



Anaerobic biodegradation of personnel care products (PCPs) wastewater in an up-flow anaerobic sludge blanket (UASB) reactor

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ABSTRACT

The influence of transient changes in influent organic loading rate (OLR) on process stability of up-flow anaerobic sludge blanket reactor (UASB) reactor treating personnel care products (PCPs) wastewater was investigated at constant hydraulic retention time (HRT) of 24 h. The OLR of the reactor was increased stepwise from 1.49 to 4.0 kg COD m⁻³ d⁻¹. The chemical oxygen demand (COD) removal efficiency and methanogenesis process was increased from 40 to 68.7% and from 52.6 to 54.7% with increasing OLR from 1.49 to 2.9 kg COD m⁻³ d⁻¹, respectively. Nevertheless, increasing the imposed OLR from 2.9 to 4.0 kg COD m⁻³ d⁻¹ caused a considerable reduction in the COD removal efficiency (45%) and methanogenesis process (38%) implying that the UASB reactor was overloaded. In a subsequent experiment; the UASB reactor was operated at optimum OLR of 2.5 kg COD m⁻³ d⁻¹ and a HRT of 24 h for a period of 156 days. The UASB reactor achieved a removal efficiency of 65% for COD_{total}; 60% for COD_{soluble}; 71.2% for TSS and 57.3% for oil and grease. Moreover, 0.3391 CH₄ g COD depleted⁻¹ d⁻¹ was produced. Accordingly, it is recommended to apply such a system at an OLR not exceeding 2.5 kg COD m⁻³ d⁻¹ and a HRT of 24 h.

Keywords: Personnel care products (PCPs); UASB; COD; Methanogenesis; Biodegradability

1. Introduction

Personal care products (PCPs) industry usually involves complex manufacturing processes that consume large amount of organic and inorganic materials and generate highly polluted and fluctuated wastewater. This wastewater normally consist of mainly soluble organics, suspended solids, detergent, anionic surfactants (ASs), oil and grease, and refractural substances [1]. In Egypt, PCPs wastewater

industry is discharged into sewerage network without any treatment which deteriorates the efficiency of the sewage treatment plants (STPs). Moreover; the presence of surfactants is responsible for causing foam in effluents of treatment plants [2]. The treatment of PCPs wastewater industry prior entering the sewerage network is required. Several conventional methods have been carried out for treatment of wastewater rich in detergents such as the coagulation–flotation process [3] and advanced oxidation process [4–7]. Although these methods have been widely applied, they have some disadvantages, i.e.

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chemical coagulation causes additional pollution due to the undesired reactions in treated wastewater and produces considerable amounts of sludge [1]. Moreover, these methods are also usually expensive and treatment efficiency for organics in a soluble form is inadequate [8]. Anaerobic treatment (AT) is one of the most advantageous technologies for the treatment of wastewaters containing organic compounds [9,10], due to its relatively low cost in comparison to physicochemical treatment methods. PCP's wastewater has been reported to be anaerobically biodegraded under certain operating conditions [11,12]. An up-flow anaerobic sludge blanket (UASB) reactor was the one of most frequently systems applied for AS removal from wastewater industry [13]. Lober et al. [14] found 40–80% removal of AS in a bench-scale UASB reactor under mesophilic and thermophilic conditions. Likely, Sanz et al. [15] found that the UASB reactor is effective for biodegradation of AS without addition of co substrate.

The aim of this study is to investigate the performance of the UASB reactor treating PCPs wastewater industry at different organic loading rates (OLRs), with emphasis on the removal efficiency of COD; BOD₅; TSS; oil, and grease. Moreover, the anaerobic biodegradability (AB) tests and anaerobic conversion processes (ACP) of PCPs wastewater were assessed.

2. Materials and methods

2.1. Wastewater characteristics

The industrial wastewater used in this study was provided from a personnel care products factory in Egypt. The company produces shampoo, toothpaste, creams, and liquid soap. The main AS used in the factory is Forayel ether sulfate (FES). Some other chemicals are also used in the formulation of different PCPs, such as: calcium carbonate; silica; sorbitol; stearic acid; niacinamide; butyl methoxy cinnamat; carbopol 980 (acrylic polymer-carbomer); dimethyl ammonium and sodium hydroxide. The mean characteristics of the PCPs wastewater industry are presented in Table 1.

