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Treatment of Phenolic Industrial Wastewater Using Activated Sludge Process Preceded by Anoxic Selector

Abd El-Ghany G. Abulnour[1], Hayam F. Shaalan[1*], Taha M. Abd El-Razek[2], Ali N. Hassan[2], Sahar S. Ali[1]
[1]Chemical Engineering and Pilot Plant Department, Engineering Research Division,
National Research Centre, Dokki, Cairo, Egypt
[2]Institute of Environmental Studies and Research, Ain Shams University, Egypt

ABSTRACT

Activated sludge process (ASP) has been widely used for the treatment of high organic load industrial wastewater. Toxic compounds (e.g phenol at high concentration) limit the application of ASP. In the present work, ASP preceded by anoxic selector was investigated for the treatment of phenolic effluents generated from a chemical and pharmaceutical industry. The parameters affect the process (phenol concentration, food to microorganism ratio (F/M), retention time and pH) were studied using batch and completely mixed activated sludge (CMAS) type reactor (with and without anoxic selector). The results showed that the maximum removal efficiency obtained by using batch process was 99.9% at 800 mg/l initial phenol concentration, F/M ratio of 0.579 (g COD/g MLVSS.d) and pH around 7. For CMAS only, the maximum applied phenol concentration was 2000 mg/l to yield effluent phenol concentration of 35 mg/l. For anoxic selector-CMAS, the maximum applied phenol concentration was 5500 mg/l to yield effluent phenol concentration of 0.01 mg/l. It was found that the performance of industrial wastewater treatment was significantly enhanced using anoxic selector. It is concluded that the use of properly designed anoxic selector prior to CMAS significantly improves the performance of the process and can tolerate much higher phenol concentration than that tolerated by the conventional CMAS. The application of the proposed system contributes to the establishment of technically viable solutions for water bodies pollution by phenolic wastewater.

Keywords: Activated sludge, selector, phenol, industrial wastewater.

INTRODUCTION

Phenols and phenolic compounds are found in effluents from industrial operations such as petrochemical, pharmaceutical, plastic and pesticide chemical industries. Many phenolic compounds are considered to be hazardous pollutants (Proudfoot, 2003; Rodr´ıguez *et al.*, 2006; Papadimitrioua *et al.*, 2007). The presence of phenols inhibits or reduces microorganisms activity in biological wastewater treatment plants and strongly reduces the biological biodegradation of phenol and other components (Gernjak *et al.*, 2003). Therefore, the development of an effective technology for the removal of phenolic compounds from wastewater is important. Biological treatment is a practical and not very expensive solution to treat this kind of effluents, compared to chemical methods. Activated sludge is considered one of the most effective and economic treatment methods for industrial and domestic wastewater because the presence of various populations of microorganisms in the activated sludge are able to degrade wide range of organic compounds (Aleksieva *et al.*, 2002; Chung *et al.*, 2003; Kumar *et al.*, 2005; Marrot *et al.*, 2006). The limits and capacity of the biological process are related to the acclimatization of the biomass to degrade phenol according to the variability of the wastewater compositions. During this acclimatization, there is a selection and a multiplication of specialized microorganisms (Marrot *et al.*, 2006). The acclimatized microorganisms to phenol increases phenol biodegradability since acclimatization process makes phenoletic bacteria predominant (Ralph and James, 1998). Numerous investigations have been carried out by unconventional methods on the basis of potential microbial abilities to degrade phenols using bacterial strains, some yeast strains and immobilization of microorganisms (Watanable and Teramoto, 1998; Loh and Chung, 2000; Pazarlioglu and Telefoncu, 2005, Yan *et al.*, 2005).

In order to avoid the occurrence of filamentous bulking, a selector can be located upstream of the aeration tank and provide a high localized F/M ratio by maintaining relatively short hydraulic retention time. In the selector, high substrate concentrations are achieved which favor the growth of floc-forming bacteria or create a substrate concentration gradient in the aerator (Martins *et al.*, 2004). The objective of this study is to treat phenolic effluents generated from a chemical and pharmaceutical industry using activated sludge process preceded by a selector technique using a pre-acclimatized sludge taken from a domestic sewage secondary treatment plant.

