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Original Paper

Anaerobic treatment of pharmaceutical wastewater using packed bed reactor

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Abstract

Typically, pharmaceutical wastewater is characterized by high chemical oxygen demand (COD) concentration, and some pharmaceutical wastewaters can have COD as high as 1100 mg/L. Due to high organic content, anaerobic technology is a promising alternative for pharmaceutical wastewater treatment. Consequently, in the present study,an anaerobic packed bed reactor was designed and employed to treat highly polluted of pharmaceutical wastewater of Samarra Drug Factory (SDI) in Samarra city (north of Baghdad) in Iraq and suggested to be added to the available wastewater treatment unit of SDI. The efficiency of the anaerobic filter with respect to bed height of filter was studied. The results showed that the first third of filter was the more effective. The filter completed the acclimation period within 34 days in which the COD removal efficiency was 85%. The results also showed that the best hydraulic retention time (HRT) was 24 hours for anaerobic stage and the removal efficiencies of COD and BOD were 87%, 90% for anaerobic stage at the 65th day of operation.

Keywords: Pharmaceutical wastewater, anaerobic treatment, packed bed reactor, COD removal.

Introduction

Wastes from industries are customarily produced asliquid wastes. (Woodard, 2006). presented a potential hazard to natural water system. Treatment of these wastes is therefore of paramount important. Wastewaters produced from pharmaceutical industries pose several problems for successful biological treatment of (LaPara, 2002). therefore an anaerobic process in many ways is ideal for waste treatment (McCarty P, 1964).

Many reactor configurations are used for the anaerobic treatment of industrial wastes and waste waters. Among them, the most common types are: completely mixed anaerobic digester, up flow anaerobic sludge blanket reactor, fluidized & expanded bed reactors & An aerobic filters of (Seghezzo, 1998). Anaerobic digestion is the decomposition of organic and inorganic matter by micro-organisms in the absence of molecular oxygen. It has been used for over a century in the treatment of domestic and industrial wastewaters as(Punal *et al.,*1999 & Fernando *et al.,* 2011). Anaerobic packed bed reactor was first proposed as a treatment process by (Young and McCarty P, 1969). The material can be arranged in various confirmations, made out of different matter (plastics, granular activated carbon (GAC), sand reticulated foam polymers, granite, quartz and stone) and can be packed in two configurations (loose or modular). The reactors can be operated in up-flow or down-flow feed of (Young & Kennedy, 1991). mode. (Ince, B K *et al.,* 2002). studied the up flow anaerobic filter of chemical synthesis based pharmaceutical wastewater.

The filter was packed with plastic pall rings have void space of 90% and specific surface area 205 m^2/m^3. They concluded that a maximum of 70% COD removal efficiency was obtained with a raw pharmaceutical wastewater at an OLR of approximately 7.5 kg $COD.m^{-3}.d^{-1}$ with HRT of 2-3 days.

Two up flow anaerobic sludge blanket (UASB) reactors with different operating temperatures, mesophilic (35±1ºC) and thermophilic (54 ± 1ºC) were used by (ISA *et al.,* 2010) to study the treatment of a non-penicillin based product factory waste water .The organic loading rate varied from 0.07to 0.45 kg $COD.m^{-3}.d^{-1}$, the highest percentage of COD removal for the mesophilic and thermophilic reactors was 95% and 93% respectively.

(Nandy and kaul, 2001). Studied the upflow anaerobic fixed film reactor for treatment of herbal-based pharmaceutical

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wastewater. The upflow reactor was fabricated from a PVC column of 0.11-m diameter and 2.25 m height having a total empty volume of 0.0124 m^3.The column base was designed to disperse the wastewater flow uniformly. The COD removal efficiencies (76-96) %were achieved for applying organic Loading rate up to 10 kg $COD.m^{-3}.d^{-1}$, while increasing organic Loading rate to 48 $kgCOD.m^{-3}.d^{-1}$ led to COD removal efficiency ranging (46-50) % 40-50% While the total COD removal efficiency of the sequential UASB+CSTR treatment system of(Sponza and Demirden, 2007). was determined as 97%.Their results indicated that the system exhibited a good removal performance for sulfamerazine.

