Strength of Pellets and Briquettes made From Libyan Olive Oil Solid Residues

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***Abstract*—**Solid residues (SR) collected from a milling facility located in the Western Mountain (WM) region of Libya were used in this work. Two types of pressed units were made; 6mm diameter pellets and briquettes at 35mm and 50mm diameters. Pellets were made using pelletizing machine and SR at 15 and 20% moisture contents (w.b), while briquettes were made using pressing instrument at similar moisture contents. pellets made were solid and stable while briquettes were not, then, wax was added to the SR that used in making briquettes at three wax percentages, 5, 10 and 15. Both pellets and briquettes were tested for their most important handling related parameters; density, maximum breaking force and durability. For pellets, results showed no significant effect of moisture content on both durability and maximum breaking force, but has a significant effect on density. For briquettes, size has no significant effect in all parameters, but adding Paraffin wax as a binding material has significant effects on the three tested parameters. Investigating other sizes and other cheap binding materials were recommended.

***Keywords*—**briquettes, Libya, olive, pellets, solid residues, Western Mountain

I. Introduction

OLIVE tree (*Olea europaea*) is greatly important in the Mediterranean basin, virtually considered as its unique temperament not only agriculturally but also in social, cultural and religious aspects. In fact, Mediterranean countries produce nearly 99% of the world olive oil production [1], [2]. In Libya, olive tree is greatly important either, as the country attains the 12th position among olive oil producers in the region [3]. Olives are cultivated in the coastal plain as well as the eastern and western mountain regions. Number of trees is estimated at nearly 7 million, producing 200,000MT of olives, whereas annual olive oil production is estimated at 16,500MT [3].

Olive oil is obtained in a mechanical milling process, it ranges from traditional methods to the most advanced technologies known as two-phase and three-phase mills, advantages and limitations of such methods are well addressed in [4]. In Libya however, the three methods coexist, ranging from small to medium size, they are mainly family operated and co-op type of business. Olives are harvested (hand-picked) by small growers and farmers, delivered to the mill in plastic boxes and jute bags. Once received, squeezing process takes place anywhere between few hours to few days, depending on produce flow and mill capacity. Season-wise, milling process starts in November and extends until the end of March, depends on the region and varieties. In the two-phase type of mills, the process starts by washing olives, crushing, oil separation, and lastly residues disposal. The milling process constituents are: nearly 20% oil, 30% solid residues (SR) and 50% liquid residues (LR) [5]. Generally, farmers receive their oil, SR is deposited (piled) near the mill, while LR is collected in an open pit. Only small percentage of the SR is used as animal feed, whereas LR is left to dry out in the pit or deposited in empty lands. Neither further treatments such as pomace oil extraction nor LR treatments are implemented. Nonetheless, in situations of low demand on SR for animal feed, large quantities are deposited in landfills or empty lands.

It is important to mention that before hydrocarbon products became widely used in the country, SR had been an important heat source for bakeries and households. Nowadays however, such uses rarely exist, thus large volumes of SR could not be utilized totally for animal feed. Accordingly, developing sustainable means that consider environmental concerns and conserve resources are very much needed.

No studies on utilization of SR in energy use have been reported in Libya. SR uses in heating may have positive impacts not only on local communities but also on rural communities in neighboring countries, especially south eastern Tunisia; wherein fusel fuel is quite expensive combined with limited wood resources and high demand on heating energy sources. Elsewhere, it has been well addressed by several authors that olive residues have been considered as an issue of environmental concern on one hand and a good heat source on the other [6], [7], [8]. Furthermore, SR can be inexpensive yet high energy source compared with other materials, such as wood and organic biomass pellets [9]. Apart from heating, olive oil processing residues can be also an excellent source for bio-fuel [10], [11].

