Research Note

Influence of the process parameters on the foam fractionation treatment of olive mill wastewater

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Abstract. Olive Mill Wastewater (OMW) is a highly organic load waste produced by the three-phase decanter system used in the processing of olives for oil that has phytotoxic and antibacterial phenolic compounds, which help this waste to resist biological degradation. Foam fractionation has not yet been studied with regard to its potential for treatment of such wastewaters. In the present study, this technique was investigated in a simple model system. The effects of different operational conditions, namely, surfactant concentration, process time, pH, and gas flow rate, were evaluated in this study. Statistical analysis of the fractional factorial design revealed that surfactant concentration, pH, and gas flow rate were the most influential process parameters. Low surfactant concentration and pH of 3-4 were found to be advantageous in terms of good Chemical Oxygen Demand (COD) removal. More than 80% of COD was removed through a single-stage foam fractionation process. This method has proved to be a feasible technique for the OMW effluent treatment.

1. Introduction

Olive Mill Wastewater (OMW) is generated by the olive oil mills during the extraction of olive oil [1]. Around 6×10^6 m^3 of OMW is produced yearly worldwide, of which 98% is produced in the Mediterranean basin [2]. The disposal of OMW is a problematic issue in the countries that are active in this field. High Chemical Oxygen Demand (COD) of OMW coupled with its phenol content inhibits the natural organic load degrading capability of the micro-flora in water bodies [3]. In most cases, these waters are dumped into the environment untreated. The inadequate and uncontrolled disposal methods of OMW to the water bodies pose an environmental concern as these effluents contain appreciable amounts of COD and BOD

concentrations and high amounts of microbial growth-inhibiting compounds, such as phenolic compounds and tannins [4-6].

In the last few years, there has been a great effort to develop new solutions to the treatment of OMW, including sedimentation [7], sand filtration [8,9], ozonation [10], membrane filtration [11-13], neutralization with addition of acid, advanced chemical oxidation (Fenton reaction) [2,14], adsorption by activated carbon, and aerobic and anaerobic digestions [15,16]. In an industrial scale, these technologies have drawbacks because they are too expensive to have a wide application, are ineffective in meeting stringent effluent standards, and could result in a huge amount of sludge [15,17-20]. A method with high throughput, low costs for operation, and a simple plant concept should be employed to eliminate costly separation steps, particularly in the early stages when large matrix problems can be expected [21-23].

Adsorptive bubble separation, including foam

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fractionation, is based on the selective adsorption or attachment of materials onto the surfaces of gas bubbles rising through a solution [21,24]. The foam fractionation of ions is similar to ion flotation, but uses an excess of surfactant or a proper frother to produce stable foam [18-20,25]. During foam fractionation, gas bubbles are introduced into a liquid, which contains surface-active substances [26]. Foam formation occurs when surface-active molecules attach to the gas-liquid interface of the introduced bubbles. The foam formation can be caused by dissolved or dispersed substances. The surface tension of the interface is decreased and the foam is stabilized. Thus, the formed bubbles create an emerging foam column above the liquid level. The entrained liquid eventually drains back into the feed solution. This causes the foam to become dryer while rising [27-29]. Coalescence and disproportionation inside the dry foam increase bubble size. Thus, the concentration of surface-active substances between lamellae and Plateau borders increases [25]. The emerging foam column can be diverted into a separate vessel, where the foam is collapsed, e.g. mechanically or by decreased pressure. The collapsed foam, called foamate, comprises an enriched solution of surface-active components [30,31]. This principle can be used for the separation of organic substances from polluted industrial wastewaters. Such process offers many advantages for the treatment of industrial wastewaters compared to other treatment processes, including low space and energy requirements; simple plant design, operation, and scale-up; and low capital and operating costs [19,32]. Not only surfactants are removed by adsorption at the air-liquid interfaces, but other components that form complexes with the surfactants also tend to be concentrated [20,21,33]. Foam fractionation has some other major advantages over other techniques in the field of OMW treatment. There are no solvents required during this process, because only air or inert gases are involved [34-37]. In terms of sustainability, this process can thus be considered as 'green' [38]. Unlike solvent extraction processes, no co-extraction of other compounds such as fatty acids and chlorophyll is required [39].

