Extraction and Determination of Physico-chemical Properties of Oil from Watermelon Seeds (*Citrullus lanatus* L) to Use in Internal Combustion Engines

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The purpose of this work was to investigate the physicochemical properties of watermelon seeds and oil and to find out if this oil is suitable and compatible with diesel engines. The results showed that the watermelon seeds had the maximum length (9.08 mm), width (5.71mm), thickness (2.0 mm), arithmetic mean diameter (5.59 mm), geometrical mean diameter (4.69 mm), sphericity (51.6%), surface area (69.07), volume 0.17 cm³ and moisture content 5.4%. The oil was liquid at room temperature, with a density and refractive index of 0.945 and 1.4731 respectively acidity value (1.9 mgNaOH/g), free fatty acid (0.95 mgNaOH), iodine value (120 mgI_/100g), saponification value (180 mgKOH/g), antiradical activity (46%), peroxide value (7.5 mEqO_/Kg), induction period (6.2 h), fatty acid: palmitic acid (13.1%), stearic acid (9.5 %), oleic acid (15.2 %) and linoleic acid (61.3%). Straight non food vegetable oils can offer a solution to fossil fuels by a cleaner burning with minimal adaptation of the engine. A single cylinder air cooled diesel engine Ruggerini RY 50 was used to measure emissions of various blends of watermelon oil (WO) and diesel fuel (WO10D90, WO20D80, WO30D70 and WO75D25). The physic-chemical properties of the oil influence the combustion process and emissions leading to the reduction of NO_x and the increase in CO, CO₂ and HC.

Keywords: Emissions, watermelon seeds oil, non food oil

The demand for energy and the instability of the price of petroleum has forced governments and local and global suppliers of energy to find alternative sources of fuels. Biomass is a key source to achieve sustainability by decreasing global warming and reduce the dependence of the fossil fuels. Non food oils are interesting fuels because they can be used in diesel engines for transportation or to produce electricity without or minor modifications of engines. Watermelon (Citrullus lanatus) is classified as a member of Cucurbitaceae family and is a popular species cultivated in Romania in summer periods. This plant prefers warm climate growing conditions and is produced on large scale in S-V region in Dolj County. The watermelon seeds which normally are discarded can be used as raw material for oil production [1]. The advantages of non food vegetable oils compared with other renewable sources are that they can be cultivated anywhere from various oil seed crops with zero lifecycle CO, emission. Vegetable oils can be stored and handled with minimal safety precaution due to the high flash points and low volatility. This potential can be used mainly in agriculture to power and operate farm machinery and equipment [2]. Although vegetable oils have comparable cetane number, energy density and heat of vaporization with diesel fuel the high viscosity and low volatility limits the use in conventional diesel engines [3]. The reactivity of the unburned fuel may lead to carbon deposits in engines, piston ring sticking, gum formation and lubricating oil thickening [4]. Yilmaz and Morton [5] investigated performance and emissions on the two diesel engines fueled with peanut oil, sunflower oil and canola oil at different load and oil preheating temperature. With the exception of preheated peanut oil the brake thermal efficiency of vegetable oils was lower and NOx were higher than diesel fuel. Daho et al. [6] studied the performance and emissions on a 667 cm3 diesel engine with blends of cottonseed oil and diesel and found that brake specific

fuel consumption was 21% lower at full load, CO emissions were higher and NOx were lower than diesel fuel. Rakopoulos et al. [7] on a direct injection diesel generator investigated blends of sunflower oil, cottonseed oil, corn oil and olive oil with diesel and found that brake thermal efficiency of the blends was the same of that with diesel. The smoke emissions were decreased and CO, unburned hydrocarbon and NOx were increased for vegetable oils. Altin et al. [8] studied on a 770 cm³ diesel engine various vegetable oils like cotton, sunflower, rapeseed, corn and opium and found that diesel fuel has the lowest fuel consumption. Shehata and Razek [9] tested in an indirect injection Diesel engine coupled to a hydraulic dynamometer with variable load sunflower oil and found that the fuel consumption and CO emissions increase with 5% and hydrocarbons emissions decreased by 34% with respect to diesel fuel. De Almeida et al. [10] studied on a naturally aspirated 70 kW diesel engine fueled with palm oil specific fuel consumption, exhaust gas temperature and emissions. The palm oil produce more CO and higher exhaust gases temperature and lower NOx emissions than diesel fuel. Thus, the purpose of this work was to investigate the physicochemical properties of watermelon oil and to find out if this oil is suitable and compatible with diesel engines.

