**ORIGINAL RESEARCH** 

# Characterization of olive mill wastewater in three climatic zones in the North of Jordan

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#### Abstract

**Purpose** Olive mill wastewater (OMW) is annually generated in large amounts in Jordan without any treatment creating major environmental and public health issues. The objective of this study is to determine changes in OMW characteristics under three climatic zones in the North of Jordan during two harvest seasons.

**Method** OMW samples were obtained from 10 olive mills, representing three climatic zones (arid, semi-arid, and semi-arid to sub-humid) during November 2017 and November 2018. Physicochemical characterization and multivariate analysis were performed.

**Results** OMW is characterized by acidic conditions (pH< 5.0), high electrical conductivity (EC) (>7 dS/m), and high total phenols (2700 mg/L) and organic loads (chemical oxygen demand [COD] 41340 mg/L). OMW in the sub-humid climate contained higher total phenols, COD, EC, Ca, and K than other climates. Principal component analysis (PCA) showed that total phenols had high loadings in favour with Ca, and TSS in arid, total nitrogen in the semi-arid, and COD in the sub-humid climates.

**Conclusion** OMW properties were markedly affected by the climate. PCA showed that climate mainly affected the organic loading of extracted components. Overall, treatment of OMW is highly recommended before any use.

Keywords Olive mill wastewater (OMW), OMW characterization, Climate, Principal component analysis

# Introduction

Olive trees cover about three-fourths of the total tree planted areas in Jordan. Most olive oil is produced in the North of Jordan (e.g Ajloun, Jerash, and Irbid) accounting for nearly 32% of total olive oil production. The olive industry generates the large quantities of solid (olive pomace locally known as Jift) and liquid waste (olive mill wastewater -OMW), locally called Zibar (Albalasmeh et al. 2019; Khdair and Abu-Rumman 2020). It is estimated that about 5500 tons of Jift and 0.2 million cubic meters of OMW is produced annually in Jordan. About 70% of total olive mills are found in the North parts of Jordan (Ministry of Agriculture 2016).

The composition of OMW by weight is 83-94% water, 4-16% organic compounds, and 0.4-2.5% mineral salts (Magdich et al. 2020). OMW is a dark acidic liquid containing moderately high levels of organic compounds (e.g. sugars, tannins, pectin organic acids and phenols). OMW also contains large amounts of phytonutrients (e.g. Na, K, P, and Ca) that could be considered as a low-cost water source and could be recycled and used as a soil fertilizer (Mohawesh et al. 2019, 2020). For the waste management, the Ministry of Environment in Jordan has designated three dumpsites in Jordan (Ayoub et al. 2018). However, these sites are lacking proper measures to prevent any pollution resulting from OMW storage/disposal. Jordan also lacks any central treatment plants or economic feasible treatment solutions for OMW, therefore, the disposal and treatment of this liquid waste is the main problem of the Jordanian olive oil industry (Khdair and Abu-Rumman 2020). In Jordan, current environmental regulations restrict discharging of OMW into the environment, however, no emergency plan is present to avoid the il-

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legal discharge of OMW (Halalsheh et al. 2021). The majority of soils in Jordan are arid characterized by poor organic matter content with vigorous degradation processes in place. The use of OMW could be recommended as a low cost soil amendment to replace or reduce the use of chemical fertilizers, restore deficit soil carbon, enhance soil fertility, and improve crop productivity (Tundis et al. 2020), and should be considered as temporary solution for OMW disposal in a water scarce countries like Jordan (Dutournié et al. 2019; Khdair and Abu-Rumman 2020). Currently, there are no suitable legislations in Jordan regarding controlled application of OMW to the soil surface (Khdair et al. 2019). Several studies showed that OMW spreading (Ayoub et al. 2018; Albalasmeh et al. 2019; Mohawesh et al. 2020) increased soil aggregate stability, improved soil fertility, and improved crop growth. Similar results in other Mediterranean countries were reported (Mahmoud et al. 2012; Mekki et al. 2013; Zema et al. 2019).

The chemical composition of OMW is highly variable depending on many factors such as climatic conditions, cultivar type, fruit maturity, and extraction method (Khdair et al. 2019; Benamar et al. 2020; Khdair and Abu-Rumman 2020). Characterization of OMW is important to estimate pollution loads, recommended application rates and mass of nutrients delivered to soil, and assessment of environmental impacts on soil and plant properties (Jones 2002; López-Pieñiro et al. 2006). Furthermore, the characterization of the spatial variation of OMW under different climate zones supplies indispensable tool that provides scientists, engineers, and environmental managers with relevant data for classifying OMW for future planning of viable and efficient treatment technologies, developing waste management plans including safe disposal for sustainable use of OMW (Khdair and Abu-Rumman 2020).