2.2. Lab scale UASB reactor

The experimental setup used in this investigation is shown in Fig. 1. The UASB reactor was designed and manufactured from polyvinyl chloride with 9.2 cm internal diameter and 150 cm height. Total effective volume of the reactor is 101. The reactor was operated and situated at the PCPs factory. The influent wastewater was continuously pumped into the reactor using a peristaltic pump. The wastewater was introduced from the

Table 1
Characteristics of PCPs wastewater

Parameters	Values
pH-value	7.7 ± 0.7
COD _{total} (mg/l)	2,576 ± 488
COD _{filtrated} (mg/l)	1,424 ± 222
COD _{particulate} (mg/l)	1,152 ± 447
VFA–COD (mg/l)	519 ± 113
TSS (mg/l)	503.2 ± 122
VSS (mg/l)	154.1 ± 16.3
TKj-N (mg/l)	14 ± 5.5
Total-P(mg/l)	6.3 ± 2.5
O&G (mg/l)	234.1 ± 99

bottom of the reactor via inlet distribution network and the outlet was collected from the top of the reactor through overflow by means of a gas–solid separator. The biogas produced was collected in gas bags with a capacity of 4.0l/bag, which was changed after being filled. The gas volume is determined by emptying the gas bag via a vacuum pump connected to a wet gas meter. The reactor was inoculated with partially digested sludge (VS = 14 g/l) from a UASB reactor treating municipal wastewater. The UASB reactor was operated at an average wastewater temperature of 22°C.

2.2.1. Operational strategy of a UASB reactor

Sludge acclimatization. The sludge was acclimatized with PCPs wastewater industry under study for three weeks. The reactor was daily batch-fed with diluted PCPs (300–500 mg COD l⁻¹). This substrate was replaced stepwise with the original wastewater by proportionally increasing the feed volume of the PCPs wastewater.

Experimental design. Continuous feeding of the reactor was started with an initial OLR of 0.7 kg COD m⁻³ d⁻¹. The HRT of 24 h was kept constant throughout the whole experimental period. The influent COD concentration was 700 mg/l for the first 7 days, and then it was increased stepwise to 1,494 mg/l (OLR = 1.49 kg COD m⁻³ d⁻¹) from 28 to 50 days. Steady state operating conditions were attained after 50 days. Cattony et al. [16] found that at least 30 days are necessary to attain steady state operating conditions for horizontal-flow anaerobic immobilized biomass (HAIB) reactor treating sulfate rich wastewater. After attaining a consistent stable biogas production condition at OLR of 1.49 kg COD m⁻³ d⁻¹, two experiments were conducted. In the first experiment; the OLR imposed to the UASB reactor was increased step by step from 1.49 to

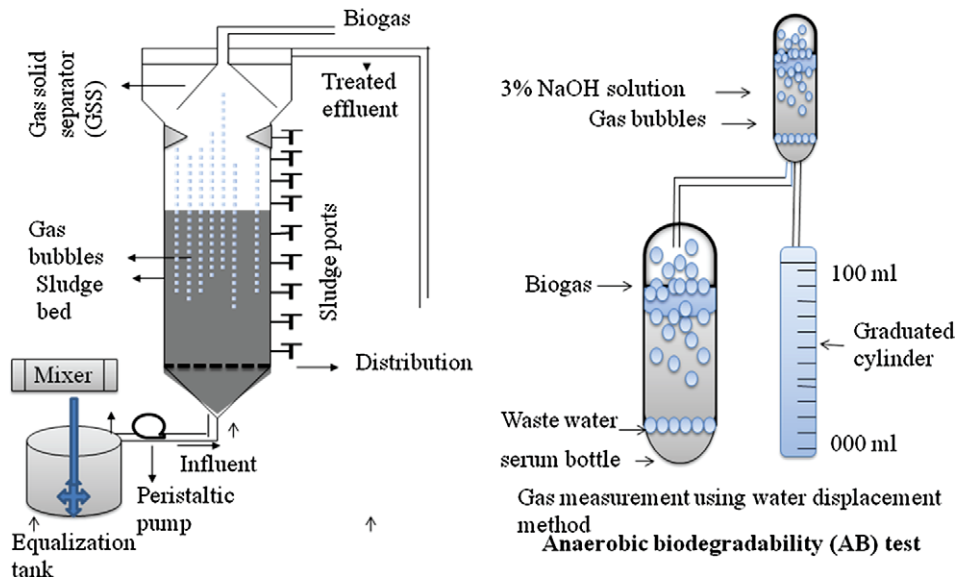


Fig. 1. UASB reactor treating PCPs industry wastewater and biodegradability test.

