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COMPARATIVE STUDY REFERRING TO PHYSICAL AND CALORIFIC PROPERTIES OF PELLETS OBTAINED FROM GRAPE BIOMASS

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Abstract

In this paper some comparative studies referring to physical and calorific properties of pellets obtained from grape biomass are investigated. The type of grape biomass that is investigated is mainly the pomace, respectively skins and seeds of grapes. Results are firstly specified to the granulometry of milled pomace, and after that to the properties of pellets. Good bulk densities are resulted for grape seed. A higher calorific value of 24.1 MJ/kg is obtained, better those wooden pellets, because this biomass contained some sugars besides of pomace waste. The main conclusion of the paper is that grape pomace pellets have good properties and there are within the limits of the European standards (EN 14961-2).

Key words: ash content, calorific value, grape biomass, pomace, pellets

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1. Introduction

The vine, known under the botanical name *Vitis vinifera L* is a shrub or liana species of the genus *Vitis*, from the *Vitaceae* family, originally from the Mediterranean region, central Europe and southwest Asia. Grapes are cultivated on every continent except for Antarctica. It grows in the sunny and southern hills of warm temperate zones. The length of the vine ropes can reach up to 35 m. There are wild vines species growing on river banks or in wet forests, with grapes of small berries with a diameter of about 6 mm and a ripening colour towards black. Wild vines (*Vitis sylvestris* or *Vitis rupestris*) are used as a rootstock for the noble species used in the wine industry (Fig. 1). There is also the noble or grafted grapevine where the berries are larger, up to 30 mm long, having colours ranging from white -yellow to red or black-purple.

Grapes, as a fruit of the grapevine (*Vitis vinifera*), are highly sought due to their special taste and flavour, regardless of their subsequent use. Grapes are considered to be one of the most popular fruits in the world. It can be consumed directly without further processing, or as a result of other processing in the form of wine, jam, juice, raisins, vinegar and seed oil. The grape berry is made up of skin, pulp and seeds. The biggest quantity of liquid can be found in the pulp, from which the juice is obtained as the main product with the most numerous uses. Grapes with high liquid content are used in winemaking and those with less liquid content are used for direct consumption, jam or raisins.

There are two types of grapes differentiated according to their colour, namely white grapes and red grapes, both varieties being sought almost in equal measure. White grapes have actually a yellowish colour, and the noble varieties belonging to this category include world-wide Pinot Gris, Riesling, Chardonnay, Sauvignon Blanc. Among the black ones we can mention Pinot Noir, Cabernet Sauvignon etc.

Grapes containt a lot of phenolics, these can be found in the skin, in pulp and tannins in the seeds.

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Grape seed oil is highly sought as it contains significant amounts of vitamin E and F as well as some mineral elements such as zinc, copper and selenium. The seeds also contain a very powerful anti-aging agent, being 50 times stronger than vitamin E and 20 times stronger than vitamin C. Also, this seed oil keeps under control some allergic reactions of the human body.

Vine-growing is one of the most extensive agricultural activities in the world, producing more than 50 million tonnes of grapes each year, of which more than 20 million tonnes are grown in Europe. If we take into account that about 75% is used in the wine industry, it results that around 27 billion litres per year are obtained. Among the wine producers, France ranks first with 4.6 million litters, followed by Italy with 4.5 and Spain with 3.8 million litres (FAOSTAT, 2014) as seen in Fig. 2.

The grape wine production flow includes the following operations:

- harvesting grapes from vineyards;
- destemming and removing leaves of the vine harvested accidentally;
- breaking grape berries;
- pressing
- fermentation
- sedimentation and settling
- maturing and bottling.

From an environmental point of view, the wine industry is environmentally friendly, accounting for only 0.3% of annual greenhouse gas (GHG) emissions. The correlation between winemaking and environmental pollution is shown in Table 1.

In order to achieve a clean production, the first step is to know the wine making process and the nature and quantity of manufacturing remains. Producers should be encouraged to reuse the remains and manage them properly. Grape pomace is the most important by-product obtained in the wine industry, generally accounting for 20-30% of the initial grapes mass. Generally, it can be said that from 1 kg of grapes you can get 0.75 l of red wine.