(Chen *et al.*, 1994). studied the up flow anaerobic filter for treatment of pharmaceutical wastewater. They used a cylindrical Plexiglas pipe within internal diameter of 0.14 m. Fire expanded clay pellets were used as packing medium filled to a depth of 2 m with an effective void volume of 15 L. They concluded that when HRT decreased from 20to 2day organic loading rate from 1 to 10kg $COD.m^{-3}.d^{-1}$).Anaerobic treatment achieved 93- 70% COD removal rate. (Hamdy *et al.*, 1992). studied the mesophilic and thermophilic upflow anaerobic filter for treatment of pharmaceutical waste resulted from pharmaceutical plant in Bombay –India, (Sachs *et al.,* 1982). used six Laboratories upflow anaerobic filter for pharmaceutical wastewater treatment while (Jennett & Dennis, 1975). used four Laboratory filters fabricated of Plexiglas's column .These filters successfully treated pharmaceutical wastewater with 70 to 96.8 % COD removal efficiency ,but (Oktem *et al.,* 2007). study the performance of a lab-scale hybrid up-flow anaerobic sludge blanket (UASB) reactor, treating a chemical synthesis-based pharmaceutical waste water, was evaluated under different operating conditions. The hybrid UASB reactor was found to be far more effective at an OLR of 8kg $COD.m^{-3}.d^{-1}$ with a COD removal efficiency of 72%. As (Morse *et al.,* 2002 and Abbas, 2005). studied the Anaerobic/ Aerobic sequence for treatment of pharmaceutical waste water. Their investigation was amoxicillin that is an antibiotic used by (NASA). The biological components of water recovery system (WRS) were an anaerobic packed-bed reactor and aerobic tubular reactor.

The anaerobic packed-bed reactor reduced total organic carbon (TOC) concentration and denitrifies the wastewater by covering nitrate and or nitrite to nitrogen gas, (Altaf and Ali, 2010). designed a sequential batch reactor after a series of experiments. The effluent met the Pakistan National Environmental Quality Standards specifications after 21days of treatment in the SBR. The changes in pH, BOD, COD,TDS,TSS,Ammonia levels, Oil and grease levels were found to be significant ($p < 0.05$).

Materials and Method

Three types of wastewater samples were taken for analysis. The first was untreated wastewater taken from equalization tank of SDF. the second was anaerobic treated taken from anaerobic filter effluent and the third was anaerobic/aerobic wastewater treated taken from acerbic reactor effluent. These tests including pH, temperature, COD, and BOD were tested according to Standard Methods for Examination of (Water and Wastewater, 1985). The discharge of untreated wastewater for the factory also was measured as shown in table (1).

Test	Min.	Max.	Average
Temp. C^o	13.2	19	16.8
pH	7.3	7.5	7.38
COD mg/l	400	1250	668
BOD mg/l	180	360	272
Q m^3/hr	14.4	33.34	18.671

Table. 1. Characteristics & discharge of untreated wastewater for Samarra Drugs Factory

Experimental Equipment

Toxic and recalcitrant wastewaters were previously believed not to be suitable for anaerobic processes, were effectively treated as described by (Chelliapan *et al.,* 2011). Since a pilot plant for the upflow anaerobic filter and aerobic reactor as sequential system was built and installed near equalization tank of the factory, as shown in figure (1).

The parts of pilot plant are explained as following:

Up flow Anaerobic Filter.

- Aerobic Stage.
- Hydraulic System
- Ground Tank of 500L volume capacity.

- Elevated Tank No.1 of 500L volume capacity.
- Elevated Tank No.2 of 250L volume capacity.

