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Determination of physicochemical properties of diacylglycerol oil at high pressure by means of ultrasonic methods

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ABSTRACT

The purpose of the paper is to address, using ultrasonic methods, the impact of temperature and pressure on the physicochemical properties of liquids on the example of diacylglycerol (DAG) oil. The paper presents measurements of sound velocity, density and volume of DAG oil sample in the pressure range from atmospheric pressure up to 0.6 GPa and at temperatures ranging from 20 to 50 °C.

Sound speed measurements were performed in an ultrasonic setup with a DAG oil sample located in the high-pressure chamber. An ultrasonic method that uses cross-correlation method to determine the time-of-flight of the ultrasonic pulses through the liquid was employed to measure the sound velocity in DAG oil. This method is fast and reliable tool for measuring sound velocity. The DAG oil density at high pressure was determined from the monitoring of sample volume change. The adiabatic compressibility and isothermal compressibility have been calculated on the basis of experimental data. Discontinuities in isotherms of the sound speed versus pressure point to the existence of phase transitions in DAG oil. The ultrasonic method presented in this study can be applied to investigate the physicochemical parameters of other liquids not only edible oils.

1. Introduction

Development and design of products and processes for new industrial applications in chemical, food and petroleum industries require knowledge of the physicochemical properties of liquids as a function of temperature and pressure.

High-pressure research of the physicochemical properties of food products has been stimulated by the rapid development of a new technology of food processing, i.e., high-pressure food processing and preservation. To optimize the parameters of high-pressure technological processes (HPP), precise knowledge of the physicochemical parameters of food is indispensable. Unfortunately, there is a lack of physicochemical parameters of food under high-pressure conditions and for various values of temperature. Knowledge of high-pressure physicochemical properties of fuels and biofuels is also essential due to the increasing operating pressure in modern fuel injection systems.

Ultrasonic methods due to their simplicity and accuracy are the most commonly used in the investigation of liquids. Furthermore, the speed of sound is currently considered one of the most useful properties due to its relationship with certain physicochemical properties. Direct measurements of physicochemical properties are very difficult to carry out accurately. In this regard, measuring the speed of sound in liquids under high pressure, provides a relatively easy and accurate manner to obtain isothermal and adiabatic compressibility and other fundamental thermodynamic parameters of liquids.

The aim of this paper is to determine the high-pressure behavior of the physicochemical parameters (such as e.g., isothermal and adiabatic compressibility) of liquids on the example of diacylglycerol (DAG) oil. To this end, ultrasonic velocity and density measurements of DAG oil over the range of pressures and temperatures have been employed.

Diacylglycerol (DAG) is very ubiquitous fat in the food industry and as such is indispensable for good condition of all living organisms. Fat metabolism in the human body associated with the consumption of DAG is more efficient then the metabolism associated with triacylglycerides (TAG) [1–3]. Vegetable oils are currently important not only as foodstuffs but also as a basic ingredient of biofuels (i.e., biodiesels).

Understanding the thermodynamic and rheological properties of liquids is important for the researchers of food [4–10]. The sound velocity technique seems to be very convenient tool for

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the investigation of such properties of liquids. This knowledge can also be very useful in the petroleum industry and in the rapidly developing high-pressure food preservation industry.

Investigation of high-pressure phase transitions in liquids using existing until now classical methods (e.g., optical ones) is difficult. Therefore, for this purpose we applied the ultrasonic method and the measuring setup, that are very convenient to measure the sound velocity at high pressures [11,12]. Discontinuities in the measured isotherms of sound speed versus pressure in DAG oil, indicate the presence of liquid to solid-like phase transformations. Similar phase transitions were also observed in triglycerides and other edible oils [13–20].

On the basis of experimental results (speed of sound and density versus pressure and temperature), isotherms of adiabatic and isothermal compressibility of DAG oil were evaluated as a function of pressure. This work can be extended to other liquids, in order to investigate the temperature effect on their physicochemical properties under high pressure.

The results of this study can be applied in mathematical modeling and optimization of new technological methods of high-pressure food processing and preservation, as well as to model the new fuel injection systems in diesel or biodiesel engines.