Fig. 1. *Vitis rupestris*
(https://ro.wikipedia.org/wiki/Vita_de_vie)

The presence of skins and seeds during fermentation enables the creation of the red colour of wine by the presence of some pomace pigments during the fermentation process. In case of white wine, first we have to separate the liquid from pomace, after which fermentation takes place. (Fig. 3). This is why the amount of pomace for white wine production is more consistent than in case of the red wine (Fig. 4). After fermentation, wine is pumped into large tanks, then stabilized after which it becomes clearer (no longer cloudy), then filtered, and finally before bottling, wine matures in oak barrels, usually in few years (Amienyo et al., 2014)

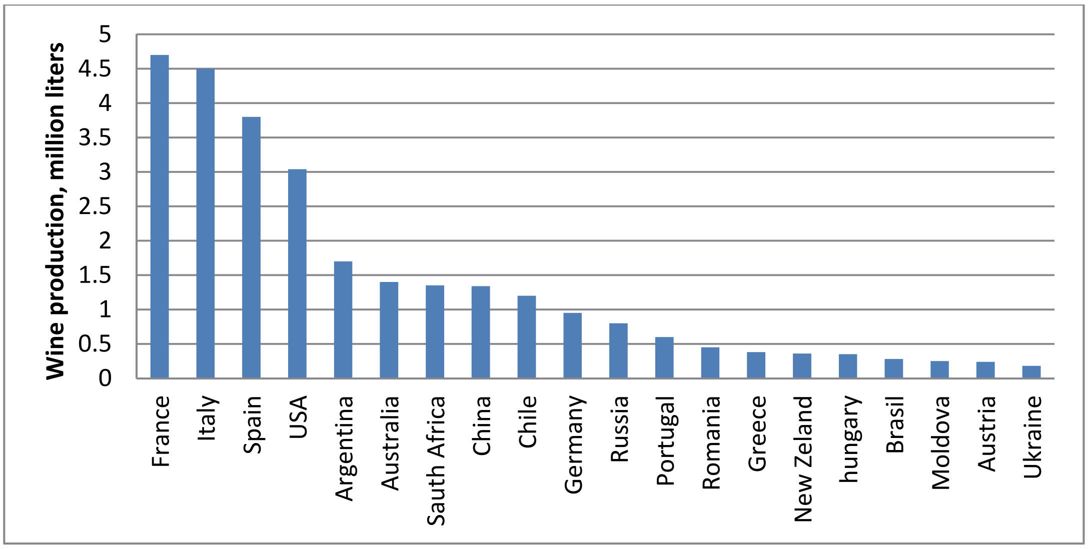


Fig. 2. World wine production at 2012 level (adapted upon Beres et al., 2013)

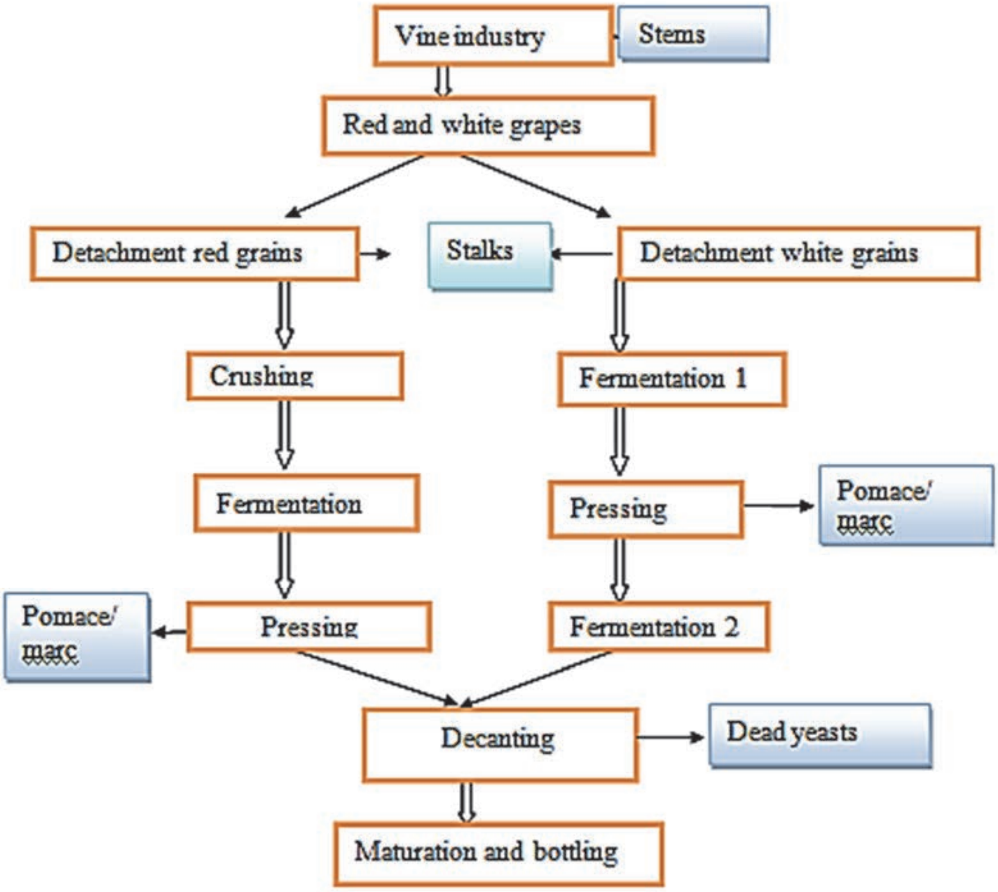


Fig. 3. The production flow for obtaining red and white wines

Table 1. Correlation between wine making process and environmental (Muhlack et al., 2018)