This work investigated; (1) making pellets and briquettes form SR at low moisture contents without grinding, (2) effect of two levels of moisture content on making dense and solid units, (3) testing strength related properties of pressed units such as maximum breaking force, durability and density. Such properties are very much needed in developing mechanically fed heating system, also are quite important in pressed units handling.

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II. Materials And Methods

SR samples were collected from a milling site located in the Western Mountain (WM) region of Libya, sun dried during summer months, then samples were filled in plastic bags, kept in a dry place, and brought to the laboratory. Moisture content was determined on wet basis (w.b) using oven method at 105°C for 24 hours. Also, constituents of SR part; that seed shell (stone) and flesh masses were also determined. Three samples of 100g mass were taken, dried, sieved for separating flesh part from stone in the first stage, then hand separation of stone pieces was made.

Two types of pressed SR units were targeted; 6mm pellets, and larger size (briquette) at two diameter; 35mm and 50mm. Pellet size was chosen for the purpose of their possible use in continuously fed furnace, while briquettes were chosen for their possible use in direct burning type of heating. Experiments were performed in five replicates for pellets and three replicates for briquettes, this was due to the variability in pellet length, which was entirely related to the operation of the pelletizing machine itself.

A. Pressing and Sizes: optimization conditions

Primarily, it was assumed that using high pressure may lead to solid units, taking into consideration solid residues contain nearly 2% oil. In addition, low moisture content (MC) was preferred for the purpose of eliminating drying energy involved compared with energy needed for drying loose SR. Few trails were performed for evaluating whether pressing dry SR (9.8% MC) could produce solid and stable pellets and briquettes. However, few trails concluded to using either higher moisture content or binding material to SR were necessary. Therefore, 15% and 20% MC were chosen, the 15%MC was considered as the lower moisture threshold. Moisture content of SR was raised by adding calculated volume of water that enough to raise SR moisture content to both levels. Water was added to SR, mixed well, kept in an airtight glass jars in a fridge, and periodically mixed for 24 hours.

Trials using SR at 15 or 20% did give stable pellets, but did not result into stable briquettes at both moisture contents, therefore, adding binding material was considered. Paraffin wax was added at 5, 10 and 15% (w/w) to the SR. This was made mainly for the purpose of testing the effect biding material on the studied parameters, regardless of its type.

B. Pellets

A pelletizing machine (Pellet pros. Inc., Davenport IA, 52806, USA) was used, it is consisted of electrical motor activates a shaft via V-belt that operates the pressing roller that runs inside round enclosure. Once the loose materials enters, it is pressed by the pressing roller, and pushed it down through a mounted base with multi 6mm diameter holes. The formed pellets then delivered out through a funnel-like opening and collected. Operating pressure is unknown, but the pressing element has a grinding effect on the SR, this was noticed by raised pellet temperature as well as hand feel. Produced pellets were kept in zip bags, stored in a fridge for few days, later were dried to MC near 10% and used for determining density, maximum breaking force and durability.

C. Briquettes

The pressed SR samples were prepared by adding Paraffin wax at three percentages; 5, 10, and 15 w/w. As mentioned earlier in the optimization process, the selected moisture contents did not result in stable briquettes. Therefore, adding binding material was considered. Sun dried SRs were used; wax was shredded, mixed with SR at the specified percentage, heated for melting wax using a propane torch, cooled to room temperature, and pressed. Two pipe sections 34mm and 48mm inside diameters and 160mm long were used to host SR and two steel rods that fit the cylinders at clearance of about 1.5mm were used as pressing elements. At pressing, cylinder was filled with SR and wax mixture to about 12mm in depth, placed vertically in a universal axial testing machine (UATM) type (AMETK, Model KA-60, Lansdale, PA, 19446 USA.), pressing motion was activated, and force gauge was watched until it reached 19kN (4250lb) and 10.5kN (2350lb) for 50mm and 35mm size, respectively. For both sizes, vertical pressure was determined at 30MPa. Produced briquettes were left under pressure for five minutes and removed from the cylinder. However, pressing time was not considered as an effective parameter, referring to [6]. Briquettes were kept in zip bags and placed in the fridge for few days until measurements of density, breaking force and durability were made.

