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Effluent Characterization and Waterbody Monitoring from An Olive Pomace Oil Extractor Industry

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***Abstract*—The environmental impact is a relevant aspect of the production of Olive oil, it is a relevant Portuguese economic sector, with job creation, maintaining rural populations, however the by-product, olive mill wastewater and olive pomace, became one important focus to the circular economy, one of the many valorizations economic viable is the olive pomace oil. The olive pomace oil industry is responsible for a secondary oil extraction based in mixture organic solvent with the olive pomace from the olive oil production. This process produces a wastewater what was physicochemical characterized -COD, BOD, TS, TSS, FOG, organic nitrogen, ammonia and phosphorus content, pH and conductivity, phenolic compounds and aromaticity, and Zahn-Wellens biodegradation test - in this study as the first step to a wastewater design for the factory. The main objective is to be able to return the treated wastewater in the river next to the factory with the required quality by the Portuguese legislation. The river water was also characterized in four different points to evaluate the factory environmental impact, the results show no large variation in the analyzed parameters when compared with the current legislation.)**

Keywords—Industrial effluent, wastewater characterization, olive pomace oil production, water body monitoring.

I. INTRODUCTION

Olive oil is a relevant Portuguese economic sector, with job creation, maintaining rural populations, its production involves also environmental aspects that cannot be neglected [1]. Mass and energy resources are consumed throughout the stages of the olive oil production chain, from the cultivation and production of olives to the final consumption of the product, as well as produced gaseous, liquid, and solid residues with impacts on ecosystems. As the circular economy principles, these residues can still be part of new industrial processes before receiving treatment and final disposal [2].

Olive oil is one vegetable oil that only needs mechanical processes to be extracted and can be considered the juice from olives. First, the olive needs some pre-treatments such as removal of leaves and stones, separation, conservation, cleaning, and washing to guarantee a good quality the final product [1]. Then the olives are milled in a process called malaxation with a hammer or toothed-disc crushers and thermal bathing producing an olive paste. Using a liquid-solid separation process, the olive oil is separated from the olive pomace, producing as by-product the olive mill wastewater (OMWW) [3, 4].

The technologies for olive oil extraction became more efficient over the decades, today most extraction occurs in a 2-phases system to reduce the amount of wastewater produced by the traditional and the 3-phase process. Nonetheless, the production of olive pomace is 3 to 5 times bigger them the olive oil [1], evidencing the importance of the olive pomace oil industry even them the bagasse has around 8 to 10% olive oil remaining [4].

The main by-product, the olive pomace, goes to a second extraction, the remaining oil percentage is removed applying an organic solvent, this olive pomace oil can be refined and used in some countries in the food and cosmetic industries and biodiesel production. After the process, the exhausted pomace serves as fuel in the industry, and the wastewaters produced need to be treated to give a destination to water and organics compounds [5].

II. MATERIALS AND METHODS

The wastewater used in this study was collected on Mirabaga, an olive pomace oil extractor industry, in Mirandela (Portugal), between April and May 2021. The amount of wastewater was delivered to the laboratory and stored as required by the Standard Methods for the Examination of Water and Wastewater, (2005) to the analysis and tests. This study was carried out at the Chemical Process Laboratory, at the Polytechnic Institute de Braganza (IPB), Portugal.

A. *Wastewater characterization*

Appling the Standard Methods for the Examination of Water and Wastewater, [6] the parameters choose to be determined were pH and conductivity, Biochemical Oxygen Demand (BOD5), Chemical Oxygen Demand (COD), total phosphorus content, ammonia and organic nitrogen content, Fats, Oils and Grease (FOG), aromaticity, phenolic compounds, alkalinity, and Total Solids (TS) and Total Suspended Solids (TSS). Zahn-Wellens Biodegradation test were conduct as the OECD guideline for testing of chemicals (1992) [7].

B. *Water characterization*

Appling the Standard Methods for the Examination of Water and Wastewater [6], the parameters choose to be determined were temperature, conductivity, dissolved oxygen and total dissolved solids were measured in situ, pH and turbidity, Biochemical Oxygen Demand (BOD5), Chemical Oxygen Demand (COD), total phosphorus content, ammonia and organic nitrogen and nitrate content, oils and grease, aromaticity, phenolic compounds, alkalinity and Total Solids (TS), Total Suspended Solids (TSS) and Total Dissolved Solids (TDS).

