DETERMINATION OF THE MAIN PROPERTIES OF GRAPE SEEDS AND IMPROVING THE MECHANICAL OIL EXTRACTION

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In this paper, in the first stage, we determined the main physical, chemical, and mechanical characteristics of grape seeds of two varieties, Riesling and Burgundy. The main parameters observed were: unit mass, dimensions (x, y, z), seed volume, humidity, oil content, and breaking strength. The second part of the paper aimed to improve the mechanical extraction of grape seed oil to increase the efficiency of oil recovery. The experiments were performed with the mechanical screw press Oil Press NF 80, made in Turkey. We created configurations by combining the parameters of two different screws (ring size 16 and 20mm), three different nozzles (nozzle diameter 7, 10, and 12 mm), and three rotational speeds (low, medium, and high). Following the experiments, it was established which of the combinations of variables has the best yield of extracted oil and energy consumption, but also which of the two varieties is the most efficient.

Keywords: grape seed oil, screw press oil, mechanical extraction oil, seed characteristics

1. Introduction

A serious environmental problem is the management and disposal of large amounts of waste produced by the food industry. Against this background, we are constantly looking for solutions for the controlled disposal of waste by transforming it into value-added bioproducts. By solving these problems, new sources of income are created that would increase the profits of the main industrial process, [1, 2].

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In the wine industry, a by-product generated in very large quantities is grape pomace which is composed of seeds, skin, and stalks. Mainly, pomace is used to make alcohol. Research over the last decade has shown that pomace is rich in nutritionally valuable compounds and biologically active compounds. Particular attention was paid to grape seeds rich in oil, phenolic compounds, antioxidants, proteins, fiber, minerals, and tannins, [3, 4].

The factors that influence the content and composition of grape seed oil are many, including the grape variety, climate, conditions of development of the vine, the degree of ripeness of the grapes, and the most important method of extracting the oil. In the literature, data on the amount of oil in grape seeds vary from 6 to 20% depending on the grape variety, [5].

In addition to the composition of healthy and nutritionally valuable fatty acids, grape seed oil is a good source of vitamin E and polyphenolic compounds that ensure the high stability of the oil [6]. The study of several grape seed varieties has shown that V. vinifera seed has the greatest potential for natural antioxidant compounds, [7 - 9]. In grape seed oil, the presence of antioxidants is also essential for the stability of polyunsaturated fatty acids and the prevention of oxidative alteration of the oil, [10 - 12].

The physicochemical characteristics of grape seeds have so far been determined for certain grape varieties. From a chemical point of view, the seeds have mainly the following composition: water (28-40% of the weight of the seed), cellulose (about 28%), oil (10-25%), tannins (4-6%), minerals 2-4%), lignin (25-28%) and other components, [11, 13-15]. From the point of view of physical characteristics, the studies were limited to determining the shape of the seed (most seeds are pear-shaped) and to measuring their dimensions, which vary as follows: total length between 4.7-7.3 mm, width between 3.3-4.5 mm, thickness between 2.4-3.5 mm, [16].

For this work, we have determined the physical properties, and the main chemical and mechanical characteristics of the Riesling and Burgundy seed. Determining these characteristics of the seeds is an important step in understanding their behavior during handling and storage, but also during processing for extraction oil. Also, sometimes needed to condition the seeds before oil extraction. Observing these properties is useful for improving the performance of extraction equipment, intending to obtain a high oil production with the best qualitative value, [17].

The most popular methods for extracting oil are cold pressing with hydraulic or screw presses and solvent extraction. Their influence on the oil refers to the oil yield (by pressing the yield is lower than by solvent extraction) and to the quality of the oil (by solvent extraction, solvent residues remain in the oil, and by cold pressing, the oil contains the highest values of seed compounds), [18].

Several studies have analyzed mechanical oil extraction, mainly for oilseeds with high oil content. In the case of grape seeds, considering that they have a low oil content, has been used solvent extraction, and other chemical and biochemical methods or combined variants these.

In this paper, we chose to use the mechanical extraction of grape seed oil because the quality of the extracted oil is very important, and the factors involved in this process were not studied in this situation for the Riesling and Burgundy varieties. Therefore, in this paper, we have analyzed the behavior of the N80 screw press, by modifying the constructive parameters such as the size of the outlet, the pitch of the auger, and the rotation speed. Do to the result obtained after performing all the experiments, we obtained the combination of parameters with the highest oil yield and the lowest energy consumption.

