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STUDY OF AN OILY WATER TREATMENT PROCESS IN A PILOT HYBRID SYSTEM COMBINING AIR FLOTATION AND A CONSTRUCTED WETLAND: DATA ANALYSIS, EFFICIENCY OPTIMIZATION AND SCALE-UP

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Abstract

In this study, we developed a hybrid system for the treatment of oily water composed of two pilot-scale prototypes combining physicochemical and biological separation methods. Oily water flowed through a dissolved air flotation (DAF) prototype in the first step. At the exit of this prototype, part of the water was saturated with atmospheric air and sent back into the DAF chamber with the aid of a centrifuge pump and the injection of microbubbles. The other part of the treated water fed a constructed wetland prototype involving floating macrophytes of the species *Eichhornia crassipes*. With the aid of a central composite rotatable design (CCRD, with two operating parameters) and the response surface methodology (RSM), a predictive statistical model was created for the operating conditions associated with an optimal oil-water separation efficiency of around 97% of the initial concentration of 120 ppm of oil, which is much higher than the concentration permitted by environmental legislation in Brazil (20 ppm). The efficiency results are discussed and interpreted based on the phenomena of physical and mechanical liquid particles. The combination of DAF and constructed wetland methods for oily water treatment without the addition of a coagulant/flocculant agent achieved quite satisfactory results and proved to be an eco-friendly technology. Hydraulic similarity laws were used to obtain scale-up correlations for designing the size of pilot plant equipment for the treatment of oily water with the proposed hybrid process.

Key words: central composite rotatable design, dissolved air flotation, *Eichhornia crassipes*, liquid-liquid separation, oily water, wetlands

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1. Introduction

Residual oily water results from the production, transportation and use of petroleum and petroleum by-products. The shearing caused by pumps, valves and other equipment disperses oil in water, forming an emulsion that can be highly stabilized by the presence of finely divided solids,

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natural surfactant substances and other reagents added during the production process of lubricating oils and fuels (Rocha e Silva et al., 2018). The discarding of this residue is only permitted after the removal of oil and suspended solids to acceptable international levels (Rocha e Silva et al., 2018). The increase in the volume of residual oily water in the past decade and the discharge of this type of effluent into the environment has become a significant concern (Al-Ghouti et al., 2019; Jafarinejad and Jiang, 2019).

Free-floating oil is easily removed with a decanter, hydrocyclone, centrifuge or flotation device capable of separating suspended oil droplets due to the difference in density (Santander et al., 2011). However, it is not enough to remove only the free-floating oil; the removal of oil in emulsion form is equally essential. Oily water is conventionally treated with different physical, chemical and biological methods (Rocha e Silva et al., 2018). Compact physicochemical systems are commonly employed for situations in which space is limited, such as offshore rigs. However, many chemical treatments are costly and produce harmful sludge. At onshore facilities, the addition of a biological post-treatment polishing step is an environmentally friendly method (Abdullaha et al., 2020; Stefanakis et al., 2016). Methods such as ultrafiltration and/or membrane filtration have been used to refine the final effluent (Lu et al., 2018; Tawalbeh et al., 2018). When there is a need for less expensive solutions, efforts can be concentrated on combining physicochemical and biological treatment technologies for the purposes of reuse or safer discharge (Rocha e Silva et al., 2015).

In recent years, a physicochemical method denominated dissolved air flotation (DAF) has demonstrated promising results in the efficient separation of liquid-liquid and non-miscible mixtures, such as oil in water. With this method, microbubbles produced with a gas (generally air) are added to the oily water and adhere to the oil droplets in a stable manner due to the hydrophobic nature of oil. This leads to the formation of an oil-microbubble system with a lower density than the surrounding water, regardless of the addition of a chemical agent to destabilize the emulsion. As separation speed is also directly proportional to the difference in the densities of the phases, the formed oil-gas system tends to migrate to the surface of the medium much faster and more efficiently, where it accumulates and can be easily removed (Rocha e Silva et al., 2018).

Constructed wetlands with surface flow are constitute a biological treatment technology inspired by natural environments, such as lakes and swamps, and can be used in the removal of excess nutrients and pollutants, such as heavy metals and oily residues (Vymazal, 2014). Such systems consist of a channel, input and output devices, and aquatic plants (macrophytes). The operational ease and considerable adaptability of the constructed wetland design favor pollutant removal mechanisms. The use of additional equipment provides greater hydraulic control of the wetlands, thereby optimizing the treatment process.

An effluent treatment system based on aquatic macrophytes may be defined as a natural process in which the plants play the role in the removal of residues (Lakatos et al., 2014). Aquatic macrophytes constitute an important community in shallow-water ecosystems with a high rate of primary production, participating in nutrient recycling and providing food for herbivores and detritivores. Floodplain lakes in Brazil are rich in aquatic macrophytes, including the abundant free-floating *Eichhornia crassipes* (Mart.) Solms (Pontederiaceae) (Villamagna and Murphy, 2010).

