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Application of Statistical Methods for the Comparative Study of the Degree of Pollution of Wastewater Collected from Three Olive Mills in Tangier-Tetouan-Al Hoceima Region (Northern Morocco)

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ABSTRACT

Olive mill wastewaters represent a severe environmental problem, especially in Mediterranean countries. Indeed, the treatment and recovery processes developed so far remain very limited and their cost is very high. However, treatment by shallow evaporation ponds in the open air remains, until now, the most used technique despite the efforts made. The volume and characteristics of this liquid effluent depend, among other things, on the nature of the extraction process used. In this context, this work aims to evaluate the effect of the mode of trituration and storage in the evaporation ponds on the quality of olive oil mill wastewaters. These effluents were collected from three olive mills located in two different provinces in Northern Morocco during the 2021–2022 olive growing season. The sampling has considered different points according to different steps of the process. The analytical work corresponded to the measurement of in-situ parameters (Temperature (T°), Dissolved Oxygen (DO), Electrical Conductivity (EC) and pH degree (pH)) and other laboratory analysis (Suspension Matter (SM), Dry Matter (DM), Fatty Matter (FM), biochemical (BOD5) and chemical (COD)demands in oxygen). Results showed a high degree of environmental degradation. Indeed, these effluents are characterized, on average, by elevated concentrations of biological and chemical oxygen demands and suspended solids reaching up to 0.43 g/L ± 0.87, 1.69 g/L ±0.71 and 30.78 g/L ±25.1, respectively. This study also showed, except for two-phase olive mill BNANDA, a slightly alkaline nature of the vegetable waters ($7.81<pH<8.52$), as opposed to what was reported in the literature, and high levels in fat content (4.73 g/L ±3.85). Moreover, the estimated volume of vegetable water released in the study area during the 2019/2020 olive growing season (350×10^3 m^3) is very high considering its equivalence to domestic water. Normalized Principal Component Analysis (ACPN) and Hierarchical Principal Component Classification (HCPC) indicate that differences in the composition of this wastewater were more pronounced between the extraction processes than by the origins of the vegetable waters (fresh or stored in the natural evaporation pond).

Keywords: olive oil mill wastewater, degree of pollution, multivariate statistical methods, ACPN, HCPC.

INTRODUCTION

The global area of olive groves is estimated, in 2020, at 11.5×10^{10} m^2, distributed in 67 countries, of which nearly 98% are located in the Mediterranean basin (Dermeche et al., 2013). The production of olive oil generates significant quantities of wastewater, formed in a short period of 3 to 4 months (Achak et al., 2009; Dermeche et al., 2013; Rajhi et al., 2018). Indeed, the serious environmental problems are associated with this effluent, when discharged into the environment, as already shown in several studies (Achak et al., 2009; Aharonov-Nadborny et al., 2017; El Abbassi et al., 2014; Suna Erses Yay et al., 2012; Zghari et al., 2018).

In Morocco, agriculture has focused on plant species that require less water and adapt to climate change and water stress. In order to ensure a sustainable increase in olive productivity, several efforts have been made to promote the olive growing sector. The Moroccan varietal range in olive cultivation consists mainly of Moroccan Picholine, Menara, Haouzia and Dahbia. This olive-growing heritage, marked by the dominance of the Moroccan picholine variety, occupies the agroecosystems and modern orchards in the north, center, and south-west (Khadari et al., 2008). This variety, characterized by phenotypic plasticity, has adaptive potential for a resilient and sustainable olive cultuvation (Terral & Ater, 2016). The area planted by the olive trees increased by 57% between 2008 and 2020, while production increased 10 times during the 2019-2020 season compared to 2007–2008 (DWRP, 2020). However, and despite efforts to modernize the sector and expand crops, olive yields remain lower than the real potential.

Generally, three crushing systems mark Morocco's olive oil production: discontinuous press crushing and three-phase systems, three-phase continuous centrifuge process (UT/Tri) and two-phase continuous centrifuge process (UT/Bi).

These modern production processes are based on a continuous extraction system comprising horizontal and vertical centrifugation. The first one separates the paste into three phases UT/Tri (oil, vegetable water and pomace) or two phases UT/Bi (oil and plastic paste) (Benyahia and Zein, 2003). Indeed, the olive oil industry generates two residues: pomace (solid to semi-solid) and vegetable water (liquid). These liquid effluents are the main harmful waste of this industry (Hanafi et al., 2009) and constitute a major environmental problem for olive oil-producing countries (Zghari et al., 2018). Some authors consider that their high polluting power is mainly due to a high load in chemical and biological oxygen demands (COD and BOD_5) (Aharonov-Nadborny et al., 2017; Kissi et al., 2001; Mouncif et al., 1995; Paredes et al., 1999; Suna Erses Yay et al., 2012). Others have associated it with phenolic and aromatic compounds that are very difficult to biodegrade (Aguilar et al., 2002; Ergüder et al., 2000; Lesage-Meessen et al., 2001).

