

Specifications of Exhausted Olive Pomace as an Energy Source: A Statistical Approach

Khalid M. Tawarah

Department of Chemistry, Faculty of Science, Yarmouk University, Irbid, Jordan

***Corresponding Author:** Khalid M. Tawarah, Department of Chemistry, Faculty of Science, Yarmouk University, Irbid, Jordan. Email: kmtawarah@outlook.com

Abstract: Samples of exhausted olive pomace were prepared from six slurry and sun-dried agglomerated raw olive pomace samples via Soxhlet hexane extraction treatment. For the case of exhausted olive pomace, the replicates of the measured percentages of carbon, hydrogen, nitrogen, ash, calculated oxygen, and the values of the gross and net calorific values were subjected to a thorough statistical analysis method. The results of the statistical analysis were reported as 95% confidence interval of a population mean. The numerical results of the statistical analysis, on dry basis, are: 49.230 ± 0.817 %, 5.888 ± 0.148 %, 1.397 ± 0.199 %, 1.797 ± 0.176 %, 40.716 ± 0.486 %, 20.069 ± 0.635 MJ/Kg, and 18.709 ± 0.605 MJ/Kg for C, H, N, ash, O, gross calorific value and net calorific value, respectively. Comparison of these results with those of the precursor raw olive pomace indicated that the exhausted pomace is enriched with N and O containing compounds. A cost analysis regarding the use of olive pomace as a cheap substitute for the highly-priced kerosene and diesel in home heating resulted in a saving of 260 USD per ton of combusted exhausted olive pomace.

Keywords: Exhausted olive pomace; Raw olive pomace; Hexane extraction; Olive mill solid residue; Biomass solid fuels

1. INTRODUCTION

The olive oil industry is a seasonal agricultural activity in countries having the climate of the Mediterranean basin. Ripe olive fruits are harvested for two main reasons. They can be processed to yield pitted or un-pitted table olives or pressed to generate olive oil. In all cases there are by-products that need to be managed in a proper way to avoid their negative impacts on the environment. According to previous statistics covering the 2014-2016 period, about 176,000 olive trees were planted in Jordan [1,2]. The main reason for the growing interest in planting olive trees is to produce olive oil and table olives in sufficient amounts to cope with the needs of the growing population in Jordan and to improve the income of many families. For example, in 2014 the self-sufficiency ratios of preserved olives and olive oil were estimated to be 114.3 and 103.4 %, respectively [3]. Recent statistics indicate that the number of olive trees in Jordan was 11.848 million trees [1]. In 2015 and 2014 olive harvest seasons, 156,639 and 118,215 tons of olive fruits were used for oil production, respectively. For the 2015 harvest season, 62.32 % of the pressed fruits was a harvest of the northern region of Jordan, while those harvested from central and southern regions were 26.23 and 11.45 %, respectively. These percentages are in accord with the geographical distribution of the number of trees in these regions [4]. There are four types of olive mills in Jordan. Based on their oil extraction method, the four types are: the traditional press mill, the two-phase mill, the two-and-half-phase mill, and the three-phase mill. The most common type is the three-phase olive mills [5]. Currently, the olive oil production is handled by 128 olive mills with regional distribution of 70%, 22%, and 8% in northern, central, and southern regions of Jordan, respectively. An input output analysis of the olive mills used in Jordan was reported [5]. The quantity and quality of the produced liquid and solid by-products depend on the type of the olive mill used for olive oil extraction [5]. For example, the percentage of moisture content of the fresh solid residue was estimated to be 26.15-28.25, 48.30-52.17, and 54.61-58.99 % for traditional mills, three-phase mills, and two-phase mills, respectively [5]. Other characteristics of the raw olive mill solid residue such as the content of residual fatty material, mineral content (ash), polyphenols, total carbon, and the carbon/ nitrogen ratio were also found to depend on the type of the olive mill [5]. The four types of olive mills also have different rates for generating the

wet solid residue. With rates expressed as % of the processed olive fruits, the two-phase mill generates the highest amount ($\approx 78\%$) while the traditional mill generates the lowest amount ($\approx 50\%$). The two-and-half-phase mill and the three-phase mill nearly generate the same amount of solid residue ($\approx 55\%$) [5]. The various stages involving the extraction of olive oil and the generation of its by-products by the traditional and the three-phase olive mills were described in reference [6] while those pertaining to the two-phase mill were described in reference [7]. In short, extraction of olive oil by using the traditional olive mills entails stone crushing and pressing of the fiber disks that contain the crushed fruits; extraction by means of three-phase mills involves decanter centrifugation yielding olive oil, water, and pomace, while extraction by means of two-phase mills involves decanter centrifugation that yields olive oil and a very wet pomace