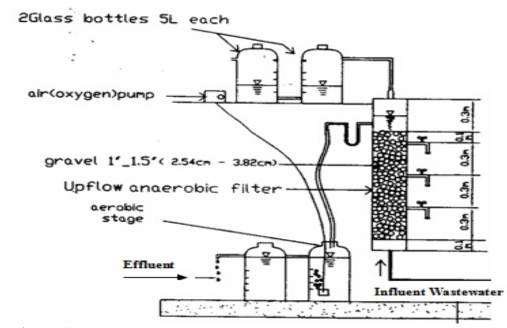

Fig. 1. SchematicDiagram of Upflow Anaerobic Filter - Aerobic Stage

The anaerobic packed bed reactor (Fig. 1) is a PVC cylindrical reactor of external and internal diameters with 0.15and 0.14m respectively was used similar to the model of Jennett and Dennis(1975).Plastic perforated plate screen was placed in the bottom of the pipe (10 cm above the base of column) for dispersion the influent wastewater uniformly through its holes upward. Three sample ports were placed at 30 cm interval throughout column height. These sample ports extended to the center of the column, so that a more representative sample of the filter contents could be obtained. The sample ports were made of 1.27cm inside diameter of P.V.C tubing were sealed in to the wall of the column by special kind of glue to give a watertight and prevent probable leakage. The column was filled with 1m height by smooth and inert gravel pass sieve opening 2.54 cm and retain sieve opening 3.82cm.The volume of packed media was 15.33 L. The gravel was well cleaned by water before placing it into the column. Porosity of this graded gravel was determined practically by using plastic cylinder volume 2L and filled with this gravel and then put quantity of water up to the Level of 2 L, the water will occupy the voids of the gravel. By measuring the water volume water graduated glass cylinder and divided this number on the 2L.It showed that the void ratio was 0.43.

The filter worked volume was 6.6 L and the specific surface area per volume ratio was 107.716m^2/m^3 these were calculated by the following equation, (Cheremisinoff *et al.,* 2000):

$$V_{worked} = V_{packed\ meadia} \times Void_{ratio} \quad ---(1)$$

$$AB = \psi A (1 - e) \quad ----(2)$$

Where: AB: the surface area presented to the fluid per unit volume of the bed when the particles are packed in a bed (m^2/ m^3)

A: the average geometric specific area of the particles (m^2/m^3) is equal to (6/d),

d: diameter of particle of packed media,

e: void ratio of packed media.

Ψ: sphericity coefficient (ψ = 1.0 for sphere particle) the upflow anaerobic filter and aeration system had operated for three runs after 34 days of seeding for reactor startup. Each run had operated for thirty days.COD & BOD were measured for upflow anaerobic filter and for aerobic stage.

The efficiency of a wastewater treatment process is defined as:

$$E = \frac{S_0 - S_1}{S_0} \times 100\% \quad ------(3)$$

In which: E: treatment efficiency %,

So & S1: influent & effluent wastewater concentration (mg/l) respectively.

Hydraulic retention time (HRT) was found to be an important key parameter which can improve the removal rate of all targeted substances Chen (1994).

$$HRT = \frac{V}{Q} \quad ------(4)$$

A linear relationship existed between COD removal efficiency and inverse of (HRT) in the void within the rock-filled reactor as shown below Young (1983).

$$E = 100 * \left(1 - \frac{\varepsilon}{HRT}\right) \quad ------(5)$$

In which: E = COD removal efficiency %,

ε=proportional coefficient=6 (ε = 4 as Young and McCarty P 1969).

The variation in ε value was belonging to temperature difference.

Experimental Work

The reactor operated as startup seeding on a substrata consisting of 1000mg/l glucose (stage1) with addition of trace nutrient (phosphates and Nitrates) and initially a feed rate of 3.3 l/d (HRT= 48 hr), which effects the COD removal (CODr). After the 16^{ths} day (stage2), the pharmaceutical wastewater was gradually replaced to ensure acclimatization that achieved in the 34th day (stage 6), table (5).

Results and Discussion

A100% pharmaceutical wastewater feeding, three runs with HRT 24, 18, 12 hr, were used to evaluate the biofilter performance, and each run tasted 30 days. The percentage of removal efficiencies for COD was calculated by using equation (3). These results are shown in the table (2) and graphically represented in figure (2) that showed the change of COD removal efficiency with time progress. It can be noted that the maximum COD removal efficiency (COD %) was 89% in the 65th day with HRT=24 hr. This may be attributed to complete of anaerobic attached biofilm.

Days	COD_{in} mg/l	*Q* l/d	COD r%	BOD r%	Days	COD_{in} mg/l	*Q* l/d	COD r%	BOD r%	Days	COD_{in} mg/l	*Q* l/d	COD r%	BOD r%
35	980	6.6	71	74	70	800	8.8	68	73	105	820	13.2	51	66
40	960	=	75	77	75	820	=	64	72	110	840	=	50	64
45	1000	=	77	79	80	840	=	61	70	115	830	=	48	63
50	880	=	82	84	85	844	=	59	70	120	880	=	44	60
55	860	=	84	87	90	740	=	55	68	125	900	=	42	61
60	750	=	86	89	95	860	=	54	68	130	1000	=	41	60
65	760	=	87	90	100	880	=	53	64	135	1100	=	39	58
Avg.	884	6.6	80	84	Avg.	826	8.8	59	69	Avg.	910	13.2	45	62