$4.0 \text{ kg COD m}^{-3} \text{ d}^{-1}$ to optimize the operational conditions of the reactor. At each loading rate, the UASB reactor was operated until a steady state performance was reached. In the second experiment the UASB reactor was operated for a period of 156 days at optimum OLR of $2.5 \text{ kg COD m}^{-3} \text{ d}^{-1}$, and a HRT of 24 h.

2.3. Calculations

The percentage of hydrolysis (H), acidification (A), and methanogenesis (M) of the UASB reactor was calculated according to Eqs. (1), (2), and (3), respectively.

$$\text{Hydrolysis (H)} = \left\langle \frac{\text{CH}_4 \text{ as COD} + \text{Effluent COD}_{\text{filtered}} - \text{Influent COD}_{\text{filtered}}}{\text{Influent COD}_{\text{particulate}}} \right\rangle \times 100 \quad (1)$$

$$\text{Acidification (A)} = \left\langle \frac{\text{CH}_4 \text{ as COD} + \text{Effluent VFA as COD} - \text{Influent VFA as COD}}{\text{Influent COD}_{\text{total}} - \text{Influent VFA as COD}} \right\rangle \times 100 \quad (2)$$

$$\text{Methanogenesis (M)} = \left\langle \frac{\text{CH}_4 \text{ as COD}}{\text{Influent COD}_{\text{total}}} \right\rangle \times 100 \quad (3)$$

2.4. AB test of PCPs wastewater

A batch AB test for PCPs wastewater was carried out according to the method described by El-

Mitwalli et al. [17]. This method evaluates the extent of ultimate anaerobic biodegradation of the PCPs wastewater based on the production of biogas. The AB was determined in duplicate for raw, paper-filtered, and membrane-filtered wastewater at a temperature of 30°C . The experiments were carried out two times for different wastewater samples to achieve representative AB values. The experiment was performed in serum bottles with a capacity 250 ml and each bottle was flushed with nitrogen gas for 5.0 min to guarantee anaerobic conditions. The bottles were fitted with gas tight septa and aluminum crimp seals. After sealing the vessels and incubating them for 1.0 h at 30°C , excess gases were allowed to release to the atmosphere. The incubation process was preceded in the dark. The experiments were carried out without inoculums addition. Therefore, the AB was determined after a long test time of 180 and 161 days for the first and the second experiment, respectively. The increase in headspace pressure in the closed bottles was used to follow the conversion process. Gas volume was measured using the water displacement method. The biogas was regularly measured by passing total biogas through 3% NaOH solution and measuring the amount of NaOH displaced. Moreover, the concentration of $\text{COD}_{\text{total}}$, $\text{COD}_{\text{filtered}}$, and $\text{COD}_{\text{particulate}}$ were measured at the start and the end of each experiment. The AB of PCPs wastewater was calculated according to the Eqs. (4) and (5),

$$\text{Biodegradability (\%)} = \frac{\text{COD as CH}_4}{\text{Influent COD}_{\text{total}}} \times 100 \quad (4)$$

$$\text{Biodegradability (\%)} = \frac{\text{COD as CH}_4}{\text{Influent COD}_{\text{soluble}}} \times 100 \quad (5)$$

2.5. Analytical methods

Monitoring of the performance of the UASB reactor treating PCPs wastewater was carried out by analyzing influent and the treated effluent, twice a week. pH, chemical oxygen demand (COD), volatile fatty acids (VFA), total suspended solids (TSS), volatile suspended solids (VSS), total Kjeldahl nitrogen (TKj-N), total phosphorous (TP), oil and grease (O&G) were determined according to APHA [18]. Due to a lack of facilities, AS was not measured. The filtrate of the 0.45 μm sterile membrane filter paper (Whatman, England) was used to determine the filtrate COD. The $\text{COD}_{\text{particulate}}$ was calculated by the difference between $\text{COD}_{\text{total}}$ and $\text{COD}_{\text{filtered}}$.