Foam fractionation is subject to various influencing parameters which, furthermore, are not necessarily independent of each other. For this reason, this process was investigated by means of Design of Experiments (DoE). DoE has shown its benefit in the establishment of foam fractionation in biotechnological applications [21-24,40] and oxidative treatment of OMW [25-27,41], for example. The simultaneous variation of several process parameters combined with statistical analysis is advantageous with regard to the smaller number of experiments compared to the conventional concept of "one factor at a time".

The scope of this study was to evaluate such a technique for the OMW treatment. The influence of pH, surfactant concentration, process time, and gas flow rate was investigated by fractional factorial design in order to find the most economical and effective process conditions, which could make this technique exploitable for treating OMW at industrial scales.

2. Material and methods

2.1. Collection and preservation of OMW

Fresh OMW was collected during the 4th quarter of 2014 from an industrial estate in Isfahan. The OMW samples were collected in 5-L plastic containers and transported immediately to the laboratory and stored at approximately 7°C. They were digested in their original form, which means that they were not subjected to any alteration. The physicochemical characterization of OMW is presented in Table 1.

2.2. Analytical procedures

The COD analyses were performed by the dichromate closed reflux Colorimetric Method using a Hach DR2010 Model spectrophotometer in accordance with Standard Methods. Total polyphenols were determined spectrophotometrically according to the Folin-Ciocalteau Method [40]. Other wastewater parameters were analyzed according to Standard Methods. All pH values were determined with a scale-reading precision of ±0.01 on HACH H260G pH meter. Nitrogen, 5N grade, was distributed by Iranian Electronic Industries (IEI). The flow rate of nitrogen was regulated by a GCR Compact Regulator of HTK Hamburg GmbH (Hamburg, Germany).

2.3. Chemicals

Cetyltrimethylammonium bromide (CTAB) was purchased from BDH and was of analytical grade. HCl (37%) and NaOH (97%) were obtained from Sigma-Aldrich (Seelze, Germany). The CTAB solution (10 mg/mL) was dissolved in pure water under slight warming.

2.4. Apparatus

The experiments were conducted in a batch system, shown in Figure 1. The column was a Pyrex cylinder with internal diameter of $d_i = 10.0$ cm. The column

Table 1. Physico-chemical characterization of OMW.

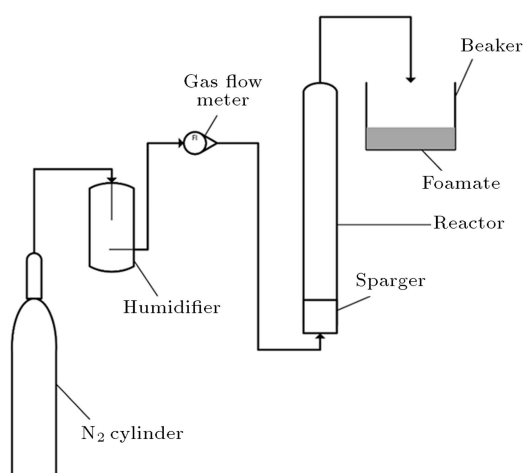| Parameter	Value
pH	4.7
COD	58.42 gL^{-1}
BOD$_5$	9.67 gL^{-1}
Total suspended solids	5.43 gL^{-1}
Total polyphenols	0.41 gL^{-1}
Biodegradability (BOD$_5$/COD)	0.165

Figure 1. Experimental setup for foam fractionation.

was equipped with a sintered glass frit (ASTM 40-60 type C). The bubbles formed by this type of frit had diameters that were distributed roughly between 0.4 and 1.2 mm. Nitrogen gas was passed through a fritted wash bottle filled with water to humidify it and minimize evaporation losses in the feed solution. To ensure good passage of the gas through the pores of the frit, the nitrogen flow was always activated in advance to fill the glass column with liquid via a funnel. Through a Π-formed glass tube, the rising foam was piped into a beaker. The schematic of the experimental setup is presented in Figure 1. All experiments were carried out at ambient temperature, $16 \pm 2°C$. After each experiment, the column was thoroughly cleaned with water and acetone and let dry before further use.

2.5. Experimental procedures
A 30-minute period was assigned to all solutions prior to beginning the experiments for equilibration considerations [41]. The volume of the feed solution was always 50 mL. The pH of the stock solutions was adjusted by adding normal solutions of either HCl or NaOH before starting the experiments. Samples analysis began at the end of each experiment by pipetting aliquots of liquid from the reactor solution.