Table. 2. The Removal Efficiencies During (HRT 24, 18 & 12 h) Anaerobic Treatment

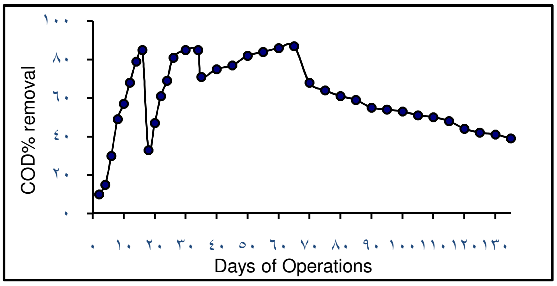

Fig. 2. Effect of HRT on COD and BOD Removal

Effect of Seeding and Startup on COD Removal

During the startup (acclimation) periods; as shown in table (3) the removal of COD decreased from 85% in the 16th day (stage 2) of operation to 33% in the 18th day (stage 3) then it increased up to 81% in the 26^{th} day (stage 6). The sudden decrease may be attributed to the existence of toxic materials and acidity during the transition stages (2, 3, 4 and 5).

Stage	Days	HRT (hr)	Substrata	COD_r %	Stage	Days	HRT (hr)	Substrata	COD_r %
1	2	48	Glucose 100%	10	**2**	16	48	20% waste 80% Glucose	85
	4	=	=	15	**3**	18	=	40% waste 60% Glucose	33
	6	=	=	30	**4**	20	=	60% waste 40% Glucose	47
	8	=	=	49	**5**	22	=	80% waste 20% Glucose	61
	10	=	=	57	**6**	24	=	100% waste	69
	12	=	=	68		26	=	100% waste	81
	14	=	=	79		30	36	100% waste	85
						34	=	100% waste	85

Table. 3. Performance of Packed Bed Reactor During Seeding and Startup

Effect of HRT on COD and BOD Removal

Different HRTs were used to determine practically the COD removal efficiency for each HRT. Table (4) showed the average COD_r% with respect to HRT and calculated the proportional coefficient (ε) according to equation 5. The average (ε) is 6.03, while (ε) in the study of (Young and McCarty P, 1969) was 4. Figure (3) showed the increase of COD removal efficiency with the increase of HRT (Barr *et al.,*1996; Chang *et al.,* 2006; Sach E F *et al*., 1987; Omer *et al*., 2008 and Selvamurugan *et al*., 2010).

No.	HRT(hr)	COD r %	ε
1	36	85	5.4
2	24	80	4.8
3	18	59	7.35
4	12	45	6.6
Average			6.03

Tables. 4. COD Removal Efficiency with Respect to HRT & Values of (ε).

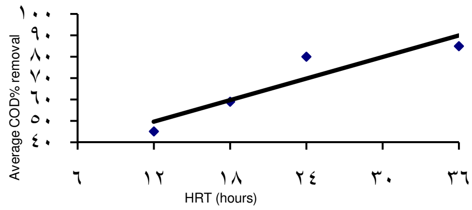

Fig. 3. The Average COD Removal with HRT during Anaerobic Treatment

Behavior of Anaerobic Filter in COD Removal with Respect to the Height.

For evaluating the height behavior of anaerobic filter in COD%, samples were withdrawn from a filter at a various heights from the ports at (30 cm, 60 cm, 90 cm, and 100 cm) from bottom to top. Tables (5) showed the COD_r and effluent of COD at different column depths, and they were represented in figures (4) to (9).All figures showed that the lower 30 cm is the most effective in COD_r% (Young *et al.*, 1989 & Weil *et al.*, 1987).

HRT & Influent COD	HRT=24 (hrs) Influent COD 760 mg/l		HRT=18 (hrs) Influent COD 800 mg/l		HRT=12 (hrs) Influent COD 820 mg/l	
Height (cm)	**COD% Removal**	**Effluent COD mg/l**	**COD% Removal**	**Effluent COD mg/l**	**COD% removal**	**Effluent COD mg/l**
30	75	190	65	280	40	492
60	86	106	66	272	42	476
90	87	96	67	264	48	430
100	68	243	68	256	51	405

Table. 5. COD Removal and COD Effluent with Respect to the Height of Anaerobic Filter