Operations of wine industry	***Environmental contamination***
Grape culture	Pesticides, fertilisers, water supply and fuel
Packaging	Glass bottles and paper labels
Winery	Electricity and water
Transport	Fuel
Waste management	Stalks, waterwaste and grape pomace

Fig. 4. Red grape pomace

In the wine industry, there have always been a number of remains left, including the vine stalks that are cleaned annually and secondly the remains resulting after the squeezing of grapes (marc or pomace). The main ingredients of the grape remain, after the wine is obtained, are the skin and the seeds. Most studies have highlighted the existence of phenols and antioxidants in fibres in grapes skins and seed oil (Beres et al., 2017). Regardless of the nature of the remains resulting in the wine industry, their amount is enormous, occupying vast spaces (which should be previously established and properly prepared). Also, the grapes remain and some small amounts of liquid go easily into fermentation and rotting process, which is why they need to be processed quickly in order not to pollute the terrain and the surrounding air.

Vine ropes are commonly used in combustion directly or transformed into briquettes and pellets (Espinoza-Tellez et al., 2020). This use of the dried vine ropes is advantageous due to good caloric power, but especially due to the high rate of energy release. The uses of grape remain after squeezing the must are multiple and start from the use as fertilizer through rapid degradation by bulk composting or during winter in the soil between vines cuttings. Another use is that of animal feed, usually in combination with other fodder plants.

In the pomace, after pressing, there is a quantity of about 70% of phenols left, for use in other high added value products. There are more flavanoids in red grapes, in skins and seeds, for health benefits (Abarghuei et al., 2010). Pomace consists of skins, stems, residual pulp and seeds. Of the total amount of dry pomace, the seeds represent 38-52% and the skins 5-10%, and the rest up to 100% is represented by stems and sediment (Brenes et al., 2015). In the last 10 years, it is estimated that about 3% of the grape pomace is reused for animal feed, other applications being the compost for vine fertilization and last but not least in the construction of houses in order to improve thermal insulation (Crang et al., 2018, Zang et al., 2017). Their storage or incineration in free field is detrimental to the environment due to phenolic compounds that increase the resistance to biological degradation (Re-Densa et al., 2011). There are other environmental issues, namely total pollution on the soil, water and air.

There are a number of uses assigned to grape pomace, namely: dietary fibres and polyphenols, in the food industry as biosurfactants, in the cosmetics industry as seed oil and antioxidants, or in the pharmaceutical industry as pomace powder (Dwyer et al., 2014). In Spain, there are three variants of pomace management: recycling, recovery and storage. Some companies have adopted the storage solution, but have to pay to the Spanish State an amount of 230 Euro/ month/m^3/h as environmental pollution taxes.

The compounds from grape pomace can be divided into seeds and compounds without seeds. The seeds represent 2-5% of the total grape mass, but 38-52% of the solid remains of the wine industry. Generally, pomace contains 40% fibre, 10-20% lipid, 10% protein, supplemented by phenolic complexes, sugars and minerals (Xu et al., 2016). The undigested fraction, mainly cellulose and pectin, is generally found in seeds and accounts for about 80% of the non-sugary grape matter. After extraction of the oil from the seeds, the obtained remains contain 46% lignin, 11% protein and 5.7% ash, usable in combustion (Prado et al., 2014, Raveendran et al., 1995).

(Mendes et al., 2013) finds an analysis of the chemical constituents of grape skins remains: cellulose 20.8%, hemicellulose 12.5% (mass), tannins 18.8%, protein 18.8%, ash 7.8% and the remainder to 100% represents liquid. The remains resulting after wine, grape juice and grape syrup are obtained (generally 20% but varying from 12% to over 35%) contain skins, seeds and water, as well as other components such as acids and polyphenols, sugar residues and even small amounts of alcohol. Grape berries remain contain appreciable amounts of phenolic acids, pigments and antioxidants. The calorific value of husk grapes is estimated at 20388 kJ/kg, compared to 15972 kJ/kg for rice husk, 18672 kJ/kg for sunflower husk or 18562 kJ/kg for apricot kernels. Also, the pomace contains in small quantities other components such as fatty acids, sugars and other lignocellulosic materials (Verna et al.2009). All of these compounds are extracted and then processed for proper use. Vegetable biomass includes the biomass obtained from the wine production, respectively from the peels and seeds of the remaining grapes after wine are obtained (Yeniocak et al.2014). This category of biomass is environmentally friendly and is a renewable source because is obtained every year in large amounts.