In order to comply with the environmental restrictions governing the handling of the produced raw olive pomace, the mill owners need to adopt certain environmentally acceptable waste management practices. However, the high water content of the fresh olive pomace is the main obstacle facing its direct use in useful applications. For example, if the pomace is intended to be used as a solid fuel, its water content should not exceed 15 % [8]. Likewise, the process of extracting the pomace olive oil requires a moisture content of about 8 %. In countries that produce relatively small amounts of olive pomace, such as Jordan, sun drying in summer time seems to be the least expensive option. The sun-dried pomace is usually used for providing heat energy for the mill facilities or sold for residential space heating purposes. The sun-dried pomace is either agglomerated into fuel blocks (usually cylindrical in shape with mass of about one Kg/block after sun drying) or sold in its loose form.

In countries that produce large amounts of olive pomace, such as Spain, the two-phase olive mill pomace (alpeorujo) is a very wet residue. Spreading such wet pomace for sun drying alone is not a practical solution; reducing its moisture content down to 10-15 % by such method is unfeasible. It is known that drying wet olive mills solid residue by conventional methods is a costly process. However, a reasonably less expensive alternative might be the use of the free solar energy. In many olive oil producing countries in the Mediterranean basin, wind energy and solar energy are two promising major natural renewable energy sources. Since the technology of converting solar energy into electricity is well developed, it might be possible to devise techniques for using solar energy in olive pomace drying. Indeed, there are research activities aiming at using solar dryers for reducing the moisture content of the olive mill solid residue [9,10]. By using a greenhouse-type solar dryer, a three-phase fresh olive mill residue, having an initial moisture of $47.3 \pm 0.2\%$, had its moisture content reduced to $24.0 \pm 2.5\%$ within three weeks and attained a moisture value of $9.6 \pm 1.9\%$ on the 56th day of the solar drying treatment [9]. A review article on drying of biomass and a study concerning analysis of energy consumption for a biomass drying process are to be found in references [11] and [12], respectively.

There are some valorization processes aiming at producing value-added products from raw olive pomace. Extracting the residual olive oil (known as olive pomace oil) and the polyphenols content of the raw olive pomace are examples of such valorization processes. The industrial process of extracting olive pomace oil yields a solid by-product known as exhausted olive pomace. The moisture content of such residue is about 10-15% [13]. However, the process of olive pomace oil production requires a drying pretreatment step. In a recent study, a microwave-assisted solvent extraction of the residual oil from wet olive pomace has been suggested as an alternative to the traditional hexane extraction method [14].

In the case of polyphenols extraction, it was reported that the polyphenols content of a wet olive pomace having a moisture of about 65% is 2-8 g/Kg pomace [13]. The interest in extracting polyphenols is largely based on their powerful antioxidant activity. It is known that exposure of edible oils to light and air shortens their shelf life and causes deterioration in their qualities. Since polyphenols are derived from natural sources, they are used in food industry instead of the undesirable synthetic antioxidants. As an example on the use of a polyphenol extract as antioxidant, we refer to the work reported in reference [15]. According to the findings in this reference, we quote: “*The enrichment of refined sunflower oil with oleuropein and hydroxytyrosol rich extract from olive pomace inhibits the deterioration of oil and improves its oxidative stability*”. It should be mentioned that one of the targets of the EU-project Phenolive [13] is to revalorise the waste of the pomace oil producers (exhausted olive pomace) by extracting its content of polyphenolic compounds.

Almost a decade ago, the management of the by-products of the olive oil extraction in Jordan was at its lowest level. Nowadays, all the produced raw olive pomace is consumed for heat generation. There are no factories in Jordan for olive pomace oil production. Also there are no means for recovering value-added products.

The goal of the present work was an identification of the main characteristics of a renewable solid biomass fuel such as exhausted olive pomace. Having a set of specifications in advance, the future marketing of imported or locally-produced exhausted olive pomace will be properly managed.

