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Optimization of petroleum refinery effluent treatment in a UASB reactor using response surface methodology

S.O. Rastegar[a], S.M. Mousavi[a,*], S.A. Shojaosadati[a], S. Sheibani[b]

[a] Biotechnology Group, Chemical Engineering Department, Tarbiat Modares University, Tehran, Iran
[b] R & T Management Department, National Iranian Oil Refining and Distribution Company, Tehran, Iran

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ABSTRACT

An upflow anaerobic sludge blanket (UASB) bioreactor was successfully used for the treatment of petroleum refinery effluent. Before optimization, chemical oxygen demand (COD) removal was 81% at a constant organic loading rate (OLR) of 0.4 kg/m^3 d and a hydraulic retention time (HRT) of 48 h. The rate of biogas production was 559 mL/h at an HRT of 40 h and an influent COD of 1000 mg/L. Response surface methodology (RSM) was applied to predict the behaviors of influent COD, upflow velocity (V_{up}) and HRT in the bioreactor. RSM showed that the best models for COD removal and biogas production rate were the reduced quadratic and cubic models, respectively. The optimum region, identified based on two critical responses, was an influent COD of 630 mg/L, a V_{up} of 0.27 m/h, and an HRT of 21.4 h. This resulted in a 76.3% COD removal efficiency and a 0.25 L biogas/L feed d biogas production rate.

1. Introduction

The annual worldwide consumption of petroleum hydrocarbons is estimated to be approximately 1012 US gallons. Oil refineries generate huge volumes of oily sludge during refining [1]. Oil and oil-derived product residues are complex mixtures of thousands of compounds with a high proportion of hydrocarbons, which possess different solubility and microbial resistances to biodegradation. Bioremediation is one of the most extensively used treatment techniques because of its low cost and high efficiency [2–4]. Biological treatment processes, in an effort to minimize cost, utilize diverse microbial communities that interact in a multitude of ways to mediate a myriad of biological reactions [5].

The biodegradation of oil and oil-derived products has been the focus of many studies. Nevertheless, these products remain an important concern due to the toxicity of their constituents [6], the large volumes of effluent waters and soils that need to be treated in compliance with environmental standards, and the need for enhanced biodegradation of the more recalcitrant petroleum compounds [4,7].

Anaerobic digestion is an interesting and effective alternative for wastewater treatment and simultaneous gas production. It has been successfully applied, and the development of new reactor designs creates increased employment. Anaerobic digestion presents a number of significant advantages over conventional aerobic wastewater treatment systems [5,8]. Anaerobic biomass granulation is regarded as instrumental in the successful operation of UASB bioreactors treating high-strength wastewaters. The success of these bioreactors has led to their use in the treatment of sewage in some tropical and subtropical countries such as India, Argentina, Brazil, and Colombia. However, many of these full-scale installations, as single stage secondary treatment units, are unable to attain higher COD removal compared with established treatment methods for the treatment of wastewaters having COD levels less than 700 mg/L [9,10].

The UASB bioreactor is one of the most popular anaerobic wastewater treatment systems and has been the most widely used high-rate anaerobic reactor for wastewater treatment throughout the world since the 1980s [11]. The UASB process is an economic and effective anaerobic treatment method for refinery wastewater. UASB bioreactors have economic advantages over aerobic treatment units as they economize space utilization and require no external input of energy and because the required mixing is achieved through wastewater upflow and rising gas bubbles, nutrient requirements (nitrogen and phosphorous) are much less

* Corresponding author. Tel.: +98 21 82884917; fax: +98 21 82884931.
E-mail address: mousavi_m@modares.ac.ir (S.M. Mousavi).

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Table 1
Characteristics of petroleum refinery effluent.

Parameter	Amount (mg/L)
BOD_5	105
COD	450
Oil and grease	56
Total N	12
Total P	0.75
H_2S	3

than those of aerobic treatment processes (by approximately half), residuals (i.e., sludge) generated by UASB bioreactor treatment are much less in volume and are well digested, there are no mechanical/moving components, post-treatment and sludge handling requirements are reduced, and both the capital costs and operating costs of plants using a UASB bioreactor are significantly less than those of fully aerobic treatment plants [8,12].