Objectives. Considering that grape pomace is a waste of winemaking, its superior use in pellets leads to the environment cleaning of waste. If the vineyard ropes has been used so far, the present paper desires to use the peels and seeds of the grapes in the form of pellets as a result of their squeezing operation. The obtained pellets will be analyzed in terms of physical properties (moisture content, bulk and effective density), calorific properties (high and low calorific values, volatile and ash content, energy density, etc.) and their environmental impact.

2. Method and materials

Variety of grape pomace types has made possible to use four types of winery biomass (called usual grape pomace or marc), namely: white grape peels; white grape seeds; red grape peels and red grape seeds (Fig. 5). Grape pomace was taken from a laboratory winery, near Transilvania University of Brasov, Romania. After drying at 105 °C in the laboratory oven (Memmert, Germany) for 3-4 hours, grape seeds and peels biomass was ground to a hammer mill to a certain granulometry.

After a conditioning in a controlled environment, 10% moisture was obtained followed by a pelletisation of biomass in pellets with a diameter of 12 mm and a length of 7-15 mm. Density of pellets were determined using the next equation (Eq. 1):

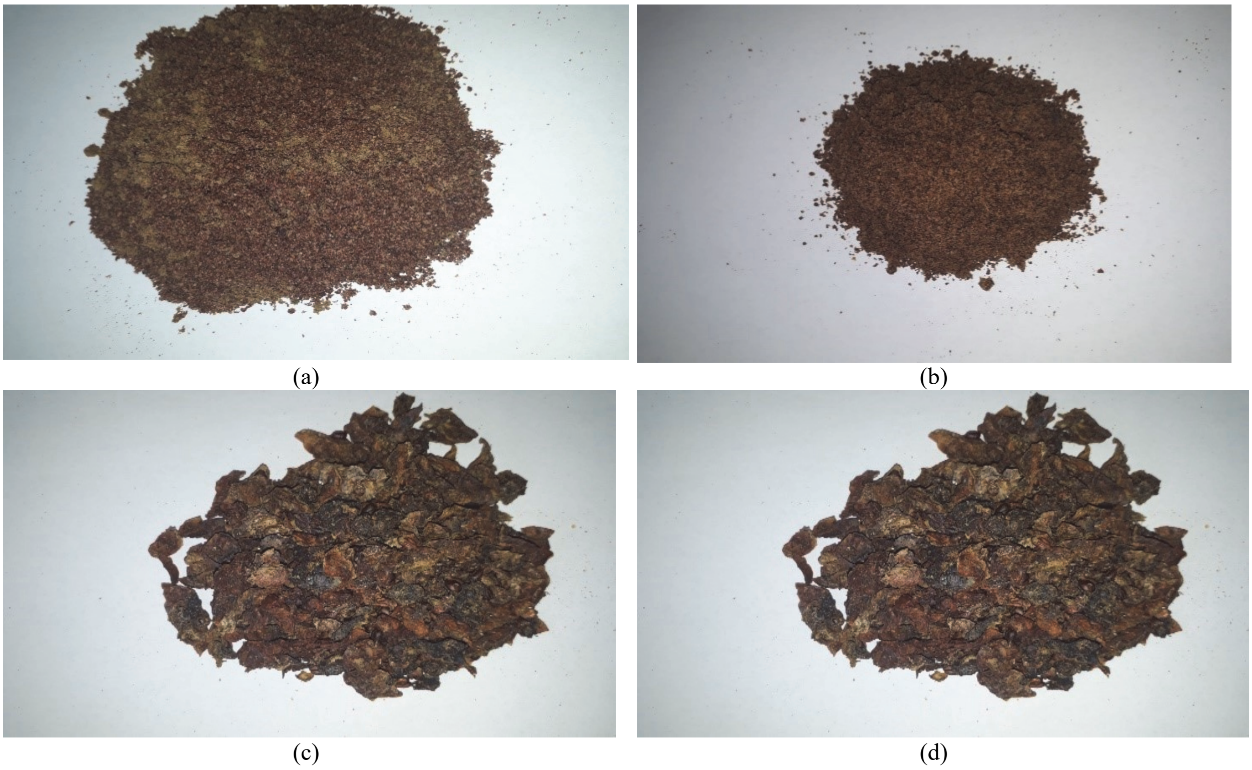
$$Pp = \frac{4 \cdot m}{\pi \cdot d^2 \cdot l} \cdot 10^6 \left[\frac{kg}{m^3} \right] \qquad (1)$$

where: m-mass of pellet, in g; D-dyameter of pellet, in mm; l-length of pellet, in mm.