These vegetable waters continue to generate many damages which can be irreversible in the absence of a legal framework regulating the spreading or the conversion. However, treatment by shallow evaporation ponds in the open air remains the most widely used technique despite the efforts made by several researchers to limit their harmful effects (Fenice et al., 2003; Gharsallah et al., 1999; Hoyos et al., 2002; Kissi et al., 2001; Leger et al., 2000; M'Sadak et al., 2015). The characteristics and composition of these residues are very variable and depend on a multitude of factors among which we must distinguish the variety of the olive tree, the climatic conditions, the water used in the process, the degree of their maturation and the olive oil extraction system (De Felice et al., 1997 and Kavvadias et al., 2017). In this regard, several studies have been carried out on vegetable waters, in their raw form or the receiving environment, and various analytical methods have been adopted to identify and quantify their substances. Therefore, this study was conducted to assess the effect of the mode of trituration and storage in the evaporation ponds on the degree of pollution of olive oil mill wastewater from three olive mills in the Tangier-Tetouan-Al Hoceima (TTA) region. The analytical work concerns the measurements of temperature (T), dissolved oxygen (DO), pH, electrical conductivity (EC), matter in suspension (SM), dry matter (DM), fatty matter (FM), biochemical demands (BOD_5) and chemical in oxygen (COD). The results were analyzed by multivariate statistical methods (ACPN and HCPC) to determine the effect of these parameters on the characteristics of these olive oil mills wastewaters.

MATERIALS AND METHODS

Methodology

For this study we process several steps:

- Collected data "sample" in our studied area;
- Physicochemical analysis, of vegetable waters generated by three mills of oil, for the characterization of T, DO, pH, EC, SM, DM, FM, BOD_5 and COD;
- Estimation and mapping of the number of vegetable waters produced in the provinces of the TTA region during the 2019/2020 olive growing season;
- The Normalized Principal Components Analysis (ACPN), allowing to synthesize the information, was used in order to identify the variables which govern and explain the similarities between the various processes and the points of acquisition of the wastewater;
- Hierarchical principal component classification (HCPC) is used to group samples with similar characteristics into stable dissimilar classes.

Presentation of study area

The Tangier-Tetouan-Al Hoceima (TTA) region, located in the extreme northwest of Morocco, covers an area of 17262 m^2, or 2.43% of the national territory. It has two prefectures Tanger-Assilah and M'Diq-Fnideq and six provinces: Al Hoceima, Chefchaouen, Fahs-Anjra, Larache, Ouezzane and Tetouan. This work was carried out in three oil mills and their evaporation ponds located in (Figure 1):

1. The commune of Ksar Bjir in the province of Larache (Morocco):
   - Two-phase olive mill (UT1/Bi) (BNANDA) (34°59'09" N 5°56'28" W);
   - Mixed olive mill (UT3/Bi & UT3/Tri) (BARAKA) (34°59'01" N 5°56'38"W);
2. The municipality of Zouada in the province of Larache (Morocco): evaporation pond (UT3/BT) (0.5 km from the olive mill) (34°59'19" N 5°56'58" W);
3. The commune of Sidi Redouane in the province of Ouezzane (Morocco): Three-phase olive mill (UT2/Tri) (BAB L'MHAJ) (34°42'06" N 5°24'35" W).

Sampling and analysis methods

This study focused on the vegetable waters of three modern oil mills located in the TTA region during the 2021–2022 olive-growing campaign (Figure 1):

- UT1/Bi/P2 and UT3/Bi/P2: fresh vegetable water taken from the direct outlet of the two-phase vertical centrifuge (UT1 and UT3);
- UT2/Tri/P2 and UT3/Tri/P2: fresh vegetable water taken directly from the horizontal centrifuge of three-phase oil mills (UT2 and UT3);
- UT1/Bi/P3, UT3/BT/P3 and UT2/Tri/P3: entry of the vegetable waters into the evaporation pond;
- UT1/Bi/P4, UT2/Tri/P4 and UT3/BT/P5: extremity of the evaporation pond;
- UT3/BT/P4: the middle of the BARAKA pond.

The physico-chemical characterization essentially concerns the measurements of temperature (T°), dissolved oxygen (DO), pH, electrical conductivity (EC), matter in suspension (SM), dry matter (DM), fatty matter (FM), demands biochemical (BOD_5) and chemical COD in (oxygen). Indeed, in situ measurements (T°, pH, EC and DO) were carried out using a HI 9828 PH/ORP/EC/DOs (HANNA) multi-parameter, at the level of each olive mills. Each result obtained represents an average of three analytical tests carried out. The vegetable water sampling was manually done using glass bottles previously cleaned according to Rodier process and placed in an icebox under a temperature of 4 °C in order to reduce the risks of precipitation, adsorption, contamination or evaporation (Rodier, 1984). Then samples were