3. Results and discussion

3.1. Influence of transient changes in influent OLR on process stability

Surfactants and detergents were reported to adversely impact anaerobic digestion [19]. As with other inhibitory substances, microbial acclimation is an important process in overcoming the inhibitory effects of organic substances [20]. Therefore, a short term experiment concerning influence of transient changes in influent OLR on process stability of UASB reactor was assessed. Table 2 shows the performance of the UASB reactor at different OLR for removal of COD and the biogas production. The average COD removal efficiency was increased from 40% (days 28–50) to 51% (days 50–67) with increasing OLR from 1.49 to 1.96 $\text{kg COD m}^{-3} \text{d}^{-1}$. The influent OLR was then increased stepwise to 2.9 $\text{kg COD m}^{-3} \text{d}^{-1}$ (67–122 d). The COD removal efficiency reached to 68.7%.

However, increasing the imposed OLR from 2.9 to 3.5 $\text{kg COD m}^{-3} \text{d}^{-1}$ caused a considerable reduction in the COD removal efficiency (54.3%) implying that the system was overloaded and indicated a shift in the methanogenic population. This is also reflected by the decrease of the methanogenesis conversion process during these periods (Table 2). These results are comparable with those obtained by Vidal et al. [21] who found that the sudden increase in COD loads substantially reduce the activity of methanogenic bacteria. Moreover, sensitivity of methanogenic bacteria to surfactant rich wastewater has been previously described by Alexander [22]. The OLR was increased subsequently to 4 $\text{kg COD m}^{-3} \text{d}^{-1}$. at days (143–166) which led to a further deterioration of the methanogenesis process resulting in low methane yield (0.221 $\text{CH}_4 \text{ g COD depleted}^{-1} \text{ d}^{-1}$) in the reactor. Nonetheless, an increase in the hydrolysis and acidification processes was occurred as shown Table 2. Based on these results, the UASB reactor treating PCPs wastewater can be successfully operated up to 2.9 $\text{kg COD m}^{-3} \text{d}^{-1}$.

3.2. Long-term evaluation of the performance of the UASB reactor treating PCPs wastewater at optimum OLR of 2.5 $\text{kg COD m}^{-3} \text{d}^{-1}$

The results presented in Figs. 2abc show the influent and effluent quality of the UASB reactor treating PCPs wastewater in terms of $\text{COD}_{\text{total}}$, $\text{COD}_{\text{particulate}}$, and $\text{COD}_{\text{filtered}}$. The effluent quality of the UASB reactor was remarkably stable at an OLR of 2.5 $\text{kg COD m}^{-3} \text{d}^{-1}$, with a good removal efficiency of 65% for $\text{COD}_{\text{total}}$; 60% for $\text{COD}_{\text{filtered}}$, and 69.3% for $\text{COD}_{\text{particulate}}$. This good removal efficiency is probably due to the adsorption and biodegradation processes in biological anaerobic sludge [23]. The relatively high values of soluble COD removal achieved in the reactor ($60 \pm 9\%$) indicate that the

Table 2
Performance of UASB reactor at various organic loading rates (OLR s)

OLR ($\text{kg COD m}^{-3} \text{d}^{-1}$)	ORR ¹ ($\text{kg COD m}^{-3} \text{d}^{-1}$)	Operational period (d)	COD (%R)	H* (%)	A** (%)	M*** (%)	L $\text{CH}_4 \text{ g COD depleted}^{-1} \text{ d}^{-1}$
1.49	0.594	28–50	40	11.2	4.1	52.6	0.5
1.96	1.003	50–67	51	16.1	10.3	66.7	0.49
2.28	1.3	68–83	57	18.9	10.2	68.9	0.46
2.58	1.585	83–101	61.3	21.2	12.1	60.7	0.378
2.9	1.97	102–122	68.7	21	12.9	54.7	0.3
3.5	1.9	122–142	54.3	21	13	43	0.26
4.0	1.8	143–166	45	23	16	38	0.22

Note: H*, hydrolysis; A**, acidification; M***, methanogenesis; ORR¹, organic removal rate.