2. Material and method

For this study, we obtained the pomace from the Riesling and Burgund varieties from INCDBH Ștefănești, Argeș County. The vineyards are found in the southern part of Romania, on plateaus with smooth slopes to the S and SE, at an altitude of 283 m compared to sea level. The specific climate of the area is a temperate continental transition, normal for Central Europe, with four seasons. The average temperatures in summer are between 22-30 ºC, and in winter between -3 and -5 ºC. The average annual rainfall is about 637 mm, according to the NMA (National Meteorological Administration).

The grape seeds were extracted from the pomace at INMA Bucharest following the methodology in Figure 1, which also includes the stage of obtaining the oil. In the stage of preparation of the pomace, the following technological flow followed:

- breaking the pulp bulbs with Detachment equipment;
- drying the pulp with belt drying equipment; The humidity of the pomace at the reception was 55.11% for Riesling and 48.52% for Burgundy, respectively. At the exit of the dryer, the pulp has a humidity of 10.2% for Riesling and 9.14% for Burgund.
- separation of pomace seeds was done with ESSS equipment. The equipment consists of three vibrating sites and has a seed extraction yield of 80% of the total weight of the pomace, [19];
- finally, the extracted seeds are prepared for entering the screw press to extract the oil.

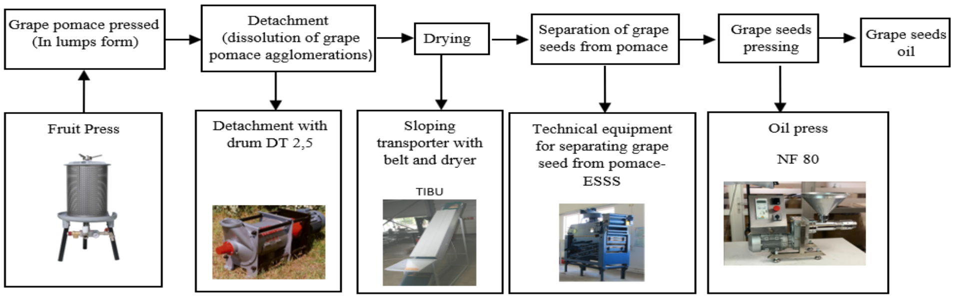

Fig. 1. Technology for grape seed separation, [19]

Seed samples were taken from each seed variety, Riesling and Burgund, one hundred seeds from random places in three tranches, and with an analytical balance (Shimadzu AW220) we recorded the mass of 100 seeds, Figure 2.

Fig. 2. Grape seeds sample

Geometric dimensions length x, width y, and thickness z (Figure 3), were measured with electronic calipers (MAFCOM) with an accuracy of ± 0.01 mm. For each variety, the geometric dimensions were determined by the average of ten measurements.

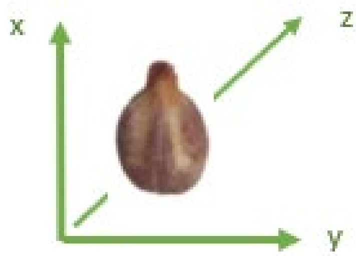

Fig. 3. Three major dimensions of grape seeds were x - length, y – width, and z – breadth.

The unit volume V_u (cm^3) and the approximate surface area S (mm^2) of grape seeds were determined using formulas (1) and (2), [20].

$$V_u = \frac{4}{3}\pi \cdot (x \cdot y \cdot z) \,/\, 1000 \tag{1}$$

$$S = 2\pi \cdot \left(\frac{y}{2}\right)^2 + 2\pi \cdot \frac{x \cdot y}{4c} \cdot sin^{-1} \cdot c \tag{2}$$

Where:

$$c = \left[1 - \left(\frac{y}{x}\right)^2\right]^{1/2} \tag{3}$$

The determination of the moisture content of the seeds was performed according to the procedure SR EN ISO 665: 2003. Each of the five samples contains 2 g of seeds powder, which were dried at a temperature of 103 ºC at atmospheric pressure until a constant weight was obtained. The method according to SR EN ISO 659: 2009 was used to determine the oil content. The grape seeds were ground to obtain 10 g of powder from which the oil was extracted using n-Hexane reagent, and the oil content was expressed as a percentage of the dry mass of the seed powder.

The laboratory compression tests were performed in the laboratory of the ISB Faculty, within the Polytechnic University of Bucharest, using a uniaxial compression tester (Hounsfield, model H1 KS) and the data were purchased and processed by QMAT software. Experiments were performed on ten seeds of each variety, chosen at random. The seeds were placed horizontally between the two plates. The moving speed of the moving plate was 1 mm/min, and the applied force was 1 kN. The results obtained are the value of the breaking force (N), the deformation at the breaking point (mm), the energy at the breaking point (J).