According to the authors' best knowledge, the results presented in this paper are a novelty and have not been reported in the scientific literature.

2. Materials and methods

In this paper we measured the time of flight of the longitudinal ultrasonic wave between the transmitting transducer and receiving transducer. In this way, we have determined the speed of sound in the investigated DAG oil sample as a function of pressure for various temperatures. From the change in position of the piston in the high pressure chamber, sample volume changes were evaluated. On this basis, knowing the density of the sample measured by pycnometer at atmospheric pressure, we determined the changes in density of the sample as a function of pressure for various temperatures. Using the measured values of density and sound velocity, we determined changes in the isothermal compressibility and the adiabatic compressibility as a function of pressure for various values of temperatures employing analytical expressions specified in Section 3.

2.1. Sample

DAG oil sample was diacylglycerol oil that is composed of 82% of DAGs (57,4% of sn-1,3 and 24,6% of sn-1,2(2,3)) and 18% of TAGs [21]. The fractions were determined by means of the gas chromatography (GC) method using a Hewlett Packard HP6890 device. The analysis was conducted following the AOCS Cd 11b-91 method. The composition of fatty acid was obtained by the use of a Hewlett Packard HP 6890 device. The analysis was performed according to ISO 5508 and ISO 5509 norms, whereas the data were analyzed with ChemStation A 03.34 software.

2.2. Ultrasonic measurements

Velocity of sound in liquids in high-pressure condition and at various temperatures was measured in the cylindrical high-pressure chamber that constitutes an essential part of a computerized ultrasonic measuring setup, see Fig. 1.

The pressure inside the chamber was measured by the manganin resistive sensor. Ultrasonic pulses in the investigated sample were generated and received by means of piezoelectric transducers. The piezoelectric transducers were manufactured from $LiNbO_3$ (Lithium niobate Y36 cut) (Boston Piezo-Optics Inc., USA), with a diameter equal to 5 mm and 5 MHz fundamental frequency. The manganin sensor and the piezoelectric transducers (that have been placed inside the chamber) were connected to an external PC computer via a multi-channel electrical lead-through.

The high pressure chamber has a capacity of 22 cm^3. Computerized ultrasonic setup was designed and constructed in the Institute of Fundamental Technological Research, Polish Academy of Sciences. The pressure sensor was calibrated by means of a standard manometer with the measurement range up to 600 MPa [22,23]. The calibration process allows to measure pressure by means of resistance changes with accuracy of 0.1 MPa. T type thermocouple was used as a temperature sensor. The increase in pressurization was stopped whenever phase transitions had begun. After the phase transition termination, when thermodynamic properties were nearly at equilibrium (at high-pressure phase of DAG oil), pressurization was continued with the same compression rate as before.

The ultrasonic setup construction provides a low level of parasitic ultrasonic signals. The sending transducer was driven by the TB-1000 (Matec, USA) pulser–receiver computer card. The pulser generated the radio frequency one-cycle tone burst with a frequency of 5 MHz and a length 0.3 μs. The longitudinal wave pulse was generated by the sending transducer and propagated in the measured sample and subsequently was detected by the receiving transducer. The PDA-1000 (Signatec, USA) digitizer card sampled and digitized the signals received by the transducer and amplified by the receiver. For each measurement, the ultrasonic signal was averaged 1024 times in order to improve the signal-to-noise ratio. The time-of-light (TOF) is the time for the signal to travel from the sending transducer to the receiving transducer. The TOF of the ultrasonic pulses through the sample was evaluated by applying the cross-correlation method.

2.3. Ultrasonic wave velocity measurement by means of the cross-correlation method

The velocity c of ultrasonic waves propagating in a liquid along the path L is given by $c = L/t_d$: where: t_d is the TOF along the path L. The path length L is assumed to be a straight line, between two ultrasonic transducers (sending and receiving ones), immersed in a measured liquid and working in a through-transmission mode, Fig. 1. Since our measurements are always differential (time difference) all extra delays in cables, electronics, etc. will be cancelled. A measurement of the time difference between those two ultrasonic pulses and travel distance yields the sound speed in an investigated liquid.