MATERIALS AND METHODS

1. Materials

Phenol was obtained from Merck, Schuchardt (min. assay 99.5%), nutrients (ammonium nitrate, di-amonium hydrogen phosphate); trace elements (manganese sulphate mono hydrate, magnesium sulphate hepta hydrate, zinc sulphate hepta hydrate, calcium chloride dehydrate, ferric chloride) and COD analysis chemicals

* *Corresponding author: hayam64@hotmail.com*

were all of laboratory grade. Industrial wastewater samples were collected from effluents of chemical synthesis of salicylate compounds from El-Nasr Company for pharmaceutical industry, Abu-Zabal, Cario, Egypt. Sludge was obtained from domestic sewage secondary treatment plant at Zenin-Giza which is pre-acclimatized for a period of 10 weeks using synthetic phenol solutions as a sole carbon source (Abulnour *et al.*, 2006).

2. Methods

(A) Sludge acclimatization

Sludge sample, taken from a secondary sedimentation tank from a large sewage treatment plant at Zenin, Giza-Egypt, was used for sludge acclimatization in a separate vessel (20 L capacity) provided with air diffusers connected to compressed air line. The sludge was aerated for 48 hours. Gradual doses of phenol were added to the acclimatization tank according to procedures described by Ali (2007). After reaching almost nil phenol concentration, a new addition of phenol with higher concentration was performed. The initial phenol concentration at each addition during acclimatization increased from 10 mg/l to 850 mg/l at the end of the investigated acclimatization period (70 days). During the acclimatization period, aeration was conducted for 22 hours/day followed by settling for 2 hours after which, samples from the supernatant were taken for COD and phenol analysis and sludge samples for MLSS determination on daily basis. The sludge containing mixed culture of different bacterial species common in domestic wastewaters was acclimatized on phenol a sole carbon source. The acclimatization aimed at prevailing bacterial strains capable of biodegradation of phenol.

(B) Batch activated sludge

Batch activated sludge system has been developed and tested for the removal of phenol from industrial wastewater using the pre-acclimatized sludge using 5 liters reactor with ceramic air diffuser connected to compressed air line to keep dissolved oxygen concentration of 2-2.5 mg/l**.** Based on the analysis of phenolic industrial wastewater, the desired concentration is maintained by diluting the industrial wastewater with distilled water. Also, COD: N: P ratio was adjusted to 150:5:1 (using doses of ammonium nitrate and di-ammonium hydrogen phosphate calculated based on COD) at pH range from 6.8 to 7.2**.** The treatment procedure included the utilization of the acclimatized sludge for the treatment of phenolic industrial wastewater at ambient temperature. The effect of F/M ratio**,** pH and retention time (RT) have been studied**.** Samples were taken from the supernatant before and after the treatment from 2 to 12 hours at 2 hours interval to measure phenol concentration and COD determination. Samples were taken from mixed liquor to measure MLSS, MLVSS and SVI. The experimental conditions may be summarized as follows:

- Reactor volume	5 liters
- Phenol concentration	200-900 mg/l
- DO	2-2.5 mg/l
- F/M ratio	0.1-0.7 d^{-1}
- RT	2-12 hours
- pH	6-10

(C) CMAS with and without selector

The treatment of the industrial wastewater containing phenol using continuous system was conducted with and without selector**.** The experimental set-up is schematically presented in Figure (1). The main components of the system may be outlined as follows:

- Feed preparation tank (20 liters capacity) equipped with pH meter and transfer pump.
- Constant head feeding system (20 liters capacity) overhead tank connected to a constant head feeding tank provided with float valve to keep water liquid at constant level in the feed tank.
- Selector of up to 2 liters capacity equipped with a mechanical stirrer.
- Aeration tank of 20 liters capacity equipped with ceramic air diffuser connected to a compressed air line.
- Settling tank of 4 liters capacity equipped with a glass air lift pump to transfer sludge to the selector or aeration tank. The settling tank is provided with a valve to waste the excess sludge. The treated effluent was separated from the sludge by settling. Samples were taken from mixed liquor to measure MLSS, MLVSS and SVI and from the treated effluent for phenol and COD determination. The experimental conditions are summarized in Table (1).

Table (1): The experimental conditions for CMAS with and without selector.