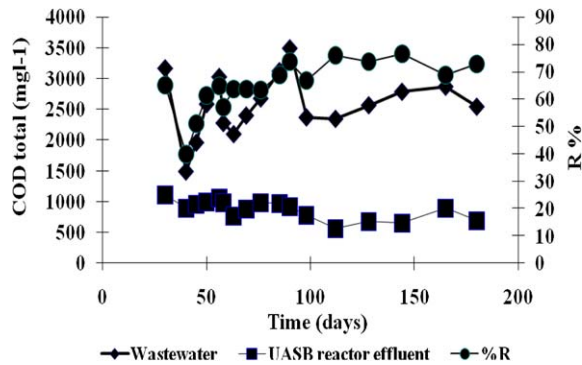


Fig. 2a. Removal efficiency of COD filtered in an UASB reactor treating PCPs wastewater.

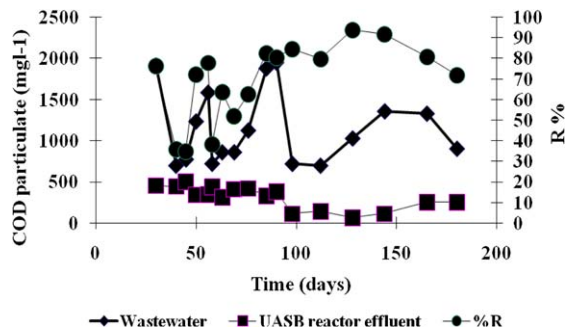


Fig. 2b. Removal efficiency of COD total in an UASB reactor treating PCPs wastewater.

surfactant did not interfere significantly in the degradation of the soluble organic matter. A higher removal efficiency of COD_{filtered} (90%) was obtained by Oliveira et al. [24] who used a horizontal-up-flow anaerobic immobilized biomass reactor (HAIB) for treatment of surfactant-rich wastewater with HRT of 12h, COD influent (550 mg/l), and AS (14 mg/l). Their results for a mass balance indicated that 28% of AS was removed by the anaerobic degradation process. The presence of AS in the wastewater fed to the anaerobic system

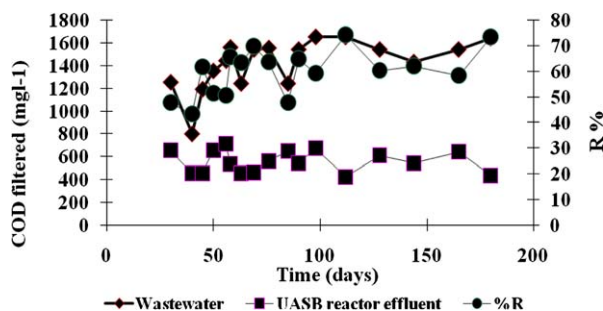


Fig. 2c. Removal efficiency of COD particulate in an UASB reactor treating PCPs wastewater.

might increase the bioavailability of other organic compounds sorbed on the anaerobic sludge enhancing their biodegradation and leading to an increase in the biogas production [12]. The results of biogas production of the UASB reactor are illustrated in Fig. 3a. The average methane production amounted to 0.341 CH₄g COD depleted⁻¹ d⁻¹ which was similar to that found by Oliveira et al. [24]. Moreover, the conversion of COD to methane was almost similar to the theoretical value (0.351 CH₄g COD removed⁻¹).

Fig. 3b shows the course of the hydrolysis, acidification, and methanogenesis processes in the UASB reactor vs. time. Methanogenesis was apparently the rate-limiting step for the overall conversion of organic matter to methane in the UASB reactor as the effluent of COD_{soluble} and VFA–COD remained relatively high in the treated effluent as shown in Fig. 4. The reactor achieved a removal efficiency of 54.9% for VFA–COD resulting in a residual value of 244 mg/l in the treated effluent. Some of these VFA could not be utilized by methanogenic bacteria in the reactor, which were important parts in the reactor effluent COD, resulting in the VFA/COD ratio increasing observably in the treated effluent (0.26) than that in the influent (0.2). This indicates that the hydrolytic–acidogenic bacteria were carried out satisfactorily and the imbalance of the process was due to the stress of methanogenic bacteria. Lissens et al. [25] showed that in a two-stage anaerobic digestion system, greater resistance toward inhibiting chemicals would be achieved.

