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Properties, Strength and Stability of Pressed Units Made From Olive Oil Milling Solid Residues

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ABSTRACT Olive oil milling operation in Libya produces nearly 40,000 metric tons per year of unusable solid residues, leading to great environmental impacts and waste of bioenergy resources. The study investigated properties of olive solid residues (SR) right after milling and producing stable encapsulated units from it. Residues were collected from milling operation sites; Yefren, Swani, Subrata and Msalata, located in the north western part of Libya. Moisture content (MC), oil content (OC), and flesh to kernel ratio (F-K ratio) were determined along the milling season. Pressed units with diameters 10, 20, 30, 40 and 50 mm were made by hydraulic press using SR at 15, 20, 25, 30 and 35% wet basis moisture content. The pressed units were tested in their braking force and density.

Results showed that mean SR moisture content from four sites ranged between 49 and 53% , F-K ratio between 1: 3 and 1:4, and oil content was 5 to 6%. Stress analysis showed that 40 and 50mm diameters combined with 30 and 35% MC exhibited best force-deformation curves, breaking force was significantly higher at ($p<0.05$ level) compared with other diameters and MC combinations. Also, unit diameter and MC significantly affected breaking force and density at ($p<0.05$ level). The study concluded to the use of SR at 30 and 35% MC combined with 40 and 50mm diameters has given the most stable units.

Keywords: Olives, solid residue, encapsulated unit, properties, strength

INTRODUCTION

Olive tree (*Olea europaea* L.) is extremely important in Mediterranean societies. Nearly 750 million productive trees are grown worldwide, yet Mediterranean region possess utmost, wherein about 98% of olive oil is produced (Avraamides and Fatta, 2007). Amongst olive producers, Libya comes in the 12th place with annual production of olives estimated at 200,000 metric tons and producing nearly 40,000 metric tons of oil. Moreover it has been estimated that olive milling operations in Libya generate more than 40,000 metric tons of solid residues.

Oil is collected in a mechanical milling operation ranges from very traditional to the most advanced technologies known as two and three phase mills. Generally, olives are washed, crushed, and squeezed, the aqueous part with its oil content is separated, while solids, mainly lignocellulosic solid husk residual known as solid residues (SR) are conveyed to the facility yard. Nonetheless, oil is collected from the aqueous part in a separate mechanical process, and liquid residues (LR) are deposited in underground reservoir or an open ditch. Both residue have great implications on ecosystems, leading to several environmental problems (Michailides et al 2011; Doymaz et al 2004). Most importantly; affecting soil properties, polluting water resources and emitting unpleasant odors (Federici et al 2011; Chartzoulakis et al 2010). Moreover, deposing olive residues in open fields may lead to reacting their oil content with phenols and other aromatics, producing harmful chemicals and bad odors (El-Hamouza et al 2007). Olive residues have been characterized as organic rich biomass with high cellulose content and considerable amounts of polyphenols and fats (Aviani et al, 2010). Several studies have dealt with olive SR, most importantly: (1) composting and improving soil properties (Tan and Markham 2008; Azbar et al 2004), (Anonymous 2008), (2) recovery of compounds with high values (Intini et al 2011); (Kalderis and Diamadopoulos 2010), (3) as heat source (Miranda et al 2008; López-Pineiro et al 2010) and (4) feeding ruminants (Sansoucy 1985).

Agricultural biomass is generally considered as a good heat source, especially in developing countries. Encapsulation certainly has many advantages, such as reducing volume (bulk density), easing handling, increased heat value, and extending burning time. Although olive milling operations in Libya generate considerable volume of SR, yet their efficient utilizations are quite limited. The current work investigated determining properties of SR right after milling, encapsulating SR to several unit size, and investigating effects of moisture content and diameter on stability and density of the units.

MATERIALS AND METHODS

Solid residue source and collection: SR and LR were collected biweekly from milling sites located in the north western part of Libya. Four sites were selected, Subrata (near coordinates: 32°47′32″N 12°29′3″E), Yefren (near coordinates: 32°03′46″N 12°31′36″E), Swani near Coordinates 32°40′10″N 013°09′24″E and Msalata (near coordinates: 32°34′56″N 14°02′24″E). Samples were collected fresh from SR piled nearby the facilities, samples were filled in polyethylene bags and transported to the department of Agricultural Engineering, Faculty of Agriculture, Tripoli, and kept frozen at -18°C until experimental procedures were carried out. However, samples used in the current study represented the beginning of the milling season, its middle and its end. LR samples were also collected from the effluent before reaching the collection tank, were used for determining oil contents only.

