. Considering ‘Salonenque’ as reference, the calculation of the deviation ε ($\varepsilon = (\mu - \mu_{\text{Salonenque}}) / (\mu_{\text{Salonenque}})$) shows that the cultivars can be classified by decreasing viscosity, as follows: ‘Aglандаu’, ‘Tanche’, ‘Bouteillan’, ‘Salonenque’ (Fig. 5).

The differences between the E_a and A parameter values for the four oils are small. Poor correlations were obtained between E_a and the MUFA/PUFA rate. However, the activation energy is correlated with the percentage of OOO ($R^2 = 0.993$, Fig. 6, Table 1). A correlation is expected between the main TAG (OOO) component which represents 31–55% of the total TAG composition (Table 1), and the energy activation, as TAGs are the molecules the oil is made of. However, the absence of significant variations in the composition in other TAGs ($\leq 5\%$) prevented us from concluding that there was a correlation between E_a and ‘OLO, PLO, POO, POP, SOO’ TAG compositions.

The dynamic viscosities (Eq. 1), calculated using the activation energy and the pre-exponential factor given by Fasina et al. [5] and by Kim et al. [6] (Table 4) for oils named unaffectedly ‘olive’ oil in those studies, were also plotted versus temperature (Fig. 4). The viscosities in this study were also found between those given by these two different authors. All these olive oils presented significant differences in viscosity for the same temperature (Fig. 4). Depending on the origin of the olive oil, the maximum

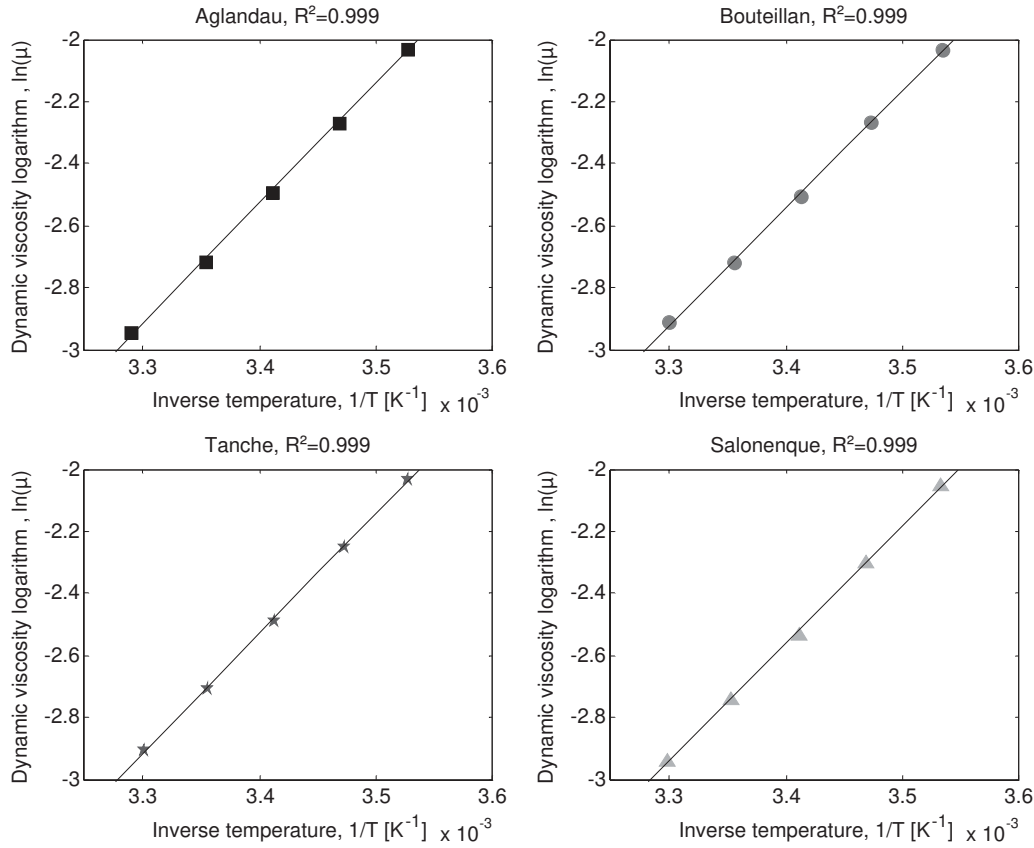


Figure 3. Variation of the dynamic viscosity logarithm ($\ln(\mu)$) as a function of the inverse temperature ($1/T$) for the four VOO samples. (Temperature range = 283–303 K)