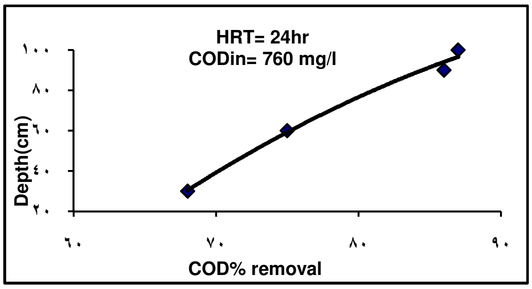

Fig. (4): COD Removal vs. the Depth of the Filter

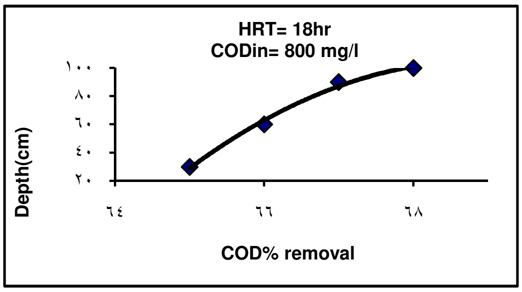

Fig. (6): COD Removal vs. the Depth of the Filter

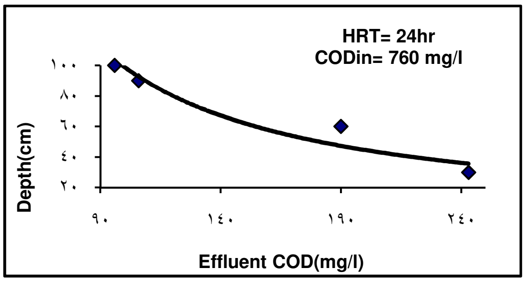

Fig. (5): Effluent COD vs. the Depth of The filter

(1st run) anaerobic treatment (1st run) anaerobic treatment

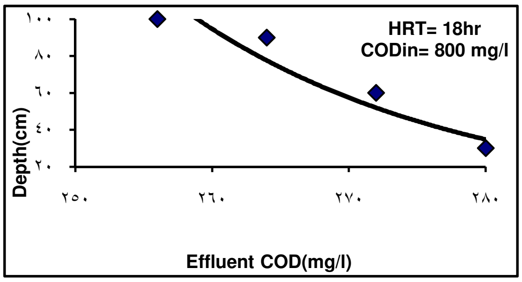

Fig. (7): Effluent COD vs. the Depth of The filter

(2nd run) anaerobic treatment (2n run) anaerobic treatment

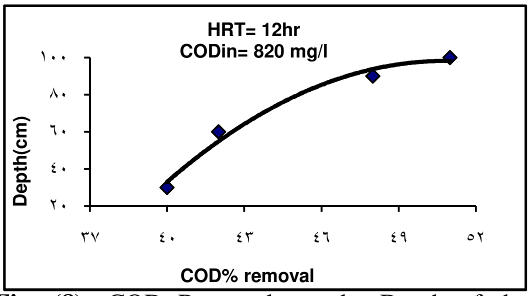

Fig. (8): COD Removal vs. the Depth of the Filter

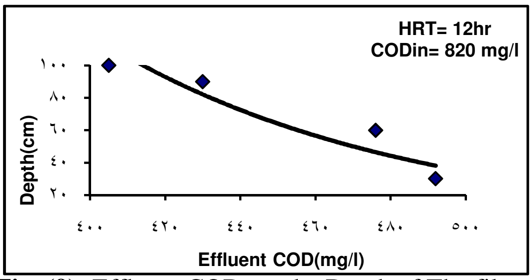

Fig. (9): Effluent COD vs. the Depth of The filter (3rd run)anaerobic treatment(3rdrun)anaerobic treatment

Design Steps for Upflow Anaerobic Filter

Two parallel circulated tank of anaerobic filter are used to improve the treatment process of SDF wastewater. Each has 6m in height and 15.4m in diameter and filled with gravel (2.54 - 3.84cm). Basic reactor design and operational data for fixed bed reactor are shown in the table (6). (Wales, 1990 & Coulson *et al.*, 1985).

Description	Unit	Data
Inert material, type	/	Gravel/ plastic
Inert material diameter	mm	20-50
Inert material, submerge	%	100
Porosity, empty bad	%	40-98
Porosity, operation	%	20-90
Specific surface	m^2/m^3	60-200
Height of reactor	m	3-6
Radius of reactor	m	5-20
Vertical velocity empty bed	m/h	0.01-0.1

Table. 6. Technical and Design Data for Fixed – Bed Anaerobic Fitter

The design steps are shown below.