Sound speed measurement is difficult and uncertain when using classical physical methods. To measure the TOF we employed the cross-correlation method [24,25] that improves considerably the accuracy of sound speed evaluation. The cross-correlation function $h(t)$, between two functions $f(t)$ and $g(t)$, is defined by Eq. (1):

$$h(t) = \int_{-\infty}^{+\infty} f(\tau)g(t+\tau)d\tau \tag{1}$$

The first received signal (the function $f(t)$) corresponds to the ultrasonic pulse, that passes the distance L between the transmitting and receiving transducers. Part of the ultrasonic energy of the first signal is reflected from the receiving transducer back to the transmitting transducer, which in turn reflects a part of the incident energy back to the receiving transducer. As a consequence, the next pulse detected by the receiving transducer (function $g(t)$) will pass an extra distance $2L$ between the transducers. In total, this signal passes the distance $3L$. The analysis of correlation

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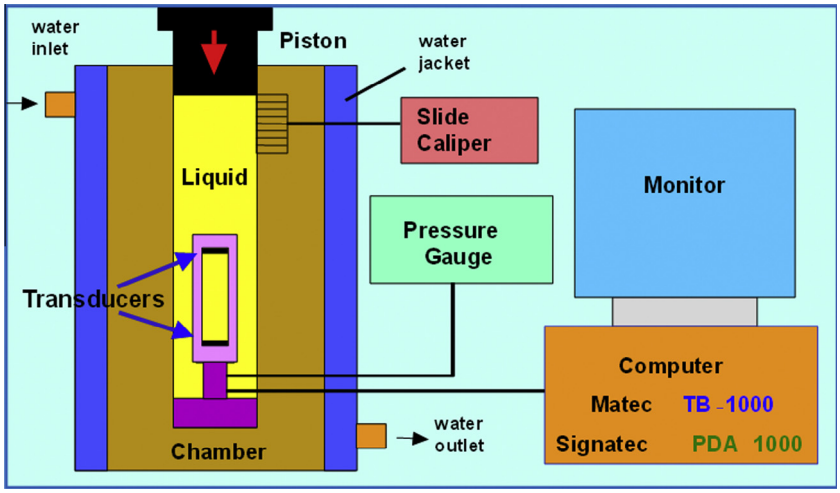

Fig. 1. Computerized ultrasonic setup for measuring the sound velocity in liquids at high pressure and various values of temperature [12,19]. Temperature of water is stabilized by refrigerated/heating circulator (Julabo Labortechnik, Germany), not presented in the graph.

provides a measure of similarity between the two considered pulses $f(t)$ and $g(t)$ shifted in time. Since these two pulses are of similar shape but different amplitude and delay, the cross-correlation function reaches its maximum at t equal to the evaluated time difference corresponding to the distance $2L$. The time delay was measured with uncertainty ±1 ns.

The distance between the transducers was determined from the calibration measurements in distilled water as the reference liquid. Knowing, with high accuracy the speed of sound in water at a given temperature (NIST data [26]) and measuring the time-of-flight, the distance between the transducers was calculated. At ambient temperature, the distance $L = 10.278$ mm. Distance L was evaluated with the uncertainty of ±10 μm.

2.4. Uncertainty analysis

According to the ISO guidelines [27] the expanded relative uncertainty of the ultrasonic velocity $\Delta U_c/U_c$, can be expressed as:

$$\frac{\Delta U_c}{U_c} = 2\sqrt{\left(\frac{\Delta U_L}{U_L}\right)^2 + \left(\frac{\Delta U_{td}}{U_{td}}\right)^2} \tag{2}$$

where: $\Delta U_L/U_L$ is the relative standard uncertainty of the path L, $\Delta U_{td}/U_{td}$ is the relative standard uncertainty of the TOF delay t_d.

The time delay was measured with a picoseconds resolution. However, due to extra systematic errors, such as diffraction contribution, the resulting relative standard uncertainty $\Delta U_{td}/U_{td}$ for the TOF delay t_d was estimated as: $\Delta U_{td}/U_{td} = \pm 0.1\%$. Similarly, the relative standard uncertainty of the path L (resulting from calibration measurements in water) was estimated as: $\Delta U_L/U_L = \pm 0.1\%$. Consequently, the expanded relative uncertainty for the sound speed in liquid equals ±0.3% at a 95% confidence level.