Experimental part

Materials and methods

The watermelon (*Citrullus lanatus*) seds were collected from local farmers from Dabuleni area. The seeds were cleaned manually by hand to remove all foreign matter such as dust, dirt, stone pieces and broken seed and washed and air-dried (at 50°C) for 24 h.

Physical properties of watermelon seed

Were selected random 100 seeds watermelon (*Citrullus lanatus*). For each seed, the three principal dimensions

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including length (L), width (W) and thickness (T) were measured using an micrometer. Some dimensional roperties of seeds were calculated based on the length, width and thickness.

The arithmetic mean diameter (Da, mm) and the geometric mean diameter (Dg, mm) of the seeds were calculated using eq. (1) and (2), [11 - 14]:

$$Da = \frac{L + W + T}{3} \tag{1}$$

$$Dg = (LTW)^{\frac{1}{3}} \tag{2}$$

The sphericity of the seeds is determined using eq. (3), [11-14,15]:

Surface area (Sa) and volume (V) of the particle were calculated using eq. (4) and (5) [14]:

$$Sa = \pi (D_g)^2 \tag{4}$$

$$\mathbf{V} = \frac{\pi (Dg)^3}{6} \tag{5}$$

Determination of the density of watermelon seed

True density is the ratio of the mass of the sample to the true volume of the particles ie excluding the volume of any internal pores.

Determination of weight of seeds were done by weighing with an analitic balance

The seed volume was determined using the liquid displacement method [11]. Toluene (C_7H_8) was used in place of water, because it is absorbed by seeds to a lesser extent and its surface tension is low, so that it fills even shallow dips in a seed and its dissolution power is low [16]. A standard pycnometric method was used to determine the volume of weighed samples [11].

The volume (V, in m³) was calculated using eq. (6) [11]:

$$V = \frac{M_{td}}{\rho_{tol}} = \frac{\left(M_{ps} - M_{p}\right) - \left(M_{pts} - M_{t}\right)}{\rho_{tol}}$$
(6)

where: M_{td} is unit mass of seed, M_t is mass of pycnometer filled with toluene; M_t , mass of pycnometer; M_{ps} , mass of pycnometer filled with toluene and sample; M_{ps} , mass of pycnometer and sample; and ρ_t , density of toluene. The true density (ρ_t , in Kg/m³) of melon seed was obtained by the following eq. (7):

$$\rho_{trus} = \frac{M_{ps} - M_p}{V} \tag{7}$$

where: ρ_{true} - true densite, Kg/m³; M_{ps} - mass of pycnometer and simple; M_{p} - mass of pycnometer.

Bulk density, ρ_{b} , is based on the volume occupied by the bulk sample as poured into a container of known volume. The seeds were not compacted in any way. The ratio of the mass and volume was expressed as bulk density.

The porosity was calculated from the measured values of bulk density ($\rho_{\rm b}$) and true density using eq. (8) [11]:

$$\varepsilon = \left(1 - \frac{\rho_b}{\rho_t}\right) 100 \tag{8}$$

Extraction of oil from watermelon seeds

The extraction of oil from watermelon seeds was carried out in a Soxhlet-type device using light petroleum solvent. The extraction time was 6 h. After oil extraction, the excess REV.CHIM.(Bucharest) ◆ 68 ◆ No. 11 ◆ 2017 http://www.r solvent was distilled off reduced vacuum using a rotary evaporator [17-19].

Characterization of oil obtained from watermelon seeds

Determination of specific gravity. The specific gravity was determined at 25 ± 0.5 °C using a 50 mL bottle pycnometer that was washed thoroughly with detergent, water and petroleum ether according to method described in AOAC (2005) [20].

The refraction Index

The refraction index, Ir, is determined with a refractometer and is altered together with the growing of oxidation degree of the oil. A bigger degree of oxidation leads to the growing of refraction index. The establishment of refraction index was done with the aid of Abbe refractometer [21].

Determination of acid value and free fatty acid value The acid value of the oil sample was determined using ASTM method [22] and the free fatty acid calculated.