The aims of this research are to examine physicochemical properties of OMW samples for two consecutive seasons (2017 and 2018), to assess variability of OMW properties, and to investigate the effect of three climate zones (sub-humid, semi-arid, and arid) North of Jordan on physicochemical properties of OMW by using multivariate analysis.

# **Materials and methods**

#### Study area

# Geography and climate of the study area

The study area included four governorates in the North parts of Jordan: Irbid (1570 km<sup>2</sup>), Ajloun (420 km<sup>2</sup>), Jerash (410 km<sup>2</sup>), and Mafraq (26,550 km<sup>2</sup>). The climate is semi-arid in Irbid, semi-arid to sub-humid in Ajloun and Jerash, and arid in Mafraq. The climate of Irbid, Jerash, and Ajloun is characterized by hot dry summers, and cold rainy winters, while Mafraq is arid





Fig. 1 Olive press (mill) locations (circles)

with hot summers, mild winters and low precipitation. Jerash and Ajloun are mountainous areas where most of winter precipitation is in the form of rain and snow. In addition, severe weather conditions of frost during winter and early spring occur in both areas whereas summer climate can be described semi-arid to sub-humid (in the preceding discussion it was labelled as sub-humid). Average annual rainfall in Irbid is (430 mm), Ajloun (650 mm), Jerash (580 mm), and less than 150 mm in Mafraq. Due to low precipitation; olive trees in Mafraq area are frequently irrigated and organic and chemical fertilizers are applied to soil, while in the other areas olive trees are rain fed only. Locations of olive mill sampling sites are shown below (Fig. 1).

#### Olive mill wastewater sampling and characterization

Three fresh OMW samples were collected in duplicates from each of the 10 olive mills (Table 1) during harvest seasons in November of 2017 and 2018, and a composite of the duplicates were made into one sample [n = 10mills × 3 samples × 2 years = 60]. Collected samples were stored in a black plastic container at 4° C and left to settle for one month before analysis and average of the duplicates was used in statistical analysis. Locations and type of phase extraction in studied olive mills are shown in Table 1.

Changes in OMW physico-chemical properties were studied under three climatic zones. Ajloun and Jerash have sub-humid to semi-arid climate, while Irbid is a semi-arid, and Mafraq an arid climate. According to the USDA order/great group classification (Lucke et al. 2013); soils of Ajloun and Jerash are Inceptisols (*Xerochrept*), Irbid Vertisols (*Chromexerert*), and Mafraq Aridisols (*Calciorthid*). Main olive cultivars are Nabaly Balady in Irbid, Souri, Romy, and Kanabisi in Ajloun and Nabbaly Muhassan and K-18 in Mafraq. Olive trees in Mafraq are complementary irrigated while cultivation of olive trees in the other governorates are rain fed. Box-and-whisker plots were used to assess the variation in physico-chemical characteristics of OMW under three different climatic zones.

Physicochemical properties were determined as the following: pH and electrical conductivity (EC) were measured using pH-EC meter, total phenols by Folin-Ciocalteau method (Vinet and Zhedanov 2011), chemical oxygen demand (COD) using a spectrophotometer at wavelength absorbance 605 nm according to the standard methods of examination of water and wastewater of the American Public Health Association (APHA) (APHA 2000), total suspended solids (TSS) by the gravimetric method, total nitrogen (TN) by the Kjeldahl method (Bremner and Mulvaney 2015), total phosphorus (TP) using spectrophotometer at a wavelength of 880 nm (Olsen and Sommers 1983), Na, Ca, and K using flame photometry (Chapman and Pratt 1982).

#### **Statistical analysis**

Descriptive statistics (mean, standard deviation (SD), coefficient of variation (cv), minimum, maximum, skewness, first or lower quartile (Q1), median (Q2), and upper or third quartile (Q3) were used to analyze related OMW properties. The Kolmogorov-Smirnov (K–S) with Lilliefors correction and Shapiro-Wilk were applied to test the normal distribution of data using Sigma plot 12.3. Pearson correlation was conducted to determine the correlation between measured OMW properties in the three studied climates.

Principal component analysis (PCA) was conducted to find loadings between measured parameters under three climates. PCA with a Varimax rotation was performed using IBM SPSS Statistics 25 to achieve individual component loadings. The KMO (Kaiser-Meyer-Olkin) criterion was considered when reaching the best result of PCA. Data were z-transformed before calculating the PCA.

Table 1 Mill locations and type of phase extraction

Governorate	Number of mills	Type of extraction	OMW produced (m <sup>3</sup> /year)*
Ajloun	2	3-phase	27000
Irbid	5	3-phase	76300
Mafraq	2	3-phase	8900
Jerash	1	3-phase	20130

\*Column 4 represents total OMW produced in each governorate (m3/year).

ANOVA test was applied to determine whether the mean values of levels differ among the three climate zones, and Kruskal–Wallis test for non-normal distributed data. One Way Analysis of variance and one way repeated measures analysis of variance using Fisher's LSD, Duncan's, and Bonferroni t-test were used to test significance differences between measured parameters under different climates.