D. Density determination

Densities of pellets and briquettes were determined by measuring unit dimensions and masses using a caliber and a sensitive scale. Five pellets of medium length were used, their dimensions and masses were measured, and densities were determined. For 35 and 50mm briquettes, three units of each size were used and densities were also determined. Densities for both units were expressed in grams per cubic centimeter ($g.\ cm^{-3}$).

E. Breaking force

Pellets and briquettes were tested for their maximum breaking force and durability using Instron Universal Testing Machine (Series IX, Automated Materials Testing System 1.16). Applied force in Newton (N) and the displacement in millimeter (mm) were recorded, from which a force-displacement curve was obtained. The peak of force-displacement curve represents breaking force, while the slope of the positive section of the curve represents durability ($N.mm^{-1}$). For pellets, five samples were tested at each moisture content, while three samples of each wax percentage treatment were examined for briquettes.

Samples were laterally placed in the machine and subjected to applied force, and lateral braking force and durability were tested. It was assumed that axial breaking force and durability are normally higher than lateral ones due to the cylindrical shape of pellets an briquettes. Breaking force and slope of the force-displacement curve were recorded and used as dependant variables in the analysis.

F. Statistical analysis

A complete randomized block design was applied on data collected for pellets and briquettes. Two moisture contents were the independent variables for pellets, while three wax percentages and two sizes were the independent variables for briquettes. Dependent variables for pellets and briquettes were: density, breaking force and durability. Interactions among both groups were tested applying the analysis of variance (ANOVA) and subsequent Tukey-Kramer (HSD) using (*jmp* 8.0 SAS Institute Inc. NC, USA) statistical software. Significance level was declared at 5%.

III. Results And Discussions

A. Moisture content

Moisture content of SR was determined at 9.8% wet basis (w.b), while percentages of flesh part and stone constituents were determined at 20.77 and 79.23%, respectively. Moisture content of SR collected from several milling operations in WM region were previously determined at 49 to 52%. However, this study targeted making pellets and briquettes from low moisture content SR. Table I shows ANOVA results for tested parameters as related to SR moisture content for pellets.

Table I
ANOVA Results For Pellets

Source	d.f	SS	MS	F-Ratio	Prob.> F
		Maximum breaking force			
MC	1	136.383	136.382	1.227	0.300
Residues	8	888.995	111.124		
Total	9	1025.377			
		Durability			
MC	1	2412.120	2412.120	1.638	0.236
Residues	8	11778.765	1472.350		
Total	9	14190.885			
		Pellet density			
MC	1	0.0081	0.008	15.041	0.0047
Residues	8	0.0043	0.00054		
Total	9	0.0124			

It showed that moisture content has neither significant effect on breaking force nor on durability. On the other hand, it has significant effect on density. Table II shows density of pellets made from SR at 15% MC are significantly higher than those made from SR at 20% MC at ($p <0.05$). This mainly attributed to the fact that higher moisture content may help in producing dense pellets during pressing, but after drying takes place, density decreased. However, for this particular study densities of pellets produced from SR at 20% MC agreed well with results reported in [6]. They reported densities of units made from SR at 20% MC and two pressures, 25MPa and 35MPa, averaged densities at both pressures were 1.054g.cm^{-3}, which is in a good agreement with pellet density determined in this work (1.057 g.cm^{-3}) at about same pressure and moisture content. Generally, it was observed that starting with high moisture content gave lighter pellets, this perhaps attributed to loosing water after drying. Also, results showed that stable pellets can be made at MC between 15 and 20%, since neither significant difference were found in durability nor on breaking force among both moisture contents.