The UASB process has proven highly effective for the treatment of medium- and high-strength wastewaters within a wide range of HRTs (3–48 h) [13]. The UASB process involves complex chain reactions, and solving the necessary equations derived on the basis of physical, chemical, and biological concepts generally requires a number of assumptions. Therefore, steady state models are generally able to predict the parameters that have been considered in mass balance relations but are unable to estimate other interrelated effluent quality parameters (responses) [14]. To overcome this problem, process modeling and optimization studies can be conducted using response surface methodology (RSM) [15]. RSM is a collection of mathematical and statistical techniques for experimental design, model development, the evaluation of factors, and the optimization of conditions. Statistical optimization reflects the role of each component of the process [16]. Response surface methodology allows not only the determination of optimum conditions but also the analysis of how sensitive the optimum conditions are to variations in experimental variables. The application of RSM to design optimization aims to reduce the cost of expensive analysis methods. Another advantage of RSM is that it is possible to make different projections, which provide graphic illustrations, thus allowing a visual interpretation of the functional relations between the response and experimental variables. Central composite design (CCD) is used extensively in building second-order response surface models [17,18]. There are some reports on the use of response surface methodology in other UASB processes. Bhunia et al. investigated statistical modeling and the optimization of biomass granulation and COD removal in UASB reactors treating low-strength wastewaters [19] and the optimization of hydrogen production in a granule-based UASB reactor [20]. The main objective of CCD is to determine the optimum operational conditions for a system or to determine a region that satisfies the operational specifications. There is no information available in the literature regarding the optimization of COD removal from petroleum refinery effluent using UASB bioreactors. Therefore, the main objective of this work is to investigate more thoroughly the phenomenon of removal of petroleum refinery effluent in a UASB bioreactor. The effects of operating parameters such as influent COD, HRT, and V_{up} on COD removal and biogas production in the bioreactor were also studied using response surface methodology.

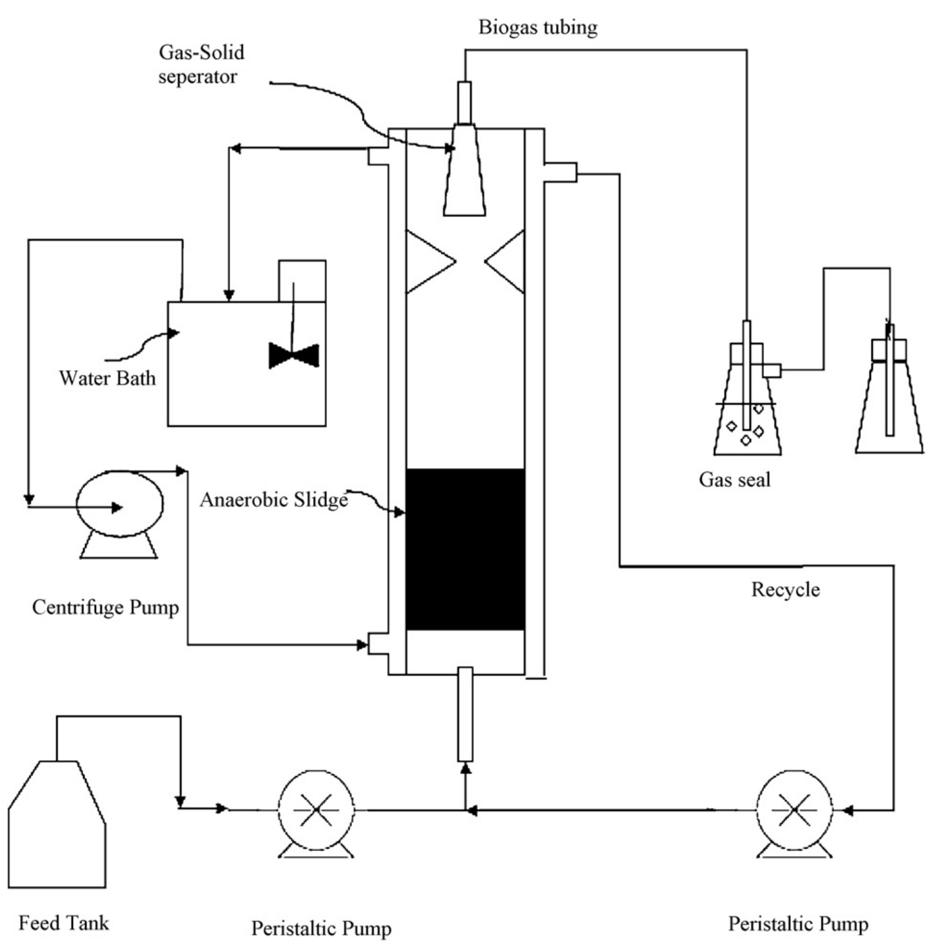

Fig. 1. Schematic diagram of the UASB bioreactor.