# **Results and discussion**

# OMW characterization under three different climates

Descriptive statistics of physico-chemical properties of OMW under three studied climates are shown in Table 2a, b, and c. All OMW samples are acidic (pH < 5.0). Average EC was  $9.11 \pm 0.75$ ,  $7.27 \pm 3.20$ ,  $6.52 \pm 2.55$ dS/m, COD 46795  $\pm$  16208, 38344  $\pm$  23076, 38204  $\pm$  17391 mg/L, and total phenols 3478  $\pm$  916, 2419  $\pm$ 1434, 2447  $\pm$  1293 mg/L, for sub-humid, semi-arid, and arid climates (SH, SA, and A), respectively. Excluding pH; EC had the lowest coefficient of variation (cv) in SH and SA, while Ca had the highest cv in SH and A climates. Among all tested parameters, TSS had the highest skewness, while TP had the lowest one. In Jordan, OMW is considered as industrial wastewater that needs to be treated before any use. For agricultural purposes, OMW has to meet Jordanian standards for industrial wastewater use JS 202/2007. The following limits were proposed (mg/L) for irrigation purposes: COD (100), total phenols (0.002), TN (45), and Na (230). Apparently, almost complete removal of COD and phenols are demanded in the case of OMW use for agricultural production (Halalsheh et al. 2021).

As shown in Table 2a, b, and c and Fig. 2a, b, OMW characteristics varied according to the climate. The OMW originating from the SH (mountainous areas) have higher total phenolic (P < 0.05), COD, EC, Ca, and K and lower Na and TN (P < 0.05) as compared to SA and A (plain areas). One-way analysis of variance and one-way repeated measures analysis of variance (Fisher's LSD, Duncan's, and Bonferroni t-test) showed that SH had significantly (P < 0.05) higher Ca, EC, and lower pH than SA climate, and no significant differences between all climates were observed for COD, TSS, TN, and K. OMW is a suspension that mainly consists on a weight basis of 80–92% water, 3–15% organic matter (e.g. oils and fats, carbohydrates, lipids, pectin, organic acids, sugars, mucilage, polysaccharides, phe-

nols, tannins and lignin), and 0.50-2% mineral content (Paraskeva and Diamadopoulos 2006; Rahmanian et al. 2014; Khdair and Abu-Rumman 2020). The dry matter in OMW, depending on the extraction method, ranges between 9.5-161.2 g/L, typical total solids (19-75 g/L), suspended solids (0.7-26 g/L), and volatile solids (17-68 g/L) (Hung et al. 2005). The majority of mills in Jordan are three phase type (71%), while the two phases (22%) and traditional ones (7%) are less common. Wastewater produced per ton of olives (L/ton) varies depending on extraction method. For example, average OMW produced per ton of olives (L/ton) were 335, 388, and 1065, while the total volumes of OMW produced were 8025, 22892, and 143775 m3/season, for traditional, two and three phases, respectively (Khdair et al. 2019). In this study, OMW from three phase mill types were characterized after being settled for a month to allow for big suspended particles to precipitate and to mimic actual conditions in storage tanks.

During the olive oil extraction, water-soluble compounds are differentially partitioned between water (OMW) and oil and the major fraction of these compounds shifts from olive pulp to OMW. It is therefore hypothesized that OMW characteristics resemble the chemical and nutritional content of olive fruits. Furthermore, factors affecting olive fruit quality can be postulated to be the same factors affecting OMW characteristics (Dermeche et al. 2013).

OMW contains different phenolic compounds and the most abundant are tyrosol, hydroxytyrosol, and oleuropein. Many studies reported that OMW phenolic contents were affected by the climate and geographical conditions (Allouche et al. 2004; Obied et al. 2005; Leouifoudi et al. 2014). For example, OMW from mountainous fields in Morocco contained higher levels of phenolic compounds as compared to the central and plain areas (Leouifoudi et al. 2014). Amaral et al. (2008) reported a strong significant positive correlation between phenolic content and COD of OMW, and it is therefore could be postulated that factors affecting both properties (organic load) could be the same. Furthermore, Visioli et al. (2002) reported that a major polyphenol compound (Oleuropein) was not identified in OMW due to its degradation to another form after late olive harvest (mature fruits). The same authors (Visioli et al. 2002) also reported that OMW coming from milling of small fruits contained higher Oleuropein than that coming from milling of large fruit varieties.