The main source of error in determining the volume was piston displacement measurement error. Piston displacement was measured using a digital caliper with an uncertainty of ±0.01 mm. This puts volume measurement relative standard uncertainty ±0.03%. The expanded relative uncertainty of the density is ±0.05%.

2.5. Volume change

Changes in the sample volume were evaluated by measuring displacement of the piston in the chamber by using a digital caliper of 0.01 mm resolution. Corrections related to the expansion of the chamber were evaluated from the Lame equations and taken into account during the data analysis. All experiments were carried out at temperatures ranging from 20 to 50 °C.

2.6. Density measurements

The density of the DAG oil sample at atmospheric pressure was determined by means of Jaulmes pycnometer in the temperature range from 20 to 50 °C. The measurements were carried out according to ISO-6883 norm (Animal and vegetable fats and oils – Determination of conventional mass per volume). Oil sample was prepared according to ISO 661 norm. DAG oil density values under high pressure (up to 600 MPa) were evaluated by measuring changes in the volume of the sample, i.e., $\rho(p,T) = m/V(p,T)$, where ρ is the density of the sample, m is the mass of the sample, and $V(p,T)$ is the sample volume at a pressure p and temperature T.

3. Results and discussion

3.1. Sound velocity

Changes in the sound velocity in the DAG oil versus pressure are presented in Fig. 2. The experiment has shown that the sound velocity augments piecewise monotonically with increasing pressure at various values of temperature. Three different parts of each curve can be seen, i.e., the low-pressure phase part (1), the phase transition part (2) and the high-pressure phase part (3), respectively.

Discontinuities in the plot indicate the existence of the high-pressure phase transition. At the beginning of the phase transition, spontaneous increase in the speed of sound at all examined temperatures was noticed, despite a drop of pressure. In the experiments the pressure was elevated by (10.0 ± 0.1 MPa) steps and stopped to attain thermodynamic equilibrium conditions. Such phase transitions were also observed in the previously studied TAGs [18] at a similar range of pressures.

At each measuring temperature, the phase transition has been noticed within applied pressure range. Pressure values when the transition begins at different temperatures are presented in the Fig. 3.

At a pressure of 600 MPa, speed of sound is almost doubled compared to its value at atmospheric pressure. The speed of sound in the DAG oil at atmospheric conditions is typical for the liquid state (e.g., in water – 1500 m/s), and after a phase transition at 500 MPa, in the high pressure solid-like phase, is raised to

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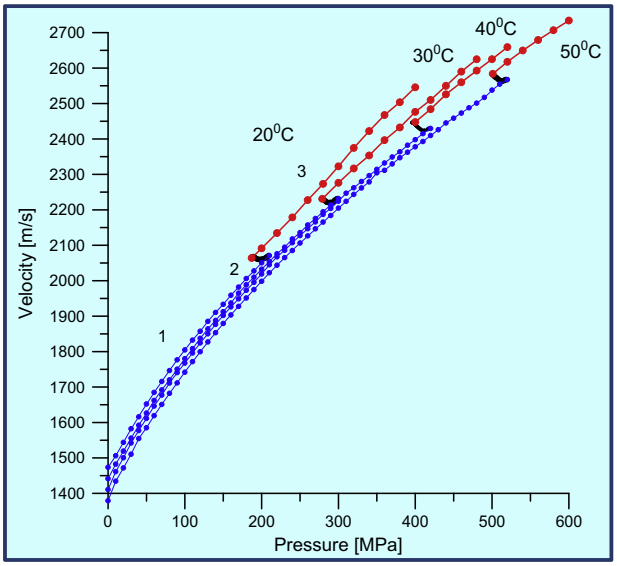

Fig. 2. Plots of sound velocity in DAG oil as a function of pressure along various isotherms (T = 20, 30, 40 and 50 °C), f = 5 MHz. (1) refers to low-pressure phase, (2) indicates the phase transition, and (3) applies to high-pressure phase.