The performance of foam fractionation is expressed by one criterion, i.e. Enrichment Factor (EF), which describes the ratio of the concentration of the target compound in the feed (COD_{feed}) minus the concentration of the target compound in the foamate ($COD_{foamate}$) to that in the feed, all subtracted from 1 (Eq. (1)). The higher the EF, the richer is the foamate in the COD.

$$EF = 1 - \frac{COD_{feed} - COD_{fomate}}{COD_{feed}}. \qquad (1)$$

2.6. Design of experiments
The influence of the 4 process parameters, i.e. pH, surfactant concentration, process time, and gas flow

rate, should be determined. Thus, based on a 2^4 plan, a fractional factorial design matrix is developed for optimization of the foam fractionation process. Blocked fractional factorial design reveals the main effects and interaction effects on the performance criteria of the 4 variables. Gas flow rate should not be lower than 20 mL/min to sustain formation and discharge of foam. Furthermore, the upper pH value is limited to pH 7. The enrichment factor is employed as the response factor for the process of foam fractionation.

3. Results and discussion

3.1. Statistical analysis
3.1.1. Enrichment factor
The influence of the input variables on each response factor is visualized in Pareto charts (Figure 2). The length of each bar represents the extent of the influence of the corresponding parameter. EF is negatively influenced by concentration of CTAB and pH. This means these two factors on a high level result in a low EF. In contrast, higher gas flow rate leads to high enrichment in the foam. Three interaction effects (AD, AB, and AC) are significant enough for EF to be taken into account. This means the effect of such a factor is influenced by the level of the second factor. The impact of the individual input variables on each response factor can be depicted in the plot of main effects. The diagram shows the calculated values of EF by the fitted model, which are obtained by increasing the factors from their minimum (left) to their maximum level (right). The slope and orientation of the graphs reflect the extent and the character (negative/positive) of the impact. Only significant effects are considered. For EF, the plot of main effects (Figure 3) clearly illustrates that high levels of two input variables (CTAB and pH) result in a decrease of EF. In contrast, changing the gas flow rate value from a low to a high level leads to high
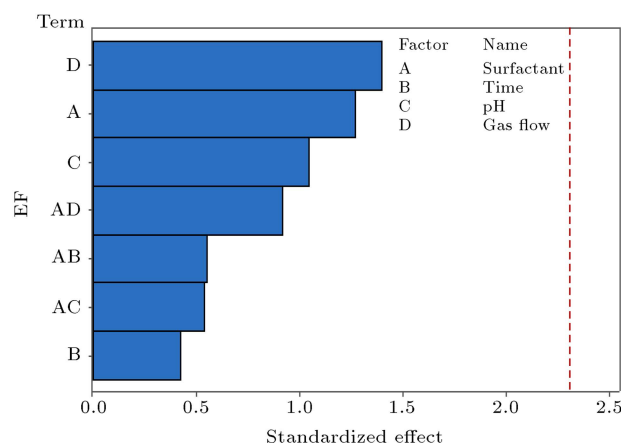

Figure 2. Pareto diagram for enrichment factor in the foamate. No significant effects are observed since no column crosses the vertical line (significance level 0.05).
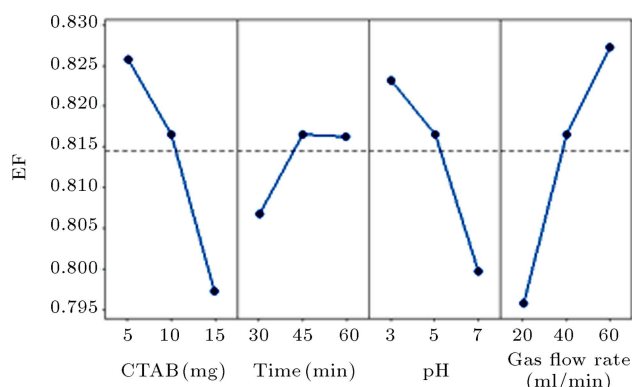
Figure 3. Plot of the main effects for enrichment factor in the foamate. Minimum (right side) and maximum (left side) levels are indicated for each input variable.

enrichment factors. Process time should be considered to remain at optimum values. As it is demonstrated, the extent of the influence and the impact of the process parameters (Figures 2 and 3, respectively) are in good agreement with each other.