Table 2a Descrip	tive statistics	of OMW in	sub-humid c	limate							
Parameter	Unit	Avg	SD	CV	Min	Max	Range	Skew	QI	Q2	Q3
hd		4.42	0.18	0.04	4.26	4.75	0.49	1.07	4.29	4.33	4.49
EC	(qS/m)	9.11	0.75	0.08	8.37	10.18	1.81	0.47	8.54	8.59	9.89
Са	(mg/L)	81.07	43.15	0.53	32.97	128.99	96.02	-0.03	42.78	82.65	122.12
Na	(mg/L)	91.18	13.44	0.15	67.69	102.88	35.19	-1.19	88.93	96.74	98.84
$COD (*10^{-3})$	(mg/L)	46.79	16.21	0.35	14.63	70.33	55.70	-0.72	36.76	51.33	56.98
ТР	(mg/L)	09.66	42.75	0.43	43.22	166.1	122.87	0.31	59.46	92.07	134.1
NT	(mg/L)	308.2	139.1	0.45	154.1	567.4	413.3	0.69	217.1	238.1	395.8
<b>T-Phenols</b>	(mg/L)	3487	916.0	0.26	1911	4710	2799	-0.28	2953	3352	4160
TSS	(mg/L)	5180	3612	0.70	1040	10920	9880	0.57	2070	4000	7840
K	(mg/L)	4999	1341	0.27	3190	6931	3741	-0.09	3749	5304	5916
Table 2b Descrip	tive statistics	of OMW in	semi-arid cli	mate							
Parameter	Unit	Avg	SD	CV	Min	Max	Range	Skew	01	Q2	03
Hd		4.58	0.24	0.05	4.20	4.94	0.74	-0.03	4.42	4.56	4.79
EC	(qS/m)	7.27	3.20	0.44	1.79	11.31	9.52	-0.65	6.06	7.65	9.42
Ca	(mg/L)	70.87	37.95	0.54	28.94	167.25	138.31	1.22	41.27	61.65	90.48
Na	(mg/L)	121.49	50.34	0.41	61.48	231.62	170.14	0.83	88.24	113.98	147.30
$COD (*10^{-3})$	(mg/L)	38.34	23.08	0.60	3.54	87.58	84.04	0.50	23.09	33.63	56.76
TP	(mg/L)	141.2	93.44	0.66	41.1	341.2	300.1	0.78	76.92	95.37	239.5
NT	(mg/L)	390.4	261.4	0.67	28.00	924.7	896.7	0.44	218.9	367.7	535.9
T-Phenols	(mg/L)	2419	1434	0.59	115.96	4437	4321	-0.25	831.25	2818	3131
SSL	(mg/L)	4430	4662	1.05	120.0	17120	17000	1.63	1340	3320	4300
K	(mg/L)	4260	2381	0.56	375.94	7440	7065	-0.39	2730	4357	6315

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Parameter	Unit	Avg	SD	CV	Min	Max	Range	Skew	Q1	Q2	<b>Q</b> 3
Hd		4.45	0.25	0.06	4.19	4.83	0.64	0.67	4.25	4.39	4.58
EC	(qS/m)	6.52	2.55	0.39	0.04	10.12	10.08	-1.2	5.7	6.5	7.2
Ca	(mg/L)	71.2	47.0	0.66	36.0	153.5	117.5	1.2	42.3	45.3	83.8
Na	(mg/L)	140.1	18.4	0.13	121.0	168.6	47.6	0.62	126.2	133.4	155.9
COD (*10 <sup>-3</sup> )	(mg/L)	38.2	17.4	0.46	15.1	61.8	46.7	0.09	25.5	34.0	55.2
TP	(mg/L)	73.3	19.2	0.26	35.5	111.8	76.3	0.06	64.2	73.2	82.5
TN	(mg/L)	418.0	142.5	0.34	266.2	644.4	378.2	0.71	301.2	385.3	481.6
<b>T-Phenols</b>	(mg/L)	2447	1293	0.53	1125.53	4592	3466	0.81	1480	2083	3016
TSS	(mg/L)	5926	3263	0.55	1880	13280	11400	1.08	3670	5120	7660
K	(mg/L)	3834	924.5	0.24	3028	5426	2398	1.18	3281	3478	4004
EC: Electrical condu standard deviation. C	ctivity (dS/m), [' 'V: coefficient of	TN: total nitrog variation, May	gen, TSS: total su v: maximum valv	uspended solid ue, Min: minin	s, TP: total phosp num value, Skew	phorus, T-pheno r: skewness, O1	ols: total phenols, ( : lower quartile, m	OD: chemical iddle quartile ()	oxygen demand median), O3: ur	1, (all in mg/L)]	. Avg: average, SD

Several reports suggest that the climate and the geographical conditions have an impact on the chemical composition of olive fruit as well as OMW. For example, Anastasiou et al. (2012) reported negative correlations between rainfall and OMW characteristics (e.g. phenols, COD, and EC). Ranalli et al. (1999) used altitude as an indirect parameter related to climate, and reported that some sterols, triterpenic alcohols and hydrocarbons were correlated negatively with altitude. Osman et al. (1994) observed that the oils from 100 m altitude were higher in phenols and unsaturated fatty acids, as well as had a higher oxidative stability and free acidity compared to 400 m elevation. Ocakoglu et al. (2009) found that phenolic profiles were influenced mainly by the cumulative rainfall. Similar results by Vinha et al. (2005) showed that geographical origin affected phenolic profiles. Furthermore, Ouni et al. (2012) found that altitude positively correlated to phenolic composition and fatty acid content of olive fruits. Compared to the Mediterranean climate; higher annual rainfall and lower temperatures (wetter and cooler climate) in China resulted in lower percentages of unsaturated fatty acids and oil content (Cheng et al. 2017).