A number of 20 pieces of representative pellets were used to determine the density of each type of grape pomace (red and white grape, skins and seed pomace). Bulk density was determined using a graduated cylindrical vessel in case of which the inside diameter was known and the height up to which the grape pomace vessel was filled and the total mass of the cylindrical vessel with the material were determined. On the basis of the data obtained, the bulk density of grape pomace was calculated with Eq. (2).

$$Pp = \frac{4(m_{fw} - m_{ew})}{\rho \cdot d^2 \cdot h} \cdot 10^6 \left[\frac{kg}{m} \right] \qquad (2)$$

where: m_{fw}- mass of full wessel, in g; m_{ew} - mass of empty wessel, in g; d - the inner diameter of the determination vessel, in mm; h - height up to which pellets are found inside the vessel, in mm.



(a) (b) (c) (d)

Fig. 5. Grape pomace (a) white ground material; (b) red ground material, (c) red raw material; (d) white raw material

The pelletizing process consisted in taking a quantity of crushed material and compacting it in a SARRAS type press (Brasov, Romania) with a capacity of 50 kg/h, provided with two pressing rollers on a 40 mm matrix. The pressure used during pelletizing was around 120 bar, and the temperature obtained was around 98 ^{0}C. The obtained pellets were cooled and conditioned at 20 ^{0}C and a relative humidity of 55%.

20 test were processed for obtaining unit density of pellets, by dividing the mass of each pellet by its volume (like a straight circular cylinder) as the EN 323:1993 specified. 10 determinations were used to determine the bulk density of each type of pellet, and for each group of pellets the arithmetic mean and standard deviation were determined as statistical parameters.

2.1. Calorific value determination

The calorific value of the pellets (EN 14918:2009) was determined using an explosion XRY-1C calorimeter (Shanhai, China) that provided and shown the testing values, using the following relationship (Eq. 3):

$$CV = \frac{k \cdot (t_f - t_i) - q_i}{m} \qquad [MJ/kg] \tag{3}$$

where: k - the calorimetric coefficient, determined with benzoic acid in the calibration stage, expressed in MJ/^{0}C; t_f - final temperature obtained in water container after testing, in ^{0}C; t_i - initial temperature obtained by termocouple before testing, in ^{0}C; q_i- calorific value obtained during the test by firing the cotton yarn and nichel yarn, in MJ.

Because the pellet contains a certain amount of moisture (usually 8-10%), the test provides for the existence of two calorific values, namely High Calorific Value and Low Calorific Value, expressed in the paper as High CV and Low CV. The difference between the two calorific values is given by the latent heat of water vaporization existing in the material to be analysed. The calorimeter software provides at the end of the test both amounts of the calorific value, namely High CV based on the data obtained during the test, and the *Low CV* based on the *High CV* and the characteristics of the tested material (hydrogen content *h)* when the test is for dry materials (Eq. 4).

$$LowCV = HighCV - 206 \cdot h \quad [KJ/kg] \tag{4}$$

Along with the increase in moisture content the calorific value will decrease, and when the humidity is zero, the two calorific powers are equal (Kuokkanen et al., 2011). If moisture content is over 8-10%, it is advisable to recalculate the calorific value, taking into account the moisture (*Mc*) and the ash content (*Ac*) based on the following relationship (Eq. 5):

$$HighCV = HighCV_c\left(1 - \frac{M_c}{100} - \frac{A_c}{100}\right)[MJ/kg]\ ] \tag{5}$$

where: *High CV* is the upper calorific value corrected, in MJ/kg; M_c-moisture content, in%; A_c-ash content in%; *High CVc* - the upper calorific value read by the calorimeter used, in MJ / kg.

2.2. Ash content determination

The ash content was determined using a calciner furnace (STP Protherm, Ploiesti, Romania) for ground and dry biomass of 0% moisture content, which was calcined at 650 °C for 3-15 hours (ASTM D2866-11 2011, ISO 1928:2009). In order to obtain the anhydrous state, grounded grape pomace was kept for at least 4 hours in a laboratory oven at 105 °C. The calcination was performed on metallic crucibles, resistant to high temperature, and the weighing was performed with an analytical balance with an accuracy of 3 decimals.Verification of the time at which the determination was made carried out by observing the state of the material on the crucible (to be completely burnt without sparkleing), and the mass difference between successive weighngs negligible below 0.01 g. In order to weight the hot melted crucible, it was cooled to atmospheric air in the first stage, followed by excite cooling. The relationship of ash content is expressed as given by Eq. (6):

$$A_c = \frac{m_{si} - m_c}{m_{sf} - m_c} \cdot 100 \quad [\%] \tag{6}$$

The obtained 10 results were statistically processed and verified that the probability of acceptance of the values exceeds 95%.