3.1.2. Effect of CTAB concentration (A)

One of the most important impact factors on EF was found to be CTAB concentration. The tested concentrations in this study were below the critical micelle concentration of CTAB, which is 9.2×10^{-4} M [39]. As a foaming agent, CTAB influenced the wetness of the foam. The higher its concentration, the "wetter" is the foam. This implies a high content of entrained bulk solution in the foam. This results in an increased discharge of liquid phase with the foamate resulting in low EF. Thus, for the optimization of the response factor, a compromise has to be found between this input variable and others which will be mentioned later.

3.1.3. Effect of process time (B)

Amongst the investigated process parameters, "time" has shown the lowest impact on the response factor. The optimum process time in this study is pointed out to be around 45 minutes. Lower process time has negative impact on the response since the time required for the adsorption to take place is not adequate. Contrary to the latter, since the foamate volume increases over time, longer process time also has negative impact on the response. This means that process time has deeper influence on the wetness of the foamate than the gas flow rate and should be monitored carefully.

3.1.4. Effect of pH (C)

The pH effect was particularly studied through experiments, which showed the COD reduction of olive mill wastewater. These results also revealed that foam fractionation was a highly pH-sensitive process. The pH value of the feed solution determines the degree of deprotonated organic pollutants. The lower the deprotonation, the higher the adsorption. For the

response factor, pH 3-4 was found to be optimum. Many organic molecules can undergo a series of autoxidation reactions in the basic to neutral conditions. Such reactions result in production of some species which, consequently, have poor interaction with the cationic surfactant. Thus, to prevent autoxidation reactions, lower pH is preferred to near-neutral and basic conditions.

3.1.5. Effect of gas flow rate (D)

Statistical analysis based on the DoE (Table 2) revealed a positive effect of gas flow rate on EF. Gas flow rate significantly influences the wetness of the rising foam and, therefore, determines the liquid fraction of the foamate. Low gas flow rates make the foam rise slowly inside the column, providing more time for the processes like coalescence and drainage of superfluous liquid. The former is based on the tendency of smaller gas bubbles to combine into one bigger gas bubble with a reduced surface compared to the aggregate surface

Table 2. Fractional factorial design in four blocks for four variables (randomized).

Run number	Block	A	B	C	D	EF	Phenolic removal (%)
1	3	0	0	0	0	0.782	91.21
2	3	−	+	−	+	0.851	99.26
3	3	0	0	0	0	0.856	99.84
4	3	0	0	0	0	0.830	99.81
5	3	+	−	+	−	0.758	88.41
6	2	+	+	−	−	0.791	92.26
7	2	0	0	0	0	0.840	97.98
8	2	0	0	0	0	0.804	93.77
9	2	−	−	+	+	0.818	95.49
10	2	0	0	0	0	0.825	96.17
11	4	+	+	+	+	0.798	93.08
12	4	−	−	−	−	0.809	94.66
13	4	0	0	0	0	0.831	96.93
14	4	0	0	0	0	0.839	97.86
15	4	0	0	0	0	0.763	89.07
16	1	0	0	0	0	0.790	92.12
17	1	0	0	0	0	0.836	97.51
18	1	+	−	−	+	0.842	98.29
19	1	−	+	+	−	0.825	96.23
20	1	0	0	0	0	0.802	93.71
Variables		−	+	0			
A: Surfactant (mg)		5	15	10			
B: Time (min)		20	60	45			
C: pH		3	7	5			
D: Gas flow rate (ml/min)		20	60	40			

of the small bubbles. Liberated water molecules can then drain off back to the feed while surface-active compounds stay adsorbed at the gas-liquid interface within the foam. Higher gas flow rates result in much more turbulence in the column; therefore, the impact between bubbles and the sample significantly increases. Although foamate volume increases, the influence of the increased impact prevails too much over this problematic angle of the experiment so that in the end, the target concentration in surface-active molecules is higher.

3.2. Optimization

Only statistically significant effects were taken into account in identifying optimum parameters for the foam fractionation process in the statistical model. As demonstrated, all main effects were statistically significant for the response factor. However, one has to keep in mind that the requested performance criterion of effective foam fractionation was a maximum Enrichment Factor (EF); therefore, the efficiency of extraction increases with the factors which have positive impact on the enrichment factor. Upper and lower limits of the input variables were determined by the corner and center points.

The foam fractionation process aims at generating foam highly enriched in the target molecules. In addition, the target should be extracted in high quantities. Therefore, in a second step, the model was used to calculate optimum parameters for achieving high EF. The response was weighted 1 in the corresponding optimization.