Residues properties determination:

Moisture content: Moisture contents of SR were determined using the oven method, Samples weighed 100g were dried at 105°C for 24 hours, and moisture contents were expressed in wet basis. SR moisture content were determined for samples represented the beginning of the season, its middle and its end.

Flesh-kernel ratio: SR constituents of kernel (seed shell) and flesh (outer part of the fruit) were determined by sieve separation. 100g of SR were dried at 70°C until sample weight stabilized, then samples were sieved using certified sieve (Giuliani Technologies srl, Torino, Italy). Ratio of flesh to kernel (F-K) was determined in mass basis.

Oil content: Oil contents of SR and LR were determined using standard Soxhlet extraction method, in similar procedures reported by (Cruz et al 2015). SR samples were dried at 70°C, grinded, 1g of SR was socked in Diethyl Ether, filtered and vacuumed for evaporating solvent. For LR 1000ml samples were taken and their water content was evaporated at 70°C, remains were collected and oil content was determined following similar procedures for SR. Oil contents were expressed in percentages (%).

Pressing units (encapsulation): SR moisture contents were adjusted to 15, 20, 25, 30, and 35% (wb). Fresh SRs were partially dried to targeted moisture contents using heated oven, time needed for reaching the desired moisture content was estimated, also a periodical sample weight was made until desired moisture content was reached. Once the desired moisture content was reached, samples were cooled, filled in polyethylene bags and kept refrigerated until pressing was carried out.

A mechanical press was used for encapsulating units. It is consisted of a 10 ton hydraulic jack mounted on a metal frame and operated by a mechanical motor. The motor turns the jack oil pressure handle via a crank connection, facilitating slow pressing motion of 0.33mm per revolutions. A threaded hole was made entering the jack's oil reservoir, facilitating fluid pressure measurement using pressure gauge. Five cylinders were machined with internal diameters 10, 20, 30, 40 and 50mm, Each cylinder was equipped with pressing rod that pressed the sample upward against metal base. Pressure was monitored until reaching 70bar (7MPa), the sample was left under pressure for three minutes, then pressure was released and pressed unit was removed. Encapsulated units were dried at room temperature for 30 days, mean room temperature and relative humidity were 24°C and 40%. Following drying, unit moisture content and relaxed bulk density were determined.

Strength test: Units were tested for their strength using CBR compression test machine (Model no. 1870-4–1457, ELE. UK)**.** The machine consisted of a ring that is subjected to deformation by compressive force. The ring is connected to a metal rod from one end and attached to the tested sample from the other. Ring deformation was read by force gauge (N) and deformation was read by another gauge in (mm). Pressed units were placed vertically and the lifting motor was operated, lifting the sample up against the metal rod. Sample was gradually subjected to compression force until sample failure occurred. Force and deformation were incrementally recorded and final breaking force was also recorded.

Statistical Analysis: Analysis of variance (ANOVA) was made using SAS statistical software applying general linear model (GLM), significant level was declared at (0.05 level), and Duncan multiple range test was applied. Independent variables were SR moisture content and unit diameter, and dependant variables were breaking pressure and relaxed density.

RESULTS AND DISCUSSIONS

SR properties; Table 1 shows means of moisture content, oil content of SR and LR, and SR flesh and kernel constituents ratio (F-K ratio). They are discussed as following:

Moisture content: It can be noticed that moisture contents of SR collected from the four milling sites ranged between 50 and 53% (w.b). Despite variations in cultivars, field practices and milling operations and their alleged effects on SR moisture content. It is apparent that such conditions had little effect of residue moisture content. Generally, moisture contents were within normal ranges and in a good agreements with values reported in the literature ranged between 50 and 75% (Doymaz et al 2004; Milczarek et al 2011). The current study values were in the lower threshold of reported values in the literature, this may be due to olives were subjected to moisture loss between harvesting and milling, since the produce had lost moisture waiting few days at the milling facility prior to milling. Other authors indicated that two phase milling operations generally generate SR with low moisture percentage (de la Casa et al 2012). Al-Widyan et al (2002) reported very low moisture content of SR of milling operations in Jordan ranging from 35 to 40% (w.b.). Generally, olive milling facilities in Libya are two phase system, and giving consideration to sources of variations, such as location, growing conditions and milling parameters, moisture content reported here can be considered as quite normal and gives good representation of SR under local conditions.