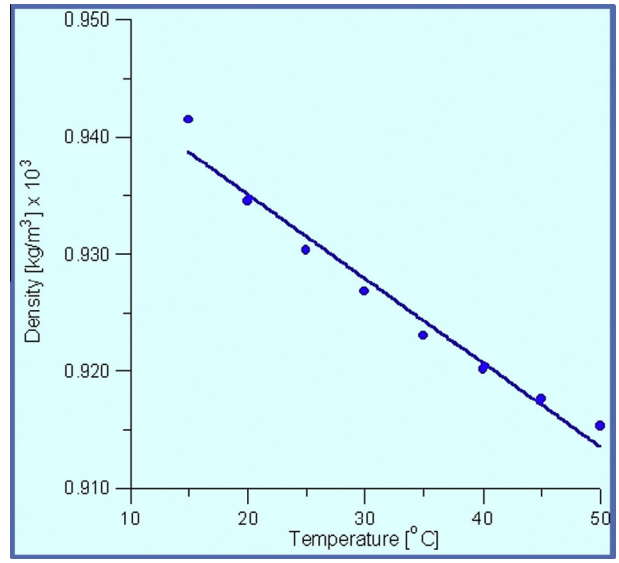

Fig. 4. Density of DAG oil versus temperature at atmospheric pressure.

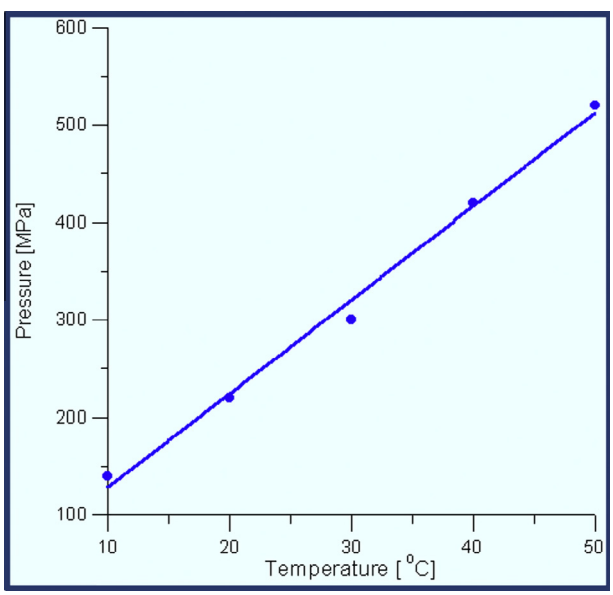

Fig. 3. Pressure values in which the phase transition in DAG oil begins at various temperatures.

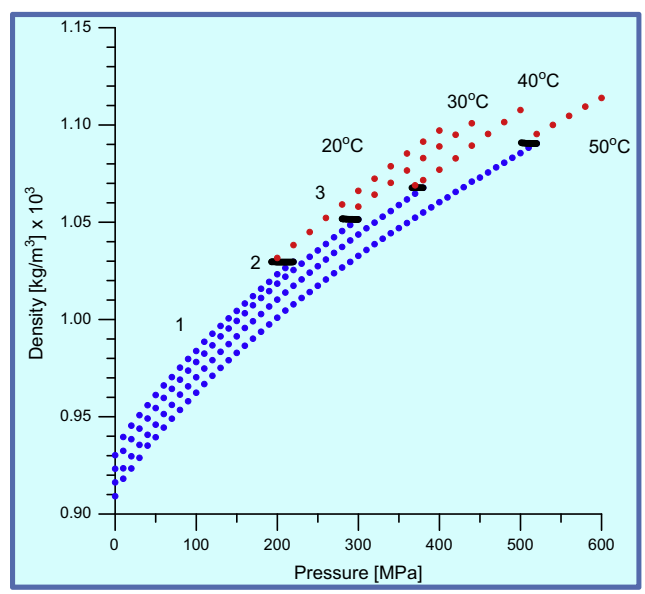

Fig. 5. Density of DAG oil versus pressure for various temperatures. (1) Refers to low-pressure phase, (2) indicates the phase transition, and (3) applies to high-pressure phase.

2600 m/s, that is similar to the sound speed value for solids (e.g., in ebonite – 2400 m/s), [28,29]. The results confirm the existence of solidified phases after phase transition.