2.3. Volatile content determination

The methodology for obtaining volatile substances content is governed by EN 15148 (2009). The volatile substances content is determined as loss of mass (less due to moisture content) when the biofuel is heated but not in direct contact with the air. The heating furnace must be electrically heated, because inside it must have a constant temperature of 900 °C, with a maximum accuracy of + -10 °C (Prado, J., 2014). Also, special crucibles are used, with a very tight plug.

Matching and sealing between the plug and the crucible is one of the elements that enable a good determination. The weighing scale must have an accuracy of at least 0.1 g. The crucibles must be disposed on shelves especially created for this determination.

At first, before determining the mass of the crucibles with caps, these are placed in the oven at 900 °C for a period of 7 minutes. The crucibles are extracted from the oven to allow them to cool down on a heat-resistant surface in the desiccator and they are weighted. Weigh 1 gram of the material to be analysed. Then 1 gram of the material to be analysed is weighted, the material is placed in the crucible. The loaded crucible and stuffed with the plug is introduced in the oven at 900 °C, where it is kept for 7 minutes. The crucibles are removed from the oven, cooled to 30-50 °C at room temperature, and finally in the desiccator at room temperature. When cooled, the crucible with plug and material is weighted with a precision nearest to 0.1 g.

The content of volatile substances is expressed by Eq. (7).

$$V_c = \left(\frac{(m_2 - m_3)}{m_2 - m_1} 100 - Mc \right) \frac{100}{100 - M_c} \tag{7}$$

where: m_1 is the mass of the empty crucible with lid, in g; m_2 is the mass of the crucible and lid and test portion before heating, in g; m_3 is the mass of the crucible with lid and contents after heating, in g; M_c is the moisture content, in %.

If dry material is used, the previous equation doesn't interfere with the moisture content Mc and we have following relationship (Eq. 8):

$$V_c = \frac{(m_2 - m_3)}{m_2 - m_1} 100 [\%] \tag{8}$$

Based on volatile content (V_c) and ash content (A_c) it is possible to determine the fixed carbon content as a percentage difference from the whole with the following realtionship (Eq. 9):

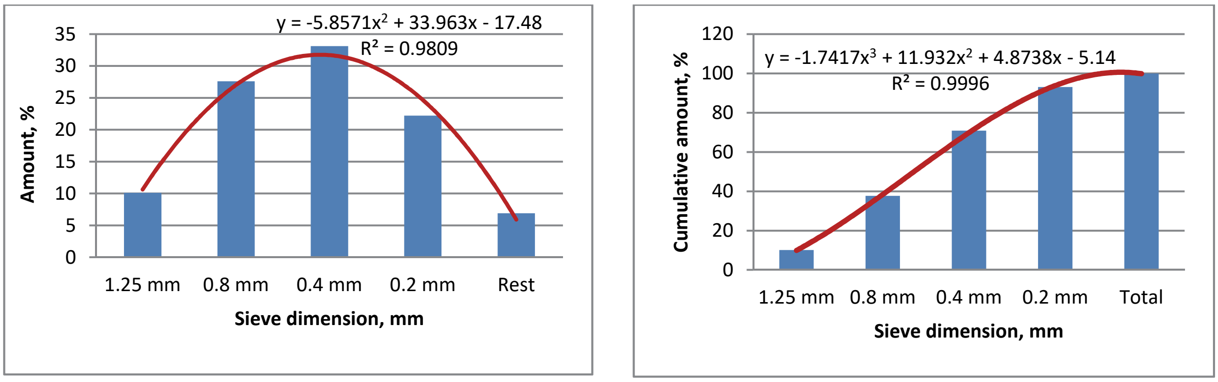
$$C_f = 100 - Ac - Vc [\%] \tag{9}$$

3. Results

Particle granulometry is important when grape pomace is transformed in pellet because when the particle size is smaller the better compaction and a higher density will be obtained. Extend the argumentation to the other end when particle granulometry is high, the weak compaction and a lower density will be obtained (Toscano et al., 2013). This granulometry of grape pomace is shown in Fig. 1 This granulometry was performed on a sorting electrical device equipped with four sorting sites with dimension of 1.25, 0.8, 0.4 and 0.2 mm.

The Gauss's shape shows a uniform and correct distribution of the values obtained, and the Pearson coefficient R^2=0.98 show that the polynomial curve of 2th degree is the best one in this case. When the cumulative values are taken in consideration de “S” form of curve is evidenced and the Pearson coefficient R^2 = 0.999 show that the polynomial curve of 3th degree is the best matched.