Experimental system / Parameter	CMAS without selector	CMAS with selector
Feed flow rate	1 l/h	1 l/h
Sludge return flow rate	1 l/h	1 l/h
DO		
- selector	-	0.5 mg/l
- aeration tank	2.5-3 mg/l	2 mg/l
- F/M:ratio		
- selector	-	0.7-1.9 h^{-1}
- aeration tank	0.9-1.8 d^{-1}	0.2-0.8 d^{-1}
HRT:		
- selector	-	2 hs
- aeration	20 hs	20 hs
Feed phenol concentration	1000-2000 mg/l	2000-5500 mg/l

3. Analytical methods

Phenol concentration was determined by a colorimetric method based on 4-aminoantipyrine at a wavelength of 470 nm using spectrophotometer (HACH DR-2000). After preparing a calibration curve for standard solutions for phenol.

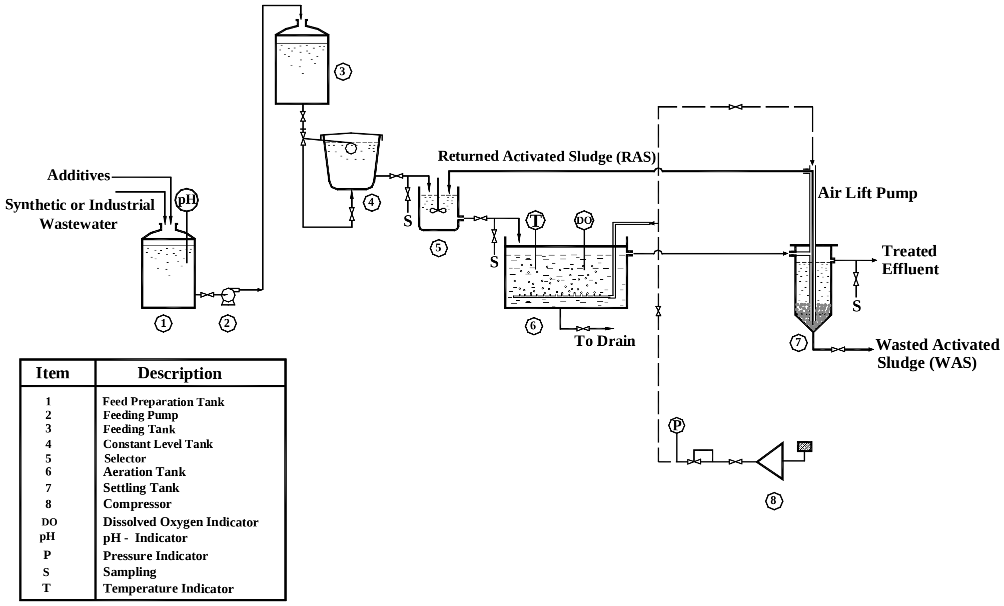

Figure (1): Schematic diagram of the experimental set-up.

Other parameters including: COD, MLSS, MLVSS and SVI were measured according to the Standard methods **(**APHA-AWWA-WPCF, 1995).

The morphology of the acclimatized sludge were identified using Transmission Electron Microscope examination at the National Research Center- Egypt by taking a sample of the mixed liquor from the acclimatized sludge to p-NP while, bacterial identification was conducted by Automated Identification System, at Agricultural Research Center, Plant Pathology Research Institute - Microorganisms Identification Unit, to identify the dominant strain in the acclimatized sludge.

RESULTS

1. Sludge acclimatization

Based on sludge pre-acclimatization using synthetic phenol solutions for 10 weeks (Abulnour *et al.*, 2006), Figure (2) shows the change of initial and final phenol concentration during the acclimatization period of activated sludge which is divided into three zones: (a) non-inhibitory zone with phenol concentration up to 400 mg/l, (b) inhibitory zone with phenol concentration ranged from 400 to 600 mg/l, and (c) acclimatized zone with concentration ranged from 600 mg/l till 800 mg/l. Figure (3) depicts the change of phenol degradation rate during the acclimatization period involving three kinetic stages, steadily increase of degradation rate for the non-inhibitory zone followed by an inhibitory zone with fluctuating degradation rate and finally a steady increase of degradation rate to a concentration of 800 mg/l. After that, a new inhibitory zone is observed.