E. crassipes, also known as water hyacinth, is one of the most widely used species in constructed wetlands due to its adaptation to hostile environments. Its low sensitivity to oil makes this species suitable for use in the treatment of oily effluents in constructed wetlands (Crema et al., 2012). The water hyacinth root system vertically fills the entire length of the wetland, increasing the contact of the effluent with microorganisms on the macrophyte roots and slowing the flow of the fluid to enhance the effect of gravity on the pollutants (Gunathilakae et al., 2018; Kadlec and Wallace, 2009).

In this work, a synthetic lubricating oil was treated in a pilot hybrid system combining a DAF prototype without addition of chemical collectors and a constructed wetland prototype involving *E. crassipes*. The efficiency of the treatment process in the single hybrid configuration was evaluated based on two operational parameters and modelled using a statistical approach. The results were interpreted considering physical-mechanical phenomena and the process was optimized by adjusting the operational conditions with the aid of a central composite rotatable design (CCRD) for maximal efficiency of the treatment process. We complete the study by dimensioning the different equipment involved in the hybrid plant to enable an industrial scale-up using dimensionless analysis.

2. Material and methods

2.1. Materials

Individuals of *E. crassipes* (higher than 20 cm) were collected from natural freshwater habitats in the city of Recife, Brazil, where the species is abundant. This macrophyte was chosen due to its tolerance to oil.

Oily water was prepared with 120 ppm of lubricating oil. The concentration of the synthetic effluent was obtained by saturating water with the oil through intense mixing with the aid of a centrifugal pump and a drum. Preliminary experimental tests demonstrated a constant oil content in the water by physical saturation, thereby achieving a synthetic effluent consisting of a constant concentration of the oil-water slurry. Motor oil (type SAE 20W-50; PETROBRAS) was used as the contaminant oil. This flex engine oil (gasoline, VNG and alcohol) consists of a paraffin-based lubricating oil, i.e., a complex mixture of hydrocarbons and additives.

2.2. Dissolved air flotation pilot unit

Fig. 1 shows the side view of the DAF pilot prototype previously designed and constructed for oil-water separation tests using a synthetic fluid (Vasconcelos et al., 2015). An oily water input duct is seen on the left of the Fig. (1). The fluid enters the lower portion of the mixture chamber (2), which is equipped with a mechanical agitator (3) driven by an electric motor (4). A test chamber was also designed for use when an auxiliary oil-in-water emulsion destabilization substance is needed (5). A static mixer is used for the destabilization of the emulsion (6). The oil-in-water emulsion comes into contact with the flow of microbubbles emitted from a diffuser (7). In the first floatation chamber (8), oily foam appears as the result of the aggregation of the air microbubbles and oil droplets dispersed in the aqueous phase. Through flotation due to differences in density, the oil foam is gathered into a collector (9) and the water passes through a second flotation chamber (10) for the interaction with an additional flow of microbubbles. The flow of microbubbles reaches the flotation chambers through distribution ducts (11) with the aid of a valve (12) for pressurization and control. The treated water is then sent through two subsequent chambers (13 and 14), the latter of which (14) has a centrifuge pump (15) that re-circulates part of the water for the production of microbubbles. At the base of this chamber, a duct (16) releases the treated water for reuse or discharge into the original aquatic environment. The floated oil is collected by ducts at the surface for a suitable treatment. All experiments were conducted for one hour.

A large part of the DAF pilot prototype, such as the flotation chamber walls, was made from transparent acrylic measuring 10 mm in thickness. The framework of the prototype was made from type L carbon steel measuring 1 inch in thickness. The prototype can hold a total volume of 1.55 m^3. Levelling of the framework was necessary to achieve greater separation efficiency of the floated oily foam and water column.

The level of liquid in the DAF prototype was monitored with the aid of the LabView computational software program (Galdino et al., 2015). Control and monitoring activities of the process were performed. Different reference values ("set points") were stipulated to define the height of the interface between the water and oily foam formed in the DAF chamber. The information from the sensor was sent to an interface through a data acquisition board. The aqueous flows were measured by the YF-S403 and YF-S201 sensors and air flow was controlled by a rotameter from 1 to 15 $L \cdot h^{-1}$.

2.3. Constructed wetland pilot prototype

As a biological step of the treatment system, a constructed wetland pilot prototype was designed involving the floating macrophyte *E. crassipes* (Ansari et al., 2020; Colares et al., 2020). The wetland prototype was based on three analogies through operational similarities (Kunes, 2012): i) rectangular channel with width twice the height of the sides and the upper portion of the liquid subjected to atmospheric pressure. The movement does not depend on internal pressure, but rather the slope of the bottom of the wetland and the surface of the liquid; ii) continuous decanter - the two phases continuously enter one end of the wetland. The length-to-hydraulic diameter ratio enables adequate separation conditions for the two phases. The flow of treated water is continuously removed from the wetland with the aid of closed conduits located at the output end of the equipment.

