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Testing an aerobic fluidized biofilm process to treat intermittent flows of oil polluted wastewater

Feroz Shaik* and Nayeemuddin Mohammed**

** Department of Mechanical Engineering, College of Engineering, Prince Mohammad Bin Fahd University, Al Khobar, Kingdom of Saudi Arabia.*
*** Department of Civil Engineering, College of Engineering, Prince Mohammad Bin Fahd University, Al Khobar, Kingdom of Saudi Arabia.*
** Corresponding Author: ferozs2005@gmail.com*

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ABSTRACT

An aerobic fluidized biofilm process of treating oil-polluted wastewater has been studied. A series of batch experiments were conducted using synthetic wastewater, and the kinetic coefficients were evaluated. The maximum rate of substrate utilization per unit mass microorganisms (K) was 1.6 $days^{-1}$, the substrate concentration at one-half the maximum growth rate (K_s) was 26 mg/L, the maximum specific growth rate (μ_m) was 1.0 $days^{-1}$, the ratio of the mass cells formed to the mass of substrate consumed (Y) was 0.61, and the endogenous decay coefficient (K_d) was 0.044 $days^{-1}$. The kinetic coefficients obtained were within the range of municipal wastewater. It was observed that up to 1500 mg/L oil (Motor oil SAE–40) could be degraded in a fluidized bed bioreactor (FBBR). The experiments, however, were limited to the oil concentration within a range of 1000–2600 mg/L. Average biofilm thickness (δ) under specific conditions was found to be 22 μm, and an average oil degradation rate of 0.053 mg oil/mg biomass/hour was measured in the FBBR. The results also support that the increase in the concentration of oil in the treatment process reduced significantly the degradation rate of non-oil carbon.

Keywords: Oil polluted wastewater; Fluidized bed bioreactor (FBBR); Water treatment; Innovative process; Motor oil SAE–40.

INTRODUCTION

There is a tremendous interest all over the world in industrial wastewater management due to concern of water scarcity and sustainable use of water (Al-Ammar et al., 2020). The ever-growing demand on water consumption emphasizes the development of cost-effective water treatment and water recycle technologies for the oil industry (Saber et al., 2014). Oil industry discharges large amounts of wastewater contaminated with hydrocarbons and creates a potential pollution hazard to the environment. The focus of the oil industry is to reduce the quantity of wastewater generation through process modifications and treat and recycle the wastewater for potential saving (Kuyukina et al., 2020). The wastewater from oil industry was characterized by high contents of oil and grease, chemical oxygen demand (COD), and biological oxygen demand (BOD), which strip the dissolved oxygen out of the receiving water surface, harming the inhabiting aquatic life (Coelho et al., 2006). Skimming tanks and traps were conventionally used to remove oil and grease from petroleum wastewater; however, their treatment efficiencies were low. Biological

treatment gained more importance for the removal of oil from wastewater due to their clean and friendly application (Abd El-Gawad, 2014).

Kuyukina et al. (2020) and Narayanan and Narayan (2019) summarize in their review the application of various biological technologies for the treatment of oil and other organic pollutants present in wastewater. A high concentrated oil and gas field wastewater was treated using a combination of sequential batch reactor (SBR) with a ceramic membrane bioreactor (CMBR). This treatment process can reduce the COD and oil content values substantially (Wei et al., 2020).

A synthetic wastewater containing diesel fuel has been treated with a bench scale membrane bioreactor (MBR). After 200-day operation, a good reduction was observed in carbon and nitrification. An irreversible membrane fouling was also found due to oil deposits (Capodici et al., 2017). A synthetic crude oil wastewater was treated using an aerated submerged fixed-film (ASFF) bioreactor. The treatment process can achieve 83 to 97% reduction in COD (Izanloo et al., 2006). The wastewater was treated using fluidized bed reactor by varying bed height, hydraulic retention time, and superficial gas velocity. The maximum COD reduction was observed at a bed height of 0.8 m. It was also observed that the percentage reduction of COD increased with an increase in superficial gas velocity (Haribabu & Sivasubramaniana, 2014).