SR and LR oil contents: Oil content of SR from the four sites were in close range. Variations may be attributed to cultivar, growing conditions and milling operation itself (Azbar et al 2004). Swani site reported the lowest oil content in SR compared with the other three sites, it may be attributed to milling mechanical efficiencies. The manager of Swani facility reported having an improved model of the two phase mill. However, El-Hamouz et al (2004), Baccar et al (2009) reported oil contents in SR ranging between 4 and 6%, and Jarboui et al (2008) reported LR oil content around 3%. Oil content in the LR of Yefren, Subrata and Swani sites were quite similar, while Msalata site exhibited relatively high content at the beginning of the season. Similarly, Subrata site recorded high values at the middle of the season. This may be attributed to operational reasons or sampling conditions. However, variations in oil content is quite normal in olive milling operation, because of involvement of several factors related to growing conditions, in addition to milling conditions that are relatively uncontrollable. Generally, values reported in the current work can be considered acceptable, yet further investigations may be encouraged.

Flesh to kernel constituent ratio (F-K ratio). Ratios of the four sites showed differences. Yefren site showed highest kernel content (1:4), Subrata and Msalata had the same ratio (1:3.5), while Swani site recorded the lowest kernel content (1:3). This may be related to geographic location, Yefren is located in the Western Mountain region (nearly 700m above sea level) at nearly 150km south western of Tripoli. In this region, olives are grown under non irrigated conditions, also cultivars are mostly local, differ from those found in the coastal region. The local cultivars are generally characterized by small fruit size and thin flesh layer. Moreover, trees may be subjected to water stress, leading to smaller fruit size and thus smaller flesh mass consistent is quite expected. Msalata is located at 80km east of Tripoli while Subrata is located nearly 60 west. Msalata trees are mostly non irrigated but cultivars are similar to those grown in the coastal area. Subrata olives are irrigated, however cultivars are similar to those in the coastal region. Similarities in F-K ratio for the two sites is highly due to similar cultivars. Sawani site on the other hand exhibited the highest flesh content, this may be due to irrigation effect, moreover, cultivars in the area are generally characterized by large fruit size, leading to high flesh content. In general, F-K analysis showed that Yefren site had the highest kernel content, the two sites away from Tripoli had similar kernel content, while Swani site had the lowest kernel percentage.

Table 1. Properties of SR and LR of the four milling sites

Site	*Mean SR moisture content (wet basis) in the season*			
	Beginning	Middle	End	Mean
Msalata	53.60	57.04	49.66	53.43 (±3.70)
Yefren	49.82	52.57	50.68	51.04 (±1.41)
Subrata	48.10	58.76	52.16	50.30 (+ 1.41)
Swani	53.26	46.73	49.64	49.88 (+2.27)
	Mean SR total fat content in the season (%)			
	Beginning	Middle	End	Mean & STD
Msalata	5.8	6.9	5.4	6.0 (± 0.62)
Yefren	5.4	7.0	5.6	6.0 (± 0.68)
Subrata	5.4	7.8	5.7	5.9 (± 1.09)
Swani	5.1	4.1	6.4	5.2 (± 0.95)
	Mean LR total fat content in the season (%)			
Msalata	4.4	2.51	2.51	3.15 (±1.11)
Yefren	1.8	0.52	1.73	1.35 (0.73)
Subrata	0.20	3.57	0.082	1.30 (± 1.98)
Swani	0.93	0.83	0.082	0.60 (± 0.46)

SR dry constituents percentages and flesh to kernel ratio

	Beginning		Middle		End		Ratio
	Flesh	Kernel	Flesh	Kernel	Flesh	Kernel	
Msalata	33	76	27	73	32	68	1:3.5
Yefren	23	77	20	80	18	82	1:4
Subrata	35	76	30	70	24	76	1:3.5
Swani	24	76	27	73	24	76	1:3

Encapsulated unit strength parameters

Effect of unit diameter on breaking pressure and density: Analysis if variance showed unit diameter had significant effect on breaking pressure of units of the four sites. For density however, effects of tested parameters were inconsistent. Table 2 shows Duncan multiple range test (MRT) results, means of vertical breaking pressure and unit density (lower). It can be noticed that unit diameter exhibited significant effect on breaking pressure at ($p<0.05$) level for the four sits, within the same site there was a linear relation between unit diameter and breaking pressure, discussions are presented as following.