The results presented in Figs. 5a and b show that the UASB reactor achieved a considerable reduction of 71.2% for TSS and 69.7% for VSS. Due to its hydrophobic character, AS is strongly sorbed to coarse suspended solids and can be easily entrapped onto the sludge bed of the UASB reactor. Oil and grease removal efficiency was 57.3% (Fig. 6). This low removal efficiency can be certainly due to the accumulation of oils in the sludge bed. Palenzuela Rollon [26] found that the removal of oils from wastewater prior to anaerobic treatment would achieve a better process stability, i.e. using a two-stage system connected in series [27]; or by a dissolved air flotation unit [3].

3.2.1. AB test

Fig. 7 shows the decrease of COD fractions' (COD_{total}, COD_{particulate}, and COD_{filtered}) concentration and concomitant increase in methane production rate. The results showed that the COD_{total} was decreased from 2,453 to 690 mg⁻¹ and the methane gas production as CH₄–COD was increased up to 1,764 mg⁻¹ after

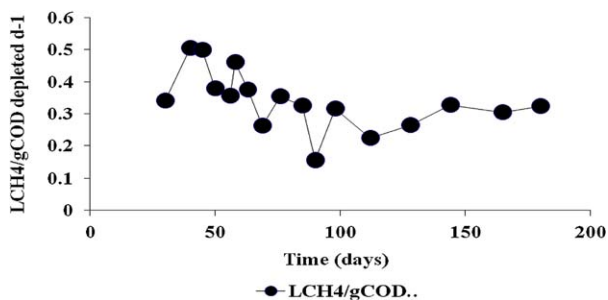


Fig. 3a. Biogas production in an UASB reactor treating PCPs wastewater.

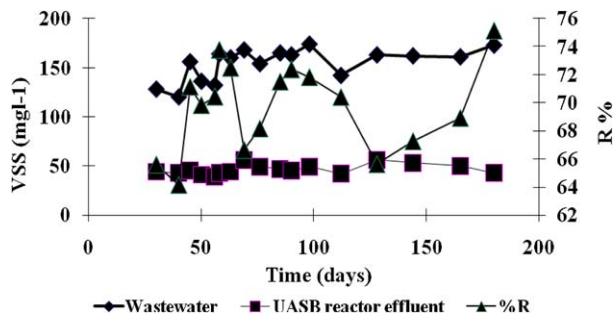


Fig. 5b. Removal efficiency of VSS in an UASB reactor treating PCPs wastewater.

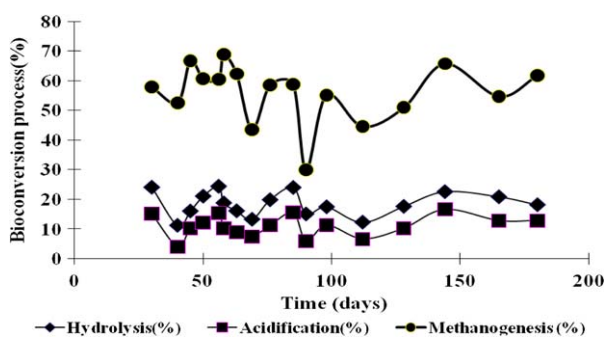


Fig. 3b. Hydrolysis, acidification, and methanogenesis process in an UASB reactor treating PCPs wastewater.

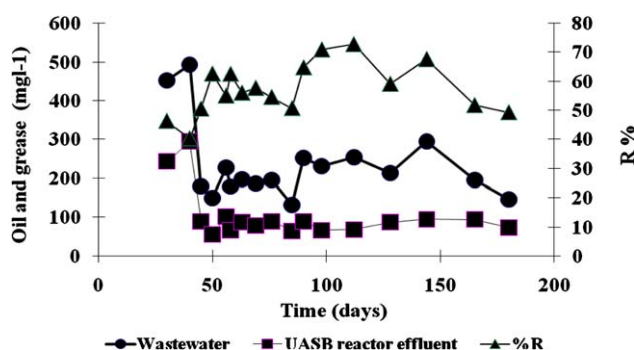


Fig. 6. Removal efficiency of oil and grease in an UASB reactor treating PCPs wastewater.