In FBBR process, the microbes attach to the media and form biofilm. Fluidization in the reactor was caused by the recirculating wastewater and/or by the air stream. Excellent mixing, enhanced mass transfer rates, enlarged surface area, and higher loading at lower hydraulic retention time were some of the advantages in FBBR operation. The increased contact between the wastewater substrates and the biofilm also allows this process to break down larger compounds that are typically more difficult to treat. It is possible in FBBR to achieve biomass concentrations in the range of 10,000 to 40,000 mg dry biomass/liter as measured in the fluidized state. In the activated sludge process, biomass concentration ranges between 2000 and 5000 mg/L.

The high biomass concentrations achieved in biological fluidized bed reactor lead to a much higher rate of substrate utilization per unit volume. The rate of reaction per unit weight of biomass remains about the same as in the activated-sludge plants (Cooper & Wheeldon, 1980; Cooper, 1981 & Zhu et al., 2017). The aerobic fluidized-bed bioreactors were proven to be an efficient process for utilizing microorganisms to degrade dissolved organic pollution in the wastewater. Representative models expressing the efficiency and functioning of the process were available. However, the performance of the process, particularly to treat oil-contaminated wastewater, is not tested exclusively. In the present study, an attempt was made to study the performance of FBBR to treat oil from synthetic petroleum wastewater.

MATERIALS AND METHODS

A batch reactor consisted of a 1000 mL glass column connected to a mixer and an air pump and contained synthetic wastewater, and a culture of microorganisms was used in this study. The reactor contained well-textured 5 g of sand with a mean diameter of 0.7 μm. Synthetic wastewater simulating municipal wastewater was used throughout this study. The reason for using it instead of the actual wastewater was to ensure uniformity in composition, which otherwise cannot be provided by municipal wastewater with fluctuating characteristics. The detailed composition of the synthetic wastewater as prescribed by Shah Alam & Angelbeck (1978) is shown in Table 1. All the chemicals and reagents were procured from local markets and used as pure as supplied. The oil used in the experiment was normal motor oil (SAE –40) with a specific gravity of 1.0595.

Table 1. Synthetic wastewater composition.

Constituents	Concentration, mg/L
Glucose, $C_6H_{12}O_6$	1000
Ammonium Sulfate, $(NH_4)_2SO_4$	500
Magnesium Sulfate, $MgSO_4.7H_2O$	100
Calcium Chloride, $CaCl_2$	7.5
Manganese Sulfate, $MnSO_4.H_2O$	10
Potassium Phosphate (Monobasic), KH_2PO_4	527
Potassium Phosphate (Dibasic), K_2HPO_4	1070

Total organic carbon (TOC) was measured using a Shimadzu TOC analyzer (LCSH/CSN, Japan). A BOD incubator (Thermo Fisher Scientific) with water analysis kit (Eutech PCD 650) for DO measurement was used for BOD estimation. Digital pH meter (JENWAY 3520) was used for pH measurement. The procedures for total solids (TS), volatile solids (VS), volatile solids, fixed on sands (SS), and oil estimation were performed according to standard methods (1992).

OPERATION OF BATCH REACTOR

The operation of the batch reactor was carried out in three phases: seeding, adapting, and testing. The synthetic wastewater was freshly prepared and poured into the 1000 mL batch reactor. In the seeding phase, a small amount of wastewater obtained from a sewer adjacent to the laboratory was added to the batch reactor to provide a microbial seeding medium in the reactor. During the adaption phase, the microbial population was allowed to acclimatize to its new environment, i.e., the synthetic wastewater. During the next two weeks, a volume of 200 mL was wasted daily from the reactor. This was replaced on each occasion by 200 mL of a mixture of 50 mL synthetic wastewater and 150 mL of tap water. At the end of two weeks, the acclimatized microorganisms tend to reach a steady-stage of growth.