2. Material and methods

2.1. Wastewater characterization

Wastewater was collected from the Tehran petroleum refinery, Tehran, Iran. The characteristics of the wastewater, including of BOD_5, COD, ammonium nitrogen, phosphate, H_2S and oil, are shown in Table 1. All experiments were conducted according to the Standard Methods for the Examination of Water and Wastewater [21]. Molasses (a by-product of the sugar industry) was used as a co-substrate.

2.2. Bioreactor configuration and startup

A laboratory-scale UASB bioreactor was used in this study (Fig. 1). The glass bioreactor column was fabricated with an internal diameter of 10 cm, a liquid height of 80 cm, and a total volume 6.28 L. An inverted funnel-shaped gas separator was used to conduct biogas to a gas collection tank. The biogas produced was collected by the water-displacement method. The UASB bioreactor was operated under mesospheric conditions (38 ± 1 °C) and temperature was maintained with an electrical heating tape (heating capacity: 40 W/m) attached to the outside surface of the reactor. The inoculum for seeding was digested sludge from a Pegah dairy industry (Tehran, Iran).

2.3. Bioreactor operation and monitoring

At start-up the reactor was operated at HRT of 3 d and V_{up} of 0.44 m/h [11]. Fresh feed was initially diluted with recycled effluent at a ratio of 1:10. Subsequently, this ratio was lowered as the HRT was reduced. Supplementary nitrogen (NH_4Cl) and phosphorous (KH_2PO_4) were added to adjust the COD:N:P ratio to 500:5:1. $NaHCO_3$ was added to maintain an influent alkalinity of 1500–1700 mg $CaCO_3$/L.

The steady state performance was evaluated under different influent COD concentrations (500–1200 mg/L) and HRTs (1–3 d). Variation in effluent COD concentration within ±3% at each condition was considered to be the criterion for steady state conditions. The biogas composition was determined using a Shimadzu GC-8A gas chromatograph. The experimental design was carried out using Design Expert Software, Windows-compatible software that provides efficient design of experiments (DOE) for the identification of vital factors that affect the process and that use RSM to determine optimal operational conditions. The results can be obtained as contour plot presentations for visualization and also as contours to illustrate the effects of system variables on responses.

2.4. Experimental design

Design-Expert 7.0 was used, and a 2^3 factorial design with six central points and six axial points was selected. The behavior of the system is explained by the following second-order polynomial empirical model:

$$y = \beta_0 + \sum_{i=1}^{n}\beta_i X_i + \sum_{i=1}^{n}\beta_{ii} X_i^2 + \sum_{i<1}^{n}\sum_{j=1}^{n}\beta_{ij} X_i X_j + \varepsilon \qquad (1)$$

where y is the response, β_0 is the constant coefficient, X_i ($i = 1 - n$) are non-coded variables, β_is are the linear interaction coefficients, β_{ii}s are the quadratic interaction coefficients, β_{ij}s (i and $j = 1 - n$) are the second-order interaction coefficients, and ε are residuals for each experiment [22].

Data were processed for Eq. (1) using the Design-Expert 7.0 program, including an ANOVA to evaluate the interaction between the process variables and the response. The quality of the fit of the polynomial model was expressed by the coefficient of determination (R^2), and its statistical significance was checked by the F-test in the same program.

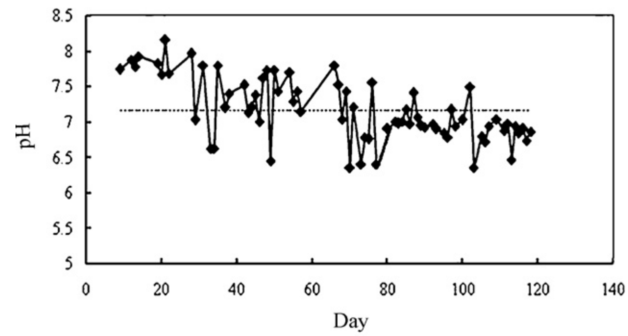

Fig. 2. pH variation during the examination of the wastewater system.