Table 3. Coefficients of the Arrhenius model

T range, 283 to ... (K)		298	303	308	313	318	323
Aglandau	R^2	0.999	0.999	0.999	0.999	–	0.994
	a^a	3954	3869	3824	3764	–	3563
	b^a	–15.98	–15.68	–15.53	–15.32	–	–14.64
	E_a^a (J/mol)	32 874	32 167	31 793	31 294	–	29 623
	A^a (Pa s)	1.15E-07	1.55E-07	1.80E-07	2.22E-07	–	4.38E-07
	Bouteillan	R^2	0.999	0.999	0.999	0.999	0.998
a^a		3860	3790	3717	3661	3586	3467
b^a		–15.67	–15.43	–15.17	–14.98	–14.73	–14.32
E_a^a (J/mol)		32 092	31 510	30 903	30 438	29 814	28 825
A^a (Pa s)		1.57E-07	1.99E-07	2.58E-07	3.12E-07	4.01E-07	6.04E-07
Tanche		R^2	0.999	0.999	0.999	0.999	0.999
	a^a	3937	3874	3807	3768	3711	3622
	b^a	–15.92	–15.70	–15.47	–15.34	–15.14	–14.84
	E_a^a (J/mol)	32 732	32 208	31 651	31 327	30 853	30 113
	A^a (Pa s)	1.22E-07	1.52E-07	1.91E-07	2.18E-07	2.66E-07	3.59E-07
	Salonenque	R^2	0.999	0.999	0.999	0.997	0.997
a^a		3843	3785	3727	3617	3532	3443
b^a		–15.63	–15.43	–15.23	–14.85	–14.56	–14.26
E_a^a (J/mol)		31 951	31 468	30 986	30 072	29 365	28 625
A^a (Pa s)		1.63E-07	1.99E-07	2.43E-07	3.55E-07	4.75E-07	6.41E-07

^{a)} Equation 2 $\ln \mu = \underbrace{\frac{E_a}{R}}_a \frac{1}{T} + \underbrace{\ln A}_b$.

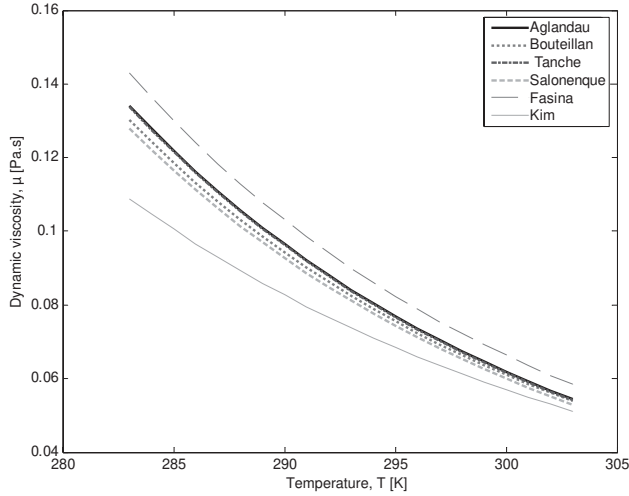


Figure 4. Variation of the dynamic viscosity (μ) as a function of temperature (T) for the four mono varietal VOO samples. Comparison with Fasina et al. [5] and Kim et al. [6] results.

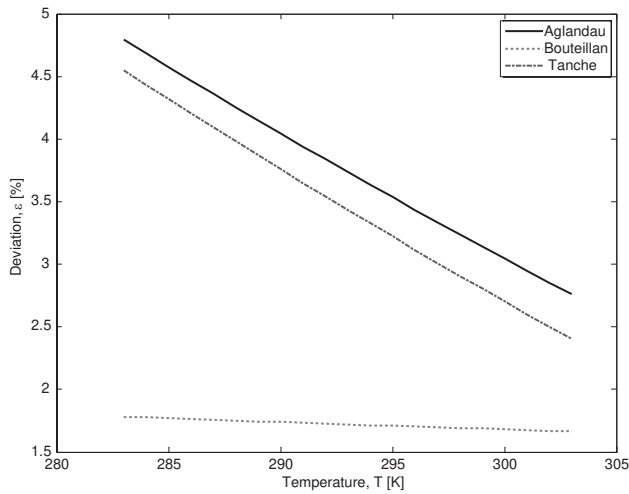


Figure 5. Dynamic viscosity deviation ε in % ($\varepsilon = \mu - \mu_{\text{Salonenque}} / \mu_{\text{Salonenque}}$); ‘Salonenque’ VOO as viscosity reference) as a function of the temperature (T) for the four VOOs.