3.2. Density

Initial density at atmospheric pressure for various values of temperatures was measured, see Fig. 4.

The DAG oil density under high pressure has been determined from monitoring changes in sample volume. The change in volume under high pressure has been assessed by measuring the displacement of the piston in the chamber by means of a digital caliper gauge. At all temperatures under consideration, there was a similar behavior of volume changes versus pressure. Significant reduction in volume was observed after phase transition at all measuring temperatures.

Initial density versus temperature (measured at atmospheric pressure) as well as change in volume versus pressure at various temperatures were used to calculate the isotherms of the density of DAG oil versus pressure, see Fig. 5.

3.3. Isothermal compressibility

Subsequent parameter which was calculated based on the measured sound velocity and changes in DAG oil volume was isothermal compressibility. Isothermal compressibility of DAG oil evaluated with Eq. (3) diminishes with increasing pressure, see Fig. 6.

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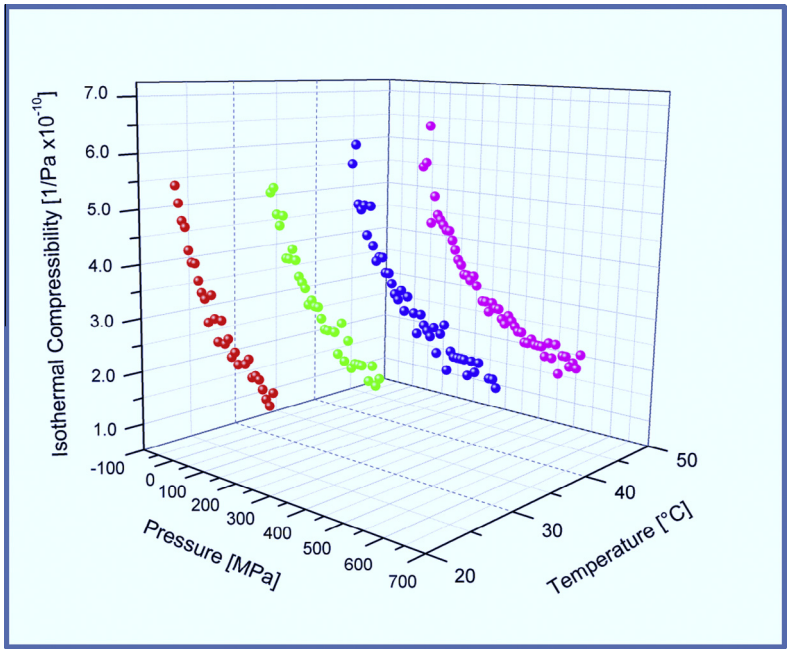

Fig. 6. Isothermal compressibility of DAG oil as a function of two variables – pressure and temperature.

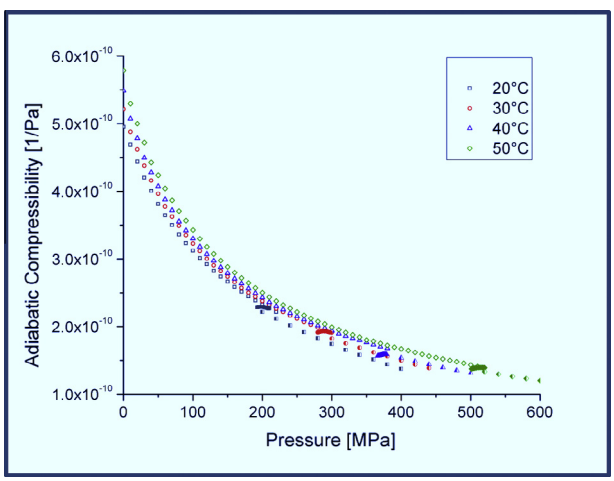

Fig. 7. Adiabatic compressibility of DAG oil versus pressure at various temperatures. Unfilled symbols denote low-pressure phase, entirely filled symbols represent the phase transformation, and semi-filled symbols represent the high-pressure phase.

The diagrams in Fig. 6 show the isothermal compressibility of DAG oil as a function of pressure for various values of temperature. Isothermal compressibility β_T is defined as follows:

$$\beta_T(p,T) = -\frac{1}{V}\left(\frac{\Delta V}{\Delta p}\right)_T \tag{3}$$

where V is the DAG oil sample volume at a pressure p. T is the temperature in Kelvin.