III. OLIVE POMACE OIL EXTRACTOR INDUSTRY

According to the traditional method of solvent extraction, the olive pomace needs to be dried to 8% moisture before the addition of an organic solvent, although it reflects a minor part of the olive pomace sold to the second extraction industry [8]. In deterrence the high percentage of water in olive pomace oil some extraction facilities who receive the three types of olive bagasse have chosen to homogenize the moisture content of the pomace to be extracted as show in the Fig. 1, mixing the three types of bagasse until they reach a humidity of around 50% before a drying process. This process is important to stop fermentation reaction and preserve the reminiscent oil from hydrolysis and enzymatic deterioration [4].

After drying, the olive pomace is mixed with a solvent for the extraction of fats. This solvent needs to have no influence on the oil quality and physical properties, also a good selectivity and extraction power. As for other vegetable oils, the most used is n-hexane [9].

The solvent/oil mixture is collected from the batch extractors and steam is injected in a desolventizer operation to eliminate the hexane residue from the solids, and the oil/hexane mixture will be distilled to recover solvent [3]. The olive pomace oil obtained, also called crude pomace oil, differs from the virgin olive oil, it is more acid, has a dark green coloration due to its high chlorophyll content and unpleasant odor and taste, needing a refining procedure to become edible [10].

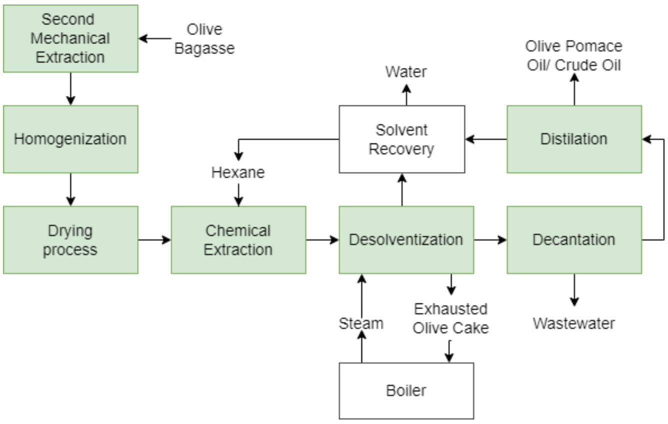

Fig. 1. *Olive Pomace Oil Production Process*

The by-products generated are biomass composed of stones and fat-free solids (or exhausted olive cake), and residual wastewater. The exhausted olive pomace can have the pulp separated from the rest and destinate to animal feed, the residue remaining is used as a fuel, reducing the fuel demand for steam production of the installation [9,10].

IV. WASTEWATER CHARACTERIZATION

Vegetable oil-producing industries are an important food industry, and as many have wastewater with specific characteristics, like color, odor, high BOD and COD, FOG is also the main pollutant in the wastewaters of the olive oil industry [11].

The first step to designing wastewater treatment is the characterization of them, identifying the pollutant composts present, estimate the volumes and the source of generation, in some cases a corrective action can be proposed to reduce or even eliminate the pollutant levels, this knowledge is also important to evaluate which of the next steps in wastewater treatments are required comparing the data to the specifications requested on the final effluent, obtained in the local legislation or equipment/process demands. Unfortunately, in wastewater treatment, zero production is close to a myth, and the best approach after trying to reduce the pollutant production is to observe valorization opportunities, and if is any, your treatment for recycling and reuse before your final disposal [9,11].

The wastewater was collected from two different locals in the factory. The first is the effluent as it leaves the process of olive pomace oil production the place and aspect can be seen in Fig. 2, (a) and (b). The second is prevenient from the second pool where the effluent is stored, the place and aspect are shown in Fig. 2 (c) and (d).

The visual aspect of the two wastewater is very distinct in color and turbidity. Effluent 1 is new, have a higher amount of BOD and suspended solids, as shown in Table 1, resulting in a lighter coloration and higher turbidity when compared to effluent 2, which is older and pass for a decantation process in the tank.