Breaking pressure: Strength of pressed unit is characterized by force or pressure necessary for its destruction (Kaķitis et al 2011). Unit strength (breaking pressure) linearly increased with diameter increase, giving significant effect at (0.05 level) of unit diameter on handling vertical pressure, also trends were quite linear as shown in Figure 1 (left).

Smaller diameters (10, 20 and 30mm) produced weaker units, this was attributed to the small surface that may led to weak binding between particles, especially with the existence of high percentage of kernel contents. Larger unit diameter indeed had larger surface area, leading to binding particles together, and thus produced stable units. For the purpose of testing SR behavior and the effects of moisture content and unit size, no binding material was used in this study. Effects of moisture content and unit diameter were clearly demonstrated (Table 2). Variations among the four sites was recorded, again this was due to the nature of the biomass and variations in SR sites as discussed in the properties section, which indeed affected strength. However, compressions among four sites was not intended, rather investigating effects of diameter and moisture content. In a previous work that reported stability of pellets and briquettes from the SR of a milling operation in the western mountain region of Libya, Fennir et al (2014) reported weak units made from rehydrated SR, and paraffin was added as binding material. In the current study however, pressing moist SR seemed to give an advantage of producing fairly stable large sizes units. Also, similar results were reported by Widyan et al (2002) and Cuenca et al (2013), but those were for grinded olive residues.

Table 2. Effect of diameter on breaking pressure and unit density

Unit diameter (mm)	Vertical pressure (kPa)			
	Msalata	Yefren	Subrata	Swani
10	6.73 [e]	10.40 [d]	10.40 [e]	7.27 [d]
20	56.67 [d]	71.73 [c]	72.93 [d]	62.33 [c]
30	101.73 [c]	176.93 [b]	128.40 [c]	83.27 [c]
40	293.60 [b]	407.93 [a]	280.07 [b]	185.53 [b]
50	446.24 [a]	424.20 [a]	447.53 [a]	271.47 [a]
	Unit density ($g.cm^{-3}$)			
10	0.84 [a]	0.90 [a]	0.91 [a]	0.86 [a]
20	0.77 [a]	0.85 [b]	0.85 [b]	0.81 [b]
30	0.84 [a]	0.80 [c]	0.83 [b]	0.76 [c]
40	0.79 [a]	0.79 [c]	0.81 [b]	0.75 [c]
50	0.74 [a]	0.75 [d]	0.79 [c]	0.71 [d]

Means with the same letter in the same column are insignificant

Mean breaking force of units of the four sites were fitted in linear model shown in Figure 1 (left). Unit diameter and breaking pressure exhibited linear relation (R^2 = 0.96), and 40 and 50mm units resisted high vertical pressure comparing with smaller units.

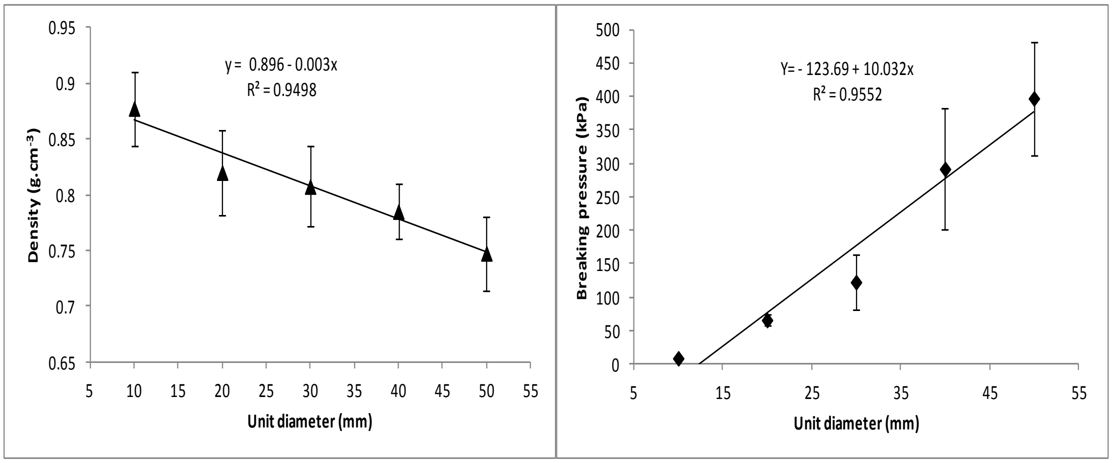

Figure 1. Unit diameter vs density (left), breaking pressure vs diameter (right)