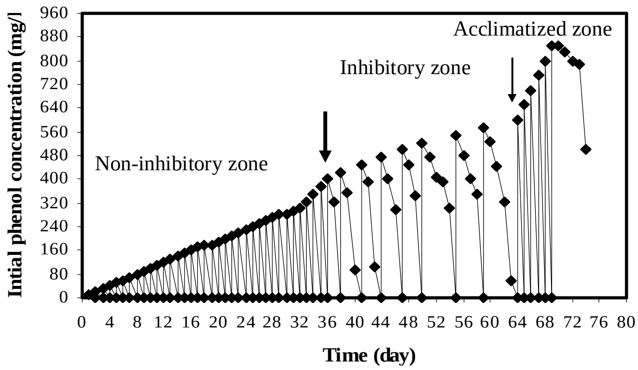

Figure (2): The change of initial and final phenol concentration during the acclimatization period of activated sludge.

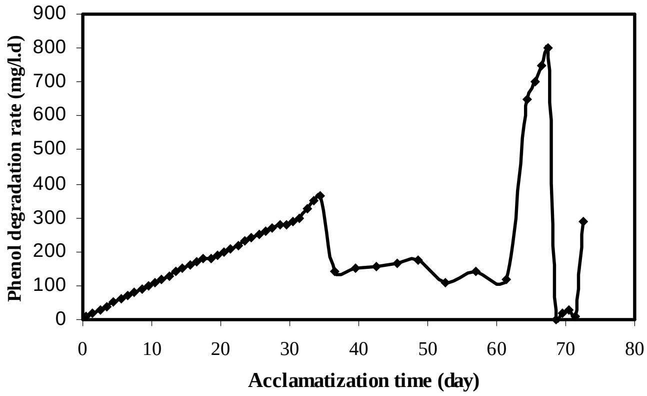

Figure (3): Change of phenol degradation rate during the acclamatization period.

2. Industrial wastewater characteristics

The physical and chemical characteristics of the studied industrial wastewater are presented in Table (2). The high phenol concentration of 5500 mg/l and high

Table (2): Characteristics of industrial wastewater

Parameter	Value
pH	1.5
Conductivity	6 mS/cm
Turbidity	420 FTU
TSS	400 mg/l
TDS	16000 mg/l
Phenol concentration	5500 mg/l
COD (dichromate)	15500 mg/l
TOC	7040 mg/l
TKN	25 mg/l

organic load are characterizing the nature of effluent from the investigated salicylate synthesis unit.

3. Batch activated sludge treatment

(A) Effect of RT on phenol degradation

The effect of RT on effluent phenol concentration is shown in Figure (4). At initial phenol concentration (Ci) of 800 mg/l and F/M ratio of 0.579 d^{-1}. The batch system results showed an increase in phenol degradation with time. Phenol was almost completely degraded within 10 hours.

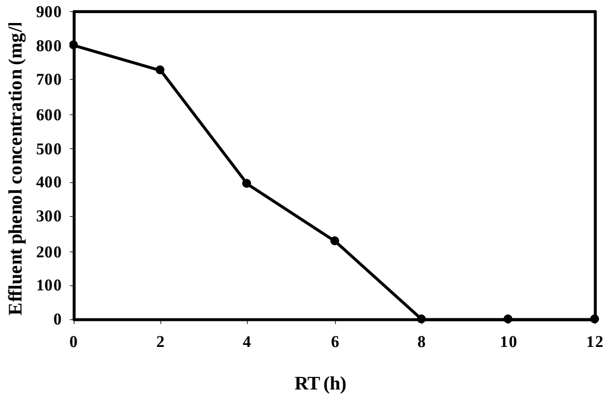

Figure (4): Effect of RT on effluent phenol concentration for phenolic industrial wastewater using batch activated sludge. (Ci = 800 mg/1, DO = 2-2.5mg/l, F/M ratio = 0.579 d^{-1}, pH = 7).

(B) Effect of initial phenol concentration

The relationship between initial phenol concentration and phenol removal efficiency is depicted in Figure (5). It is noted that, the phenol removal efficiencies are ranging from 97% to 99.9 % at phenol concentrations from 200 to 800 mg/l. Further increase in phenol concentration up to 900 mg/l, reduces the removal efficiency to 55**%**.

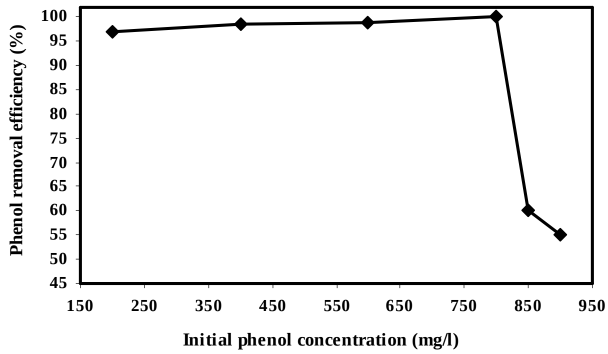

Figure (5): Effect of initial phenol concentration on phenol removal efficiency for phenolic industrial wastewater using batch activated sludge. (F/M ratio = 0.579 d^{-1}, pH = 7, DO = 2-2.5 mg/l, RT = 10 hs).