3. Results and discussion

3.1. Reactor performance at the start-up

The COD and pH of the refinery effluent were measured over 120 d of reactor operation (Fig. 2) and a variation in pH during the examination of the wastewater system was observed. The effluent pH changed when feed was added due to the fewer initial pH of the feed (pH 6), but most of time effluent pH was relatively constant near 7. Adjustment of the pH, which varied from 6.5 to 7.7 throughout the experiment, was not necessary as this range is suitable for anaerobic microbes. Outlet pH was neutral (approximately 7), and the performance of the reactor was stable. Fig. 3 shows variations in influent and effluent COD over the 120 d. Steady state conditions were achieved after the 100th d.

3.2. Functions of HRT and OLR in COD removal

After start-up period, as shown in Fig. 4, COD removal efficiency was examined at different hydraulic retention time. HRT was gradually increased from 10 to 60 h, keeping the OLR constant at 0.4 kg/m^3 d by mixing influent with effluent, the maximum removal efficiency was 81% at an HRT of 48 h. Removal efficiency tended to level off at 81% after this HRT. The rate of biogas production also increased with HRT; the biogas production rate was 559 mL/h at an HRT of 40 h and a COD of 1000 mg/L. Fig. 5 shows COD removal on the basis of OLR. OLR was increased from 0.2 to 1.2 kg/m^3 d, keeping HRT constant 48 h, and COD removal increased from 51% to 76%.

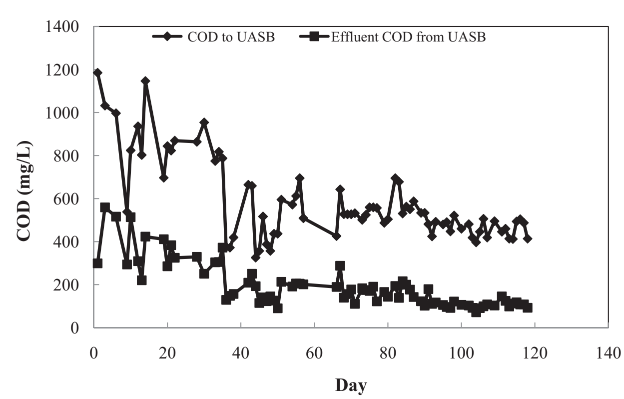

Fig. 3. COD variation during the examination of the wastewater system.

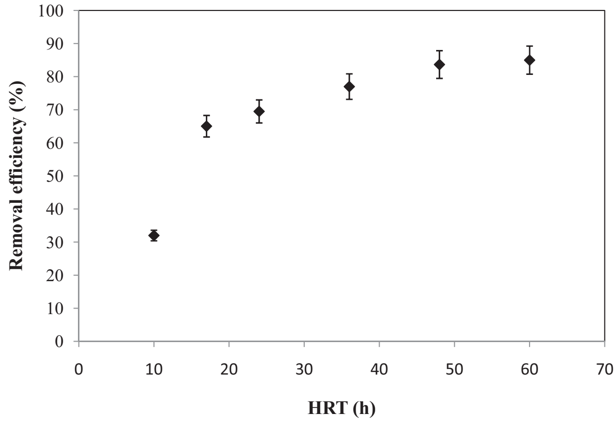

Fig. 4. COD removal percentage on the basis of hydraulic retention time at a constant OLR (0.4 kg/m^3 d).

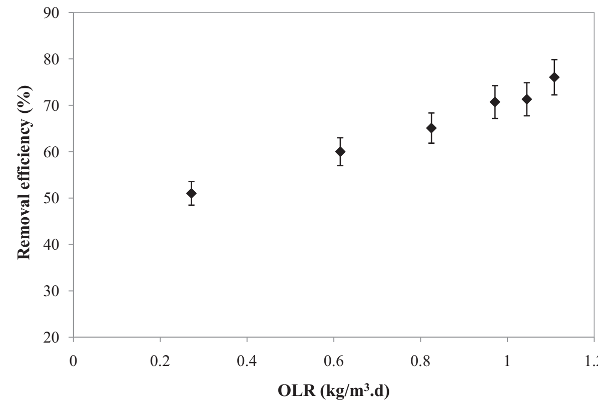

Fig. 5. COD removal percentage on the basis of the organic loading rate.

3.3. Statistical analysis

Influent COD, HRT, and V_{up} for a three-factor-five-level CCD design were used to determine the optimal values. The ranges and levels of the variables investigated in this study are listed in Table 2. Each factor was varied at five different levels while the other parameters were kept constant. Once the desired ranges of the variables had been defined, they were coded to lie at ±1 for the factorial points, 0 for the center points, and $\pm\alpha$ for the axial points. In this case, central composite designs for three independent variables each at five levels was employed to fit the model. A total of 20 experiments were required for this procedure. The CCD is shown in Table 3, which shows the experimental conditions and their responses. The ANOVA for the reduced quadratic model for COD removal and the reduced cubic model for the biogas production rate are shown in Table 4. These models are significant at the *p*-value less than 0.05 at 95% confidence interval.