Density: Bulk density was determined after slow drying of units at room temperature. Relaxed density was determined after 4 weeks of drying at room temperature. Certainly units were subjected to relaxation (expansion) during slow drying, as dimensions increased from their original. Generally, significant effect of diameter on density at ($p<0.05$) was recorded. Looking at density values shown in Table 2, density linearly decreased as unit diameter increased. Mean density of 10mm units of the four sites was (0.878 $g.cm^{-3}$) and this values reduced as unit diameter increased, reaching (0.748 $g.cm^{-3}$) for 50mm units. The difference between two densities is nearly 14%. This was attributed the relaxation effect of the units. Indeed, small units had small relaxation effect that larger units. It is well known that biomass may exhibit expansion after pressure release and moisture evaporation, leading to reduction in bulk density (Davies and Abolude 2013; Al-Widyan et al 2002).

Again, mean densities of the four sites residues were fitted in linear model in relation to diameter (Figure 2 left). As can be observed, the linear model fairly represented the experimental data (R^2 = 0.95). Investigations addressing relations between pressed biomass density and unit diameter are quite few. Kakitis et al (2011) briefly reported unit diameter and length in relation to durability and density. Indeed, investigation of the linear relation between unit diameter and its density worth further investigations.

Effect of pressing moisture content on breaking pressure and density

Table 2 shows effect of SR moisture content on breaking pressure and density. Moisture content had significant effect on breaking pressure at 0.05 level, again its effect on density was not fairly consistent. Discussions on the effect of moisture content on breaking pressure and density are presented as following:

Breaking pressure: Moisture content improved breaking pressure. Biomass moisture content perhaps the most important influential factor in pressing as water has a great impact on lignin plastification (Krizan et al 2009), however, the highest content of lignin is in the kernel part that can be obtained by steaming or high temperature treatment (Rodríguez-Gutiérrez et al 2014). In the current study however, fresh olive residues were used, they attained their normal moisture content and other constituents. Moisture content of the SR were about 50%, it was reduced to the moisture contents (15, 20, 25, 30, and 35%) by drying. Contrary, in other investigations (Al-Widyan, et al 2002; Fennir et al 2014) sun-dried SR were used and the wetting process was made artificially that may affected some properties.

In general applied moisture content range significantly improved unit strength and relations were fairly linear. Indeed, variations are quite expected because of the biomass nature. Referring to the F-K ratio reported in a previous section, the SR used contained kernel part (solid, thick, and large size) which was more than the fine part (soft, thin, and powdery). The later part indeed responsible for absorbing moisture and binding large particles with the effect of pressure. It has been reported that the finer the particle size the higher the breaking pressure or force (Davies and Abolude 2013). Nonetheless, SR contain high percentage of coarse kernel part, with less tendency to bind, and thus binding effect was mainly due to the flesh part.

Moisture content effect was fairly demonstrated by linear relations that exhibited improved unit strength as moisture content increased from 15 to 35%. Mean breaking pressure versus moisture content was fitted to linear model shown in shown in Figure 2. Giving considerations to the nature of the SR biomass, good representation of the moisture content versus breaking pressure was fairly demonstrated (R^2 = 0.77).

Table 2. Effect of moisture content on handling vertical pressure and density

Moisture content (%)	Breaking pressure (kPa)			
	Msalata	Yefren	Subrata	Swani
15	75.60 [c]	155.60 [c]	186.53 [b]	70.13 [d]
20	163.93 [b]	181.20 [c]	154.53 [b]	[c] 97.47
25	240.87 [a]	246.80 [b]	175.80 [b]	150.00 [b]
30	164.00 [b]	270.47 [a]	167.53 [b]	121.33 [b]
35	259.60 [a]	237.13 [b]	254.93 [a]	170.93 [a]
	Unit Density ($g.cm^{-3}$)			
15	0.77 [a]	0.88 [a]	0.90 [a]	0.83 [a]
20	0.79 [a]	0.82 [b]	0.83 [b]	0.80 [b]
25	0.75 [a]	0.79 [b]	0.81 [b]	0.75 [c]
30	0.79 [a]	0.80 [b]	0.84 [b]	0.75 [c]
35	0.70 [a]	0.70 [b]	0.81 [a]	0.76 [c]

Means with the same letter in the same column are insignificant

Density: Moisture content exhibited significant effects on encapsulated unit density of some sites, while others showed no significant effect, densities ranged between 0.7 and 0.9 $g.m^{-3}$. Those were lower than values reported in the literature for oil residues (Al-Widyan, et al 2002), yet in the current study neither grinding SR nor binding agent were used.