Twenty five milli liter diethyl ether with 25 mL alcohol and 1 mL phenolphthalein solution (1%) was mixed and carefully neutralized with 0.1 M NaOH. One to ten gram of the oil was dissolved in the mixed neutral solvent and titrated with aqueous 0.1 M NaOH, shaken constantly until a pink color which persists for 15 sec was obtained. The acid value was calculated using eq. 9 [23]:

$$Acid value = \frac{Titre (mL) \times 5.6}{Weight of sample}$$
(9)

The free fatty acid value was calculated from the relation: Free fatty acid = Acid value $\times 0.503$

The iodine value

The iodine value represents the quantity of iodine, expressed into grams, which is added by 100 g of lipids. It is a measure of the unsaturated fatty acids grade which enters in the structure of animal and vegetal lipids. The iodine value was calculated using the Hanus method. The iodine value was calculated according to eq. 10 [21]:

$$I_{i} = \frac{\left(V_{m} - V_{p}\right) \times t \times f \times 100}{m_{p}} \tag{10}$$

where: V_m - volume of the solution of sodium thiosulphate 0.1 N used in the titration of sample control, mL; V_p - volume of the solution of sodium thiosulphate 0.1 N used in the titration of analysed sample, mL.; t - titre of the solution of sodium thiosulphate 0.1 N in relation with the iodine (0.01269 mg/mL); f - factor of the solution of sodium thiosulphate 0.1 N, m_p - mass of the analysed sample.

Saponification index

The saponification index (Is) represents the quantity of potassium hydroxide expressed in miligrams, necessary for the saponification of fatty acids from a gram of fat. The saponification index is calculated using eq. 11 [21].

$$I_s = \frac{\left(V_m - V_p\right) \times t_{KOH}}{m_p} \tag{11}$$

where: t_{KOH} - titre of the solution of KOH = 28.055 mg KOH/mL; m_p - mass of sample submitted to the saponification, g; V_m - volume of the solution of KOH used in the titration of sample control, mL; V_p - volume of the solution of KOH used in the titration of analysed sample, mL.

Antiradical activity of oil. The conventional DPPH method uses methanol as solvent but does not dissolve

oils. Proper organic solvent for both DPPH and oil samples is isooctane [24] Two milliliters of 0.01 mM DPPH in isooctane were mixed with two g oil sample in caped test tube and after 30 min standing in dark, the absorbance of the sample mixture was measured at 517 nm using UV-Vis spectrophotometer Cary -50. Free radical scavenging activity from DPPH method is calculated with the aid of the formula [24, 26]:

$$DPPH_{scavenging \ capacity \ (\%)} = \frac{\left(A_{control} - A_{sample}\right)}{A_{sample}} \times \frac{100}{(12)}$$

where A - absorbance at 517 nm.

Peroxide value

The standard method prescribed by the Association of Official Analytical Chemists was performed to measure the peroxide value. In this procedure, the oil samples weighing 2 g were taken individually in different conical flasks and then a solution of acetic acid and chloroform in the ratio 3:2 is added. Later, the solution of saturated potassium iodide of 0.5 mL is mixed up with samples in every flask. All the flasks are then undisturbed for 5 minutes. Now the distilled water measuring 15 mL is added to each flask and then titrated with a sodium thiosulfate solution of 0.1N until the yellowish color disappears. Finally, 0.5 mL of starch is added and the titration is continued till the end point where the mixture turns colorless. The peroxide values are calculated from the expression [20, 26]:

Peroxide values =
$$\frac{(V_2 - V_1) \times T \times 1000}{m}$$
(13)

where: V₁- volume of 0.1N blank; V₂ - volume of 0.1N $Na_2S_2O_3$; T - normality of $Na_2S_2O_3$ 0.1N); m - mass of oil taken.

Induction period stability through the Hadorn-Zurcher method (Rancimat)

This method consists in the oil oxidation in accelerate conditions. The method permits the establishment of the induction period, which corresponds, with the initiation step of the oil auto-oxidation. To determine the stability in oxidation it was used an installation, which used oxidized oil samples (10 g) at a temperature of 110°C [19, 27-29]. Through the oil samples, it was bubbled air with a debit of 8 L per hour. Because of the oxidation reactions, which take place in a reactor, the formed volatile acids are trained by the air current and absorbed in the measurement cell where there is bidistilled water. The measurement of the solution conductibility is done with a conductometer of Radelkis type. In the beginning, we notice a slow increasing of the solution conductibility, after that it appears a sudden increasing of this because of the formation of volatile acids. The induction period is considered the interval until the moment of the suddenly curve's change.

Fatty acid compositions

The fatty acid compositions were investigated using apparatus Focus GC gas chromatograph coupled with DSQ II quadrupole mass spectrometer.

¹ μL of oil was analyzed on Focus GC gas chromatograph coupled with DSQ II quadrupole mass spectrometer. Column used for analysis was a capillary column, Thermo TR-WaxMS with 0.25 mm in thickness, 0.25 mm ID and 30 m length. The column oven temperature was programmed to 270°C through the following steps: 40 to 100°C at a rate of 5°C×min⁻¹, held at 100°C for 5 min, 100 to 200°C at the rate of $10^{\circ}C \times min^{-1}$, held at 200°C for 7 min and the last steps was 200 to 270°C at the rate of 8°C × min⁻¹. The column was maintained at 270°C for 13 min. During the analysis process the injector temperature was set at 200°C. The interface temperature between GC and MS was kept at 260°C and the ion source was set at 220°C and 70 eV ionization energy of impact ionization was used to fragment the elements from capillary column.