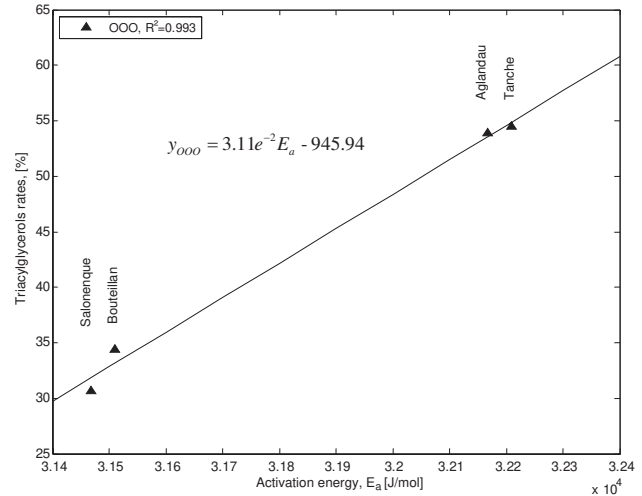


Figure 6. OOO rates in the four cultivars as a function of the activation energy (E_a) (Arrhenius type model).

deviation could reach 22% at 20°C. The compositions in MUFA and PUFA of [5, 6] are given in Table 4 for comparison. Both SFA and (MUFA + PUFA) compositions of the olive oil used by Fasina et al. are in the composition range for the four French VOOs, whereas the olive oil used by Kim et al. has an FA composition outside that composition range. This point may explain the differences highlighted in Fig. 4.

A relationship (Eq. 3) was developed by Fasina et al. [5] that relates viscosity to temperature and FA percentage:

$$\mu = A' \exp\left(\frac{B'}{RT}\right) + y_{FA} \exp\left(\frac{C'}{RT}\right) \quad (3)$$

where y_{FA} (%) is the percentage by weight (MUFA or PUFA) of total FA. A' , B' and C' are constants given in Table 4. This correlation was established using 12 different vegetable oils (almond, corn, sesame, sunflower, etc.), including olive oil. To test the predictive capacity of this model, viscosities

Table 4. Literature data

		Kim et al.		Fasina et al.		
Fatty acid composition		%	%	$A'^{(a)}$	$B'^{(a)}$	$C'^{(a)}$
	Saturated	21.9	16.88			
	Unsaturated	78.1	83.12			
	Monounsaturated	69.9	76.06	3.31E-05	3.55E + 04	5.17E + 03
	Polyunsaturated	8.2	7.06	2.31E-04	3.12E + 04	-1.55E + 04
A^b (Pa s)		1.18E-06	1.85E-07			
E_a^b (J/mol)		26 900	31 900			

^{a)} Equation 3.

^{b)} Equations 1, 2.

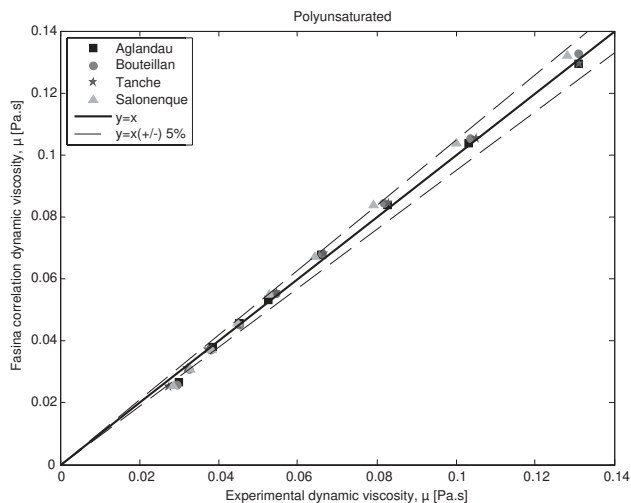


Figure 7. Comparison between experimental and calculated dynamic viscosities using Fasina [5] correlation with Monounsaturated (a) or Polyunsaturated (b) parameters.

calculated using (2) are compared to raw data on Fig. 7. The small deviations between the points and the first bisector indicate that French VOO viscosities could be well estimated by this correlation, with an accuracy of 5–10% using polyunsaturated or monounsaturated coefficients, respectively. This information is sufficient for many applications such as equipment design and storing and handling process. However, the same approach using Kim’s data shows significant differences (more than 20%).

4 Conclusions

A first approach of the effects of TAG compositions and temperature on the viscosity of VOO has been presented. As expected, for a given temperature, the dynamic viscosities measured were different depending on the composition. However, the deviations between the four VOOs tested were small (smaller than 5%). These deviations are not adequate to conclude on the sensory feeling in relation with viscosity. Knowing the PUFA/MUFA composition, VOO viscosity could be well estimated using the correlation that was established for edible vegetable oils. The accuracy obtained is satisfactory to provide the data that are required in engineering process calculations (± 5 –10%). However, PUFA and MUFA are not the constituents of the oil. As a result, this correlation limits the possible analysis of the mechanism that governs the viscosity dependence. The Arrhenius model describes the effect of temperature on viscosity. The energy activation (E_a) is highly correlated with the rate of OOO, the main TAG component in olive oil.

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The authors have declared no conflict of interest.

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