3.4. COD removal

The model equation for coded values in a quadratic model fitting the experimental results for COD removal can be seen in Eq. (2).

$$\text{COD removal } (\%) = 68.06 + (6.5A) + (3.29B) - (2.65C) - (0.63AB) + (0.87AC) - (0.16A^2) \quad (2)$$

where A is HRT in h, B is V_{up} in m/h, and C is influent COD in mg/L. Fig. 6 shows the actual and the predicted COD removal efficiencies. R^2 and adjusted R^2 (R^2_{adj}) were found to be 0.96 and 0.94, respectively indicating that actual and predicted COD removal efficiencies were in agreement. Fig. 7 shows the COD removal efficiency contour plots. There is clearly in Fig. 7A a combined effect of influent COD and HRT on COD removal at a constant V_{up} (0.3 m/h). The maximum COD removal (>75%) was observed for an influent COD of 640 mg/L and HRT of 22 h.

Fig. 7B shows the combined effects of HRT and V_{up} on COD removal at constant influent COD (850 mg/L). COD removal

Table 2
Experimental range and levels of independent test variables.

Variable	Low axial ($-\alpha$)	Low factorial (−1)	Center point (0)	High factorial (+1)	High axial (+α)
Influent COD (mg/L): A	500	642	850	1060	1200
HRT (h): B	10	13	17.5	22	25
V_{up} (m/h): C	0.1	0.18	0.3	0.42	0.5

Table 3
Experimental plan and results.

Run	HRT (h)	V_{up} (m/h)	Influent COD (mg/L)	COD removal (%)	Biogas rate (L biogas/L feed d)
1	22	0.42	642	78	0.18
2	22	0.18	642	72	0.14
3	17.5	0.3	850	68	0.15
4	17.5	0.1	850	61	0.1
5	17.5	0.5	850	74	0.44
6	13	0.42	642	68	0.17
7	10	0.3	850	55	0.21
8	22	0.42	1060	73	0.38
9	25	0.3	850	81	0.48
10	17.5	0.3	850	67	0.14
11	13	0.18	1060	54	0.52
12	17.5	0.3	850	69	0.14
13	17.5	0.3	850	68	0.14
14	17.5	0.3	1200	64	0.64
15	17.5	0.3	850	69	0.13
16	22	0.18	1060	70	0.33
17	13	0.18	642	63	0.13
18	17.5	0.3	500	73	0.07
19	17.5	0.3	850	69	0.13
20	13	0.42	1060	63	0.39

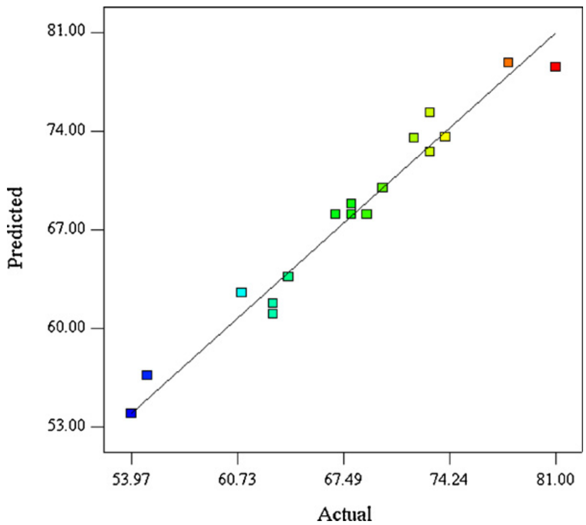

Fig. 6. Actual vs. predicted values of COD removals.