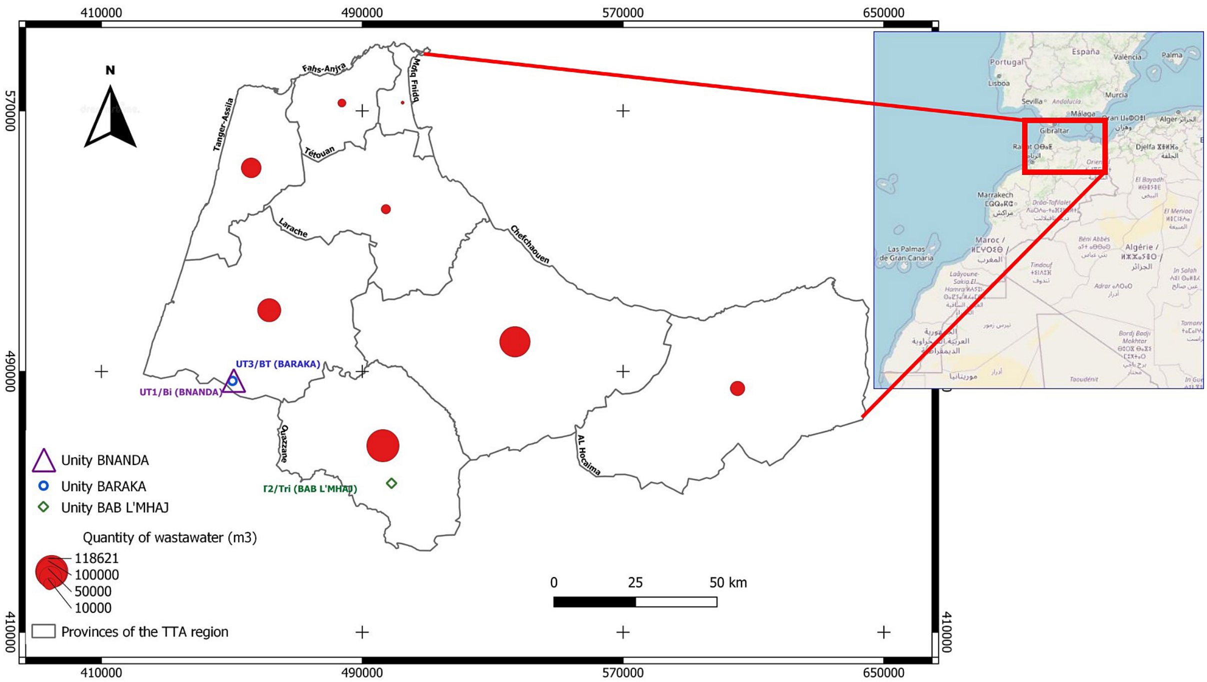

Figure 1. Location of the three oil mills in the study area and the quantity of wastewater produced in the provinces of the TTA region in 2019/2020 companies

transported to the laboratory in coolers and stored at 4 °C for immediate physico-chemical tests. Those latter were carried out at the Marine Environment and Natural Risks Laboratory, according to the Rodier analysis protocol (Rodier, 1984).

Suspended Matters (SM) were determined by a filtration of a known volume of water to be analyzed. The dry matter (DM) was determined by evaporation of a 20 ml sample in a crucible at 105 °C for 24 hours. The chemical oxygen demand (COD) is determined by oxidation in an acid medium by excess potassium dichromate at a temperature of 148 °C of oxidizable materials under the conditions of the test in the presence of silver sulfate as catalyst and mercury sulfate (Aissam, 2003). The BOD_5 is determined according to the respirometric method in a chamber thermostated at 20 °C, in the dark and for 5 days. The liquid / liquid extraction of Fatty Matter (FM) was realized by 100 ml of chloroform and 75 ml of methanol from 100 ml of samples. The chloroformical fraction was recuperated and evaporated in a rotary-evaporator and then weighed.

Evaluation of the quantity of vegetable water produced in the TTA region

In the TTA region, the area planted by the olive tree, which is gradually changing, is around 5166.77x10^4 m^2/year between 2007 and 2019. The provincial distribution of the area occupied by the olive tree shows that 85% of it is located in the two provinces of Ouezzane (44%) and Chefchaouen (41%). Indeed, the geographical distribution of crushing units in the TTA region is marked by a wide variation depending on the number and type. It is generally observed that modern types represent the majority of existing units in the TTA region, and are concentrated mainly in the provinces of Ouezzane, Chefchaouen, Larache and Tetouan. Semi-modern units are especially located in the two provinces of Tangier and Al Hoceima and represent respectively 25% and 20% of all units (DWRP, 2020).

Several studies have evaluated the quantities of vegetable water produced for the different techniques of olive oil extraction. These variable quantities were estimated by Achak et al. (2009), 1000 kg of vegetable water/ 1000 kg of olives for modern units. While for others, pressing 1000 kg of olive using modern methods produces an average of 1500 kg of liquid by product (Benyahia & Zain, 2003; Léger, 1999; Vitolo et al., 1999). Thus, the volume of vegetable water produced depends on the trituration process and therefore on the quantities and temperature of the water added along the process.

Statistical methods of data processing

The methodology adopted in this study is based on a combination of Standardized Principal Component Analysis (ACPN) and Hierarchical principal component classification (HCPC). This multi-varied approach was carried out on 10 samples and 9 variables (electrical conductivity (EC), pH, electrical conductivity, grace matter, dry matter, suspended matter, chemical oxygen demand and biological demand oxygen) using R software version 4.0.5 (2021-03-31). This analysis makes it possible to synthesize and classify a large amount of data to extract the main factors that are at the origin of the simultaneous evolution of the variables and their reciprocal relationship. These results were obtained using the packages: FactoMineR, factoextra, ggplot2, Hmisc, and corrplot. It makes it possible to highlight the similarities between two or more chemical variables during their evolution.

RESULTS AND DISCUSSION

Results of the physico-chemical analyzes of vegetable waters

The physico-chemical results obtained from the samples collected from the three oil mills located in the provinces of Ouezzane and Larache during the 2021/2022 olive growing campaign have been summarized in Table 1.

The results of the physicochemical analyzes carried out on the vegetable waters studied (Table 1) showed Temperature values varying from 17.13 to 29.71 °C for all samples from the three oil mills in the TTA region, with an average of 21.13± 5.08 °C. Indeed, for the fresh vegetable waters (P2), excepted for BNANDA olive mill UT1/Bi/P2 (19.22 °C), the highest values were recorded in the vegetable waters UT2/Tri/ P2 from BAB L'MHAJ mill of Ouezzane (29 °C), UT3/Bi/P2 (29.71 °C) and UT3/Tri/P2 (26 °C) (BARAKA). These results are within the range of the limit values for direct discharges. They are related to the use of hot water (>= 30 °C) for the humidification of the olive paste. In addition, the

Table 1. Statistical characteristics of Physico-chemical analyzes (SD: standard deviation, Mean: and GLVDD: General Limit Values For Direct Discharge (Official Bulletin No. 6199 of 28/10/2013)