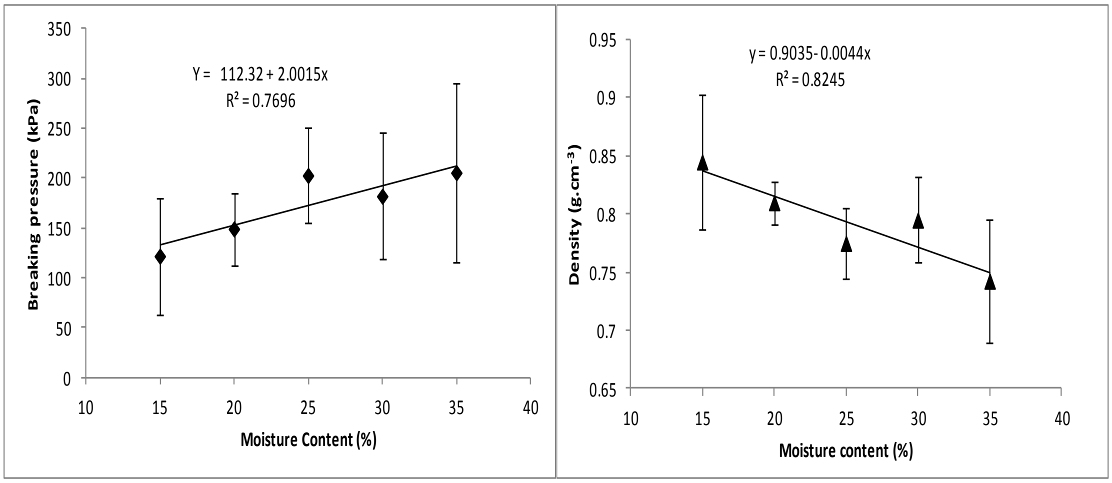

Figure 2. Moisture content (MC) vs breaking pressure (left), MC vs density (right)

Since similarities in SR properties of the four sites was observed, mean densities of the four sites were fitted in a linear model as presented in Figure 2 (right). It can be observed that moisture content exhibited inverse linear correlation with unit density (R^2 = 0.82). Reduction in density may be attributed to evaporation of moisture from units in addition to relaxation effect, giving larger volumes compared with that recorded at pressing. Indeed, larger units had larger relaxation volume, leading to reduced density, while smaller units had smaller relaxation volume, and hence higher density was recorded.

For small units, adding binding materials may produce dense units, such process has been suggested (Wakchaure and Mani 2009). Increased relaxed volume and decreased density have been also reported (Davies and Abolude 2013; Tembe et al 2014). Several authors suggested that solid and stable briquettes made from biomass could be quite difficult to obtain without binding material. The common binders used are mainly starchy such as cassava, sugary such as molasses. In the current study however, it is suspected that oil content may had negative effect on the coherence of olive SR used, since oil content was determined at nearly 6%, yet such effect needs further investigation. Nonetheless, the basic relations between SR fresh moisture content and pressed units strength and density has been demonstrated

CONCLUSIONS

Properties of fresh olive milling SR were determined for four milling sites located in the north western region of Libya wherein most of olive production takes place. Residues were pressed into encapsulated units with 10, 20, 30, 40 and 50 mm in diameter, those were made from SR adjusted to 15, 20, 25, 30, and 35% moisture content (w.b). The following conclusions are made.

- Fresh SR from the four site were quite similar, mean moisture content was about 50%, and contain nearly 6% of oil. Also, LR oil content ranged between 0.6 and 3% for the four sites.
- SR flesh and kernel ratios (F-K) were determined for the four milling sites, they ranged between 1:3 and 1:4.
- Small units (10, 20, and 30mm) in diameter were weak, larger units (40 and 50mm) were fairly stable, effect of unit diameter and moisture on unit strength and density were significant, such effects were governed by linear correlations models.
- The study presented good information on pressing olive milling solid residues for the purpose of reducing bulk density and their possible use for heating.

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