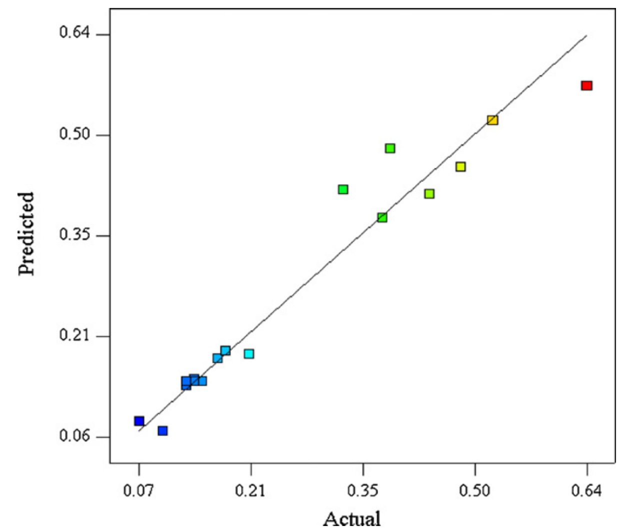

Fig. 8. Actual vs. predicted values of biogas production rates.

efficiency decreases with decreasing HRT at a constant V_{up}. Under constant HRT, a slight increase in COD removal efficiency was observed for higher V_{up}. This is confirmed by Eq. (2), which shows that HRT has a greater affect than V_{up} on the response.

3.5. Biogas production rate

The model equation for coded values in the cubic model fitting the experimental results of biogas production can be seen in Eq. (3):

$$\text{Biogas production rate (L biogas/L feed d)} = +0.14 + (0.08A) + (0.10B) + (0.14C) - (0.027AC) - (0.02BC) + (0.062A^2) + (0.065C^2) + (0.035B^2) - (0.10A^2B) - (0.1AB^2) \quad (3)$$

where A is HRT, B is V_{up} and C is influent COD. Fig. 8 demonstrates very good agreement between the experimental and predicted values. R^2 and R^2_{adj} were found to be 0.95 and 0.90, respectively. A maximum biogas production rate of >0.54 L biogas/L feed d was observed at an HRT of 25 h and an influent COD of 1060 mg/L at the constant V_{up} of 0.3 m/h.

The effect of influent COD and HRT on the biogas production rate is shown in Fig. 9A. According to this figure amount of biogas will be increased by HRT increasing from 13 to 25 h at a constant influent COD or by influent COD increasing from 640 to 1060 mg/L at a constant HRT. Fig. 9B shows combined effect of influent COD and V_{up} on the biogas production rate at a constant HRT of 17.5 h. With influent COD increasing from 640 to 1060 mg/L and V_{up} increasing from 0.2 to 0.5 m/h, the biogas production rate increased. With increasing the influent COD at a constant HRT, the amount of OLR will be increased and consequently because of increasing biodegradability of the substrate and sufficient microbial community the biogas production will be increased [23,24]. Influent COD had a stronger effect on the biogas production rate than V_{up}, because influent COD has a larger coefficient than V_{up} in Eq. (3).

The produced biogas, as analyzed by gas chromatography, had a CH_4/CO_2 ratio of 12–1. Approximately 80% of the gas produced was methane, and the rest was carbon dioxide with trace amounts of H_2S and H_2O. As more CH_4 was produced than CO_2, it can be