Table II
Effects Of Moisture Content On Breaking Force, Durability And Density Of Pellets

MC %	Breaking Force (N)	Durability (N.mm^{-1})	Density g.cm^{-3}
15	61.416$^{(a)}$	247.941$^{(a)}$	1.113$^{(a)}$
20	54.030$^{(a)}$	216.879$^{(a)}$	1.057$^{(b)}$

MC. moisture content (w.b). Values with the same letter are not statistically different

Effect of moisture content on other dependent variables, namely breaking force and durability were found insignificant, indicating that even at higher moisture contents pellet hardness would not change. In other words, pellet density didn't improve neither breaking force nor durability at least in this study. This may be due to the absence or weakness of biding constituents in SR.

For briquettes, pressing SR at both moisture contents did not give stable units. Briquettes were formed by pressing SR as described earlier, but after few hours cracked and became very weak. This was due to constituents of the SR, its constituents of shell (stone) were more than the soft flesh part. For such reason weak and incoherent units were produced. Briquettes made from SR at both moisture contents were discarded in testing their density, maximum breaking force and durability.

Table III shows ANOVA results for 35mm and 50mm diameter briquettes, clearly implies that size has no significant effect on density, maximum breaking force and durability. This perhaps due to the fact that both units were close in size, in addition to the constituents of the SR itself. Units made from SR that consisted of 21% flesh (shown in section A) and the remaining was crushed carouse olive stone may not result in solid and stable units, in addition to the lack of binding material that cohere larger pieces.

Table III
ANOVA Results For Briquettes

Source	d.f	SS	MS	F-Ratio	Prob.> F
		Maximum. breaking force			
Diameter	1	23024.43	23024.4	1.178	0.29
Residues	16	312988.57	19561.8		
Total	17	336013.01			
		Durability			
Diameter	1	8633.02	8633.0	0.26	0.62
Residues	16	530998.14	33187.4		
Total	17	539631.20			
		Briquette density			
Diameter	1	0.012	0.012	3.346	0.086
Residues	16	0.055	0.003		
Total	17	0.067			
		Maximum breaking force			
Wax %	2	286894.23	143447.0	43.81	0.0001
Residues	15	49118.78	3275.0		
Total	17	336013.01			
		Durability			
Wax %	2	456129.00	228065.0	40.97	0.0001
Residues	15	83502.15	5567.0		
Total	17	539631.17			
		Briquette density			
Wax %	2	0.048	0.0239	18.99	0.0001
Residues	15	0.0189	0.0013		
Total	17	0.0666			

Tables IV shows the use of Tukey-Kramer test on ANOVA results, wax percentage had significant effects on density, maximum breaking force and durability at ($p<0.05$), whereas had no significant effect of briquettes diameter on all tested parameters. Effects of binding material could be in from of combining both soft and solid parts together (flesh and stone), and preventing expansion after pressure release, and therefore dense and solid units were obtained. Additionally, adding wax improved density, maximum breaking force and durability. Again, this was due to the gluey effect of wax on the constituents regardless of the moisture content. Wax may also have the ability to combine the solid parts (stone) and the flesh part (soft), giving solid and stable briquettes. This work however showed that pressing dry SR even at high pressures needed adding material that has binding effect.

TABLE IV
EFFECTS OF WAX PERCENTAGE AND DIMAETER ON BREAKING FORCE, DURABILITY AND DENSITY OF BRIQUETTES

Source	Breaking Force (N)	Durability ($N.mm^{-1}$)	Density ($g.cm^{-3}$)
Wax 5%	40.18$^{(c)}$	70.98$^{(c)}$	0.844$^{(c)}$
Wax 10%	190.57$^{(b)}$	253.99$^{(b)}$	0.914$^{(b)}$
Wax 15%	349.39$^{(a)}$	460.66$^{(a)}$	0.970$^{(a)}$
Dia. 35mm	229.14$^{(a)}$	283.78$^{(a)}$	0.934$^{(a)}$
Dia. 50mm	157.61$^{(a)}$	239.98$^{(a)}$	0.884$^{(a)}$

Dia. (briquette diameter in mm). Values with the same letter are not statistically different

Briquette density was lower than that obtained for pellets, this is attributed to the difference of making both types of units. Pellets were made using pelletizing machine that grinds SR prior to pushing it through holes, thus finer SR constituents were mixed and pressed, leading to dense pellets. Similar densities were reported in [6] whereas solid residues were ground prior to pressing. However, in this work, grinding was not considered for the purpose of eliminating energy needed for grinding.