3.4. Adiabatic compressibility

The elastic properties of liquids can be characterized by bulk modulus K. The bulk modulus K is a measure of the resistance of the liquid subjected to uniform mechanical compression. Bulk modulus is a very important physicochemical parameter of liquids, since it characterizes the behavior of fuels in diesel engine injectors. Using the measured isotherms of sound speed and density, bulk modulus K was evaluated. The bulk modulus is an inverse of the adiabatic compressibility β_s, namely:

$$\beta_s = \frac{1}{K} = \frac{1}{\rho c^2} \tag{4}$$

where ρ is the density of DAG oil, c is the speed of sound in DAG oil.

Using Eq. (4), the evaluated density values and the measured values of the speed of sound, the bulk modulus K was calculated. The K values for the DAG oil (at atmospheric pressure) are similar to those values of bulk modules for other liquids as reported in the literature [30] ($2.2 * 10^9$ Pa for water, and $8.2 * 10^8$ Pa for methanol). The dependence of the bulk modulus K on the pressure is the reciprocal of the pressure dependence of the adiabatic compressibility. For this reason, only the adiabatic compressibility graphs are reported in this paper.

The dependence of the adiabatic compressibility β_s on pressure is presented in Fig. 7. As can be seen, adiabatic compressibility of DAG oil in the low-pressure phase decreases monotonically. The transition into the solid-like state results in a substantial change in compressibility. Upon completion of phase transformation, in the solid-like phase, the adiabatic compressibility is lower than that in the low-pressure phase.

4. Conclusions

The origin of this study was motivated by the lack of sufficient information on physicochemical parameters of liquids under high pressure and at various temperatures. Knowledge of these parameters is necessary in mathematical modeling and optimization of high-pressure technological processes in food, petroleum and chemical industry [31–33].

The fundamental goal of this work is to evaluate the influence of temperature and high pressure on physicochemical properties of liquids. The method used for this purpose is based on ultrasonic velocity measurements under high pressures up to 600 MPa and at various values of temperature. Measurements of sound velocity and density in DAG oil as a function of pressure and temperature enable to evaluate several useful physicochemical parameters,

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such as e.g., adiabatic and isothermal compressibility, whose direct measurement, in particular under high pressure is very difficult.

The application of the ultrasonic wave method enabled both the detection of phase transitions in DAG oil and investigation of their kinetics. The phase transition, that is identified by a spontaneous change in volume, corresponds to the reduction of the molecular oscillation [34]. Measurements of sound velocity and density confirmed the occurrence of pressure induced phase transition previously observed in DAG oil by optical methods [35]. Discontinuities in the curves of speed of sound and density versus pressure indicate a phase transition in the DAG oil and the formation of a new high-pressure phase that exhibit properties of the solid-like medium. During the phase transition, the bulk modulus in DAG oil augments and approaches the values typical for solid-like media.

The results of this work can be utilized in the numerical modeling and optimization of new methods of high-pressure food processing and conservation [36,37]. Presented in the paper, the ultrasonic method can also be used to investigate (at high pressure conditions) the composition, purity, and quality of other liquid foodstuffs (e.g., fruit juices), not only the fats and oils. On the other hand, this ultrasonic method can also be applied in the petroleum industry in designing efficient oil production processes [38]. Moreover, the speed of sound in fuels provides important information about the behavior of fuel injection systems for diesel engines [39]. Bulk modulus (or adiabatic compressibility) changes with pressure and temperature have a significant impact on the operation of fuel injection systems [30]. The study of acoustical parameters of liquids as a function of pressure is of great importance also in tribology [40]. Speed of sound and density measurements can be applied for evaluation of high-pressure physicochemical properties of various liquids, e.g., industrial solvents [41], drilling muds [42], or polymers [33]. The detection of phase transitions is very important in biofuels [32].

To the authors' best knowledge the results presented in this paper are a novelty and have not been reported in the scientific literature. Hitherto, similar measurements of physicochemical properties of liquids have not been performed in such a wide range of pressures.

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