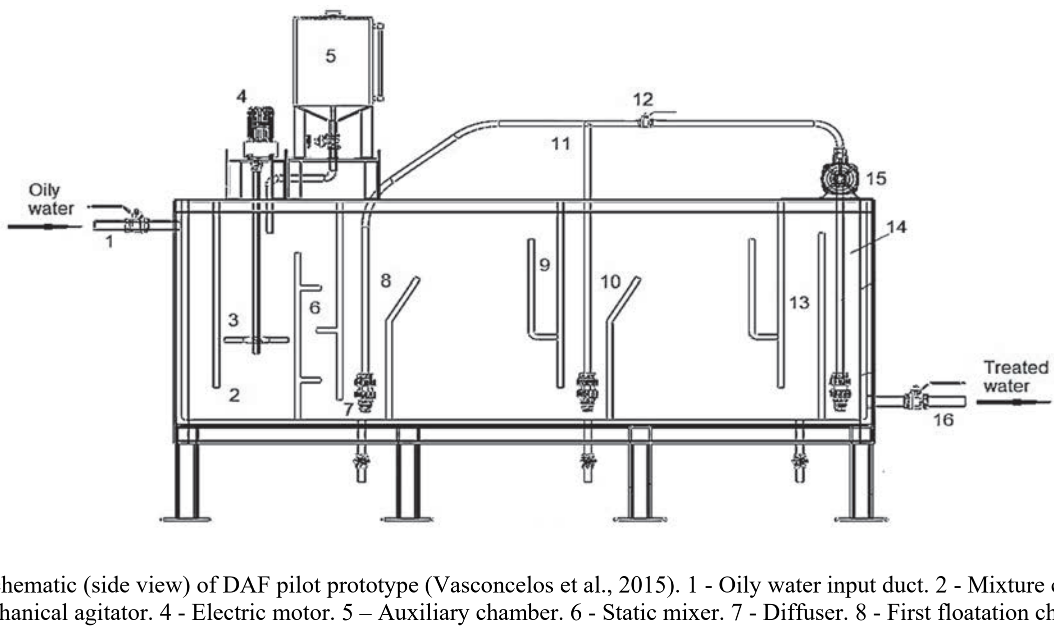

Fig. 1. Schematic (side view) of DAF pilot prototype (Vasconcelos et al., 2015). 1 - Oily water input duct. 2 - Mixture chamber. 3 - Mechanical agitator. 4 - Electric motor. 5 – Auxiliary chamber. 6 - Static mixer. 7 - Diffuser. 8 - First floatation chamber. 9 – Collector. 10 - Second flotation chamber. 11 - Distribution ducts. 12 - Pressurization and control valve. 13 and 14 - Subsequent chambers. 15 - Centrifuge pump. 16 – Duct

A length-to-hydraulic diameter ratio greater than tenfold enables excellent separation efficiency; iii) a semi-continuous reactor characterized by the maintenance of the organic phase in contact with the macrophyte shoots (batch system) and a continuous flow of the aqueous phase. First-order kinetics were adopted based on previous studies to enable the estimation of the kinetic constant of the reaction and define the period of pruning of the plants (macrophyte density). The number of seedlings was based on the kinetics of a first-order reaction (Lakatos et al., 2014).

Fig. 2 displays a partial view of the constructed wetland pilot prototype. Four wetland units were constructed with plates of transparent polycarbonate measuring 10 mm in thickness (dimensions: 3 m in length, 0.6 m in height and 0.8 m in width). After installation in an open location, the wetland prototypes were filled with water to a height of 0.4 m and each tank was stocked with 40 seedlings of the species E. crassipes.

Fig. 2. Wetlands constructed in transparent polycarbonate with floating macrophytes

The height of the runoff liquid was maintained at 60% of the height of the channel to avoid overflow in cases of excessive rainfall. The macrophyte density in each wetland was between 30 and 40 seedlings per meter squared (Kunes, 2012). The liquid is fed into one of the ends with the aid of a perforated duct to enable the even distribution of the oily water. The removal of the treated liquid (aqueous phase) is performed using a perforated duct at the base of the wetland to avoid including the oily surface layer.

2.4. Combination of prototypes

For the simultaneous tests of the physicochemical (DAF) and biological (constructed wetland) treatment of oily water, a hybrid system was designed combining both methods. Three horizontal chambers were used as wetlands and an extra chamber was built to ensure total oil absorption by *E. crassipes*, if necessary. For such, an auxiliary oily water production system was designed using the recirculation of all treated water, favoring the reuse of the water. The water exiting the DAF prototype entered the wetland prototype and the water exiting the wetland was sent back to the oily water production system prior to feeding the DAF prototype (Fig. 3). However, a control system was needed for the implantation of the hybrid system. The recirculation pump was adjusted for the same aqueous flow from the DAF entrance.

For the operation of the prototypes to occur in a static state, a level sensor was installed in the treated water chamber of the DAF prototype to monitor maximum and minimum water levels. When the level in the chamber reached the programmed minimum, the re-circulation pump was switched off. When the level reached the programmed maximum, the mixture pump or feed pump was switched off. The oil content in the liquid of the DAF prototype was maintained at 120 ppm throughout the tests.