Experimental setup

Engine performance experiments were performed using a four stroke single cylinder type Ruggerini RY 50, air cooled, naturally aspirated, direct injection diesel engine. Further general information about the technical details of the engine can be found in table 1.

Table 1	
ENGINE SPECIFICATIONS	

Manufacturer	Ruggerini
Model	RY 50
Configuration	Single cylinder vertical
Туре	Direct injection diesel
Displacement	224 cc
Bore	69 mm
Stroke	60 mm
Compresion ratio	21:1
Power	3.5 kW
Speed	3600 rpm
Type of cooling	Air cooling
Max. torque	10,4@2400 rpm
Weight	28 kg

The schematic diagram of experimental setup is shown in figure 1.



Fig. 1 Schematic diagram of experimental setup

The engine used for this study has no modifications and is coupled with a generator (Type Ce 160s, 110V, 44A) and the speed of the engine was kept 1000 and 2000 rpm engine speed range at full load condition. The engine was loaded using a series of electrical resistance elements at 20, 40, 50, 60, 80, and 100%. Voltage, current and power factor of the produced electricity was measured by a voltmeter, energy meter and wattmeter. The fuel consumption is measured by an electronic balance that measures gravimetric consumption. Exhaust gases were analyzed with a gas analyzer type Stargas 898 (table 2).

The ambient conditions were monitored by a barometer and a thermometer. The engine ran on neat diesel for at least ten minutes prior to switching to vegetable oil blends to reach the operation temperature. At the end of a specified load test, the engine is allowed to run using petrodiesel fuel without no load to make sure that the fuel system is cleaned from any residuals remained from the previously tested fuel. The blends of watermelon oil and diesel fuel were mixed based on volume basis in WO10D90

Equipment name	Method	Measurement	Range		Resolution	Accuracy
STARGAS 898	Non-dispersive Infrared Spectroscopy	со	0-15 Vol%	0.00 1	010% 10.0115%	±0.002% abs./ ±3% rel. ±5% rel.
	Non-dispersive Infrared Spectroscopy	CO ₂	0-20 Vol%	0.01	0.0016.00 % 16.0120.00%	±0.03% abs./ ±3% rel. ±5% rel.
	Non-dispersive Infrared Spectroscopy	нс	0-30000 ppm	1	02000 ppm 200115000 ppm 1500130000 ppm	±4ppm abs./ ±3% rel. ±5% rel. ±8% rel.
	Electrochemical detection	O ₂	0-25 Vol%	0.01	0.0025%	±0.1% abs./ ±3% rel.
	Electrochemical detection	NO	0-5000 ppm	1	04000 ppm 40015000 ppm	±25ppm abs./ ±5% rel. ±5% rel.

 Table 2

 TECHNICAL SPECIFICATIONS STARGAS 898

(10% watermelon oil and 90% diesel fuel), WO20D80 (20% watermelon oil and 80% diesel fuel), WO30D70 (30% watermelon oil and 70% diesel fuel) and WO75D25 (75% watermelon oil and 25% diesel fuel).

Results and discussions

Physico-chemical properties

The density of the watermelon oil influences the total fuel consumption and brake specific fuel consumption. Also the higher density of the oil affects the injection system (injection timing, spray penetration and spray characteristics). High viscosity of the oil increases the fuel droplet size and affects the atomization property with the increase of the shoot emissions. In general the higher viscosity of the fuel leads to higher NOx production. The combustion behavior and ignition is influenced by the fatty acid composition of the raw material. The nature of fatty acids (palmitic, stearic, oleic and linoleic) affects the tendency to polymerization which may lead to the obstructions of the injector holes. The watermelon oil can be defined as bi-unsaturated (iodine number between 100 and 150). Peroxide value of oil is higher than diesel fuel. The physic-chemical properties of the oil affect atomization, combustion and injection timing in diesel engines.