2. DESCRIPTION OF THE RAW OLIVE POMACE SAMPLES

The raw olive pomace samples (precursor of the exhausted olive pomace samples) considered in the present work are given in Table 1. They were collected over a period of three olive harvest seasons (2009-2011). The samples were derived from the olive residues of five three-phase olive mills located in north Jordan. Samples described as loose form or as compressed fire logs were collected during summer time from the drying yards of the olive mills. Samples directly collected from a mill operation line (described as slurry) were obtained in November of the specified olive harvest season. Each collected sample had its initial mass within the 2-6 Kg mass range. The physical states of samples included mill operation line slurry, loose sun-dried raw pomace, and sun-dried compressed fire logs (agglomerated fuel blocks). The slurry samples were dried in the laboratory at the ambient temperature under convenient ventilation. Before conducting a measurement, the moisture of the samples was brought down to about 10% by prolonged air drying.

Table1. Description of precursor and lipid-depleted olive pomace samples

Precursor samples (raw olive pomace)					Lipid-depleted samples (exhausted olive pomace)
Sample symbol	Collected sample mass/Kg	Olive harvest season	Sample physical state	Sample source	Sample symbol
KT120	2.7	2009	3 briquettes	Al-Kfarat region	KT120h
KT121	2.9	2010	3 briquettes	^a Yarmouk mill, pile top	KT121h
KT122	6.1	2010	5 briquettes	^b Al-khair mill	KT122h
KT123	5.3	2010	5 briquettes	Yarmouk mill, pile bottom	KT123h
RR1	2.5	2011	Slurry	Al-Kfarat region, olive mill line	RR1h
RR2	4.0	2011	Slurry	Irbid region, olive mill line	RR2h

^{a,b}: Two neighboring olive mills near Nuiemeh intersection, Amman-Irbid high way.

3. EXPERIMENTAL PROCEDURE

The laboratory air drying of the slurry samples resulted in a mass loss of about 50 % after one month of air drying. However, prolonged air drying had brought their moisture level down to about 10 %. On the other hand, the non-slurry samples, either in loose form or as compressed fire logs, had been sun-dried in the mill drying yard by the mill work people. Their moisture content was about 10 to 15 % by the time when they were brought to the laboratory. These samples were also air-dried under the ambient laboratory conditions. An amount of about 500 g of each raw olive pomace sample was pulverized for use in the planned measurements. All measurements were carried out on samples in powder form with particle diameter less than 750 µm. The moisture content of a test sample was determined by drying at 110°C for about 15 hours in a conventional drying oven. About two grams of the test sample were used for moisture determination. In most cases, the moisture content was less than 10 %. The oven-dried test samples were stored in capped plastic tubes and placed in a desiccator for use in subsequent measurements. The mineral component of a sample (ash) was determined by

placing about one gram of the test sample in a porcelain crucible followed by burning at 600 °C for 5 hours in a muffle furnace. However, some samples were found to attain complete burning after three hours. At least three runs per sample were carried out for ash determination. The percentages of carbon, hydrogen, and nitrogen (CHN content) were determined using an elemental analyzer (Euro Ea 3000, Perkin Elmer, USA). Duplicate determinations were carried out for the CHN analysis. The heat of combustion of a test sample (gross calorific value, GCV) was measured using an adiabatic oxygen bomb calorimeter (IKA Calorimeter System C 2000 Basic, Japan). About one gram of a sample was used for each calorimetric measurement.

The exhausted (de-oiled) olive pomace samples were prepared from their precursor raw olive pomace samples via soxhlet hexane extraction process. About 3-5 g of a raw olive pomace and 150 ml of hexane (95% n-hexane) were used in each soxhlet extraction experiment. The non-slurry samples had their extractives in the range of 6-10% while the slurry samples had extractives in the 16-21% range. The difference in the amount of the extracted matter between slurry and non-slurry samples might be due to the fact that the slurry samples were not affected by weather conditions as the non-slurry samples before being brought to the laboratory. Other details of the hexane extraction step were given in reference [16].