(C) Effect of F/M ratio

The phenol removal efficiency dependence on F/M ratio was shown in Figure (6). Starting with F/M = 0.1 d^{-1}, the phenol removal efficiency reached its maximum value (99.9%) at F/M = 0.579 d^{-1} then, it was sharply decreased for higher F/M ratio and reached 58% at F/M = 0.7 d^{-1}.

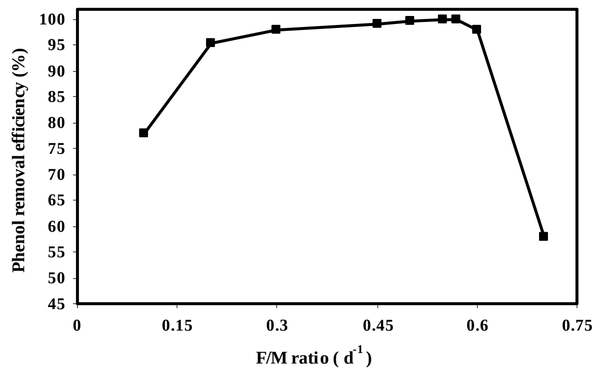

Figure (6): Effect of F/M ratio on phenol removal efficiency for phenolic industrial wastewater using batch activated sludge. (Ci= 800mg/l, pH= 7, DO= 2-2.5mg/l, RT= 10 hs).

(D) Effect of pH

The maximum phenol removal efficiency of more than 99% was achieved for the industrial wastewater at pH ranging from 6 to 7, and more likely at 6.8 which is confirmed by the previous results reported for treatment of synthetic phenolic solutions (Ellis *et al.*, 1998).

4. CMAS system without selector

For CMAS system without selector, the optimum F/M ratio obtained was 1.2 d^{-1} at phenol concentration 1200 mg/l and HRT = 20 hours as shown in Figure (7). The effect of changing initial phenol concentrations from 1000 to 2000 mg/l on effluent phenol concentration is shown in Figure (8). The minimum effluent phenol concentration of about 5 mg/l was obtained at phenol feed concentration of 1200 mg/l and the maximum effluent phenol concentration was 35 mg/l at initial phenol concentration of 2000 mg/l.

5. CMAS system with selector

The results of applying higher feed phenol concentrations with CMAS preceded by anoxic selector with selector retention time (SRT) of 2 hours and DO = 0.5 mg/l revealed that, for high phenol concentrations (3500 and 5500 mg/l), the maximum attained phenol removal efficiencies were about 94% and 93..3% at F/M ratios of 1.2 and 1.3 h^{-1}, respectively, as shown in Figure (9).

The effluent phenol concentration after the whole treatment of phenol feed concentration of 5500 mg/l and HRT= 20 hours at pH= 6.8 was 0.01mg/l. SVI measurements of mixed liquor in the aeration tank, showed that by increasing initial phenol concentrations from 2000 to 5500 mg/l, the SVI increased from 50 to 87 ml/g (Fig. 10). These results reflected the positive effect of applying anoxic selector on the settle ability of sludge at high phenol load (5500 mg/l).

DISCUSSION

Based on sludge pre-acclimatization using synthetic phenol solutions for 10 weeks, the maximum attainable readily degradable phenol concentration under the investigated conditions is about 800 mg/l. The Automatic Identification System examinations showed that the prevailing bacterial strain in the acclimatized sludge is *Corynebacterium nitrilophlious sp*. This strain has been reported among bacterial strains utilizing phenol as a sole carbon source (Vesel? *et al.*, 2007).