The experiments for extracting the oil were performed with the N80 screw mechanical press, produced in Turkey, located at the National Institute of Research – Development for Machines and Installations Designed for Agriculture and Food Industry – INMA Bucharest. This press is powered by a 0.70 kW electric motor, (Figure 4). The maximum working capacity of this equipment is 12 kg / h.

Fig. 4. Oil screw press, N80

For this paper, a series of parameters related to the construction of the equipment were taken into account in order to increase the extraction performance of the oil from grape seeds, figure 5. These included:

- a cylindrical pressing chamber with mash with size 1mm;
- two screws with step size of 16 and 20 mm, labeled as R8 and R10;
- three different nozzles with diameters of 7, 10 and 12 mm, labeled as N7, N10 and N12.

Fig. 5. Screw press equipment components

In this experiment, three levels of screw rotation speed were chosen; low, medium and high speed, namely 220, 290 and 355 rpm for R8 screw and 115, 180 and 255 rpm for R10 screw. The speed limit range for different screws was determined by preliminary tests. Screw R10 allowed lower speeds to be applied than screw R8 without locking during pressing. Dependent variables, such as oil content, oil recovery, and time required for pressing, were monitored by measuring oil weight.

The efficiency of oil recovery was calculated using formula (4), by dividing the weight of crude oil extracted by the weight of seeds processed in a series, being expressed as a percentage. The values in the calculation were compared with the values obtained by chemical determinations of the oil content of grape seeds.

$$O_\eta = \frac{Q_o}{Q_S} \times 100 \tag{4}$$

where: O_η as oil recovery efficiency in % (w/w); Q_o - quantity of crude oil extracted from grape seeds sample, in g; Q_S - quantity of grape seed sample, in g.

The model of the tests for this work was established below in Table 1. Two sets of experiments related to the two screws with different pitches were performed, for which combinations were made between the rotation speed and the outlet nozzle of the groats, for both varieties of grape seed.

Table 1

The set of experiments for each screw, with the variation between the outlet nozzle and the speed

I. Screw R8	II. Screw R10
N7_ω220 N7_ω290 N7_ω355	N7_ω115 N7_ω180 N7_ω255
N10_ω220 N10_ω290 N10_ω355	N10_ω115 N10_ω180 N10_ω255
N12_ω220 N12_ω290 N12_ω355	N12_ω115 N12_ω180 N12_ω255

In the first set, the 16 mm pitch auger was used, which was kept constant while the outlet nozzle (three nozzles of 7, 10, and 12 mm) and the rotation speed were varied (three rotation speeds were chosen 220, 290 and 355 rpm) resulting in nine sets of tests.

In the second set of experiments the screw's pitch was changed from a pitch of 16 to one of 20 mm and also the exhaust nozzles and rotation speeds were varied between 7, 10 and 12 mm diameter and 115, 180 and 255 rpm speeds.

For each experiment, a batch of 5 ±0.05 kg of grape seeds was used, with the average dimensions determined for each grape variety. Before starting the pressing process, the housing in the compression zone was heated with an electrical device to 90 °C. This prevented the press from jamming at the beginning of the operation. The temperature adjustment was made from the press control panel. Initially about 3 kg of grape seeds were introduced to check that the equipment operates in its optimal parameters. The oil and grit were weighed after each experiment and analyzed in the laboratory. The collected data was analyzed statistically as standard using medium values, and regressions using Excel as a data analysis tool.

3. Results and discussions

The geometric characteristics of the grape seeds for both grape varieties were determined, and the results are shown in Table 2 as averages between the 10 randomly selected seed measurements.

Table 2

Basic geometric characteristics of grape seeds

Variety	Length [mm]	Width [mm]	Thickness [mm]	Mass of 100 seeds [g]	Volume Vu [cm³]	Surface area S [mm2]
Riesling	5.54±0.03	3.43±0.02	2.53±0.02	26.60±0.05	0.20±0.005	51.61±0.02
Burgund	6.21±0.03	3.97±0.02	2.70±0.02	33.45±0.05	0.280±0.005	67,84±0.02

It was observed that all the characteristics, such as dimensions (x, y, z), mass, unit volume, and surface area are considerably larger in the case of the Burgund variety than in the Riesling variety. In terms of the dimensions of the two varieties of seeds, the length and width show the largest differences with values of 0.68 and 0.54 mm, while the thickness has a difference of about 0.16 mm.