An electric mixer was used to ensure uniformity in the reactor, and oxygen was provided through an air diffuser placed inside the reactor and connected to an air pump. In the testing phase, the kinetic coefficients governing the growth of the microorganisms contained in the batch reactor were determined. All experiments were done with synthetic wastewater without any addition of oil. Sand concentration in the cylinder was nearly 5000 mg/L. Upon reaching steady state, measurements of the total organic carbon (TOC), total solids (TS), volatile solids (VS), and volatile solids fixed on the sands (SS) were made. For the determination of total solids, the sample was dried at 105 ^{o}C. At the end of the three days required to reach a steady state, measurements of TOC and SS were made. The cycle of variation of the nutrient concentration was then repeated until enough experimental data was recorded for the

determination of the coefficients: K = maximum rate of substrate utilization per unit mass microorganisms, T^{-1}; K_s = substrate concentration at one-half the maximum growth rate, M/L^3; μ_m = maximum specific growth rate, M/L^3T; Y = ratio of the mass cells formed to the mass of substrate consumed, M/M; K_d = endogenous decay coefficient, T^{-1}. These coefficients were determined according to the method described in Metcalf & Eddy (1984). Five samples were tested for TOC and BOD_5. The BOD_5 was found to be, on average, about two times of TOC value in mg/L.

PILOT STUDIES ON FBBR

Having determined the kinetic coefficients (K, K_s, Y, μ_m, K_d) from the smaller batch reactor using synthetic wastewater only without any oil, a pilot scale aerated FBBR as shown in Figure 1 was tested for the degradation of oil (SAE-40). The reactor consists of a 1.77 m Plexiglas column with an inside diameter of 14 cm. The effective volume of the reactor was 23 L. The base of the column has a cone shape, and a small ball was placed to block the cone to prevent the sand particles from escaping out of the reactor. Sand was used as the filtration medium. Local desert sand available in Al-Seeb (Muscat, Oman) area was used. Sieve analysis performed on the sand showed a mean diameter of 0.7 μm, an effective size of 0.48 μm, and a coefficient of uniformity of 1.458. The density and porosity of the sand were found to be 2.626 and 0.42, respectively.

The sand was tested for weight loss by heating a sample of 20 g to a temperature of 105 °C for one hour and weighing before and after the heating operation. It was then dried in the muffle furnace at 600 °C for 15 minutes, and its resulting weight was recorded. No significant weight change was noted, which means that the sand was free from organic contaminants. A small pump was used to deliver the feed from the continuously mixed contents in the reservoir to the recycle line connected to the column. The inflow was nearly 2 liters per hour. The effluent discharged from the top of the column through a submerged pipe was transferred to a settling tank having a capacity of 12 L. Effluent from the reactor was collected at the top of the settling tank.

A large portion of the activated biomass, mixed with the feed solution, was pumped by the means of a centrifugal pump to the column through the recycle line connecting the settling tank with the column. Air was injected into the bottom of the settling tank to promote oxygenation and mixing. The experimental unit was initially charged with 345 g of raw virgin sand. The amount of sand, which can be effectively kept fluidized, was determined by several initial trials testing the capacity of air flow and underflow pump capacities. The feed solution used throughout the experimental work consisted of the same synthetic wastewater used in the batch reactor.

The column and the settling tank were filled with tap water into which 200 mL of the effluent of the batch reactor was transferred and diluted to promote microbial growth. After two weeks of acclimatization of the microbial population, the formation of biofilms became evident on the fluidized media in conformity with the observations made by Shieh and Li (1989). The experimental unit was then ready for the steady state experimental work. A total of nine steady state experiments was conducted. The amount of sand in the column for all the experiments was 345 g. For each experiment, the temperature of the bulk liquid, pH of the effluent flow, depth of fluidized bed, and TOC initial and TOC final of a steady state were measured. Experiments were run every alternate day on the assumption that 48 hours was adequate for the microbial population to adapt to the applied feed and flow conditions. The experiments were conducted at batch test. The unit was operated with synthetic wastewater as a continuous system with very low oil concentration of 50 mg/L. The oil was fed every four hours at an amount equal to four-hour inflow of oil at 100 mg/L. For a hydraulic inflow of 2 L/hour, every four hours an oil amount of 400 mg was added in the reactor from the top. This small amount of oil addition ensured that the biomass remained acclimatized to oil environment.