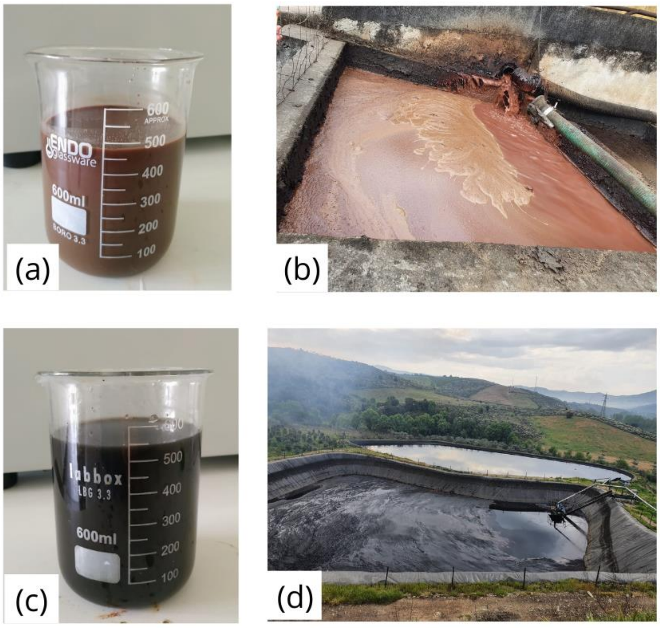

Fig. 2. *Industrial effluents and effluents collection sites*

TABLE I. OLIVE POMACE OIL WASTEWATER CHARACTERIZATION

Parameter	***Unit***	**Effluent**	
		1	***2***
pH	-	4.21	4.97
Conductivity	mS/cm	9.30	8.80
BOD_5	g O_2/L	48.40	15.01
COD	g O_2/L	126.64	28.52
Organic Nitrogen	g N/L	1.48	0.21
Ammoniacal Nitrogen	mg N/L	73.13	19.87
Total Phosphorus	g P/L	0.54	0.33
FOG	g/L	4.62	2.63
Phenolic compounds	g/L	7.82	1.42
Aromaticity	g/L	51.66	11.74
Alkalinity	g/L	3.77	4.71
TS	g/L	54.97	23.65
TSS	g/L	11.40	3.72

The polluting power of this effluent can be attributed to the high organic load, observed in the large concentration of BOD5 and COD, organic nitrogen, and phosphorus content. Ranade and Bhandari [11] attributed this to the presence of lipids, sugars, tannin, pectin and lignin, the high concentration of phenolic compounds as well as phosphate salts.

The BOD5/COD ratio is a good indicator of the biodegradability of wastewater, for effluent 1 this ratio is 0.38 and for effluent 2 is higher, 0.53, due to the degradation and sedimentation while stored. A higher ratio (>0.3) indicates a high biodegradable organic compounds fraction, and a biological treatment may be effective. The biodegradability is confirmed by the Zahn-Wellens test shown in Fig. 3.

In the Zahn-Wellens test, activated sludge from the local domestic wastewater treatment unit is used to degrade an amount of the two effluents (R1 and R2). The third reactor (C) for control is used to validate the results using ethylene glycol as a control substance to determine the sludge quality and adaptability. A blank reactor with the same conditions as determined for the OECD Guideline [7], was settled with no biodegradable substance to reduce influences in the COD determination by a colorimetric method due to parallel reactions and variations in the activated sludge. Due to the good adaptability of the activated sludge, in 14 days all the effluents were complete degraded.

Another crucial aspect is its acidity due to the presence of organic acids, capable of changing the pH of a waterbody if discarded without treatment. The high among of organic and inorganic compounds, that work as nutrients, can destabilize the natural condition on the recipient waterbody, favoring the growth of algae in the process acknowledge as eutrophication.

When compared with the Emission Limit Values in the discharge of wastewater present in Decree-Law No. 236/98 of 1st August [12], all parameters analyzed in the characterization of both effluent 1 and 2 are above the established limits, this confirms the need to implement a treatment unit for industrial effluents.

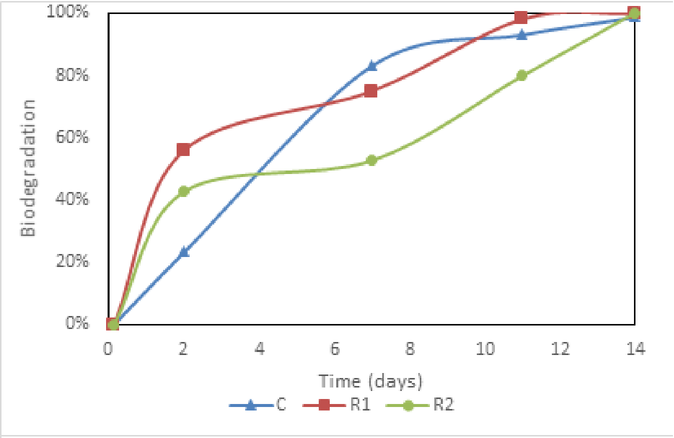

Fig. 3. *Zahn-Wellens Biodegradation Test Curves*

Analyzing previous works with effluents with a polluting load close to that found in effluents and from nearby matrices, a promising route for treatment is the reduction of solids and organic matter by pre-treatments such as coagulation/flocculation and flotation, followed by a biological reactor, followed by tertiary treatments such as filtering and polishing the effluent until the emission limit values are respected in their entirety.