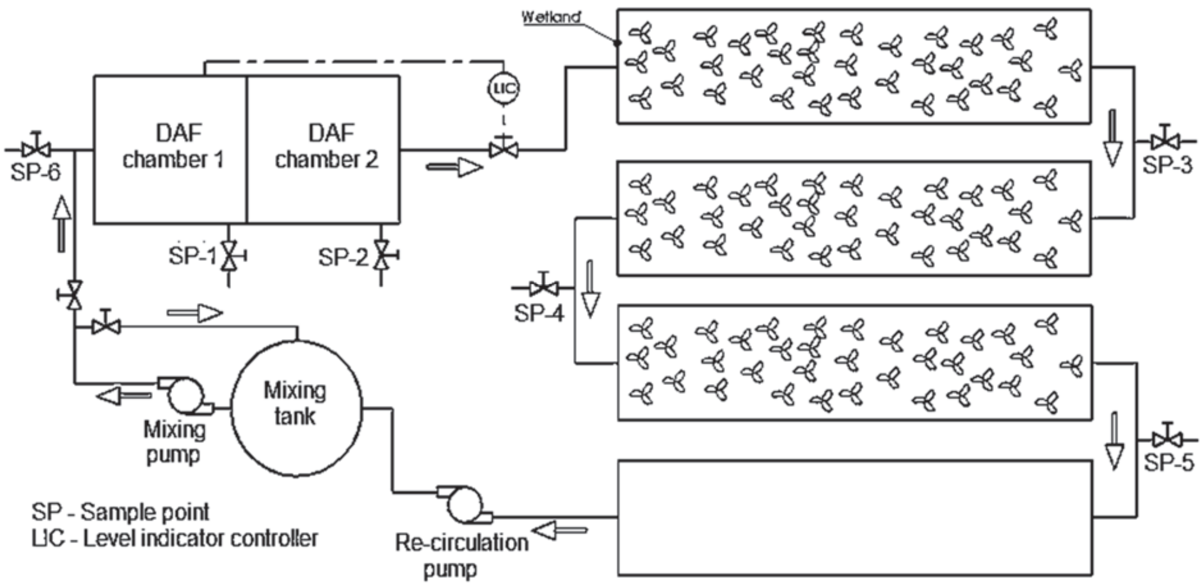

Fig. 3. Schematic of control strategy for combination of DAF and wetland prototypes

2.5. Quantification of oil removal

Oil was measured at the beginning and end of the wetlands to quantify the amount after the DAF and after the combination of the DAF and wetlands, respectively. The amount of oil in the water after treatment was determined gravimetrically as the amount of material after extraction with hexane. The oil removal rate (%) was calculated using (Eq. 1):

$$Oil\ removed = \frac{O_i - O_r}{O_i} \cdot 100\% \quad (1)$$

in which: O_i - initial oil in water, mg·L^{-1}; O_r - remaining oil in water after treatment, mg·L^{-1};

2.6. Experimental factorial design and response surface methodology

Response surface methodology (RSM) was used in conjunction with a central composite rotatable design (CCRD), which requires fewer tests than a full factorial design to establish functional relations between two operating variables (Bezerra et al., 2008). RSM was used with the aid of the Statistica software program (StatSoft®, version 12). The CCDR was employed with the ratio between the air and water outflows for microbubble production (X_1) and the ratio between the feed outflow of the wetland prototype and water for microbubble production (X_2). A total of $2^k + 2k + n_0$ experiments were needed, in which k is the number of independent variables and n_0 is the number of repetitions at the central point so that the planning could be characterized as rotational, totaling 36 runs for 12 assays (three runs per assay). Table 1 displays the independent variables and respective calculated levels.

Physical meaning of X_1: The numerator is the flow of air aspirated by the centrifugal pump. This liquid flow of the pump is controlled by a valve connected to a computer for digital monitoring. The aspirated air passes through a rotameter, which measures the flow and keeps the pressure and flow of the recirculating water from the centrifugal pump constant. Since the water flow of the centrifugal pump is constant (denominator) and maintains the pressure invariable, $X_1 \cdot 10^3$ increases from 0.24 to 1.39 with the control of the gas rotameter valve and the air is distributed in the DAF system in the form of microbubbles. As the gas flow increases, X_1 increases; the gas retention (hold-up) in the DAF cell increases and the size of the microbubbles is affected by coalescence. These two quantities (hold-up and bubble size) directly affect the interfacial area of gas/liquid (oil) contact, but in an antagonistic way. As the bubble size increases, the interfacial area of gas/oil contact increases. As X_1 is increased, the gas hold-up (the amount of gas present per unit of liquid volume) increases, which tends to increase the interfacial area, favoring the efficiency of oil/water separation. At the same time, bubble size also increases due to the coalescence of the bubbles, reducing the interfacial area of gas/oil contact. The result of these two antagonistic phenomena defines the final value of the interfacial area.

Physical meaning of X_2: The numerator is the flow of the liquid leaving the wetland tanks and returning to feed the DAF system, considering the flow of the recycled liquid in the mixing tank negligible in comparison to the flow of liquid leaving the wetland. The denominator is the constant flow of water from the microbubbles produced by the centrifugal pump. If X_2 increases from 0.11 to 0.39, the flow rate of the wetland recirculation liquid is increased and the turbulence in the DAF system is increased. The increase in the flow rate of the wetland recirculation liquid also leads to a decrease in the contact time between the microbubbles and oil droplets. Oil removal efficiency depends on the interfacial area (hold-up and microbubble size) and microbubble/oil droplet contact time.