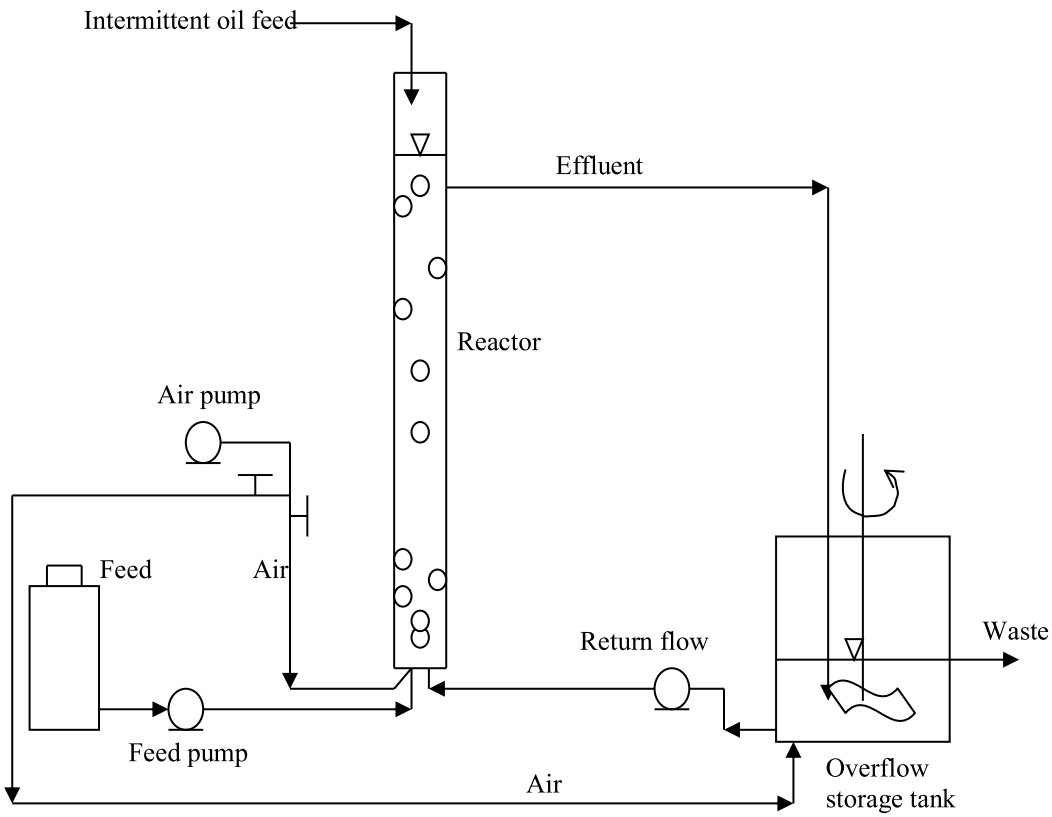

Figure 1. Schematic diagram of pilot FBBR.

Once a steady or pseudosteady state in the continuous process is reached, the inflow pump is stopped. The reactor is spiked with oil to a desired concentration. The air supply and return flow continue. After one hour of oil addition, samples were taken and analyzed for the initial state of the reactor. After a period of one detention time, which is equal to the hydraulic detention time of the previous operating steady state, the final samples were taken and analyzed to determine the final state of the system. Thus, the experiments at this section of the tests were operated like batch reactors spiked with a large single dose of oil added at the start of each experiment. From the experience, it was found that the added oil broke up in minute globules within an hour and got completely mixed and suspended with reactor contents. Hence, the initial sample for analysis was taken after one hour of the oil-spike.

RESULT AND DISCUSSIONS

Kınetıc Coefficıents from Batch Reactor Data

The experimental data determined on samples collected from the batch reactor are shown in Tables 2 and 3. In these experiments, only synthetic wastewater was used without any addition of oil.

Table 2. Experimental data obtained from batch reactor.

θ (days)	S_o (mg/L) TOC*	S (mg/L) TOC	X (mg/L) VS	Rate of degradation mg/mg/hour
1.0	82	28	65.41	0.034
1.1	115	27	98.24	0.034
1.1	118	23	114.35	0.031
1.25	115	19	111.36	0.029
1.5	124	22	91.06	0.03
2.0	136	13	115.09	0.022

* BOD_5 = approximately 2 times of TOC

where θ = hydraulic detention time; S_o = substrate concentration in the reactor at the beginning of θ; S = substrate concentration in the reactor at the end of θ; X = concentration of microorganisms in the reactor effluent at the end of θ. The kinetics coefficients K, Ks, Y, K_d and μ_m (maximum growth rate) were calculated using equations 1 and 2 (Metcalf & Eddy, 1984).

$$(X\theta/(S_o - S)) = ((K_s/K)(1/S)) + (1/K) \quad (1)$$

$$(1/\theta c) = (Y((So - S)/X\theta)) - K_d \quad (2)$$

where θ_c = Biomass in reactor / Biomass wasted per day, (day^{-1})

Table 3. Data for calculation of kinetic coefficients.