4. DESCRIPTION OF THE STATISTICAL ANALYSIS PROCEDURES

The data subjected to the statistical analysis procedures were: percentages of ash, nitrogen, hydrogen, carbon, oxygen, and values of GCV, and NCV (net calorific value). The sequence of the steps of the statistical procedure was as follows. Identification of the statistical distribution of the population from which the sample was drawn, calculation of sample arithmetic mean (\bar{X}), calculation of sample standard deviation (S), application of Chauvenet's criterion for rejecting wild data points (outliers), recalculation of sample mean and sample standard deviation for data sets that had points rejected by Chauvenet's criterion (criterion was applied once), calculation of the standard error of sample mean ($S/(\sqrt{n})$), calculation of the mean absolute deviation (MAD) of the sample, finding the t-statistic corresponding to 95% confidence level, and calculating the 95% confidence interval of the population mean (μ). Full details of these statistical steps were given in a previous publication [4]. However, an example on the application of these steps will be given in the results and discussion section.

5. RESULTS AND DISCUSSION

5.1. Detailed Statistical Analysis of the Hydrogen Content of the Exhausted Olive Pomace Samples

As an example of illustrating the details of the statistical procedure followed in the present study, the percentages of the hydrogen content of the exhausted pomace samples were used for this purpose. Based on the arguments given for the statistical analysis of raw olive samples in a previous study [4], the group of exhausted olive pomace samples listed in Table 2 were assumed to represent a grand sample drawn from a certain population. The next step in the statistical analysis was identification of the type of the probability distribution of the population from which the grand sample was drawn. In all cases, a normal probability distribution was first assumed for testing the data at hand. Retaining or rejecting such assumption depends on the features of the Normal Quantile-Quantile plot (Normal Q-Q plot) of a given data set. Construction of a Normal Q-Q plot, which is a scatter plot, requires the generation of a set of values for the P_i - Z_i pair as given in Table 2. The headings of the third, fifth, and sixth columns given in Table 2 are explained as follows. The column with heading "Ranked data" contains the data in an ascending order, the column with heading " P_i " contains the proportion of data points below the data point of rank " I ". The values of P_i listed in Table 2 were calculated according to the following equation [17].

$$P_i = (i-0.5)/n \quad (1)$$

The symbol Z_i , heading of sixth column in Table 2, stands for the value of Z-score of the standard normal probability distribution at a probability equal to P_i . The values of Z_i can be obtained either from the Z-Table or by using Excel command =NORM.S.INV (value of P_i). This set of Z_i values is called rank-based Z-score values and are usually used as values for the X-coordinate of a point in graphing the Normal Q-Q plots as shown in Fig. 1.

The assumption that the hydrogen data were drawn from a population having a normal probability distribution was retained based on the following features of the diagonal line of Fig.1 (a) The correlation coefficient, r , has a value of 0.9713 ($=\sqrt{R^2}$), which is higher than its critical value, $r(n = 14, \alpha = 0.05)$, which is 0.9343, where α is the significance level of the comparison, (b) the slope of the diagonal line is 0.2502%, which is matching the standard deviation of the hydrogen data with value equal to 0.2554%, (c) the intercept of the diagonal line is 5.8880 which is matching the arithmetic mean of the data, \bar{X} , with value of 5.8880%.

Table2. The hydrogen content of the pomace samples used for illustrating the details of the statistical procedure followed in the present study with total number of data points, n , equal to 14 points

Sample ID	%H	Ranked data	Rank	Pi	Zi	D
KT120h	5.410	5.410	1	0.0357	-1.8027	1.871
	5.416	5.416	2	0.1071	-1.2419	1.848
KT121h	6.020	5.692	3	0.1786	-0.9208	0.767
	5.835	5.763	4	0.2500	-0.6745	0.489
KT122h	5.900	5.835	5	0.3214	-0.4637	0.207
	6.134	5.899	6	0.3929	-0.2719	-0.043
KT123h	5.763	5.900	7	0.4643	-0.0896	-0.047
	5.943	5.915	8	0.5357	0.0896	-0.106
RR1h	6.283	5.943	9	0.6071	0.2719	-0.215
	6.148	6.020	10	0.6786	0.4637	-0.517
	5.692	6.074	11	0.7500	0.6745	-0.728
	5.899	6.134	12	0.8214	0.9208	-0.963
RR2h	6.074	6.148	13	0.8929	1.2419	-1.018
	5.915	6.283	14	0.9643	1.8027	-1.546