The oil content of the seeds is presented in table 3 for each of the two varieties of grapes. Grape seeds contain a small amount of oil compared to oilseeds such as sunflower, or rapeseed. However, the rich content of antioxidants and bioactive compounds, makes grape seed oil an important resource worth exploiting. An important factor in oil extraction is the moisture of the seeds before pressing. In the literature, it is recommended that the humidity of the seeds be between 6-8% for the best possible efficiency. For this work, the seeds were dried by natural ventilation together with the pomace, then mechanically with the help of a dryer conveyor.

Table 3

Moisture content and oil content of grape seeds

Variety	Moisture content [%]	Oil content [%]
Riesling	9.76	8.36
Burgund	9.61	9.64

In Table 3, the values for oil content and humidity are the averages of the three experimental repetitions. The Burgundy variety has a higher oil content than the Riesling variety, the difference being in this situation of 1.29 percent.

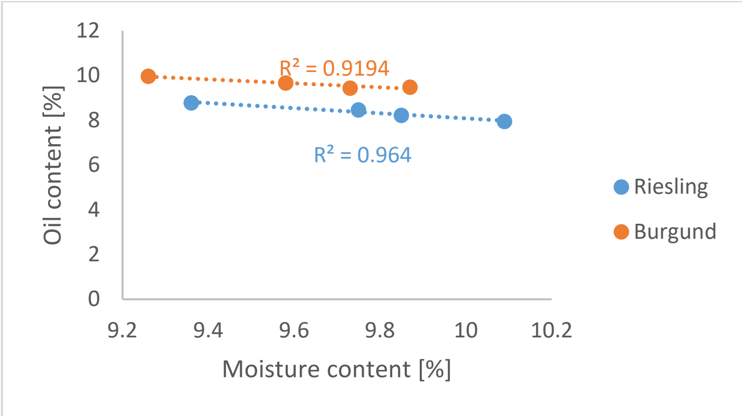

Fig. 6. Dependence between oil content and moisture for grape seeds

Statistical analysis of the results obtained in the case of oil content (OC) showed a high correlation with moisture content (MC) which can be expressed as a linear function, which has R^2 = 0.964 for the Riesling variety, respectively R^2 = 0.919 for Burgund variety:

- for the Riesling variety:

$$OC_R = 19.52 - 1.144\ MC \qquad (6)$$

- for the Burgund variety:

$$OC_B = 18.155 - 0.886\ MC \qquad (7)$$

Regarding the mechanical characteristics of the grape seeds, the breaking force, the deformation at the breaking point, and the energy at the breaking point were registered, according to Table 4. On average, the breaking force was recorded 11.11 Nm for the Riesling variety and 13.04 Nm for Burgundy; for the deformation at the breaking point, 0.41 mm and 0.42 mm were registered, respectively.

The energy consumed during the mechanical testing of grape seeds was obtained from the QMAT software of the Hounsfield equipment and the averages were 0.0028 for Riesling seeds, respectively 0.0034 J for Burgundy seeds.

Table 4

Mechanical characteristics of grape seeds

Variety	Breaking Force [N]	Deformation at the breaking point [mm]	Energy at the breaking point [J]
Riesling	11.35	0.338	0.0029
	7.25	0.34	0.0021
	9.2	0.452	0.0025
	8.41	0.315	0.0019
	16.14	0.619	0.0037
	12.5	0.363	0.0032
	7.2	0.288	0.0019
	11.7	0.394	0.0034
	16.14	0.61	0.0036

	11.25	0.336	0.0032
Variety	**Breaking Force [N]**	**Deformation at the breaking point [mm]**	**Energy at the breaking point [J]**
Burgund	9.12	0.305	0.0024
	9.57	0.321	0.0026
	9.61	0.326	0.0023
	11.31	0.489	0.003
	12.78	0.297	0.0032
	12.98	0.371	0.0041
	13.67	0.382	0.0037
	14.77	0.378	0.0036
	18.24	0.637	0.0048
	18.35	0.679	0.0043

Figure 7 shows a polynomial tendency of the deformation at the breaking point with respect to the breaking force, respectively $R^2 = 0.8342$ for Riesling and $R^2 = 0.8115$ for Burgund.