Sample points	T (°C)	pH	EC (s/m)	SM (g/L)	DM (g/L)	FM (g/L)	DO (g/L)	BOD_5 (g O_2/L)	COD (g O_2/L)
UT1/Bi/P2	19.22	6.3	0.31	1.76	23.34	1.12	0.0057	0.16	1.21
UT1/Bi/P3	17.43	6.09	0.24	7.39	30.25	0.46	0.0024	0.27	1.75
UT1/Bi/P4	17.34	5.04	0.26	10.91	43.67	0.45	0.0034	0.8	1.76
UT2/Tri/P2	29	8.52	0.92	42.42	103.9	4.86	0	0.05	1.54
UT2/Tri/P3 (P4)	20.14	8.38	1.28	61.07	92.98	2.74	0	2.8	1.21
UT3/Bi/P2	29.71	7.81	0.44	65.77	34.57	4.78	0.00075	0.045	0.81
UT3/BT/P4	17.28	7.98	0.69	16.54	51.7	4.33	0.0011	0.042	2.32
UT3/BT/P3	18.06	8.52	0.67	64.26	52.82	9.06	0.0002	0.024	1.63
UT3/BT/P5	17.13	8.09	0.69	18.02	58.12	12.04	0.0011	0.041	1.31
UT3/Tri/P2	26	7.86	0.70	19.66	25.87	7.41	0.002	0.046	3.36
Mean	21.13	7.46	0.62	30.78	51.72	4.73	0.0017	0.43	1.69
SD	5.08	1.21	0.32	25.10	27.41	3.85	0.0018	0.87	0.71
GLVDD	**30**	**5.5-9.5**	**0.27**	**0.1**	**0.03**	**0.03**	**-**	**0.1**	**0.5**

temperatures of the vegetable waters collected from the evaporation ponds ranged from 17.13 °C to 20.14 °C, with maximum value measured at the BAB L'MHAJ oil mill. This explains the zero dissolved oxygen content.

The pH represents the intensity of acidity or alkalinity and measures the concentration of hydronium ions. The results have shown a range of variation from 5.2 and 8.52, with an average of 7.46±1.21, oscillate between acid to alkaline status. The acid vegetable waters (pH<7) were collected at different points of two phases oil mill BNANDA, which reveals the richness of these vegetable waters in organic acids (phenolic acids) which makes difficult the biological purification of this effluent (Capasso et al., 1992; Aggelis et al., 2003) modifying, thereby, the favorable conditions for microorganism's development (Khoufi et al., 2000). However, alkaline values (pH> 7) were recorded at points P2, P3, P4 and P5 of BAB L'MHAJ mill UT2/Tri and BARAKA oil mill UT3/BT. These values are different from what were generally was reported in previous studies, although they remain within the range of general limit values for direct releases (5.5 <pH< 9). Nevertheless, these results are comparable to those reported by Martínez-Gallardo et al. (2021), where vegetable waters ware studied at low humidity and stored for a long time in evaporation pounds.

Electrical conductivity is closely related to the concentration of dissolved substances and their nature. The results displayed values oscillating between 0.24 m/s and 1.28 m/s with an average of 0.62 ± 0.32 m/s. High values corresponded to Wastewater collected from the evaporation pond of the BAB L'MAHAJ three-phase mill UT2/Tri/P3 (P4) (1.28 m/s). except the vegetable waters collected from BNANDA pond UT1/Bi/P3 (0.24 m/s), these values are 3 to 5 times higher than Moroccan standard given by the general limit values for direct discharges (0.27 m/s). However, these values are on average comparable with those obtained by Rais et al. (2017), 0.78 ± 0.006 m/s. These high salt contents are due, on one hand, to the natural richness of the vegetable waters in dissolved mineral salts and, on the other hand, to the practice of salting the olives before crushing for conservation.

The vegetable waters collected from the three crushing units seems to be heavily loaded with suspended solids. According to the analyses, the SM values were between 1.76 g/L and 65.77 g/L, which exceed up to 660 times the general limit values for direct discharges (0.1 g/L). However, these values remain lower than those obtained in previous research (Al-Malah et al. 2000; Assas et al. 2002; Hanafi et al., 2009; Zaier et al., 2017). In addition, values obtained for vegetable water collected from natural evaporation ponds were lower than those of fresh vegetable water, which could be explained by the setting in the ponds.

The fat content present in vegetable water depends on the olive oil extraction system. The leveles obtained in our study showed a viscous aspect related to the presence of the oily fraction varying between 0.45 and 12.04 g/L, with an average of 4.73 g/L±3.85. Indeed, the average value is 150 times higher than the national standard with a maximum value was recorded at the extremity of the BARAKA evaporation pond of the mixed mill UT3/BT/P5. In fact, this is due to the lipid layer formed on the surface of the pond at this part, which could limit natural evaporation.

The dry matter concentration of our samples oscillates between 23.34 and 103.9 g/L, with an average of 51.72±27.41 g/L. These values are on average compared to those reported by Achak et al. (2008), 56.75±2.51 g/L. However, our values remain lower than the maximum value of centrifugation wastewater reported by Di Giovacchino et al. (1988), which is 161.2 g/L. This fact could be explained by the use of hot water to mix the paste required by the centrifugation system, which results in a dilution of the vegetation water produced.

The quantity of dissolved oxygen in the wastewater collected oscillates between 0 and 0.0057 g/L. Per the process system, the highest values were recorded in the vegetable waters of the BNANDA two-phase mill, which correspond to the lowest temperatures. The range of variation of the results obtained in our study is more extensive than that obtained in other studies (0.0001 to 0.0041 g/L) (Bouknana et al., 2014; Sassi et al., 2006).