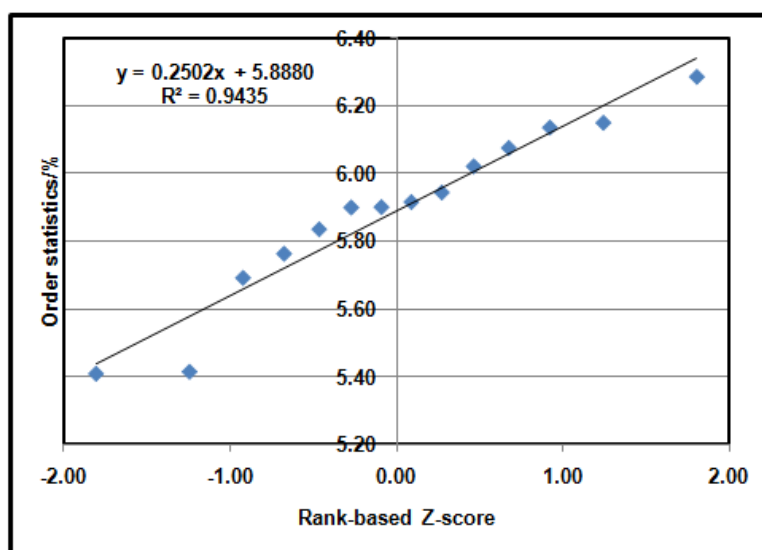


Fig. 1. The Normal Q-Q plot of the hydrogen percentage data

5.2. Statistical Analysis of the Percentages of Carbon, Nitrogen, Ash, Oxygen, and the Values of GCV and NCV of the Exhausted Olive Pomace Samples

When the percentages of carbon, nitrogen, ash, oxygen, and values of gross and net calorific values were subjected to the same statistical procedure applied for the hydrogen data, a normal probability distribution was confirmed for all cases. The normal Q-Q plots of these parameters are given in Figures 2-4. The percentages of oxygen were found by difference.

The correlation of the standard deviation and the arithmetic mean of the experimental data to the slope and intercept of their corresponding Normal Q-Q plots was found to be linear in both cases. The linear correlation of the samples means versus the intercepts of their Normal Q-Q plots gave a correlation coefficient exactly equal to unity. However, the linear correlation of the standard deviations versus slopes of the Normal Q-Q plots gave a correlation coefficient equal to 0.9999 (rounded to 4 decimal places). These findings constitute an evidence for the adequacy of retaining the assumption that all the data regarding the characteristics of the exhausted olive pomace samples were drawn from populations having normal probability distributions.

The net calorific values were calculated from their corresponding experimental values of GCV as J/g on dry basis according to the following formula. [16,18]:

$$NCV = GCV - \{(2442)(\%H/100)(9.01) + (92.7)(\%S) + (42.6)(\%N)\} \quad (2)$$

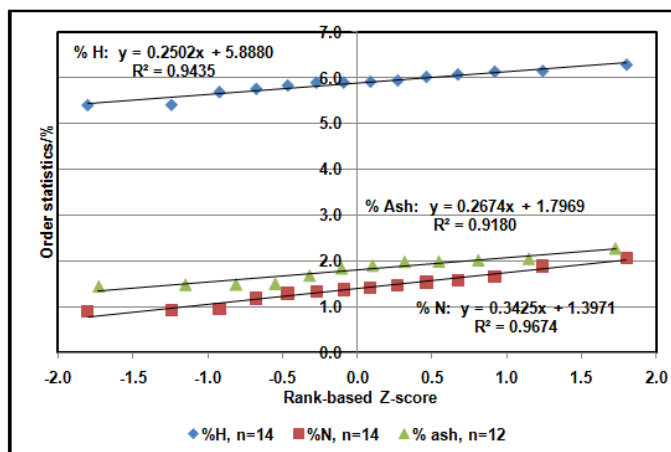


Fig. 2 The Normal Q-Q plots of the hydrogen, ash, and nitrogen percentage data.