Amaral et al. (2008) studied OMW microbiological and physicochemical characteristics under two different malaxation periods. The first involved collecting OMW after immediate washing and processing and the second was after storage of processed mill olive fruits for various periods of time inside the milling machine. Significant higher yeasts and heterotroph counts were observed in the second scheme. Relative high values of K, P, and reduced and oxidized N forms were observed in general. OWW collected during the second campaign was richer in K and P due to the solubilization of these compounds in the waters left in deposit for some time.

Mafraq has the highest Na and TN and this could be attributed to fertilizer and organic matter use. As described earlier, in Mafraq (arid climate), N fertilizers are applied during irrigation of olive trees, while in Ajloun, Jerash (SH), and Irbid (SA) olive trees are rain fed. Several studies reported that nitrogen fertilization of olive trees increased N accumulation in olive fruits and decreased polyphenol content (Fernández-Escobar et al. 2006; Dag et al. 2009; Tekaya et al. 2013). Furthermore, Greven et al. (2009) reported a decrease in phenol content with increasing irrigation level. Therefore, variation in OMW Irrigated and fertilized olive grove also induce variability in OMW properties



Fig. 2a Spatial changes of tested parameters under three different climates

(Allouche et al. 2004; Obied et al. 2005, 2008; Morillo et al. 2009; Dermeche et al. 2013).

The physico-chemical and biological characteristics of OMW are expected to be a reflection of olive fruits properties (Hanifi and Hadrami 2008; Obied et al. 2008). In SH areas higher rainfall may solubilize more salts (EC, Ca, and K) into the soil solution and higher uptake and translocation into olive fruits could be expected, therefore this may explain the higher salt contents in OMW in Ajloun and Jerash areas (SH).



Fig. 2b Spatial changes of tested parameters under three different climates

Elabdouni et al. (2020) showed that variability of physico-chemical properties of OMW in the province of Al-Hoceima, Morocco was mainly due to extraction method, olive cultivar, degree of maturation, cultivation systems, and olive storage prior to extraction. Gómez-Rico et al. (2008) and Menz and Vriesekoop (2010) showed that fruit maturation affected the composition of volatile organic and phenolic compounds in the olive fruit and therefore this could be postulated to have a direct effect on OMW properties. Irrigation of olive trees was reported to decrease TP levels in fruits (Jose Motilva et al. 2000), therefore a decrease in the phosphorus levels in OMW could be expected.

Obied et al. (2008) showed that olive cultivar was the most dominant factor causing variation in the phenolic content of OMW. Both organic and inorganic composition of OMW are expected to be affected by fruit quality (their level in the fruit water), stage and amount of added water during extraction, olive paste malaxation time and temperature, and the extraction efficiency of fruit solutes and particulate matter (TSS) into the wastewater phase (Aviani et al. 2012). Frost during winter was reported to significantly increase phenolic levels in olive fruits (Morelló et al. 2006). Phenolic compounds, produced by olive trees are soluble in water, and therefore washed away with OMW (Pierantozzi et al. 2012). These findings could explain the higher levels of T-phenols in OMW of Ajloun and Jerash (sub-humid), both areas are known to suffer from frosting days during winter. Moreover, Fernández-Escobar et al. (2015) reported that rain fed olive trees annually removed higher amounts of N, K, and Ca and lower phosphorus and magnesium from soil. Our results show that OMW contained higher N and lower P levels in rain fed (arid and semi-arid) as compared to SH areas.