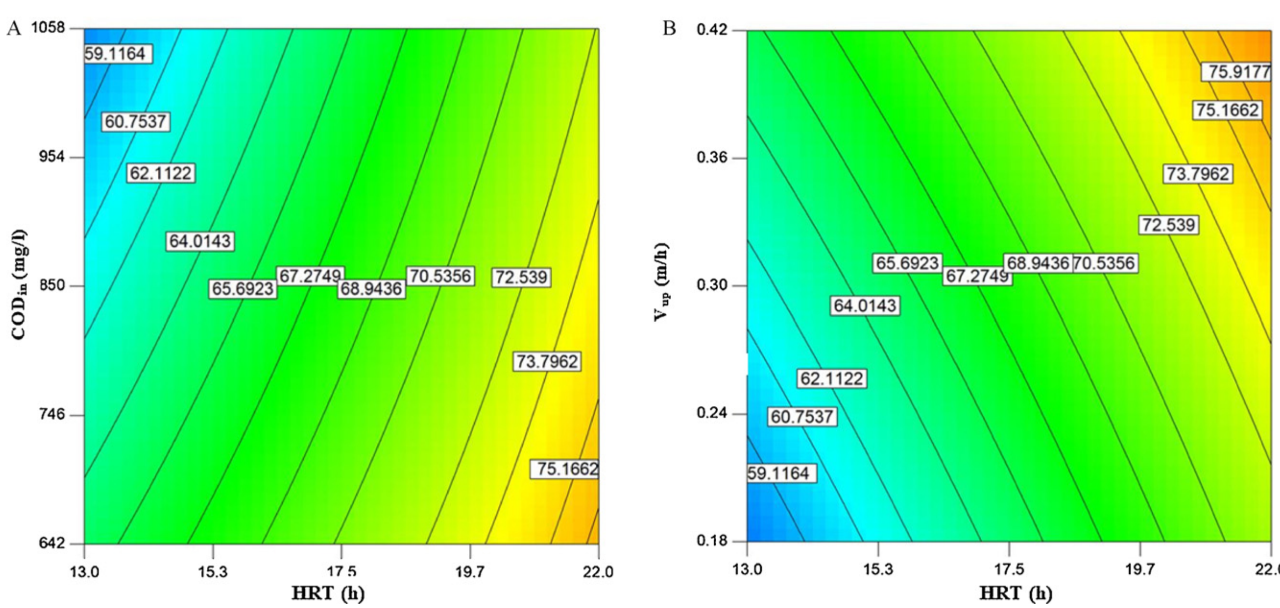

Fig. 7. Contour plots of the interactive effect for COD removal (A) effect of HRT and influent COD and (B) effect of HRT and V_{up}.

Table 4
ANOVA for response surface models applied.

Response	Model	ANOVA					
		Source	Sum of square	DF	Mean square	*F* value	Prob > *F*
COD removal (%)	Reduced quadratic model	Model	829.05	6	138.18	60.08	<0.0001
		A	576.44	1	576.44	250.66	<0.0001
		B	147.38	1	147.38	64.09	<0.0001
		C	95.62	1	95.62	41.58	<0.0001
		A^2	0.37	1	0.37	0.16	0.6965
		AC	6.12	1	6.12	2.66	0.1267
		AB	3.13	1	3.13	1.36	0.2647
		Residual (R^2 = 0.96, $R^2_{adj} = 0.94$)	29.90	13	2.30		
Biogas production rate (L biogas/L feed d)	Reduced cubic model	Model	0.5	10	0.05	18.48	<0.0001
		A	0.036	1	0.036	13.42	0.0052
		B	0.058	1	0.058	21.29	0.0013
		C	0.28	1	0.28	103.44	<0.0001
		AC	0.006	1	0.006	2.23	0.1697
		BC	0.003	1	0.003	1.18	0.30
		A^2	0.055	1	0.055	20.11	0.0015
		C^2	0.061	1	0.061	22.48	0.0011
		B^2	0.018	1	0.018	6.52	0.0311
		A^2B	0.034	1	0.034	12.47	0.0064
		AB^2	0.035	1	0.035	12.89	0.0058
		Residual (R^2 = 0.95, $R^2_{adj} = 0.90$)	0.024	9	0.0025		

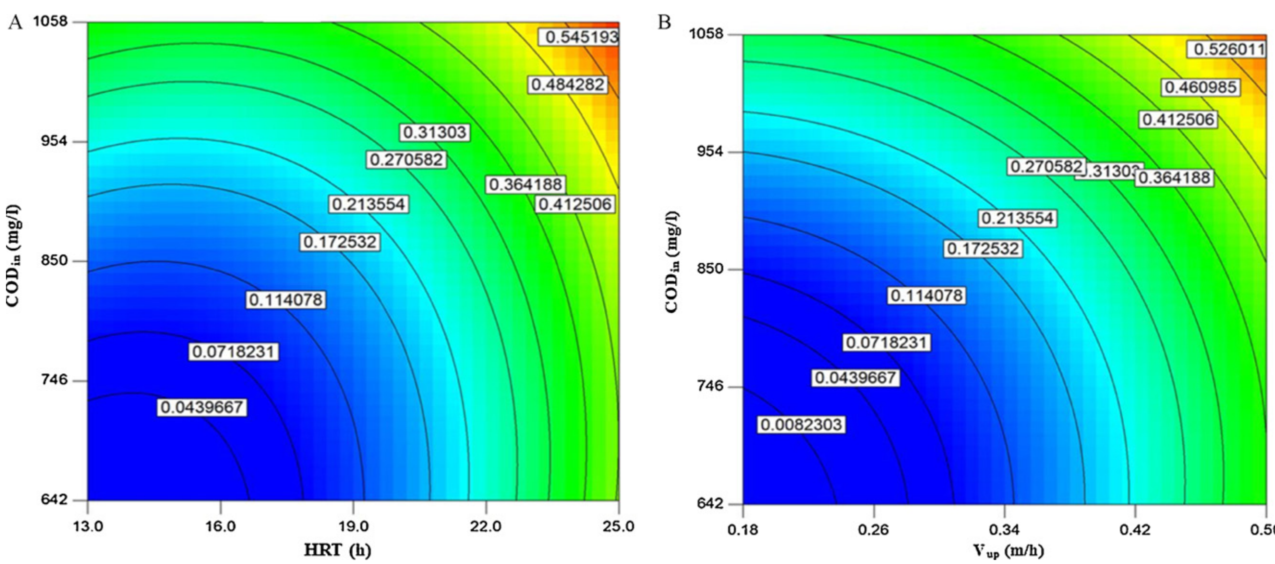

Fig. 9. Contour plots of the interactive effect for the biogas production (A) effect of HRT and influent COD and (B) effect of V_{up} and influent COD.