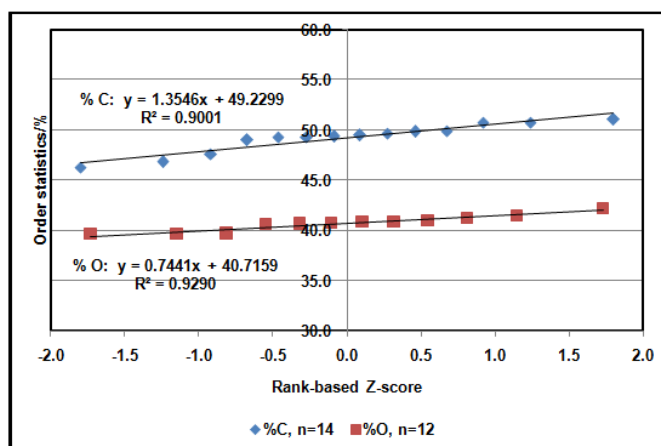


Fig. 3 The Normal Q-Q plots of the carbon and oxygen percentage data.

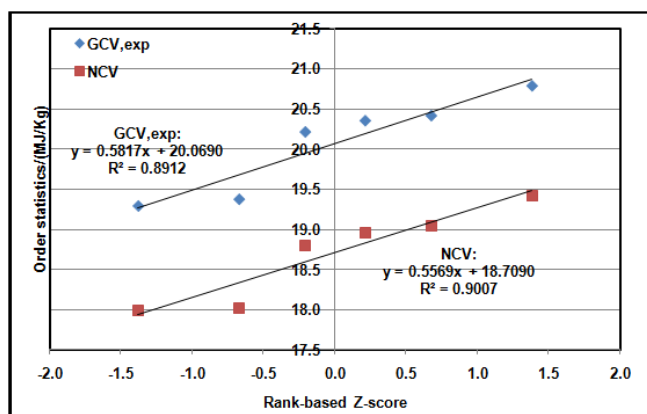


Fig. 4 The Normal Q-Q plots of the gross and net calorific values.

5.3. Checking for Wild Data Points: Chauvenet's Criterion

Before reporting the 95% confidence interval of a certain population mean, the corresponding data set needs to be checked for the possible existence of wild data points (outliers). The most common statistical method used for statistical rejection of wild data points is Chauvenet's criterion [19, 20]. According to Chauvenet's method, for a dataset of n measurements of a sample drawn from a normal probability distribution, one can define a probability band around the sample mean that contains a certain fraction of the measurements. The probability, P , of having "N" data points contained within the bounds of this probability band, is given by

$$P = 1 - 1/2n \tag{3}$$

If one considers one half of the standard normal probability distribution curve (one tail), then $1/4n$ is the probability (P^*) of having data points outside the bounds of the probability band either to the right of the upper bound or to the left of the lower bound (i.e. the $1/2n$ probability of being outside the bounds of the probability band is associated with points equally scattered above and below the bounds of the probability band). To implement this condition, the absolute standardized deviation, D , of each data point, X_i , needs to be calculated and compared with the absolute Z-score associated with the $1/4n$ probability. In the last column of Table 2 are listed the values of the standardized deviations for the hydrogen data. Any data point having an absolute standardized deviation larger than the absolute value of Z-score should be rejected. The absolute value of the Z-score can be determined by using Excel Formula=ABS (NORM.S.INV($1/4n$)) or looked up from the Z-table. For the case of the data shown in Table 2, where $n = 14$, the absolute value of Z score is 2.100. This means that for 14 measurements, the probability band around the mean of the data is enclosed between $Z = +2.100$ and $Z = -2.100$. The absolute standardized deviation, D , of an observation was calculated according to:

$$D = |(X_i - \bar{X})S| \tag{4}$$

In the case of the hydrogen data, all absolute values of D are less than the absolute value of Z . This means that there were no outliers in the hydrogen data. The same finding was obtained for the rest of the other parameters.

5.4. Evaluation of the Confidence Interval of a Population Mean

A confidence interval gives an estimated range of values which is likely to include an unknown population parameter. The estimated range can be calculated from a given set of sample data [21]. The evaluation of the confidence interval of the population mean (μ) requires knowledge of a specified confidence level, the standard error of the sample mean, and a distribution statistic. The Normal distribution and Student's T- distribution are usually used; the choice depends on the sample size [22]. In the present study, a t-statistic was used in all cases. The following expression was used for evaluating the confidence interval of the population mean based on a Student's T-distribution [23].

$$\mu = \bar{X} \pm (t)(S)/\sqrt{n} \tag{5}$$

In Equation (5) " n " is the number of data points remaining after application of Chauvenet's criterion, (S/\sqrt{n}) is the standard error of the estimate of the mean [24], " t " is a statistic obtained from a t-table at 95% confidence level and $n-1$ degrees of freedom, or by using Excel command = CONFIDENCE.T (alpha; standard deviation; size). The product $(t)(S/\sqrt{n})$ determines the bounds of the confidence interval.