Table 3 Avera	age monthly	y temperatu	ıres (C°), rain	ıfall (mm), and l	long-term ra	uinfall deptl	n (mm) in thi	ee climatic zon	es (studied a	area)		
		S	ub-humid				emi-arid				Arid	
	Тел	np (°C)	Rain	fall (mm)	Ter	np (°C)	Rain	fall (mm)	Теі	() dm	Rair	fall (mm)
	Min	Max	Study period	30 years record	Min	Max	Study period	30 years record	Min	Max	Study period	30 years record
Jan	3.2	8.7	123	166	5	13	91.3	101.45	7.5	14.5	24.3	26.8
Feb	3.6	10.1	114.4	120	Г	15	97.4	94.85	6	18	22.7	35.0
Mar	5.8	12.9	103	77	6	18	60.3	73.9	11	21.5	7.1	23.0
Apr	8.9	17.9	32.2	31	12	23	32	28.85	13.5	25.5	37.8	14.3
May	12.8	23	9	8	16	28	13.2	9.95	18.5	30	2.4	7.9
Jun	15	25.5	0	0	19	32	2.1	1.35	21.5	32.5	0.0	0.8
Jul	16.5	26.7	0	0	20	34	0.2	0.1	23.5	35.5	0.1	0.0
Aug	16.3	27.2	0	0	21	34	0.5	0.25	23.5	34	0.1	0.1
Sep	15.6	26	0	2	19	32	2.3	1.5	23	33	0.2	3.4
Oct	13.6	22.6	16	19	17	27	21.9	17.55	19.5	27.5	7.4	10.5
Nov	8.8	15.7	09	65	12	20	34.4	42.4	15	22	2.4	12.5
Dec	4.3	10.2	114.3	123	8	14	72.9	9.77	12	18	19.4	20.1
Total			568.9	611			428.5	450.1			123.6	154.4

Average monthly temperatures rainfall during the two studied seasons in addition to 30 years' records are shown in Table 3. During the studied period average rainfall records were 569 mm (SH), semi-arid (429 mm), and arid (124 mm), while 30 years' records were 611 mm (SH), 450 mm (SA) and 154 (A). This study reported similar values of OMW properties as compared to local and regional studies (Table 4) (Vlyssides et al. 2004; Erses Yay et al. 2012; Ayoub et al. 2014; Rusan et al. 2016; Albalasmeh et al. 2019; Meftah et al. 2019; Mohawesh et al. 2020). Relationships between physicochemical characteristics of OMW were examined using two statistical tools (Pearson correlations and PCA). Table 5a, b, and c show moderate to strong correlations between most of OMW properties. For SH areas, TP was highly and positively related to total phenols, COD and pH, EC with K and pH, and Na with Ca, while TN was negatively correlated with total phenols and TP, and Ca with COD, TN, TSS, and TP. For SA areas, positive moderate to strong correlation between total phenols and COD, TP, TSS, TN, K and pH, while COD with TSS, Ca, K, Na, pH and EC. In addition, arid areas showed positive correlations between total phenols, TSS and Ca, in addition COD with TP, and EC, and negative correlations between TN, Ca and Na.

PCA was used to establish the combination among most influencing factors of OMW physicochemical properties. The KMO (Kaiser-Meyer-Olkin) criterion considered to reach the best result of PCA was (0.61). Results of PCA of related OMW properties and their respective loadings of all collected data for maximum variance are presented in Table 6 and 7. Ca and Na were removed for not reaching the required loading.

The eigenvalues values of the two extracted components were >1.0. The data set was z transformed and varimax rotation was conducted. The PCA-Model reached a KMO-criterion of 0.60 with a total explained variance of 75.77%. PC1 explained 33.96% of the total variance with eigenvalue of 2.72 and had high loadings in favour of K, EC, and TN. PC2 explained 21.62% of the total variance dominated by pH and COD, PC3 (explaining 20.2% of the total variance) was loaded with TP, T-phenols, and TSS. In the first factor (PC1), it can be seen that all extracted parameters may relate to the EC or dissolved salts in OMW. The second factor (PC2) is related to the organic load in OMW, and PC3 is related to TP, T-phenols, and TSS. Moreover, TSS is loaded in both PC3 (0.58) and PC2 (0.51), which could explain

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Parameters	111	(Ayouo et al. 2014)	al. 2015)	(INUSAIL CL AL. 2016)	(AI-L334 2018)	et al. 2019)	et al. 2020)	(5007) 2007)	(IMERNI 51 al. 2013)	et al. 2020)	ánme em r
	<b>UUI</b>	Jordan	Jordan	Jordan	Jordan	Jordan	Jordan	Palestine	Tunisia	Morocco	
Hq		4.91	4.6	4.7	4.63	4.22	4.85	5	5.0	4.7	4.5
EC	(qS/m)	7.64	12.7	7.6	19.89	7.87	8.68		8.1	17.34	7.6
TSS	(mg/L)	69835	20700	1236.2		1277		86840	39400	6146	6906.0
COD	(mg/L)	58614	40000	118800	12000	26550	43099	163500	53300	159537	40538.6
T-Phenols	(mg/L)	2269	360	1666.7	1340	1813	1247	6800	8600	4300	2719.1
NI	(mg/L)	544	ı	96.8	360	ı	372	110	500	ı	374.6
TP	(mg/L)	245	ı	369.5	4120	230	ı	ı	36	ı	115.0
Na	(mg/L)	59.7	0.68	ı	297.9	68.9	45.2		800		138.0
$\mathbf{K}^+$	(mg/L)	2783	25.18	2441.8	6366.3	1668	968		860		4362.3
Ca	(mg/L)	294	2.11	ı	ı	317	117	ı	006	ı	88.5