concluded that the conditions for the growth of H_2-utilizing and CO_2-utilizing bacteria were present. Because of the low incoming COD, bacteria will consume the produced CO_2 in biochemical reaction pathways. This result is similar to results reported in other work at low COD levels [25].

3.6. Process optimization

With multiple responses, regions where requirements simultaneously meet the critical properties, or "sweet spots," need to be found. The best compromise can be found visually by or overlaying critical response contours on a contour plot. Graphical optimization produces an overlay plot of the contour graphs to display the area of feasible response values in the factor space. The shaded portion, which indicates the zone of possible response values in the factor space and graphical optimization, is shown in Fig. 10. The optimum region was identified based on two critical responses, a COD removal of 75% and a biogas production rate of 0.2 L biogas/L feed d. The reason for selecting these two responses was that they were considered the most important for the viable representation and

Table 5
Optimum condition verification and additional experiments.

Influent COD (mg/L)	HRT (h)	V_{up} (m/h)	COD removal (%) (model)	COD removal (%) (experiment)	Biogas (L/L feed d) (model)	Biogas rate (L/L feed d) (experiment)
630	21.4	0.27	75.0	76.3	0.2	0.25
620	21.4	0.27	74.9	78.3	0.2	0.24

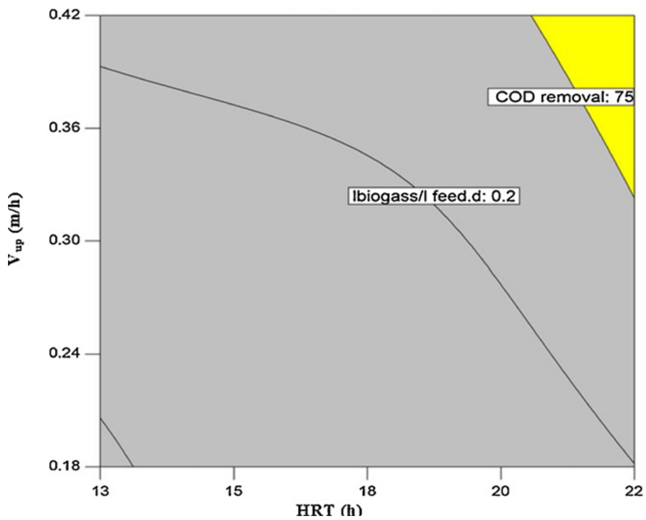

Fig. 10. Overlay plot for the optimum region.

optimization of the anaerobic treatment process. COD removal represents the substrate metabolized in anaerobic digestion and biogas production represents methanogenic activity [24,26].

Model validations at two experimental combinations were used for validation of the statistical model. The results of analysis indicated that the experimental values were in good agreement with the predicted values (Table 5). Under these conditions, the experimental response for COD removal was 78.3%, and the response for the biogas production rate was 0.25 (L biogas/L feed d). These results confirmed the validity of the model, and the experimental values were determined to be quite close to the predicted values.

4. Conclusion

The UASB bioreactor was found to be a successful biological treatment system, achieving a high COD removal efficiency for the treatment of petroleum refinery effluent. The results are summarized as follows:

1. When the system was operated at a high HRT (48 h) and influent COD (500 mg/L), COD removal was 81%.
2. The rate of biogas production increased when HRT increased; the biogas production rate was 559 mL/h at an HRT of 40 h and an influent COD of 1000 mg/L.
3. Efficient factors including HRT, influent COD, and V_{up} were modeled and optimized using response surface methodology.
4. The best models for COD removal and biogas production rate were the reduced quadratic model and the cubic model, respectively.
5. The optimum region based on two critical responses was at an influent COD of 630 mg/L, a V_{up} of 0.27 m/h, and an HRT of 21.4 h. At these conditions, the COD removal efficiency was 76.3% and the biogas production rate was 0.25 L biogas/L feed d.

Acknowledgement

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