The biological oxygen demand (BOD_5) of our vegetable waters oscillates between 0.024 and 2.8 g O_2/L. The maximum was recorded in the evaporation pond of the BAB L'MHAJ three-phase mill (2.8 g O_2/L). This latter exceeds 28 times the Moroccan standard set at 0.1 g O_2/L. Indeed, for the three-phase continuous system, the values reported in the literature are up to 24 times higher than the maximum value obtained in this study, they vary within the range (5.65–67.5 g O_2/L) (Achak et al., 2008; Bouknana et al., 2014; El Hassani et al., 2009; Eroğlu et al., 2008; M'Sadak et al., 2015; Khoufi et al., 2007; Rais et al., 2017; Vlussides et al., 2004; Yaakoubi et al., 2010). However, values lower than ours have been reported by Dogruel et al. (2009), i.e. 0.015 g O_2/L.

The chemical oxygen demand (COD) makes it possible to assess the concentration of organic and mineral matter, dissolved or suspended in water, through the quantity of oxygen necessary for their total chemical oxidation. In this case of study, the vegetable waters displayed values of COD varying from 0.81 to 3.36 g O_2/L which highlights that all values obtained for all points exceed greatly the Moroccan standard (GLVDD = 0.5 g O_2/L). However, the values obtained remain lower than those obtained in several studies carried out on vegetable water from the centrifugation system (Achak et al., 2019; Borja et al., 1992;Di-Giovacchino et al., 1988; Elabdouni et al., 2020; Ergüder et al. 2000; Hamdi, 1993c; Tsioulpas et al. 2002 & Yaakoubi et al., 2010). Indeed, vegetable waters need a very high oxygen rate for their oxidation, which shows that these vegetable waters can cause very significant pollution (Hanafi et al., 2009).

Estimation and mapping of the number of vegetable waters produced in the provinces of the TTA

For the calculation of the quantity of liquid waste, produced during the 2019/2020 olive-growing season, in the provinces of the Tangier-Tetouan region, we have adopted:

- For discontinuous three-phase oil mills 0.50 m^3/1000 kg;
- For continuous three-phase oil mills 1.2 m^3/1000 kg;
- For two-phase continuous oil mills 0.25 m^3/1000 kg.

In 2020, the area planted with olive trees amounts to 170×10^7 m^2 in the TTA region. The provincial distribution shows that it is located up to 65% at the level of the two provinces, Ouazzane and Chefchaouene, with respectively 35% (58852×10^4 m^2) and 30% (52235×10^4 m^2). The overall quantity of vegetable water discharged by industrial units is estimated at approximately 350×10^3 m^3/olive growing season. The proportional circle map showed that the most of this quantity is produced in the provinces of Ouazzane (33%), Chefchaouene (29%), Larache (16%) and Tetouan (11%) (Figure 1).

Principal component analysis results

The correlation matrix between the physicochemical parameters with their significance, obtained by the Hmisc and corrplot packages of the R software, is illustrated in Figure 2. Results showed that pH is significantly negatively correlated with dissolved oxygen DO ($r = -0.8$ and $p = 0.0056$) and positively with electrical conductivity ($r = 0.79$ and $p = 0.0067$), while the dry matter is positively and significantly correlated with electrical conductivity ($r =-0.8$ and $p = 0.005$). In addition, SM ($r = 0.67$ and $p = 0.035$) and FM ($r = 0.67$, $p = 0.027$) are significantly and positively correlated with hydrogen potential, while

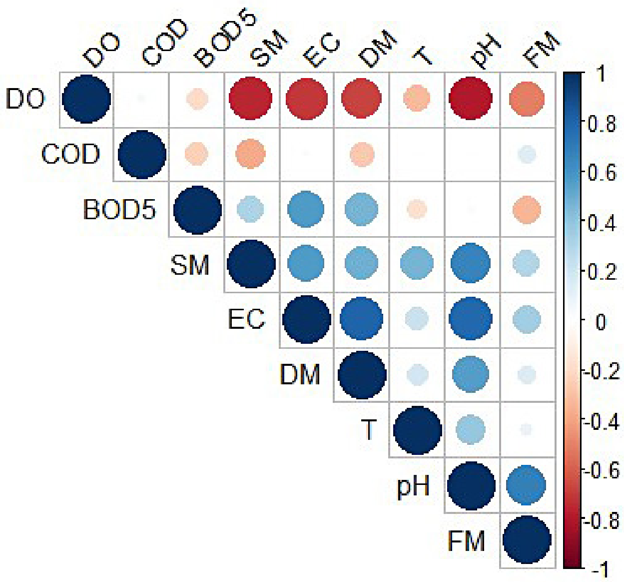

Figure 2. The correlation matrix graph

dissolved oxygen is correlated negatively and significantly with electrical conductivity (r = -0.71 and p = 0.021), suspended matter (r = -0.76, p = 0.011) and dry matter (r = -0.67 and p = 0.033).

The storage efficiency in evaporation ponds was assessed by monitoring the rate of variation of the parameters studied analytically (Table 2). The results show that the effect of storage differs from one mill oil to another and from one parameter to another. For the two-phase mill, the storage eliminated 60% of the FM, 20% EC, however the concentration of COD, BOD5, SM and DM increased by 45%, 234%, 420% and 58% respectively. This explains the consumption of oxygen which has decreased by 49%. Concerning the three-phase mill, the reduction was recorded for the parameters T (30%), FM (44%) and COD (21%), however, an extreme increase was recorded for the parameters BOD_5 (5,5‰), DM (795%), SM (44%) and EC (39%). In the mixed mill, an increase in concentrations was recorded, for the vegetable waters collected from the evaporation pond, for the DM (79%), FM (39%) and EC (20%) parameters. While, the reduction only concerned the T (37%), SM (23%), DO (43%), BOD_5 (22%) and COD (16%) parameters. In parallel, the comparative study, carried out by Achak et al., 2008, between the characteristics of the raw vegetable waters and those decanted, revealed a reduction in the concentrations of DM (43%), COD (8.73%) and BOD_5 (7.17 %), while the electrical conductivity increased by 14%.