Considering these lower and upper limits of the experimental design, values of process parameters for achieving highest EF were calculated (Table 3). "Desirability" reflects the experimenter's acceptance of the degree by which the desired optimization can be fulfilled. It can vary from 0 to 1, with 1 corresponding to the optimum for all response factors. In our optimization process, desirability reached 0.930.

If all the factors are taken into account, one can get the following equation by using a linear parameter

Table 3. Predicted optimum values for the operational parameters to achieve maximum enrichment in the foam.

Factor	Lower limit	Upper limit	Optimum for maximum EF
A: Surfactant (mg)	3.00	21.00	7.29
B: Time (min)	15.00	50.00	42.00
C: pH	2.00	6.00	3.00
D: Gas flow rate (ml/min)	20.00	80.00	55.00
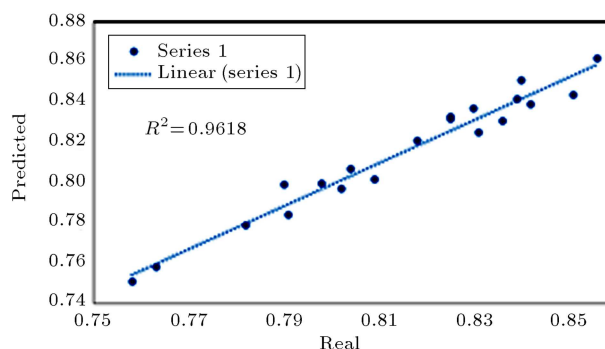
Figure 4. EF values: Predicted vs. real.

model based on the actual factors:

$$EF = 0.790 + 0.0005 \text{ A} + 0.00138 \text{ B} + 0.0020 \text{ C}$$

$$- 0.00055 \text{ D} - 0.000107 \text{ AB} - 0.00078 \text{ AC}$$

$$+ 0.000133 \text{ AD}. \tag{2}$$

The analysis of variance (ANOVA) table of the statistical analysis is provided in Table 4. As can be observed, although the P-values of the factors, with complete agreement with Pareto chart, indicate that there is no significant effect from any of the four factors, the P-value of the model shows the significance of the model itself. It can be emphasized that the predicted values obtained from the empirical model (Eq. (2)) are in very good agreement with actual experimental data.

The observed mean values of the experiments were compared with the predicted values (Figure 4). The figure shows that the model is acceptable and the proposed empirical model is suitable for predicting the EF value. The regression squared (R-sq.) of the model is equal to 0.9618 and is in good agreement with the adjusted R-sq., which is 0.9080. The F-value is 35.11, which shows that the model equation is a safe model for the purpose of system navigation.

4. Conclusion

Several methods have so far been proposed for OMW treatment; however, very little research has been performed regarding the treatment of OMW using foam fractionation technique. In this study, a simple low-cost method was proposed for treating the OMW produced by the olive oil mills. The overall results of this study indicate that the application of foam fractionation is a feasible method to partially treat olive mill wastewaters, allowing to achieve a significant decrease of COD. Design of experiments was used to find optimum experimental conditions for effective COD removal (high EF) process. Results prove that more than 80% of the OMW COD can be removed through a single stage in fractionation process. This

Table 4. ANOVA of the statistical analysis.

Source	Sum of squares	R^2	Adj. R^2	F-value	P-value
Model	3.983E-003	0.9618	0.9080	35.11	0.043
A: Surfactant	2.420E-004	0.8820	0.8715	9.17	0.149
B: Time	1.800E-003	0.9492	0.9122	4.18	0.482
C: pH	1.250E-003	0.9211	0.8956	8.71	0.241
D: Gas flow rate	6.912E-004	0.8677	0.8273	11.40	0.107
AB	2.383E-003	0.9019	0.8887	5.98	0.519
AC	3.379E-004	0.9887	0.9530	5.07	0.503
AD	2.764E-003	0.9535	0.9133	7.39	0.587
BC	5.945E-004	0.8891	0.8664	2.66	0.811
BD	1.913E-003	0.9114	0.8976	3.79	0.634
CD	3.566E-003	0.8940	0.8566	1.90	0.890
Curvature	3.333E-006				
Residual	0.011				
Lack of fit	1.932E-003				
Pure error	8.650E-003				
Cor. total	0.015				

study also pointed out that low concentrations of surfactant in acidic conditions with a proper gas flow rate were favorable in this process.

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