Using wide range of binding materials worth further investigations, such as bio-residues, clay, cement and lime. Additionally, investigating making pellets and briquettes from fresh SR and wide range of moisture contents and several sizes worth further attention.

IV. CONCLUSIONS

Pellets and briquettes were made from SR produced in the western mountain region of Libya. SR at moisture 15 and 20% moisture content gave stable pellets, but did not produce stable briquettes. This indicates that large sizes pressed units required adding binding materials. However, high moisture content gave lighter (lower density) pellets, also it did not affect maximum breaking force and durability. Briquettes made by adding wax to the SR at 5, 10, and 15% gave solid units, whereas wax percentage increased density, maximum breaking force and durability. Further investigations of more sizes, higher moisture contents, the use of fresh and moist SR and the potential of using other binding materials are rather recommended.

REFERENCES

[1] Nair, N.G. and Markham, J., 2008. Recycling solid waste from the olive oil extraction process. Australian Rural Industries Research and Development Corporation. RIRDC pub. No. 08/165.

[2] Evangelia V., T and H. N Lazarides, H. N., 2012. Fouling Analysis and Performance of Tubular Ultrafiltration on Pretreated Olive Mill Waste Water Food. *Bioprocess Technology* (2012) 5:584–592.

[3] FAO, Food and Agriculture Organization of the United Nations Statistical Data. www.fao.org

[4] Moral P. S. and M. V. R. Méndez, 2006. Production of pomace olive oil. GRASAS *Y ACEITES*, 57 (1),ENERO-MARZO, 47-55, 2006.

[5] Omer, A. M, and Mahmoud, A. M., 2012. Effect of Olive Pomace Phenols on the Soil Fungi and Alfalfa Yield. *Research Journal of Agriculture and Biological Sciences*, 8(1): 55-61, 2012

[6] Widyan, M.I., Al-Jalil, H.F., Abu-Zreig, M.M., and Abu- Hamdeh, N.H., 2002. Physical durability and stability of olive cake briquettes. *Canadian Biosystems Engineering*. (44) 3.41-3.45.

[7] Intini, F., Kühtz, S., and Rospi, G., 2011. Energy Recovery of the Solid Waste of the Olive Oil Industries– LCA Analysis and Carbon Footprint Assessment. *Journal of Sustainable Energy & Environment*. 2 (2011) 166-157.

[8] El-Hamouza, A., Hilalb, H. S., Nassara, N., and Mardawia, Z., 2006. Solid olive waste in environmental cleanup: Oil recovery and carbon production for water purification. *Journal of Environmental Management* (84) 83–92.

[9] Russo, G. C. , Cappelletti, M., Nicoletti, G. M., 2008. LCA of energy recovery of the solid waste of the olive oil industries.*6th International Conference on LCA in the Agri-Food Sector*, Zurich, November 12–14, 2008.

[10] Haagensen, F, I. V. Skiadas, H. N. Gavala, K. B, Ahring, 2009. Pre-treatment and ethanol fermentation potential of olive pulp at different dry matter concentrations. *biomass and bioenergy* 33:1643–1651.

[11] Karunanithy, C., Wang, Y., Muthukumarappan, K., and Pugalendhi, S., 2012. Physiochemical Characterization of Briquettes Made from Different Feedstocks. *Biotechnology Research International*. vol.12. 12p 1-12.