3. Results and discussion

In this section, we discuss the results obtained in the different stages of the experimental tests using the CCRD, with an evaluation of the effect of the two operational parameters investigated (X_1 and X_2). We demonstrate from these studies the efficiency of the DAF (Y_0) stage and overall efficiency when combining the DAF and constructed wetlands (Y_1) for the separation of a synthetic oil/water mixture.

A statistical model was created and efficiency is discussed based on the physical and mechanical phenomena of the suspension of the liquid particles. In a final step, dimensionless analysis was applied to dimension the equipment involved in the hybrid oil/water mixture separation process without the addition of coagulants/flocculants for operation on an industrial scale based on data obtained in the pilot system studied in this work.

3.1. Efficiency of DAF and hybrid system: statistical analysis

The planning matrix obtained from the experiments is shown in Table 2.

Table 1. Real and coded values of independent variables in CCRD for oil-water separation efficiency

Variables	***Levels***				
	-1.41	***-1.00***	***0.00***	***+1.00***	***+1.41***
Ratio between air and water outflow for microbubble production ($X_1 \cdot 10^3$)	0.24	0.40	0.80	1.20	1.39
Ratio between feed outflow of wetland prototype and water for microbubble production (X_2)	0.11	0.15	0.25	0.35	0.39

Four factorial experiments, four axial experiments and four central points were analyzed. The repetitions at the central points are used to estimate the experimental error in a design of this type. In each assay, separation efficiency (%) was the average of three runs, including each assay of the central point. At these same central points, there was a considerable increase in efficiency in reducing the residual oil content in the treated effluent comparing the water-oil separation efficiency values obtained without (Y_0) and with (Y_1) the connection of the wetland to the DAF prototype. The maximum experimentally obtained rate was 98%, occurring at the central point. The decrease in separation efficiency to higher or lower levels than those at the central point was performed to determine an optimal point.

Table 2. Experimental CCRD matrix with coded variables and response values for oil-water separation efficiency

Assays	$X_1 \times 10^3$	X_2	*Separation efficiency (%) DAF (Y0)*	*Separation efficiency (%) DAF + wetland (Y1)*
1	-1.00	-1.00	23.00	44.00
2	-1.00	+1.00	70.00	88.00
3	1.00	-1.00	26.00	45.00
4	1.00	1.00	59.00	80.00
5	-1.41	0.00	44.00	62.00
6	1.41	0.00	25.00	45.00
7	0.00	-1.41	15.00	33.00
8	0.00	1.41	62.00	82.00
9	0.00	0.00	75.00	96.00
10	0.00	0.00	77.00	98.00
11	0.00	0.00	78.00	98.00
12	0.00	0.00	74.00	97.00

The estimated effects with the DAF + wetland treatment (Y_1) are displayed in Table 3. The p-value was lower than 0.05 for every factor analyzed. Table 4 displays the results of the analysis of variance (ANOVA), indicating that a model can be created with lower lack-of-fit results and smaller experimental error. Therefore, a quadratic model was proposed using the response surface method that satisfactorily fit the experimental data with a regression coefficient (R^2) of 97.5% between predicted and observed values (Fig. 4). All linear and quadratic terms exhibited considerable statistical significance within the 95% confidence interval. The $F_{calculated}/F_{critical}$ ratios were favorable and experimental errors remained around 1.0%. The final empirical model of the response to the non-coded experimental variables is presented in Eq. (2). The predominant factors of this quadratic model are the quadratic ratio between air and water outflows for microbubble production ($X_1 \cdot 10^3$) and the linear and quadratic ratios between the feed outflow of wetland prototype and water for microbubble production (X_2), as shown in the Pareto Chart (Fig. 5).

$$Y_1 = -144.18 + 198.15 - 120.01 \cdot X_1{}^2 + +1136.98 \cdot X_2 - 1811.23 \cdot X_2{}^2 - 56.25 \cdot X_1 \cdot X_2 \quad (2)$$