Table 3 contains the results of the full statistical analysis concerning the specifications of the exhausted olive pomace on dry basis. The numerical data displayed in Tale 3 are the outcome of the combined steps of the applied statistical procedure which included the normality testing, application of Chauvenet's criterion, and the determination of the 95% confidence interval of the population mean.

Table3. Final results of the statistical analysis regarding the specifications of exhausted olive pomace on dry basis

Analysis	Ash %	N %	H %	C %	O %	GCV/(MJ/Kg)	NCV/(MJ/Kg)
Initial number of data points, n	12	14	14	14	12	6	6
Statistic of normality test, r ($\sqrt{R^2}$)	0.9581	0.9836	0.9713	0.9487	0.9638	0.9440	0.9491
Critical value of r (0.05, n)	0.9267	0.9343	0.9343	0.9343	0.9267	0.8880	0.8880
Outcome of normality test	O.K	O.K	O.K	O.K	O.K	O.K	O.K
Number of rejected data points	0	0	0	0	0	0	0
Final number of data points	12	14	14	14	12	6	6
Sample arithmetic mean, \bar{X}	1.7969	1.3970	5.8880	49.2299	40.7159	20.0690	18.7090
Sample standard deviation, S	0.2764	0.3453	0.2554	1.4159	0.7646	0.6052	0.5764

Percentage relative standard deviation, $(S/\bar{X}) * 100/\%$	15.3820	24.7172	4.3376	2.8761	1.8779	3.0156	3.0809
Standard error of sample mean, (S/\sqrt{n})	0.07979	0.09229	0.06826	0.37842	0.2207	0.2471	0.2353
Mean absolute deviation, MAD	0.2368	0.2614	0.1891	0.9593	0.5529	0.4940	0.4643
t-statistic, 95% confidence level	2.20	2.16	2.16	2.16	2.20	2.57	2.57
95% confidence interval of population mean, $\mu = (\bar{X} \pm t * S/\sqrt{n})$	1.7969 ± 0.1756	1.3970 ± 0.1994	5.8880 ± 0.1475	49.2299 ± 0.8174	40.7159 ± 0.4858	20.0690 ± 0.6351	18.7090 ± 0.6049

6. AN OVERVIEW OF THE EFFECT OF HEXANE EXTRACTION ON THE CHARACTERISTICS OF OLIVE POMACE

Industrially speaking, the purpose of subjecting the raw olive pomace to the hexane extraction step is to recover the residual amount of olive oil associated with the solid parts of the pressed olive fruits. The outcome of the extraction is exhausted pomace and olive pomace oil. Hexane is a nonpolar low-boiling solvent that can solubilize the nonpolar lipid material from the olive pomace matrix. The hexane extractives contain the lipid material (waxes, oils) and other nonpolar ingredients. In Table 4, a sample-per-sample comparison was made. A column heading of type KT120h/KT120 indicates a ratio whose numerator is a property of the exhausted pomace while the denominator is a property of the raw pomace. It should be mentioned that the statistical means of raw olive pomace given in the third row of Table 5 were taken from previous results [4].