In batch activated sludge treatment, the RT (10 hours) is lower than that reported by other studies in which RT reached 25 hours for complete phenol degradation starting with 500mg/l using a pure culture of immobilized cells in a batch fluidized bed bioreactor (Gonzalez *et al.*, 2001). Also, maximum removal efficiencies (ranging from 97% to 99.9%) at phenol concentrations from 200 to 800 mg/l in this study are better than that obtained by Hellal *et al.* (2006) who concluded that phenol was degraded to undetected limit at initial phenol concentration below 400 mg/l and significant inhibition occurred above 500 mg/l. Also, Chung *et al.* (2003) reported that phenol could be degraded only up to 600 mg/l. The optimum attained F/M ratio of 0.579 d^{-1} is in good agreement with that reported by Ellis *et al.* (1998) where the recommended F/M is higher than 0.5 d^{-1}.

The performance of phenolic industrial wastewater treatment using CMAS with anoxic selector is enhanced by increasing the ability of the pre-acclimatized activated sludge to degrade phenol concentration from 5500 mg/l (feed) down to 0.01 mg/l (treated). While, in case of using CMAS without selector, the maximum treated phenol feed concentration was 2000 mg/l giving an effluent phenol concentration of 35 mg/l. The values of SVI were not significantly affected by the process type. The application of the anoxic selector could improve the applicability of activated sludge for the treatment of industrial wastewater with high phenol content. These results could be attributed to the bio sorption and transformation of phenol to intermediate biodegradation products by the specialized enzymatic activities responsible for phenol degradation (Wanner and Grau, 1989, Argaman *et al.*, 2000, Izzo *et al.*, 2005). A properly designed anoxic-CMAS could contribute to endeavors targeting abatement of water bodies' pollution by industrial phenolic effluents.

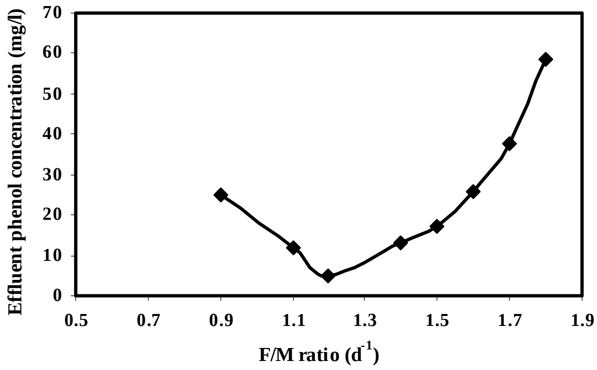

Figure (7): Effect of F/M ratio on treated industrial effluent phenol concentration using CMAS system without selector. (Ci = 1200mg/l, HRT = 20hs, DO = 2.5-3mg/l).

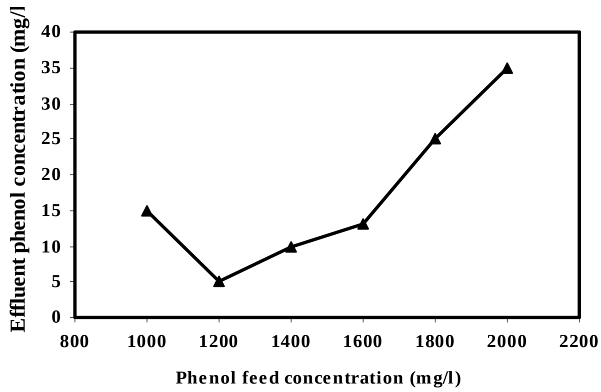

Figure (8): Effect of feed phenol concentration on effluent phenol concentration using CMAS without selector. (DO = 2.5-3mg/l, F/M = 1.2 d^{-1}, HRT = 20hs).

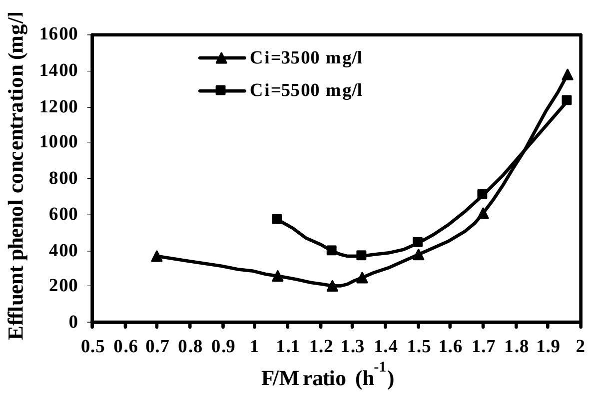

Figure (9): Effect of F/M ratio on treated industrial effluent phenol concentration using CMAS with selector. (SRT = 2hs, DO = 0.5mg/l).