$Q_{max} = Q_{peak} = 800 \frac{m^3}{day}$ Peak wastewater discharge

HRT = 24 hr (1day) (Experimental)

Volume of filter = $Q_{max} \times HRT$

∴ V = 800 m^3 (equation 4) (Copper *et al.*, 2010).

Use two filters

Each Volume = 400 m^3

Use gravel (2.54 - 3.82) cm (Experimental)

Gravel void ratio = 0.43 (Experimental)

Worked reactor volume = 400/0.43 = 930 m^3

Choose height of filter 5 m table (5)

Area = 186 m^2, A=πr^2,

Thus, r =7.7 m, d= 15.4 m

Use 0.5m space in the bottom;

And 0.5 m space in the top of circular tank anaerobic filter.

Total height of filter = 6m.

Max. COD influent= 1100 mg/l (Experimental)

Organic load $= Q \times C = 400 \frac{m^3}{day} \times 1.1 \frac{kg}{m^3} = 440 \frac{kg}{day}$

$$\text{Volumetric Organic load} = \frac{440 \frac{\text{kg}}{\text{d}}}{400\ \text{m}^3} = 1.1\ \frac{\text{kg}}{\text{m}^3.\text{day}}$$

Conclusion:

In this research Anaerobic treatment showed more improvement to COD removal reached to 87% with 24 hours anaerobic HRT treatment in the 65th day of the operation. In packed column the lower third (30cm) of upflow anaerobic filter height shows to be the most effective in COD removal and HRT is very important indicator for upflow anaerobic filter in removing COD &BOD. The removal efficiencies were 87%, 90%, achieved respectively in the 65th day of Operation with (HRT=24 hrs). It was observed that the removal efficiencies decreased with the decrease of HRT.

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المعالجة اللاهوائية لمياه الفضلات الناتجة عن الصناعات الدوائية باستخدام المفاعل اللاهوائي ذو الحشوة

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الملخص

تتميز مياه الفضلات الناتجة من الصناعات الدوائية على الأغلب بارتفاع تركيز المتطلب الكيمياوي للاوكسجين (COD) وقد يصل تركيز المتطلب الكيمياوي للاوكسجين (COD) لمياه الفضلات غير المعالجة إلى ١١٠٠ ملغم\لتر. نظرا لارتفاع تركيز المحتوى العضوي فان تكنولوجيا المعالجة اللاهوائية يعد اختيارا واعدا لمعالجة مياه الفضلات الناتجة من الصناعات الدوائية.وبناءا على ذلك فقد تم استخدام مفاعل لاهوائي ذو حشوة (نمو ملتصق) باتجاه جريان من الأسفل إلى الأعلى حيث تم تصميمه لمعالجة مياه الفضلات الناتجة من معمل أدوية سامراء الواقع في مدينة سامراء شمال العاصمة العراقية بغداد ب ١٢٠ كم حيث تم الاقتراح بإضافتها إلى وحدات معالجة مياه الفضلات الهوائية الموجودة حاليا للمعمل. تم دراسة كفاءة المفاعل اللاهوائي(المرشح اللاهوائي) في إزالة المواد العضوية (COD) نسبة إلى ارتفاع الحشوة. بينت الدراسة ان الثلث الأول للمفاعل كان أكثر كفاءة في الإزالة. تم انجاز فترة الأقلمة بمدة تشغيل مستمرة لمدة ٣٤ يوم حيث حقق المفاعل كفاءة إزالة ثابتة مقدارها ٨٥%.كما بينت الدراسة بان أفضل فترة بقاء هيدروليكي HRT التي حققت أفضل كفاءة في الإزالة للمواد العضوية في مياه الفضلات الدوائية كانت ٢٤ ساعة حيث حققت إزالة مقدارها ٨٧% للمتطلب الكيمياوي للاوكسجين (COD) و٩٠% للمتطلب البيوكيماوي للاوكسجين (BOD) في اليوم ٦٥ من بدء التشغيل المستمر.

الكلمات الدالة: مياه الفضلات الناتجة من الصناعة الدوائية، المعالجة اللاهوائية، المفاعل اللاهوائي ذو الحشوة، ازالة المتطلب الكيمياوي للاوكسجين.