V. WATERBODY QUALITY

To ensure the environmental safety of the river that receives the effluent of the factory, the water was collected in four different points of the river Tua (Fig. 4), located in the district of Braganza, Portugal.

The first upstream from the factory to observe the water quality before the factory, the second sample collected in the place the factor realize the wastewater. The third and fourth points were downstream the factory to evaluate the depuration of a pollution charge the factory relies on the river. Point four also corresponds to a pluvial beach accessible to the local population used for recreation and other activities.

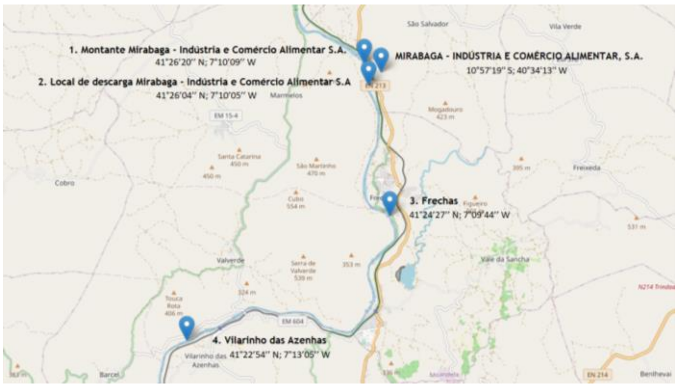

Fig. 4. *Tua river sampling points localization*

TABLE II. RIVER WATER CHARACTERIZATION IN DIFFERENT LOCATIONS

Parameter	**Unit**	**Collected point**			
		1	**2**	**3**	**4**
Temperature*	ºC	16.2	18.8	17.3	17.5
Conductivity*	µs/cm	47.3	56.5	46.5	45.9
TDS*	mg/L	26.9	31	26.3	25.7
Dissolved Oxygen*	%	95.4	87.1	92.5	94.1
pH	-	6.4	6.14	6.2	6.17
Turbidity	NTU	12	27	11	14
BOD_5	mg O2/L	< 10	30.57	< 10	< 10
COD	mg O2/L	13.58	86.07	15.23	24.27
TSS	mg/L	6.9	28.9	5.2	7.5
TDS	mg/L	61.7	111.3	52.4	49.4
Ammoniacal Nitrogen	mg/L	0.44	0.31	0.27	0.34
Nitrates	mg/L	0.69	0.39	0.09	0.51
Total Phosphorus	mg/L	0.02	0.01	0.02	0.03
Phenolic Compounds	mg/L	<DL	<DL	<DL	<DL
Aromaticity	mg/L	13.16	23.17	13.53	14.76

*: parameters measured in the local where the samples were collected
<DL: lower than the method detection limit.

The impact of industrial wastewater disposal is to be seen in point 2, the data shows a raise in parameters COD and BOD when compared with point 1, as noted in table 2. No effluent characterized previously in this article is released in the waterbody, this alterations in the values were due to the water used to cool the streams out the separation processes and the water recovered in the solvent recuperation process, the effluent pass for a cooling step before being pour in the river.

The waterbody has a good depuration capacity, with the parameters returning lower levels of organic matter as seen in point 3. In point 4, some attributes increase again due to the human activity present in the area.

The water quality was assured comparing the results with the Portuguese water legislation, Decree-Law No. 236/98 of 1 August [12], considering the maximum recommended value for irrigation and bathing waters, none of the parameters obtained values higher than those allowed.

VI. CONCLUSION

In order to obey the Portuguese legislation, the olive pomace oil extractor industry studied cannot dispose of industrial effluents as it is produced due to the amount of organic matter and other substances. The effluent must be treated before disposal, to this, a wastewater treatment plant must be constructed and dimensioned based on the amount produced daily and the characterization proved by the article. To provide an excellent environmental management to this factory, the waterbody must be monitored as required by current legislation, in the parameters determined by it and sampling frequency required for the maintenance of the quality validated in May 2021.

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