The analysis of the distribution of the samples of vegetable waters in the factorial planes makes it possible to highlight the similarities and dissimilarities existing between the samples and to identify the effect of certain parameters on their elementary composition. The results of

Table 2. The rate of variation of the parameters between the fresh outlet of the wastewater and the evaporation pond of the three oil mills

Parameter	UT1/Bi (%)	UT2/Tri (%)	UT3/BT (%)
T	-9.54	-30.55	-37.21
pH	-11.67	-1.64	+4.61
DO	-48.95	0.0	-42.75
SM	+419.88	+44	-22.88
DM	+58.35	+794.89	+79.39
FM	-59.15	-43.63	+39.07
EC	-19.62	+38.90	+19.77
BOD_5	+234.37	+5500	-21.61
COD	+45.65	-21.21	-15.86

the PCA indicate that the factorial axes PC1, PC2 and PC3 represent 79.8% of the total inertia. The PC1 axis, the main axis of inertia, expresses 47.3% of the variance and opposes the hydrogen potential and the electrical conductivity to the dissolved oxygen. Indeed, this axis would discriminate the vegetable waters collected according to their acidity, electrical conductivity and their dissolved oxygen content. The PC2 axis, which expresses 19.8% of the total variance, separated the waste waters studied according to their lipid content and their biological oxygen demands (Figure 3a). Finally, the third axis of the PCA explained 11.8% of the total variance and mainly illustrated the relationship between the process and the chemical oxygen demand of the collected vegetable waters (Figure 4a). The PCA has therefore made it possible to show a strong relationship between the variables studied and the extraction process, however, the differentiation of the vegetable waters according to the collection point (fresh outlet or natural evaporation pond) could not be distinguished by principal component analysis (Figure 3b & Figure 4b).

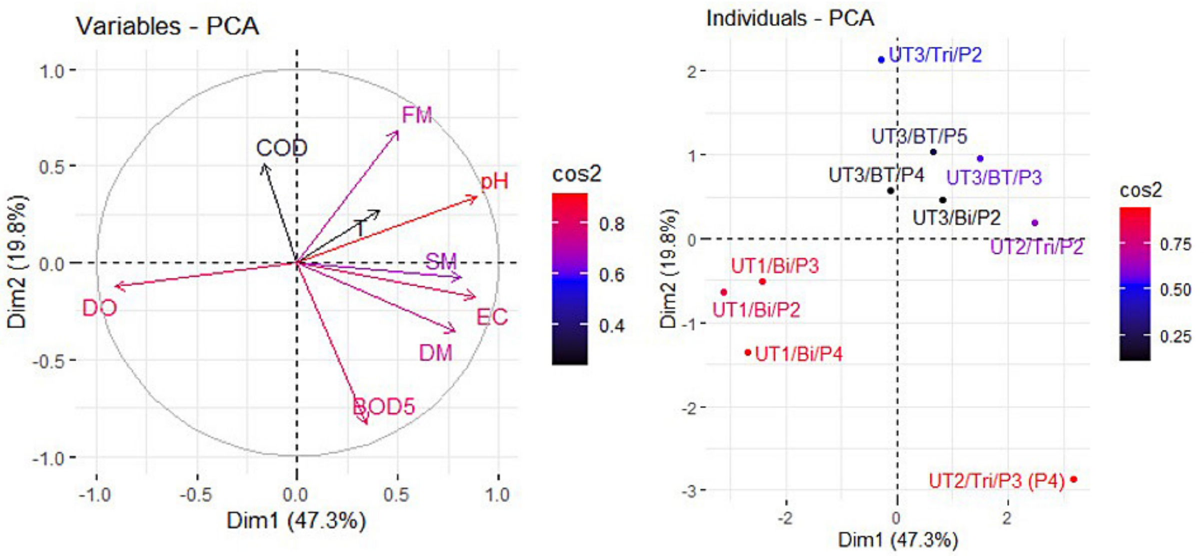

Figure 3. PCA projections for factorial plane PC1 – PC2: a. different variables (on the left); b. different samples collected from three oil mills (in the right)