Steady state No.	Volume wasted, mL	M* mg/day	θ_c =X/M Day	$1/\theta_c$	1/S	$(S_o - S)/ X\theta$	$(X\theta)/ (S_o - S)$
1	470	30.7	2.13	0.47	0.0357	0.826	1.21
2	440	43.2	2.27	0.44	0.037	0.814	1.23
3	430	49.2	2.32	0.431	0.0435	0.755	1.32
4	375	42.8	2.6	0.385	0.0526	0.689	1.45
5	400	36.4	2.5	0.4	0.0455	0.748	1.34
6	280	32.2	3.57	0.28	0.0769	0.534	1.87

* M = Biomass wasted per day

The values of K_d and Y were obtained from Figure 2. The coefficient of determination was found to be 0.98. The values of K and K_s were determined from Figure 3. The coefficient of determination was found to be 0.998. The value of maximum growth rate (μ_m) was determined by equation 3 (Metcalf & Eddy, 1984).

$$\mu_m = Y K \quad (3)$$

The parameter values found were Y= 0.61; K_s= 26 mg/L; K= 1.6 day^{-1}; K_d = 0.044 $days^{-1}$; μ_m = 1.0 $days^{-1}$. The values obtained for Y, K_s, and K_d lie within the range reported in Metcalf and Eddy (1984) for municipal wastewater, which are Y = 0.4–0.8 mg VSS/mg BOD_5, K_s = 25–100 mg/L BOD_5, K_d = 0.04–0.08 $days^{-1}$. The value of K was 1.6 day^{-1}, which was slightly lower than the range 2– 10 day^{-1} as reported in Metcalf and Eddy (1984).

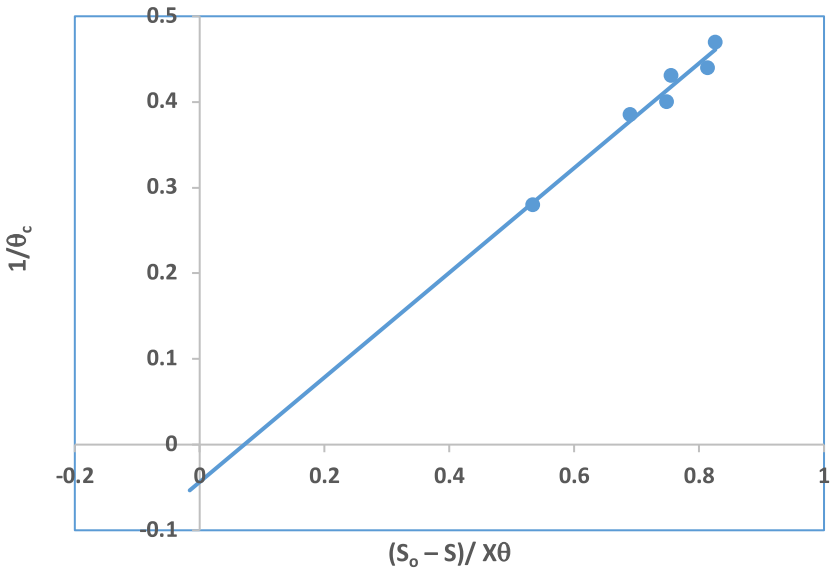

Figure 2. Relationship between ($1/\theta_c$) vs. ($S_o - S$)/Xθ.