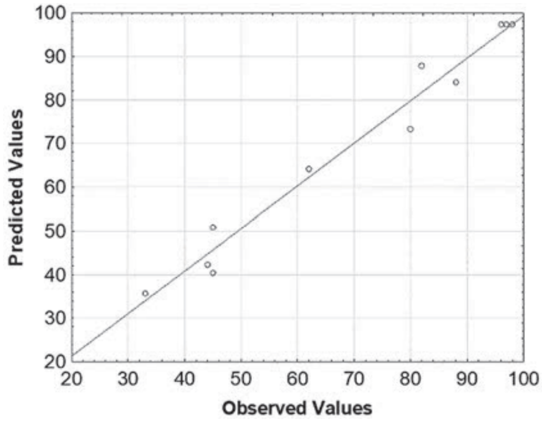

Fig. 4. Predicted versus observed values of proposed model

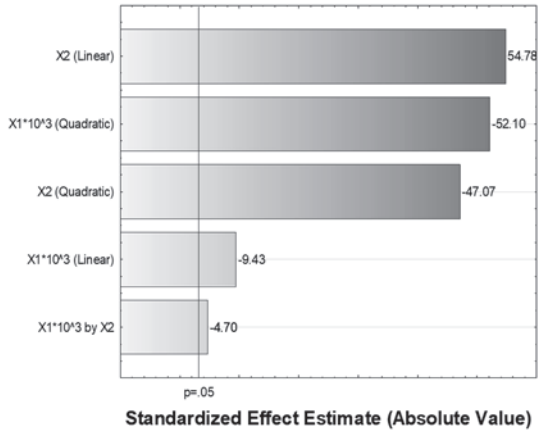

Fig. 5. Pareto chart for estimate of effects

Based on the model, 3D (Fig. 6a) and 2D (Fig. 6b) surfaces were built to analyze the region of greater separation efficiency. Thus, the central region (points 0,0) of the CCRD (points 0.00, 0.00) exhibited greater oil removal even without the wetland. This may be possible due to the correct combination of air and water flows to produce microbubbles with small diameters and high uniformity in terms of the Sauter mean diameter. The measurement of the diameter and uniformity of microbubbles was described by Brasileiro et al. (2020). Therefore, some adaptations can be made to the hybrid system to measure the size of microbubbles as the result of the flows of water, oil and air.

3.2. Efficiency results of DAF and hybrid system: Physical and mechanical analysis

Analyzing the "effect" column in Table 3, it is evident that the effect is negative as X_1 increases linearly (-6.34) and even more negative as X_1 increases in quadratic mode (-38.40). Thus, as X_1 increases, oil removal efficiency tends to be compromised by the reduction in interfacial area. According to the physical meaning of X_1, the increase in microbubble size due to coalescence was apparently preponderant compared to the increase in the hold-up of the gas (Chu et al., 2019).

Table 3. Estimated effects of DAF + wetland treatment

	Effect	*Standard Error - Pure Error*	*t(3)*	*p-value*	*-95% Confidence Limit*	*+95% Confidence Limit*
Mean	97.33	0.48	203.38	0.00	95.80	98.85
$X_1 \cdot 10^3$ (Linear)	-6.34	0.67	-9.43	0.00	-8.48	-4.20
$X_1 \cdot 10^3$ (Quadratic)	-38.40	0.74	-52.10	0.00	-40.75	-36.06
X_2 (Linear)	37.27	0.68	54.78	0.00	35.11	39.44
X_2 (Quadratic)	-36.22	0.77	-47.07	0.00	-38.67	-33.78
$X_1 \cdot 10^3 \cdot X_2$	-4.50	0.96	-4.70	0.02	-7.55	-1.45

Table 4. ANOVA for validation of mathematical model*

Source of variation	*Quadratic sum*	*Degrees of freedom*	*Quadratic mean*	*F*	*P*
X_1 (L)	81.54	1	81.54	88.95	0.00
X_1 (Q)	2488.13	1	2488.13	2714.33	0.00
X_2 (L)	2750.73	1	2750.73	3000.79	0.00
X_2 (Q)	2030.73	1	2030.73	2215.34	0.00
$X_1 \cdot X_2$	20.25	1	20.25	22.09	0.02
Lack of fit	166.42	3	55.47	60.52	0.00
Experimental error	2.75	3	0.92		
Total	6834.67	11			

**95% confidence level*

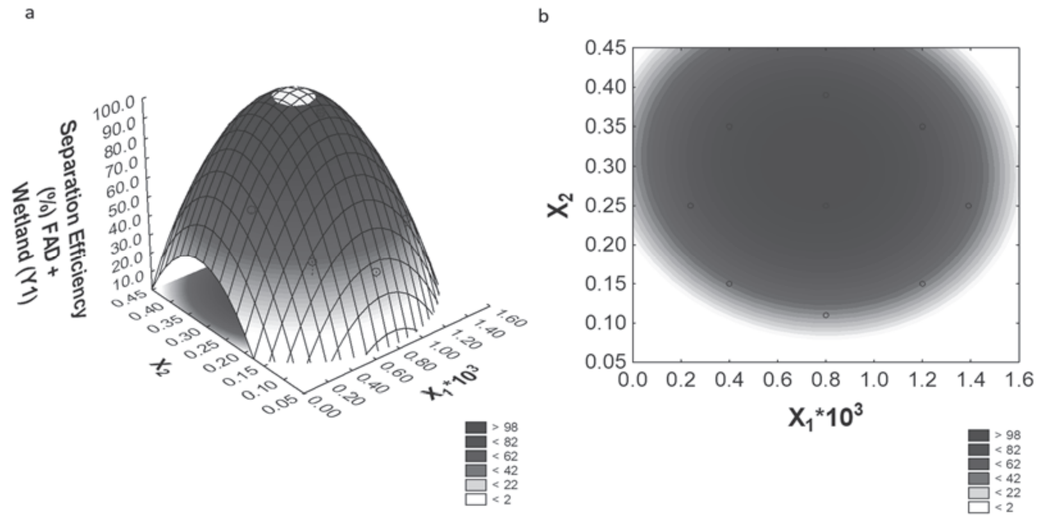

Fig. 6. Results of: a- response surface and b-contour plot for separation efficiency based on ratio between air and water outflows for microbubble production ($X_1 \cdot 10^3$) and ratio between feed outflow of wetland prototype and water for microbubble production (X_2)

Table 2 shows that oil droplet removal efficiency is greater higher at the central values of X_1 (intermediate gas flows) in both the DAF system and the combined DAF + wetland system. This air flow provides an increase in the hold-up without favoring a considerable increase in microbubble size, reaching a balance between the two antagonistic effects, the optimization of which enables the maximum possible interfacial area and, consequently, maximum efficiency (Prakash et al., 2018).