Bulk density of grape pomace was different, depending on the type of pomace, but also on granulometry. In Fig. 6 we see the bulk density of a raw pomace, in shredded form. Greater bulk densities are observed for grape seed (regardless of color) than grapes than grapes, with 113% for red grapes and 105% for white grapes. Grape pomace had a bulk density different from that in brute condition, with 30.6% for red seeds and 6.3% for larger white seed, because by shrinking the dimension these particles are better placed, empty spaces being fewer (Fig. 7, Fig. 8). Moreover, by a better arrangement of the small particles in the structure of the pellets obtained a higher density than in the case of non-bodied particles, which is also the cause of the shredding operation of the pomace grape before pelettization.



(a) (b)

Fig. 6. Granulometry of biomass resulting from shredded pomace: (a) simple graph; (b) cumulative graph

The density range of these four types of pellets when the moisture content was 10% was 0.8-1 g/cm^3, with some variation from a type of grape pomace to another ones (Fig. 9). Even if the values obtained are small, the higher compatibility towards the native state of the pomace of more than 110% in the case of red skins, make it possible to use the efficiency of the biomass in combustion.

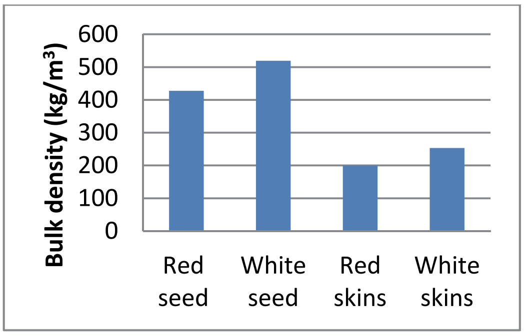


Fig. 7. Bulk density of raw pomace

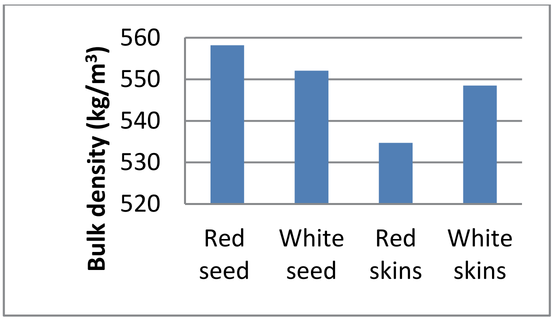


Fig. 8. Bulk density of grounded pomace

Generaly, pellets obtained from grape seeds had o higher density as skin pelets, with 3.5 % in the case of red grapes and with 29.3% in the case of white grapes. The calorific value of the biomass obtained from the grape biomass is devided related to pomage types and superior and inferioar ones. The usual values of 24.1 MJ/kg , respectively 23.6 MJ/kg are found within the pellets obtained from the seeds of the red grapes (Fig. 10).