Engine emissions Carbon monoxide (CO) emissions



Fig. 2. CO emissions for various oil-diesel blends at different load

CO emissions are mainly due to the incomplete combustion of the fuel. The cause is attributed to the poor quality of the combustion process affected by the high viscosity and low volatility of the watermelon oil. Through blending with diesel fuel the viscosity was reduced and the emissions are decreased (fig. 2). Mohanty et al. [30] on a diesel engine using blends of polanga oil and diesel

Seeds		Oil	Diesel fuel standard	
Properties	Value	Properties	Value	-
Moisture content, % (w.b.)	5.4	Refraction Index	1.4731	-
Length, mm	9.08	Acid value, mgKOH/g	1.9	-
Width, mm	5.71	Free fatty acid, mgNaOH/g	0.95	-
Trickness, mm	2.0	Iodine value, mgI2/100g	120	-
Arithmetic mean diameter, mm	5.59	Saponification value, mgKOH/g	180	-
Geometric mean diameter, mm	4.69	Antiradical activity, %	46	-
Sphericity, %	51.6	Peroxide value, mEqO ₂ /Kg	7.5	
Surface area, mm ²	69.07	Induction period, h	6.2	-
Volume, cm ³	0.17	Fatty acid	r I I I	-
True density, Kg/m ³	803	Palmitic acid, %	13.1	-
Bulk density, Kg/m ³	399.8	Stearic acid, %	9.5	-
Porosity, E, %	50.3	Oleic acid, %	15.2	-
Specific gravity	856	Linoleic acid, %	61.3	-
-	-	Density, Kg/m ³	945	820-845
-	-	Viscosity 40°C, mm ² /s	11.6	2-4

 Table 3

 VALUES OF PHYSICO-CHEMICAL ANALYSIS OF WATERMELON SEEDS AND OIL

fuel reported an increase in CO emissions of vegetable oil blends compared to that of diesel fuel. Fontaras et al. [31] tested blends of 10% of three vegetable oils (rapeseed, sunflower and cottonseed) and recorded emissions over NEDC (New European Driving Cycle). An increase of CO and HC emissions were recorded for all blends tested.





Fig. 3. CO₂ emissions for various oil-diesel blends at different load

In the literature are contradicting reports for CO₂ emissions of vegetable oils [33-34]. The quantity of emissions gives information's about the quality of combustion since in complete combustion is a lower quantity of CO and a higher quantity CO₂. Higher values of CO₂ were emitted by watermelon oil blends compared to that of diesel fuel (fig. 3). However the CO₂ released into the atmosphere by the oil is almost null since is compensated by the CO₂ absorbed during the growth of the watermelon. Shehata and Abdel Razek [35] studied in direct injection diesel engine neat sunflower oil and at 1500 rpm and 50% load and found an increase in CO and CO₂ emissions.

Hydrocarbons (HC) emissions





The variation of HC emission of watermelon oil, diesel fuel and blends with respect to engine load are given in Fig. 4. The HC emission presents a similar trend to CO and CO_2 . Due to the high viscosity of the oil and nonhomogeneous combustion conditions the incomplete combustion occurs. In addition the low heat value of watermelon oil may cause the extinction of the flame in various part of the combustion chamber (walls, dead volumes, etc). Devan and Mahalakshmi [36] evaluate emissions and performance of a single cylinder diesel engine fueled with blends of neat poon oil and diesel and found a slight increase in HC and CO emissions. Similar results on increase in HC emissions with pure vegetable oil and their blend with petrol-diesel were reported by various researches [37-40].

Nitrogen Oxide (NOx) emissions



Fig. 5. NOx emissions for various oil-diesel blends at different load

Comparison of NOx emissions for various blends of watermelon oil and diesel fuel is presented in figure 5. The reduced amount of NOx emissions is attributed to the low heating value of oils, lower fuel air mixing rate and worst atomization conditions due to the higher viscosity which reduces the premixed burning rate and reduces the incylinder temperature responsible for high NOx emissions. Mubarak and Kumar [38] found a reduction with 31% for NOx emissions using waste vegetable oil compared to that of diesel fuel. Kawasaki et al. [41] using a direct injection diesel engine operating for 32 h found a higher CO, HC and smoke emissions and lower NOx emissions.

Conclusions

Watermelon seeds which were discarded were used to produce oil. The culture of watermelons is strongly developed in areas with sandy soils like Mehedinµi, Galaµi, Dolj, Braila, Timiº, Arad, Bihor. The physical and chemical characteristics of the oil have a great influence in the performance and emissions of internal combustion engines. A single cylinder diesel engine was used to test various blends of watermelon oil and diesel at 20, 40, 60, 80 and 100 load of the engine. According to engine test results the NO_v decreased and CO, CO_v and HC increased. Further research is necessary to get a better understanding of the influence of the fatty acid composition and physical chemical properties on the combustion and emission behavior of watermelon oils. Preheating the oil can reduce the viscosity and density of the oil closer to diesel fuel improving the atomization in the combustion chamber and reducing emissions.

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Manuscript received: 8.04.2017