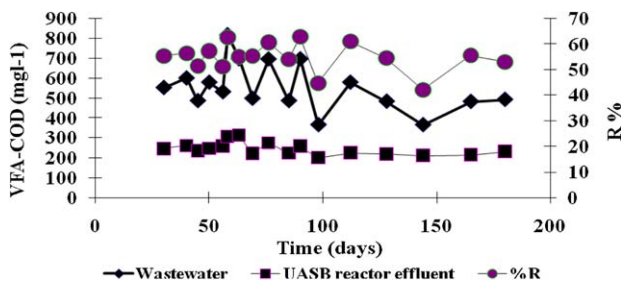


Fig. 4. Removal efficiency of VFA-COD in an UASB reactor treating PCPs wastewater.

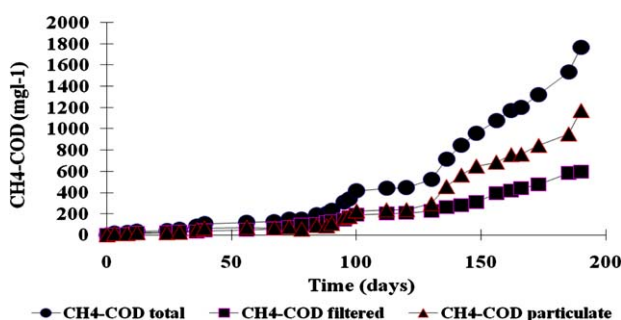


Fig. 7. Anaerobic biodegradability (AB) test of PCPs wastewater.

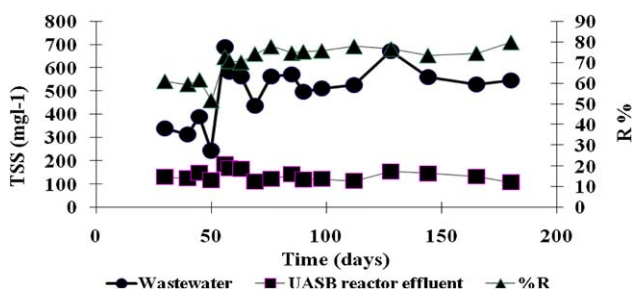


Fig. 5a. Removal efficiency of TSS in an UASB reactor treating PCPs wastewater.

180 days. The calculated AB for COD_{total} was 71.9%. The COD in the particulate form had the highest AB (81.2%) and the COD in the soluble form was relatively lower (AB=59%). Low degree of anaerobic degradation of the soluble COD can be certainly due to the stabilization of the ester bond by the adjacent sulfonate group (FES). Likely, low AB of 40% for methyl ester sulfonates (MES) was found by Garcia et al. [28]. This was not the case for AB of dialkyl sulfosuccinates (di-C₈-SS) and monoalkyl ethoxy sulfosuccinates (C12 (EO)₃-SS) where the AB was higher (73 and 76%)

respectively. Differences between MES and sulfosuccinates can be attributed to their molecular structure. The ester bonds in the sulfosuccinates are easily hydrolysable either chemically or enzymatically. This enables cleavage into non-surface active fragments and is consistent with the high AB. Remde and Debus [29] investigated the AB of fluorinated surfactant. They found that a fluorinated surfactant was easily degraded (91%) under anaerobic conditions during the incubation period of 60 days.

4. Conclusions

The results obtained indicated that the UASB reactor has a great potential in treating PCPs wastewater with stable operation and satisfactory removal performance at loading rate not exceeding $2.5 \text{ kg COD m}^{-3} \text{ d}^{-1}$. The COD removal efficiency and methanogenesis process was reduced from 68.7 to 54.3% and from 52.6 to 38%, respectively, when the loading rate was increased from 1.49 to $4.0 \text{ kg COD m}^{-3} \text{ d}^{-1}$. At optimum loading rate of $2.5 \text{ kg COD m}^{-3} \text{ d}^{-1}$; the UASB reactor achieved a removal efficiency of $\text{COD}_{\text{total}}$ (65%) and $\text{COD}_{\text{filtered}}$ (60%). Moreover, $0.341 \text{ CH}_4 \text{ g COD depleted}^{-1} \text{ d}^{-1}$ was produced. In addition, the reactor provided a considerable reduction of 71.2% for TSS and 69.7% for VSS. AB test of PCPs wastewater amounted to 71.9% for $\text{COD}_{\text{total}}$, 81.2% for $\text{COD}_{\text{particulate}}$ and 59% for $\text{COD}_{\text{filtered}}$.

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