Fable 4 Main characteristics of OMW in current study, Jordan, and different countries

	<b>T-Phenols</b>	COD	ТР	TSS	TN	Ca	K	Na	рН
T-Phenols									
COD	0.38								
TP	0.65*	0.71*							
TSS	0.44	0.33	0.30						
TN	-0.58*	-0.02	-0.51	0.03					
Ca	0.10	-0.54*	-0.34	-0.47	-0.54*				
Κ	-0.46	0.30	0.14	-0.19	0.50	-0.75*			
Na	-0.09	-0.63*	-0.47	-0.50	-0.41	0.96*	-0.67*		
рН	0.09	0.43	0.58*	-0.46	-0.27	-0.23	0.60*	-0.29	
EC	-0.38	0.17	0.27	-0.47	-0.02	-0.35	0.81*	-0.28	0.82*

 Table 5a Pearson correlation matrix for studied parameters in sub-humid climate

 Table 5b Pearson correlation matrix for studied parameters in semi-arid climate

	<b>T-Phenols</b>	COD	ТР	TSS	TN	Ca	К	Na	pН
T-Phenols									
COD	0.31								
ТР	0.48*	0.21							
TSS	0.38*	0.52*	0.38*						
TN	0.63*	0.18	-0.08	-0.05					
Ca	0.07	0.68*	0.21	0.44*	-0.29				
Κ	0.75*	0.43*	0.24	0.28	0.89*	-0.08			
Na	0.18	0.62*	-0.19	-0.01	0.36	0.59*	0.34		
pН	0.68*	0.52*	0.14	0.17	0.90*	0.01	0.95*	0.53*	
EC	0.14	0.58*	0.16	0.47*	-0.25	0.67*	-0.13	0.20	-0.01

 Table 5c Pearson correlation matrix for studied parameters in arid climate

	<b>T-Phenols</b>	COD	ТР	TSS	TN	Ca	K	Na	рН
T-Phenols									
COD	-0.08								
ТР	0.22	0.72*							
TSS	0.60*	0.27	0.32						
TN	-0.33	0.53	0.27	0.21					
Ca	0.96*	-0.28	0.06	0.44	-0.51				
Κ	0.04	0.55	0.37	0.45	0.93*	-0.17			
Na	0.04	0.13	0.16	-0.04	-0.68*	0.11	-0.69*		
pН	0.15	-0.15	-0.45	0.25	0.04	0.15	0.11	-0.12	
EC	-0.07	0.91*	0.65	0.43	0.76*	-0.29	0.80*	-0.15	-0.04

T-phenols: total phenols, COD: chemical oxygen demand, TP: total phosphorus, TSS: total suspended solids, TN: total nitrogen, Ca: calcium, K: potassium, Na: sodium, EC: electrical conductivity. \*: significant at 0.05 probability level.

that this parameter is well correlated or connected to organic loads (T-phenols and COD) present in OMW.

Table 6	5 Rotations of s	um squared loa	adings of all data
PC	Eigenvalues	Variance %	Cumulative %
1	2.717	33.962	33.962
2	1.729	21.618	55.580
3	1.616	20.195	75.774

a D C 11 1 4

PC: Principal component

Table 7 Rotated component matrix of all data

Parameter		Component	t
	1	2	3
Κ	0.915	0.137	0.197
EC	0.884	0.033	0.184
TN	0.814	0.082	-0.248
рН	-0.078	0.900	0.076
COD	0.343	0.758	0.197
TP	-0.050	0.225	0.810
T-phenols	0.556	-0.113	0.670
TSS	0.036	0.505	0.576

K: Potassium, EC: electrical conductivity, TN: total Nitrogen, COD: chemical oxygen demand, TP: total phosphorus, T-phenols: total phenols, TSS: total suspended solids

PCA was run again to identify underlying factors for each climate zone (arid, semi-arid, and sub-humid). The PCA-Model reached a KMO-criterion of 0.53. Respective rotation sum squared loadings and rotated component matrix for each climate are shown in Table 8 and 9. Table 8 shows that the total explained variance was 85.23, 75.0, and 84.44% for arid, semi-arid, and sub-humid climates, respectively. Two factors were extracted for studied climates, with eigenvalues values of the three extracted components >1.0.

For arid climate; PC1 explained 50.55% of the total variance with an eigenvalue of 3.54 and had high loadings in favour of pH, K, TN, and COD. PC2 was dominated by T-phenols, Ca, and TSS accounting for 34.68% of the total variance. For semi-arid climate; PC1 explained 46.1% of the total variance with an eigenvalue of 2.77 and had high loadings in favour of Ca, COD, pH and TSS. PC2 was dominated by TN and T-phenols accounting for 28.92% of the total variance. For sub-humid climate; PC1 explained 54.25% of the total variance with an eigenvalue of 2.71 and had high loadings in favour of EC, K, and pH. PC2 was dominated by TN and T-phenols accounting for 30.18% of the total variance.