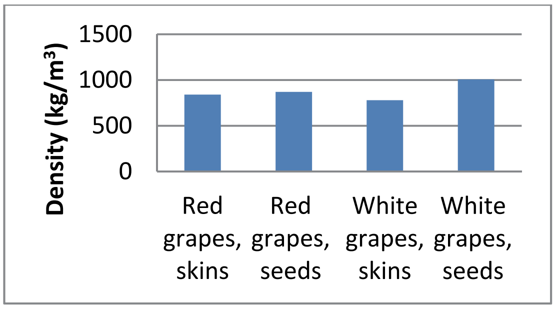


Fig. 9. Density of pellets obtained from grape pomace

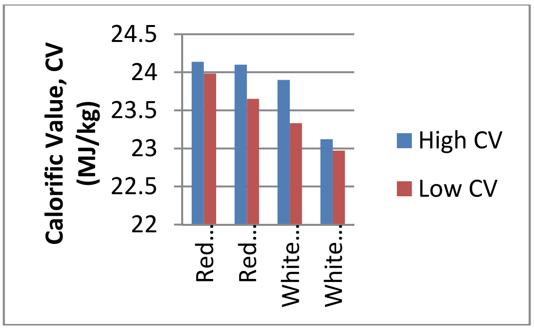
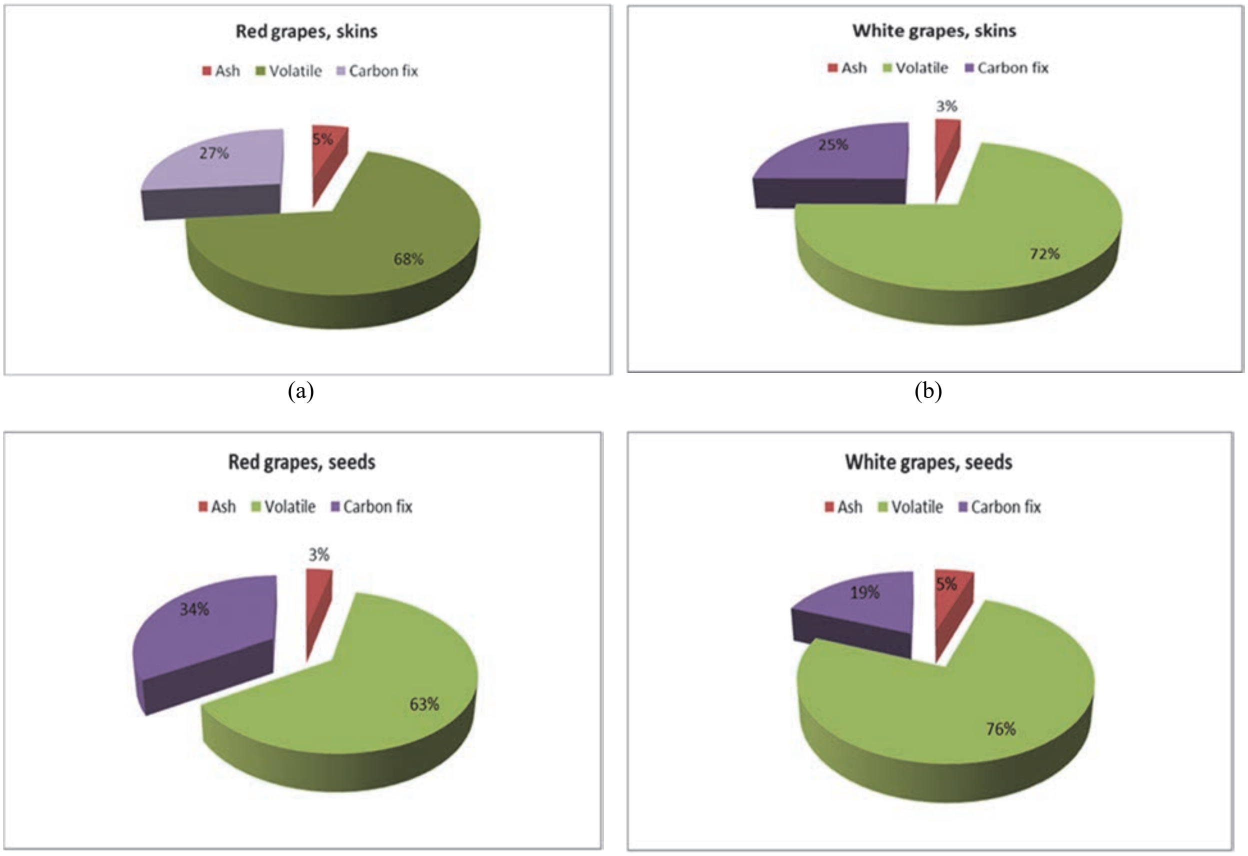


Fig. 10. Calorific value of grape pomace

These inferior and superior calorific values are higher than those of wood (18.6 MJ/kg) or bark (18.4 MJ/kg) and are closer to those of inferior coals (24.5 MJ/kg) (Dumitrascu et al., 2018). The reason for these increases in calorific power is that this type of biomass contains waste of sugars.

The ash content of biomass in grapes is between 3.1-5.3%. These values are higher than of wood biomass with maximum 1.2% (Griu et al., 2016), but lower than wheat straws (with 12-18%, and even 24%) or than wood bark (4.1-6%) as is specified by (Dumitrascu et al., 2018). Volatile content has varied between 62.7-75.8%,the highest values being obtained for the pits of white grapes, and the smallest for the seeds of the red grapes (Fig. 11). The volatile content of pomace grape is found inside the values expressed by other authors for wood and other kind of vegetable biomass (Raveendran et al., 1995).



(a) (b)

(c) (d)

Fig. 11. Content of ash, volatile and carbon fix content: (a) for skin of red grapes; (b) for skin of white grapes (c) for seeds of red grapes; (d) for seeds of white grapes

4. Conclusions

The overall conclusion of the paper is that grape pomace pellets characteristics is within the limits of the European standards (EN 14961-2) regarding to the determined tests (pellet density over 800 kg/m^3, calorific value over 18 MJ/kg, volatile matter content and ash content under 6%). This low-cost biomass is abundant and easy to process for efficient combustion. Getting pellets from this grape pomace is environmental friedly and does not require additional glue adhesives and additives to improve their physical and calorific properties.

Calorific values of grape pomace are superior to wood waste, because it has a superior calorifc value, an increase of 10-18%. Due to this other calorific characteristics of the pellets of this type of biomass will be superior to wood or vegetable remains.

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