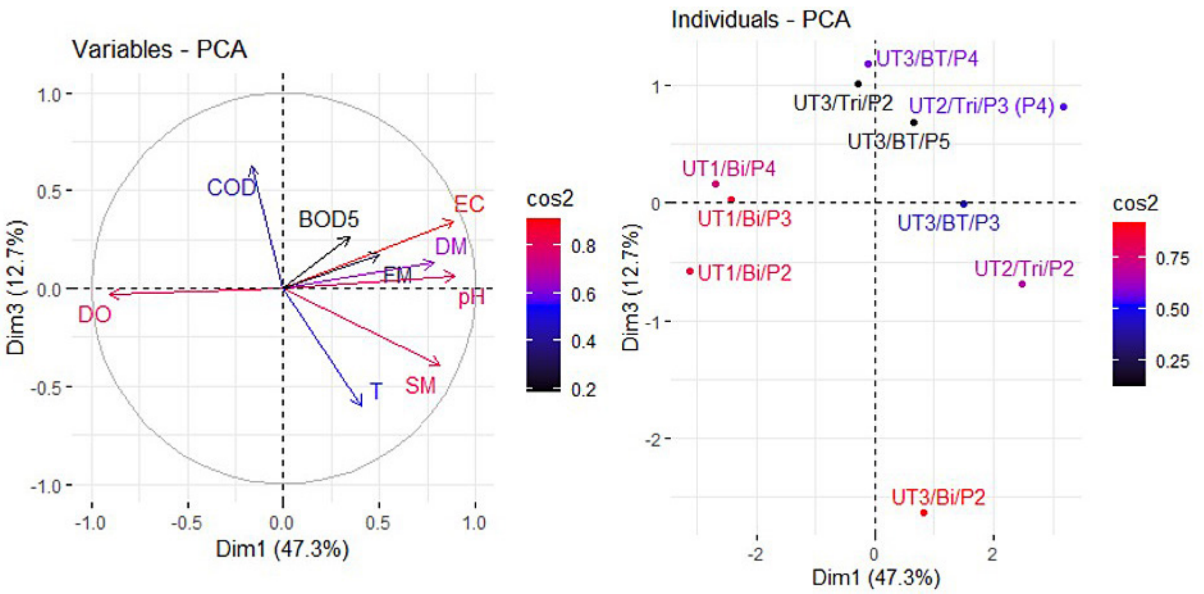

Figure 4. PCA projections for factorial plane PC1 – PC3: a. different variables (on the left); b. different samples collected from three oil mills (in the right)

Hierarchical classification on principal component results

The dendrogram carried out on the 10 samples of vegetable waters collected from the three crushing units of the provinces of Ouezzane and Larache reveals indications of the level of similarity between the samples forming two main statistically distinct classes (Figure 5). This dissimilarity was specially oriented by the differences between the values of the parameters: pH, EC and FM. The first class is made up of the sampling points P2, P3 and P4 belonging to the BNANDA two-phase crushing system at Ksar El Kbir. The vegetable waters of this class are characterized by their acid character (<6.3), low temperature (<19.22), a minimum electrical conductivity between 0.24 and 0.31 s/m (<average CE (0.62 s/m) and low-fat content which does not exceed 1.12 g/L (the average value is 4.73 g/L). The second class is made up of the rest of the samples collected from the BAB L'MHAJ three-phase oil mill of Ouazzane and the BARAKA mixed mill of Ksar El Kbir which includes the two types. The vegetable waters of this class are essentially characterized by their alkaline character and their fat content which exceeds the average value (4.73 g/L > 157xGLVDD). This class is made up of two groups. The first group is made up of the two points UT2/Tri/P3 & P4 of the BAB L'MHAJ three-phase oil mill. The latter is characterized by a maximum content of BOD_5, dry matter and electrical conductivity. Whereas, the second group is made up of the samples of vegetable water collected from point P2 (fresh exit) of BAB L'MHAJ and the BARAKA mixed oil mill. Indeed, this group can be divided into three subgroups. The first subgroup contains the points UT3/Bi/P2 and UT2/Tri/P2, characterized by maximum temperatures, alkaline wastewater and a high content of suspended matter. Whereas, the vegetable waters of the second subgroup, constituted by the two points UT3/Tri/P2 and UT3/BT/P4, are alkaline, saline and characterized by a high chemical oxygen demand. Finally, the third group is made up of the points UT3/BT/P3 and UT3/BT/P5, characterized by very alkaline wastewaters (> 8), a low BOD_5 content, a high electrical conductivity and a maximum fat content. Indeed, the presence of lipids in vegetable waters forms an impenetrable layer that prevents the penetration of light and limits the capacity for self-purification, resulting in the deterioration of the quality of water resources (Zghari et al., 2017).

DISCUSSION

The olive industry production generates 2 types of residues: vegetable waters (liquid) and the pomace (solid). Their corresponding quantities have been well related in the literature to the nature of the equipment used in the production and the extraction methods, whether traditional

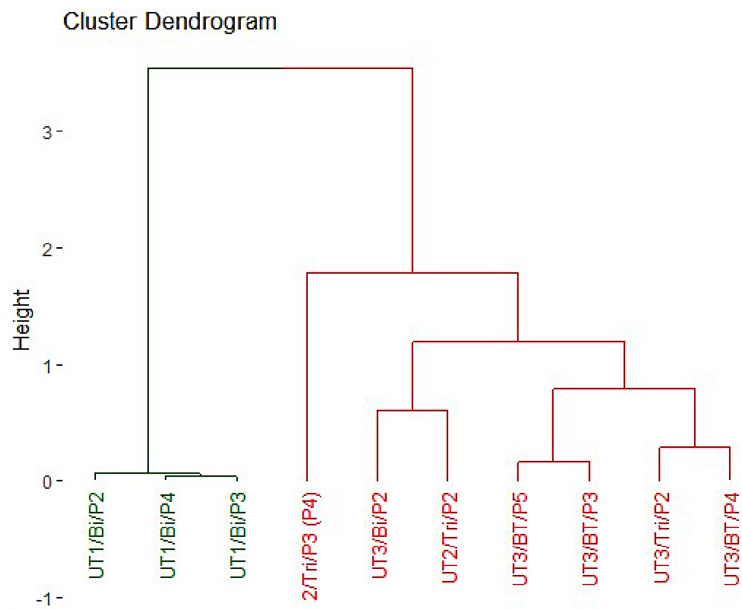

Figure 5. Dendrogram of the hierarchical component principal classification (HCPC) of wastewater collected from the three oil mills in the TTA region

or modern. Innumerable research has studied the environmental aspect of their discharges directly to the environment without any pretreatment process. Therefore, standards have been set to fight this problem and eliminate the risk of contaminating the environment.