Table 8	Rotations	of	sum	squared	loadings	for	three
studied	climates						

staatea enni	ares		
PC	Eigenvalues	Variance	Cumulative %
Arid			
1	3.539	50.554	50.554
2	2.427	34.676	85.230
Semi-arid			
1	2.766	46.101	46.101
2	1.735	28.919	75.021
Sub-humid			
1	2.713	54.251	54.251
2	1.509	30.184	84.436

Table 9 Rotated component matrix for three studied climates

Parameter	Component	
	1	2
Arid		
pН	0.963	-0.006
K	0.916	0.111
TN	0.885	-0.252
COD	0.811	-0.064
T-phenols	-0.100	0.972
Ca	-0.315	0.920
TSS	0.472	0.745
Semi-arid		
Ca	0.873	-0.244
COD	0.836	0.223
pН	0.829	-0.072
TSS	0.727	0.205
TN	-0.166	0.903
T-phenols	0.249	0.874
Sub-humid		
EC	0.958	-0.065
K	0.922	-0.121
pН	0.827	0.413
T-phenols	-0.385	0.849
COD	0.336	0.773

The outcome of PCA reveals differences in extracted components under different climates, however to simplify discussion; the focus will be on total phenols and TN as they have significant effect on plant/crop growth. As shown in extracted components in all studied climates (Table 9), total –phenols, in PC2, had high loads with TSS in arid climate, with N in the semi-arid, and with COD in the sub-humid climate.

PCA revealed that climatic factors (temperature and rainfall) affected OMW properties. Moreover, soil-related factors, soil type, and cultivar may also have contributed to differences in OMW properties.

Exploitation alternatives of OMW for the production of value-added compounds is important for the industry and agriculture and could effectively contribute to mitigation of greenhouse gases emissions and their consequent unfavorable impacts including global warming and climate change. OMW contains high levels of phenolic and other natural antioxidants, antimicrobial, and anti-inflammatory compounds that could be considered as a potential source of high value-added natural products. These products are of great significance for the biofuel, pharmaceutical, cosmetic, feed and food, and agriculture industries (e.g. fertilizers and soil conditioners (Gullón et al. 2018).

The high content of organic matter and the low content of nitrogen, volatile acid sugars, polyalcohol, and fats, makes OMW an attractive resource for the production of bioenergy and alternative biofuels, such as methane or ethanol given that oily residues or phenols responsible for antimicrobial activity must be first treated or diluted (Dermeche et al. 2013; Ahmed et al. 2019). OMW can be used in the renewable energy and biofuels industry (biohydrogen, bio-methane, bioethanol, biodiesel production). For example; biohydrogen, a renewable energy carrier with a future potential to replace fossil fuels, is produced through a wide range of reactions, including direct and indirect biophotolysis, photo-fermentation, and dark-fermentation of OMW. Moreover, methane can be produced via aerobic or anaerobic pretreatment of OMW followed by a two-phase anaerobic digestion process (Borja et al. 1995; Malode et al. 2021; Ahmed et al. 2021). High organic content of OMW is considered a suitable feedstock for ethanol and biodiesel production via fermentation and thermal process of OMW given the removal or reduction of phenolic compounds (Massadeh and Modallal 2008).

Furthermore, OMW is regarded as a suitable substrate source for the production of enzymes by fungi. For example, polysaccharides extracted from OMW can be used in cosmetic formulations or as a supplement and thickening compound (Petri 2015). Biophenols also have potential beneficial effects on human health. These bioactive compounds have a major role in prevention and treatment of chronic diseases (e.g. obesity, type 2 diabetes, cardiovascular) as well as improving aging processes and prolonging lifespan (González-Burgos and Gómez-Serranillos 2021). Furthermore, OMW also contains bioactive compounds with potential benefits to food health. Ciriminna et al. (2016) reported that phenols in OMW strengthen the beneficial effects of foods themselves and extend their shelf life. Makri et al. (2018) reported that OMW containing silage was found to be effective in improving animal welfare and productivity.

# Conclusion

This study showed that climatic factors (rainfall and temperature) affected OMW characteristics. OMW of arid climate is characterized by lower total phenols, COD, and EC as compared to sub-humid ones. PCA served as an excellent exploratory tool in analyzing and correlating complex physico-chemical OMW properties from different climatic zones. It is highly recommended to treat the high organic loads in OMW using biological, physico-chemical and advanced oxidation processes, combination treatments, and bioconversion into high-added products prior to any recommendation of use. More studies are needed to evaluate the use of OMW under different soils and climatic conditions.

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# **Compliance with ethical standards**

**Conflict of interest** The authors declare that there are no conflicts of interest associated with this study.

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