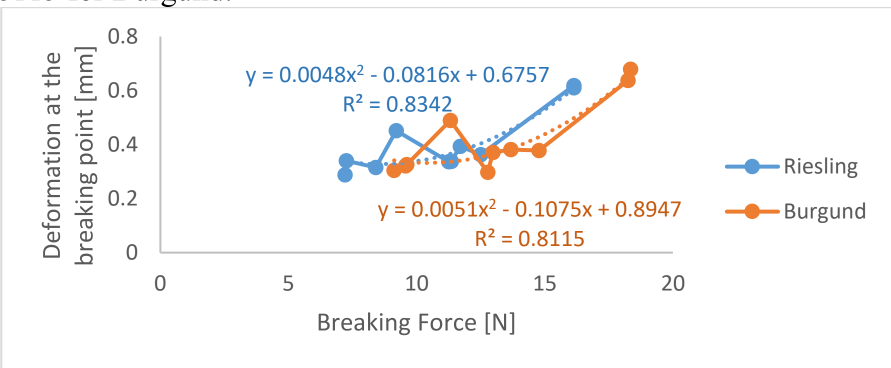

Fig. 6. Dependence between deformation and breaking force

After the characteristics of the grape seeds were determined, the pressing process for oil extraction followed, figure 8. In the first phase the pressing chamber was heated to avoid blocking the material, after which the press was fed with material without its outlet nozzle. be attached. The screw press worked until the first drops of oil appeared which showed that the press was ready to work. The press was configured according to the first set of tests and fed with five kilograms of grape seeds. The results of each set of tests can be found in table 5.

Fig. 8. The experimental process

Table 5

Grape seed oil recovery results for each set of tests

Variety	Setup name	Extracted Oil [g]	Oη [%]	Time [min]
Riesling	**Screw R8**			
	N7_ω220	966	19,32	39,71
	N7_ω290	932	18,64	36,48
	N7_ω355	903	18,06	29,78
	N10_ω220	935	18,7	41,05
	N10_ω290	913	18,26	38,97
	N10_ω355	906	18,12	28,64
	N12_ω220	901	18,02	38,51
	N12_ω290	861	17,22	37,03
	N12_ω355	852	17,04	27,9
	Screw R10			
	N7_ω115	956	19,12	39,83
	N7_ω180	842	16,84	36,59
	N7_ω255	791	15,82	29,87
	N10_ω115	944	18,88	41,17
	N10_ω180	834	16,68	39,09
	N10_ω255	787	15,74	28,73
	N12_ω115	841	16,82	38,63
	N12_ω180	741	14,82	37,14
	N12_ω255	708	14,16	27,98
Burgund	**Screw R8**			
	N7_ω220	987	19,74	39,77
	N7_ω290	953	19,06	36,53
	N7_ω355	924	18,48	29,82
	N10_ω220	956	19,12	41,11
	N10_ω290	942	18,84	39,03
	N10_ω355	927	18,54	28,68
	N12_ω220	917	18,34	38,57
	N12_ω290	908	18,16	37,09
	N12_ω355	894	17,88	27,94

	Screw R10			
	N7_ω115	975	19,5	39,89
	N7_ω180	851	17,02	36,64
	N7_ω255	789	15,78	29,91
	N10_ω115	948	18,96	41,23
	N10_ω180	841	16,82	39,15
	N10_ω255	795	15,9	28,77
	N12_ω115	864	17,28	38,68
	N12_ω180	768	15,36	37,20
	N12_ω255	717	14,34	28,03

At first glance, the best oil recovery was achieved with a 16 mm diameter screw for both Riesling and Burgundy. This meant that better compaction of the material is created, but also a higher pressure when the diameter of the screw is smaller. At the same time, the time in which the grape seed was pressed is longer in the case of the screw with a diameter of 20 mm.

During pressing, the pressure was reduced as the rotational speed increased and the nozzle diameter increased. A higher rotation speed means a higher flow of grape seeds and a lower oil extraction because the time in which the oil drains from the solid is less.

4. Conclusions

In this study, in terms of main properties of the grape seeds, Burgund seeds had the highest values, mainly oil content 9.64%. This does not necessarily mean that the seeds of red varieties are better, but in this situation, they may have won due to the larger size of the seeds.

A disadvantage of Burgund seeds was the higher resistance to presses, on average the breaking force was 13.04 N compared to 11.08 N in the case of the Riesling variety, which led to higher energy consumption.

Extraction of the oil with the N80 screw press was the most efficient using the screw with a diameter of 16 mm and a nozzle with a diameter of 7 mm, unlike other combinations. Considering the two sets of tests for each variety of grape seeds, the highest values of crude oil obtained were 966 g for Riesling seeds and 987 g for Burgund. The yield of extracted oil compared to the quantity of processed seeds was 19.74% and 19.32%.

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