The X_1 quadratic effect is even more detrimental, as the effect of coalescence (increase in microbubble size) increases when the square of the effect of the air flow increases. This phenomenon leads to a tendency to reduce oil removal efficiency, as the effect of microbubble size apparently predominates over gas hold-up.

Again, analyzing the "effect" column in Table 3, the increase in X_2 led to two different behaviors. In the linear analysis, the effect was +37.27, indicating that the increase in X_2 in a linear mode tends to favor efficiency by increasing the interfacial area of the microbubbles due to the turbulence created by the increase in the net flow of wetland recirculation. This flow from the wetland leads to a reduction in the microbubble size and gas hold-up. The effect of the microbubble size seems to be always preponderant over the effect of hold-up regarding oil removal efficiency but without an apparent effect on the reduction in the contact time between the microbubbles and oil droplets (Chu et al., 2019).

When X_2 varies quadratically, however, the effect is -36.22. In this case, efficiency is hampered by the increase in X_2 likely due to the drastic reduction in the microbubble/oil droplet contact time and the reduced biosorption of oil by the macrophytes. Such conditions are favored by the degree of turbulence due to the high liquid flow in the wetland. Even with the increase in the interfacial area under quadratic conditions of wetland flow, the contact time was very short, compromising oil/water separation efficiency (Ansari et al., 2020; Colares et al., 2020).

Oil removal efficiency fit the values of the central point (intermediate wetland flow values) for both the FAD system and hybrid system (DAF + wetland) (Table 2). Under these conditions, the results indicate that the flow of the wetland is able to reduce the hold-up of the gas less drastically without compromising the reduction in microbubble size due to turbulence and enabling sufficient contact time between the microbubbles and oil droplets to increase the interfacial area and achieve optimal oil/water separation efficiency. With the central values of the net wetland recirculation flow, the largest effects were obtained with the inclusion of the wetland, increasing oil removal efficiency from 77 to 98% (Test 10), which is around 20%, as most of the oil was removed in the DAF system.

According to Yin et al. (2017), *Eichhornia crassipes* is able to absorb oil in wastewater, which exerts an influence on macrophyte growth (Özbay, 2016) and, consequently, the control of the species. Moreover, residual plants in wetlands can be tritured to obtain biosurfactants (saponins) (Gutiérrez-Morales et al., 2017).

3.3. Scale-up correlations

Inertia and gravity have been identified as phenomena that affect the DAF flotation chamber fluid dynamics the most (Kiss and Lakatos, 1996). Analyzing the fluid dynamics of this equipment by dimensional analysis, leads to the following scale-up model (Eq. 3):

$$Q_r = \frac{Q_m}{Q_p} = \frac{{L_r}^3}{{L_r}^{1/2}} = {L_r}^{5/2} \tag{3}$$

where: subscript *m* and *p* = scale-up model and prototype, respectively; Q_r = ratio of the scale-up model and prototype volumetric outflows; Q_m = scale-up model volumetric outflow, m^3 s^{-1}; Q_p = prototype volumetric outflow, m^3 s^{-1}; L_r = ratio of the geometric dimensions of the scale-up model and prototype; L_m = geometric dimension of the scale-up model, m and L_p= geometric dimension of the prototype, m.

According the scale-up criteria, the hydraulic behavior of optimized-rectangular wetlands with continuous decanters depends on the turbulent Reynolds number (Re_t). The application of the scale-up criteria (Ludwig, 1999), leads to Eqs. (4-5):

$$(Re_t)_m = (Re_t)_p \tag{4}$$

and:

$$Re_t = \frac{\nu_c \cdot D_H \cdot \rho_c}{\mu_c} \tag{5}$$

where: ν_c = surface velocity of continuous phase (water) (m s^{-1}); D_H - hydraulic diameter, m; ρ_c = density of water, kg m$^{3\ -1}$ and μ_c = absolute viscosity of water, kg m^{-1}·s^{-1}.

The hydraulic diameter can be estimated as Eq. (6):

$$D_H = 4 \cdot R_H = 4 \cdot \frac{Outflow\ area}{Wet\ perimeter} = \frac{4 \cdot h \cdot b}{2 \cdot (h+b)} \tag{6}$$

where: *h* is the liquid depth at the section, in m, and *b* is the liquid width at the section in m.

The most efficient and economic section is given by Eq. (7):

$$D_H = 2 \cdot h \tag{7}$$

Considering than h<<b, also, replacing the velocity by the ratio of outflow (Q) and the cross-section area (A), led to Eq. (8):

$$\nu_c = \frac{Q}{A} = \frac{Q}{0.7854 \cdot h \cdot b} = \frac{Q}{1.5708 \cdot h^2} \tag{8}$$

Therefore, Eq. (9) gives the Reynolds number (Re_t):

$$Re_t = \frac{Q \cdot 2h \cdot \rho_c}{1.5708 \cdot h^2 \cdot \mu_c} = \frac{2 \cdot Q \cdot \rho_c}{1.5708 \cdot h \cdot \mu_c} \tag{9}$$

and for an operation within the optimized criteria: $\text{Re}_t < 5000$

The pilot structure preliminary design was based on scale-up correlations obtained from Eqs. (3-7). For the commercial scale hybrid oily water treatment unit, calculations were done using estimated data of a thermoelectric plant. The maximum volume of the use of it per day was around 100 thousand liters during the period of November 2017 and September 2018. Then, it was estimated the average effluent outflow of 5 m^3 h^{-1} to be treated as well as the lung tank volume of 100 thousand liters for effluent storage, in case of maintenance shut-down.