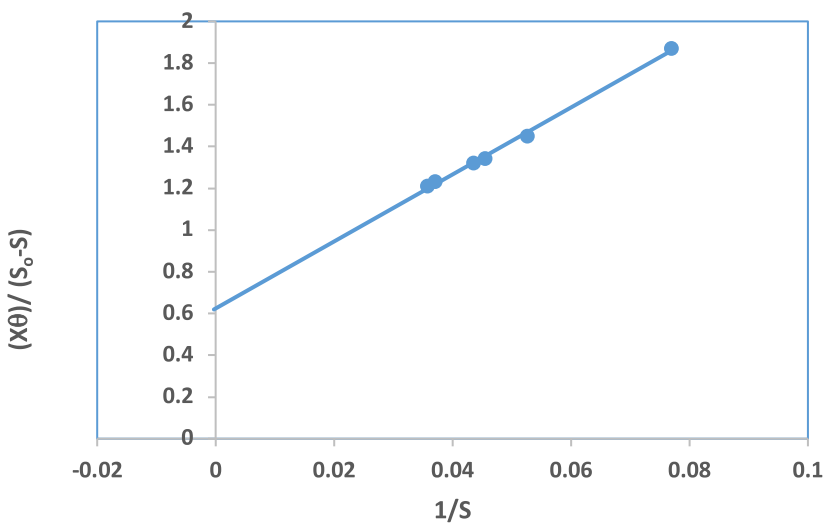

Figure 3. Relationship between (Xθ)/(So-S) and (1/S).

Estımatıon of Bıo-Fılm Thıckness

Experiments without the oil addition were conducted in a one-liter column. Due to aeration limitation, only 5 g of sand could be kept suspended. In these experiments, sand contents were kept more or less by returning sand in the wasted volume back into the unit. The wasted volume was allowed to settle for 5 min. The supernatant was discarded, when settled sand was gently returned to the operating reactor. Mean volatile fraction of total solids was found to be 0.6. Based on the total solids, 5000 mg/L of sand with a mean diameter of 0.7 μm, approximate sand surface area of 0.004 m^2/g, sludge density of 1.3 g/L, and negligible nonattached suspended solid portion, the biofilm thickness on sand was determined. The values of biofilm thickness (δ) for six experiments are shown in Table 4. Mean biofilm thickness was 6.4 μm.

Table 4. Estimated biofilm thickness.

Steady state No.	Total Solids, mg/L	Biofilm thickness (δ), μm
1	108.5	4.2
2	167.7	6.5
3	190.6	7.3
4	185.6	7.2
5	151.8	5.8
6	191.8	7.4

Degradatıon Of Oil in FBBR

Table 5 shows the initial and final concentrations of oil (SAE –40, Sp. Gravity = 1.0595) along with initial and final concentrations of other carbon sources measured in several steady state batch reactor experiments having a detention time of 12 hours. The experiment was carried out at room temperature, which remained within the range of 25 to 26 oC. The pH of reactor water was found to be within the range of 6.8 to 7. The solids concentration in the reactor, ratios of degradation rates of oil and non-oil substances and ratio of influent TOC, and oil concentration were shown in Table 6. Table 6 also contains the biofilm thickness estimated considering total dry solids attached to sands, which was about 1.667 times the volatile solids attached to the sand. The calculations were based on available surface area of sands of 0.7 μm diameter to be 0.004 m^2/g and the sludge specific gravity was 1.3. The mean 'δ' value was 22 μm, which was well within expected range of 20-40 μm (Jeris et al., 1981).

Total organic carbon (TOC) shown in Tables 5 and 6 indicates TOC available from non-oil sources only. It is evident that when synthetic wastewater was the only source of food for microorganisms, the average rate of the TOC degradation was 0.03 mg TOC/mg/hour (Table 5). However, the average rate of TOC degradation fell to 0.02 /hour (Table 5), when oil was present in the reactor. The presence of oil had a retarding effect on the utilization of non-oil source TOC by microorganisms. One of the reasons of such an outcome may be due to the attachment of oil globules on biofilm. It may reduce the transfer of substances from bulk liquid into the biofilm. The oil property of not mixing with water may enhance the retarding effect. The availability of additional food from oil sources may also have effects on the microbial utilization of synthetic wastes.

The kinetic parameters determined from the data (Tables 2, 3, and 4) collected from batch experiments using synthetic wastewater only had values, which compare very closely with values reported elsewhere (Metcalf & Eddy, 1984). The finding indicated that the process of sand fluidization, the composition of synthetic wastewater, and microbial activities in the reactor were quite normal as expected. Estimated mean biofilm thickness in the experiments with synthetic wastewater as main food source was found to be 6.4 μm (Table 4) when it was 22 μm (Table 6) in experiments where oil was present. The attachment of oil globules onto the biofilm of the sand may cause the film to be thicker. The thicker biofilm may have benefits as well as adverse effects on the overall performance of the process. The favorable effect may include enhanced activity due to excess biomass. Adverse effects were expected from reduction of effective surface area as the sand-biomass matrix gets larger and inefficiency in the substrate transport from bulk liquid to the biomass. The reduction in the TOC degradation rate as oil was added supports the assumption (Table 5).