In Morocco, the sustainable development of the olive industry requires an environmental control of the olive oil production processes, and a better management and valorization of the liquid by-products generate (vegetable waters). This latter, and in the absence of a legal framework regulating the spreading or conversion, continues to be discharged directly into the surrounding environment, causing hence many damages that could be irreversible. Furthermore, and as it was advanced, the volume of vegetable water produced depends on the trituration process and therefore on the quantities and temperature of the water added along the process.

In that context, this work come to assess the effect of the mode of trituration and storage in the evaporation ponds on the degree of pollution of olive oil mill wastewater from three olive mills in the Tangier-Tetouan-Al Hoceima (TTA) region using several physicochemical parameters (in situ and laboratory analysis).

Consequently, this work the volume estimated of vegetable water released in the study area during the 2019/2020 olive growing season (350×10^3 m^3) is very high considering its equivalence to domestic water. Indeed, it was established in literature that $1m^3$ of vegetable water is equivalent to 200 m^3 of domestic wastewater (Rocha et al., 2018; Tosti et al., 2016) and causes pollution equivalent to 1500 people in a single day (Ranalli, 1991).

On another hand, physicochemical results obtained showed a clear degradation of sample status. First of all, the fresh vegetable waters seem to show values above the standards fixed for all parameters except for Temperature and pH. These results highlighted the degraded quality of the vegetable water rejected into the basin's storage. On another hand, it has been verified a significant load of oxidizable material traduced by values of BOD_5 and COD higher than standards fixed, mostly non-biodegradable ($COD/BOD_5>3$) for all samples except for UT1/Bi/P4 and UT2/Tri/P4 representing a biodegradable matter, they correspond to the furthest points in the ponds for both two phase (BNANDA) and tree phase (BAB L'MHAJ) industries. This pattern seems to differ for the mixed unit of BARAKA. In fact, for this latter, the load of oxidizable material seems to be the highest in the evaporation pond (P3, P4, P5) where both discharges emanating from biphasic and triphasic ones are mixed. These results are consistent with those observed by El Rhaouat et al. (2014), however other studies have highlighted the biodegradable nature of vegetable waters (Gnagne and Brissaud, 2003; Zaier et al., 2017; Zerhouni, 2003). It also corresponds to the highest values of Fatty Matter (FM) concentrations and the lowest values of Dissolved Oxygen (DO) concentrations. This result remains reasonable and could be explained by the fact that the fatty matters have a low density and thus remain in the surface of vegetable waters which prevent the atmospheric dissolution of DO on one hand and the penetration of light on another hand stopping hence the photosynthesis reactions and DO production. The low concentration of DO could be also resulting from reactions of oxidation of mineral matters traduced here by EC, SM and DM values as it was displayed by the correlation matrix which highlighted a significant and negative correlations between DO and EC, SM and DM. In addition, Temperature value is also an important factor that might influence the decreasing of DO's values. Actually, results had already shown values of DO negatively correlated with Temperatures, where the minimum of DO concentrations corresponds to the maximum of Temperatures: UT2/Tri/P2 (0 mg/l; 29 °C); UT3/Bi/P2 (0.75×10^{-3} g/L; 29.71 °C); UT2/Tri/P3 (0 g/L; 20.14 °C). This status would lead to the establishment of a degraded environment deteriorating, even more, with the evaporation process. Low levels of dissolved oxygen have also been recorded in similar studies (Bouknana et al., 2014. Fakhaoui & Pattee, 1993).

At this level of results discussion, it appears difficult to clarify the degree of pollution abatement between the fresh vegetable water and the evaporation ponds water for all units. It has been shown different pattern of variation depending on the type of trituration process. The BNANDA (UT1/Bi) and BAB L'MHAJ (UT2/Tri) unities showed almost the same pattern of variation except for CE and COD parameters. However, the unit of BARAKA (UT3/BT) which have both two phase and tree phase process showed different pattern of variation although all of them remain polluted.

On another hand, the very varied nature of the results makes it difficult to interpret and compare between all trituration procedures, which is why we used multivariate statistical analysis (ACPN and HCPC) to detect resemblance between the different points of analysis. Results shown in Figures 3-4-5 displayed a very interesting information about similarity and resemblance. Actually, the statistical analysis isolated the oil mill of BNANDA two phase process from the other ones, notably the three phase system of BAB MHAJ and the mixed oil mill of BARAKA. Indeed, the application of these statistical analyses, used in several other works, has been able to group samples with similar characteristics into distinct classes (Ahoussi, 2008; Akatumbila et al., 2016; Alilouch et al., 2020; Yao and Ahoussi, 2021).

CONCLUSIONS

The wastewater collected in the three oil mills located in two provinces of the TTA region of northern Morocco has a very heterogeneous composition. The Physico-chemical results obtained showed a clear degradation of the vegetable waters studied which displayed values higher than the standards fixed for all the parameters with the exception of the temperature and the pH. These effluents have a significant load of oxidisable and, in most cases, non-biodegradable. In fact, the multivariate statistical analysis (ACPN and HCPC) could show the effect of the extraction system used on the composition of the vegetable waters produced. However, a differentiation of their compositions according to their nature (fresh or stored in evaporation ponds) could not be distinguished. In conclusion, this work has shown that the variability of the composition of the vegetable waters studied is influenced by the olive oil extraction process, the storage techniques and especially the practices used throughout the mixing process, which is the most important element. Additional work must be carried out by controlling certain parameters (olive variety, temperature and quantity of the added water, etc.) in order to design sustainable and ecological strategies, for the management of these discharges, responding to this environmental concern.

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