The volume of the DAF prototype flotation chamber scale-up model was of 1.5 m^3 (dual chamber), which combining with the outflows of microbubbles and effluent to be treated (Q_m), totalizing 2 m^3 h^{-1}, led to Eq. (10):

$$\tau_m = \frac{V_m}{Q_m} = \frac{1.5}{2} = 0.75\ h \tag{10}$$

Assuming the scale-up model was built on the basis of 1:5 scale, leads to the following estimation for the prototype average residence time (Eq. 11):

$$\tau_p = \frac{{L_r}^{5/2}}{{L_r}^3} \cdot \tau_m = \frac{\tau_m}{{L_r}^{1/2}} = 0.75 \cdot \sqrt{5} = 1.68\ h \tag{11}$$

Prototype volume was re-calculated, the value obtained was 8.4 m^3 (4.2 m^3 for each flotation chamber). Based on the 1:5 scale, the commercial DAF unit met the dynamic similarity requirements. The maximum permanence time and practical maximum outflow ($Re_t \geq 5000$) are related themselves. Then, the maximum permanence time at the physical model of the wetlands in polycarbonate (Re_t =725; Q_m = 1 m^3 h^{-1} and h = 0.4 m), could estimate by dividing the equipment volume of liquid by the maximum permissible outflow. For a prototype (laboratory) model (volume: 0.96 m^3 per unit; length: 3 m), the maximum permanence time, in each unit, was estimated in 3456 s (~ 1 h). Considering the scale-up model for an industrial equipment (outflow: 5 m^3 h^{-1}), the Reynolds number of 5000 required the water height (a) of 0.6 m and tank width of 1.2 m, leading the wetland volume of 21.6 m^3 and length of 30 m (single wetland). In this case, the permanence time was estimate in 4.32 h. The pilot wetland was designed for lateral water height of 1.0 m with width of 2.0 m, leading Reynolds turbulence number on the order of 2500 (well within safety criteria). Fig. 7 illustrates a pilot prototype designed with the aid of the SolidWorks® software program.

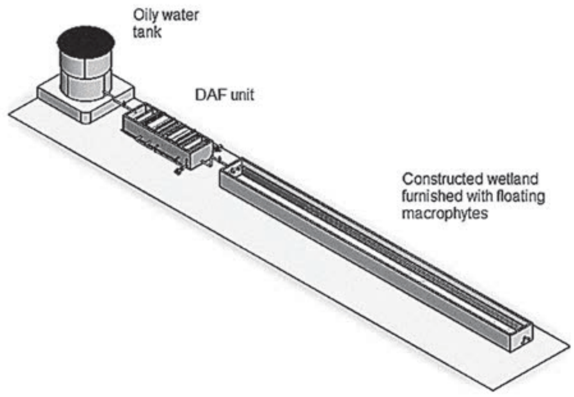

Fig. 7. Isometrics in SolidWorks of commercial DAF-wetland hybrid prototype

4. Conclusions

The combination of DAF and constructed wetland showed quite satisfactory results and was able of achieving an oil removal rate of 98% without the addition of any coagulant agent to assist in the coagulation or flocculation of the oil particles. In addition to efficiency, the system does not employ chemical additives that may cause problems in reusing the recovered water and have considerable degrees of toxicity to the flora or fauna in water bodies that receive the discharge.

The central values of variables X_1 (effect of the gas flow aspirated by the centrifugal pump with generation of microbubbles) of $0.80 \cdot 10^{-3}$ and X_2 (effect of the flow of recirculation liquid from the constructed wetland) of 0.25 in the experimental design carried out within the studied ranges of variation exhibited the highest efficiency regarding oil removal in both the DAF and combined DAF + wetland systems, with a significant contribution of the wetland in the removal of synthetic oily effluent (20% increase in efficiency). The proposed statistical model was able to predict the efficiency values (Y_1) as a function of the operational parameters evaluated (X_1 and X_2) and their interactions, with a regression coefficient (R^2) of 97.50%.

A rigorous analysis based on dimensionless criteria was proposed to extrapolate the hybrid process studied on a pilot scale to an industrial scale, with the dimensioning of the equipment involved in the process. This work proposes an innovative, efficient technology with a relatively low investment and operational cost that is clean and friendly to the environment. The automation of the prototypes both individually and together favored this high yield and the standardization of the process. However, adaptations of the system should be tested in future studies for the treatment of different types of oily waters, such as effluents with a high degree of emulsification (food industry) and those produced by mechanical metal industry facilities (oil-water-graphite), so that this hybrid process can become a suitable candidate for commercialization in the national and international market.

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