From the results of oil degradation (Table 5), it was quite evident that motor oil (SAE-40, Sp. Gravity 1.0595) could be degraded up to 1500 mg/L. It has been reported that, in conventional activated sludge process, the oil concentration up to 500 mg/L can be treated utilizing microorganisms (Metcalf & Eddy, 1984). The improvement in oil degradation using the FBBR appears to be quite significant and encouraging. Average oil degradation rate of 0.053 mg oil/mg/hour (Table 5) was about 2.5 times more than that of synthetic wastewater expressed in TOC (0.02 mg TOC/mg/hour). It was noted that the results represent an initial oil concentration range of 1000-2600 mg/L. As the oil concentration increased, the difference between oil and TOC degradations was more pronounced. It shows that, as the main bioactivity was limited in the biofilm, the increasing oil concentration facilitates oil globules to be more attached to the biofilm. This causes the microbes to come more in touch with oil. It was noted that the findings are specific to a specific brand of motor oil, which is even slightly heavier than water.

Table 5. Degradation of oil in batch steady state reactors.

Steady State No.	Total Solids, mg/L	Total VS, mg/L	Initial Oil conc., mg/L	Final Oil conc., mg/L	Initial waste-water TOC, mg/L	Final waste-water TOC, mg/L	Rate of oil degradation, mg/mgVS/h	Rate of waste-water TOC degradation, mg/mgVS/ h
1	5500	3300	2500	1000	420	80	0.038	0.0087
2	3000	1890	2600	1500	410	95	0.052	0.015
3	1000	600	2000	1500	400	186	0.07	0.03
4	1000	600	1600	900	383	175	0.097	0.029
5	2500	1550	2000	1000	404	102	0.055	0.017
6	1500	900	2000	1400	410	108	0.055	0.027
7	2000	1200	1400	1000	400	96	0.028	0.022
8	4625	2775	2250	500	420	98	0.053	0.01
9	2000	1200	1000	500	405	90	0.035	0.022

Table 6. Degradation rate ratios of oil and wastewater TOC.

Steady State No.	Total solids attached to sand, mg/L	VS attached to sand, mg/L	Biofilm thickness (δ), μm	Input: Oil/wastewater TOC (mg/mg)	Degradation rate ratio: Oil/wastewater TOC
1	3667	2200	47	5.95	4.4
2	2100	1260	27	6.34	3.5
3	667	400	9	5	2.36
4	667	400	9	4.17	3.35
5	1723	1033	22	4.95	3.3
6	1010	600	13	4.88	2.06
7	1325	800	17	3.5	1.3
8	3080	1850	39	5.36	5.5
9	1315	800	17	2.47	1.6

CONCLUSION

The steady state biofilm thickness (δ) in a FBBR process using synthetic wastewater and motor oil having a concentration of 1000–2600 mg/l seems to be significantly affected by the concentration of oil. Average 'δ' under specific conditions was found to be 22 μm. The batch reactor experiment was successful in determining the kinetic coefficients and operating environment of the process, since the values obtained fell within the reported range of municipal wastewater. The degradation rate of non-oil source carbon (synthetic waste) decreased as oil was introduced in the FBBR process. Average oil degradation rate of 0.053 mg oil/mg biomass/hour was measured in a FBBR, when the oil input range is 1000–2600 mg/L. The results corresponded to volatile solids attached to sand ranging from 400 to 2200 mg/L. Motor oil (SAE-40, Sp. Gravity 1.0595) could be degraded up to a concentration of 1500 mg/L in FBBR process under specific experimental condition. The present studies were carried out with known oil content. However, the composition of industrial oil wastewater will be complex and highly variable in the oil content and TOC. The present study is not an adequate treatment or even pretreatment for high oil content wastewater. Further, studies need to be carried out with highly variable industrial oil wastewater to validate the application of this technology at large commercial scale.

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