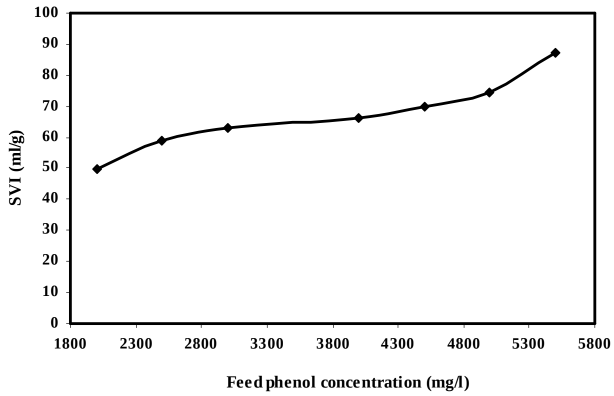

Figure (10): Effect of phenol feed concentration in industrial wastewater on SVI using CMAS with selector. (SRT = 2hs, HRT = 20hs).

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معالجة سوائل الصرف الصناعى الفينولية بإستخدام طريقة الحمأة المنشطة المسبوقة بوحدة انتقاء محدود الأكسجين

عبد الغنى جمال ابو النور[1]، هيام فهيم شعلان[1]، على ناصر حسن[2]، طه عبد الرازق[2]، سحر سعد على[1]

[1]قسم البحوث الهندسية، المركز القومى للبحوث، الدقى، القاهرة، مصر

[2]معهد الدراسات والبحوث البيئية ، جامعة عين شمس، القاهرة، مصر.

الملخص العربـــى

تستخدم طريقة الحمأة المنشطة على نطاق واسع فى معالجة الأحمال العضوية الكبيرة فى سوائل الصرف الصناعى. و تحدد المركبات الخطرة (مثل الفينول بتركيزات مرتفعة) تطبيق طريقة الحمأة المنشطة. وفى هذا البحث تم دراسة إستخدام الحمأة المنشطة مسبوقة بوحدة إنتقاء محدود الأكسجين لمعالجة مخلفات فينولية ناتجة عن صناعة كيميائية دوائية. وقد تم دراسة العوامل المؤثرة (تركيز الفينول ونسبة الغذاء/الكتلة الحية وزمن الإحتجاز والأس الهيدروجينى) على عملية المعالجة بإستخدام مفاعل ذو دفعات وبإستخدام الطريقة المستمرة للحمأة المنشطة باستخدام مفاعل كامل التقليب (بإستخدام وبدون إستخدام وحدة الإنتقاء).

وقد أوضحت النتائج أن أقصى كفاءة إزالة للفينول بلغت 99.9% لتركيز فينول إبتدائى 800 مجم/لتر ونسبة الغذاء/الكتلة الحية تساوى 0.579 جم (COD)/جم (MLVSS). يوم فى المفاعل ذو الدفعات. أما بالنسبة لنظام المعالجة المستمرة بالحمأة المنشطة فى المفاعل الكامل التقليب بدون إستخدام وحدة الإنتقاء فإن أقصى تركيز للفينول أمكن إستخدامه هو 2000 مجم/لتر ويصل تركيز الفينول فى السوائل الناتجة عن المعالجة 35 مجم/لتر. وفى حالة إستخدام وحدة الإنتقاء ذو الأكسجين المحدود مع مفاعل الحمأة المنشطة الكامل التقليب فقد أمكن التوصل إلى إستخدام تركيز للفينول حتى 5500 مجم/لتر ووصل تركيز الفينول فى السوائل المعالجة 0.01 مجم/لتر. ويوضح ذلك أن إستخدام وحدة الإنتقاء ذات الأكسجين المحدود يؤدى إلى تحسين كفاءة معالجة الصرف الصناعى بدرجة كبيرة.

ويُستنتج من البحث أن إستخدام وحدة إنتقاء ذات اكسجين محدود ذو تصميم مناسب قبل المعالجة بطريقة الحمأة المنشطة ذات التقليب الكامل يؤدى إلى تحسين كبير فى اداء المعالجة والتمكن من معالجة تركيزات أعلى من الفينول عن تلك التى يمكن أن تعالجها الطريقة التقليدية، ويسهم النظام المقترح فى إستتباب حلول فنية قابلة للتطبيق لمعالجة تلوث المسطحات المائية بالمخلفات الفينولية السائلة.