The ratios given in Tale 4 indicate that converting raw olive pomace into its lipid-depleted form via hexane extraction, generates a solid residue (exhausted olive pomace) with lower heating values. Also the carbon, hydrogen, and mineral contents (ash) had been reduced. The general trend in the nitrogen and oxygen contents indicate an enrichment of compounds containing these elements. This enrichment is likely to be due to the insolubility in hexane of polar compounds such as amino acids, polyphenols, hemicellulose, cellulose, and lignin. The same trend is also maintained when the ratios were derived from results of the statistical analysis as evident in the fourth row of Table 5. Comparison of the ratios given in the last two rows of Table 5 leads to the conclusion that the main differences between statistical and mean-based calculations are in the heats of combustion and the hydrogen and ash contents. In the mean-based calculations method, the first step was to get the average of the replicates of each individual sample, then the overall average used in calculating the ratios was calculated from these individual averages with statistical weights being taken care of. However, in the statistics-based method, all individual measurements of a certain property were taken collectively. A check for the possible existence of wild data points was performed as in the case of the hydrogen data displayed in Table 2. In a previous study concerning raw olive pomace [4], one outlier measurement was detected in ash data (n = 48), GCV data (n = 19), and NCV data (n = 15). Such an outliers check is not possible in a mean-based method. The ash ratios given in Table 5 are examples on these arguments. The low value of the statistics-based ratio of ash percentage (0.585) is due to the rejection of samples KT120h and KT122h from the statistical mean of the exhausted pomace (1.797). Their precursors, KT120 and KT122 were also rejected on statistical grounds in a previous study [4] because of their unusually high ash content. However, ash percentages of KT120 and KT122 were included in the mean-based ratio in this study. The final point to be discussed in this section is the financial profitability of using exhausted olive pomace as a substitute for liquid fossil fuels (kerosene and diesel) in home heating in Jordan. Based on the cost analysis procedure regarding the use of raw olive pomace as a substitute fuel [4], it was found that about 260 USD/ton can be saved in home heating for the same amount of heat obtainable from liquid fuels. This saving is based on the 2019 winter prices. Prices were 120 USD/ton, 0.79 USD/L, and 0.79 USD/L for olive pomace (10% moisture), diesel, and kerosene, respectively. It should be emphasized that in real life applications, differences in burning efficiencies might change these savings. The European requirements [8] consider a biomass material suitable for use as a solid fuel if its ash is less than 4% ant its NCV is higher than 16 MJ/Kg. The exhausted olive pomace considered in the present work fulfills these requirements since its %ash is 1.8% and its NCV is 18.71 MJ/Kg as given in Tale 5. For the sake of

comparison with other sources of information, the data given in Table 6 were collected. Except for the ash content, all other characteristics are in good agreement.

Table4. Non-statistical, mean-based ratios data for paired samples of exhausted and raw olive pomace on dry basis

Paired samples	KT120h/KT120	KT121h/KT121	KT122h/KT122	KT123h/KT123	RR1h/RR1	RR2h/RR2
% N ratio	1.145	1.011	0.941	1.149	1.393	1.615
% C ratio	0.957	0.955	0.998	0.959	0.950	0.924
% H ratio	0.865	0.912	1.009	0.987	0.890	0.844
% Ash ratio	0.779	0.837	0.709	0.671	0.683	0.591
% O ratio	1.147	1.087	1.093	1.077	1.102	1.160
GCV ratio	0.959	0.938	1.134	0.944	0.895	0.863
NCV ratio	0.965	0.939	1.145	0.941	0.895	0.863

Table5. Data for comparing statistics-based ratios with mean-based ratios

Category	%N	%C	%H	%Ash	%O	GCV/(MJ/Kg)	NCV/(MJ/kg)
Statistical mean, Exhausted pomace	1.397	49.230	5.888	1.797	40.716	20.069	18.709
Statistical mean, Raw pomace	1.079	52.322	6.787	3.070	35.271	22.269	20.542
Statistics-based ratio	1.295	0.941	0.868	0.585	1.154	0.901	0.911
Mean-based ratio	1.209	0.957	0.918	0.712	1.111	0.955	0.958

Table6. Characteristics of a European exhausted olive pomace and the exhausted olive pomace prepared in the present study

Property	Phenolive project [13]	Present work
NCV/(MJ/Kg)	18.983	18.709
%Ash	4.7	1.797
%C	49.4	49.230
%H	5.9	5.888
%N	1.0	1.397
%O	38.8	40.716

7. CONCLUSIONS

The present work provided unprecedented statistics-based typical specifications for exhausted olive pomace that can be implemented for controlling the possible marketing of this material in Jordan.

Because of its reasonable net calorific value, the utilization of exhausted olive pomace for home heating should be encouraged to avoid the use of the highly-priced kerosene and diesel fuels.

Subjecting raw olive pomace to the hexane extraction process resulted in a solid residue enriched with nitrogen and oxygen compounds. However, the gross and net calorific values, and the contents of carbon, hydrogen, and minerals of the solid residue were lower than those of raw olive pomace.

A cost analysis regarding fuels used for space heating in Jordan had shown that about 260 USD can be saved per each ton of combusted exhausted olive pomace instead of liquid fossil fuels giving the same amount of heat. This amount of saving indicates the benefits of using the exhausted pomace as a substitute for the highly priced liquid fossil fuels.

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