ARCHIVES OF ENVIRONMENTAL PROTECTION
vol. 39 no. 2 pp. 69 - 80 2013

VERSITA
PL ISSN 2083-4772
DOI: 10.2478/v10265-012-0028-2

EFFECT OF HYDRAULIC RETENTION TIME AND FILLING TIME ON SIMULTANEOUS BIODEGRADATION OF PHENOL, RESORCINOL AND CATECHOL IN A SEQUENCING BATCH REACTOR

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Keywords: Phenol; resorcinol; catechol; BR; hydraulic retention time; biodegradation.

Abstract: In the present study, treatment of synthetic wastewater containing phenol, resorcinol and catechol was studied in a sequencing batch reactor (SBR). Parameters such as hydraulic retention time (HRT) and filling time have been optimized to increase the phenol, resorcinol, catechol and chemical oxygen demand (COD) removal efficiencies. More than 99% phenol, 95% resorcinol and 96% catechol and 89% COD removal efficiency was obtained at optimum conditions of HRT = 1.25 d and fill time = 1.5 h. The heating value of the sludge was found to be 12 MJ/kg. The sludge can be combusted to recover its energy value.

INTRODUCTION

Phenol, a derivative of benzene, is an important product and raw material for manufacture of a variety of products. Phenol and its derivatives are used in the wide range of industries like pesticides, wood preservatives and as an intermediate in the pulp and paper [1]. Phenolic compounds are produced in industrial processes such as gasification, coking, oil refining, phenolic resin manufacturing and paint stripping [2]. The Ministry of Environment and Forests (MOEF), Government of India has listed phenol and other phenolic compounds on the priority-pollutants list. The MOEF has set a maximum concentration level of 1.0 mg/L of phenol in the industrial effluents for safe discharge into surface waters [3]. Several processes such as adsorption, chemical oxidation, photochemical oxidation and biological treatment have been used for the removal of phenolic compounds from wastewaters [4–13]. Biological treatment of phenol is generally favoured over other processes in terms of lower cost as well as the possibility of complete mineralization. The conventional treatment technology employs activated sludge process (ASP) as a secondary treatment facility. ASP is prone to bulking of sludge, and is very sensitive towards variation in chemical oxygen demand (COD) and biological oxygen demand (BOD) loading. It also requires large area for its installation.

Sequencing batch reactor (SBR) is one of the best available techniques for the biological treatment of industrial wastewaters. It can be applied to treat wastewaters having high concentration of COD, BOD, phenolic compounds and other hazardous pollutants [12, 13]. SBR process is a sequential suspended growth (activated sludge (AS)) process in which all major steps of conventional ASP occur in the same tank in sequential order. It is characterized by a series of phases namely fill, aeration, settle, draw and idle phase [14].

Various studies have been reported for treatment of phenolic wastewater in SBR (Table 1) [15−27]. Sarfaraz *et al*. [16] conducted studies on the anoxic phenol removal using granular denitrifying sludge in SBR at different cycle lengths and influent phenol concentrations. Results showed that removal exceeded 80% for an influent phenol concentration of 1050 mg/L at 6 h cycle length. Beyond this, there was a steep decrease in phenol and COD removal efficiencies. Chan and Lim [18] evaluated the performance of the SBR treating synthetic phenolic wastewater at influent phenol concentrations in the range of 100–1000 mg/L. Tomei *et al.* [19] studied the biodegradation kinetics of 4-nitrophenol (4NP) in a lab-scale sequencing batch reactor fed with the 4-NP as the sole carbon source. The experimental results showed that both long feed phase and high biomass concentration are effective in reducing the substrate concentration peak and then improving the process efficiency. Yu and Gu [28] evaluated the performance of the two SBRs with aerated fill and unaerated fill treating synthetic phenolic wastewater. Sahinkaya and Dilek [29] investigated the biodegradation kinetics of 4-chlorophenol (4-CP) and 2,4-dichlorophenol (2,4-DCP) separately in batch reactors and mixed in SBRs.

Most of the studies reported in the literature are done on single phenolic compounds (Table 1). Disposal of sludge generated in SBR is also important; however previous investigators have scarcely dealt with this aspect of wastewater treatment. The aim of the present work is to explore the possibility of using SBR for the treatment of synthetic phenolic wastewater containing three phenolic compounds namely phenol, resorcinol and catechol. In this study, the effect of hydraulic retention time (HRT) and fill time have been investigated on phenol, resorcinol, catechol and COD. Excess sludge wasted during the idle phase has been characterized for its proximate, elemental and thermal analysis; and heating value to evaluate its disposal option along with energy recovery.

MATERIALS AND METHODS

Wastewater

All the chemicals used in the study were of analytical reagent grade. The synthetic wastewater used in the present study was made by dissolving the following compounds (concentration in mg/L) in distilled water: phenol (200), catechol (200), resorcinol (200), glucose (150), $Na_2HPO_4 \cdot 12H_2O$ (100), NH_4Cl (150). Synthetic wastewater thus prepared had initial COD of 700 ± 20 mg/L.

Seed Sludge

Activated sludge was collected from sewage treatment plant located in Rishikesh, Uttaranchal, India. The sludge was first screened for the removal of coarse and bigger particles and then it was aerated for 1−2 d [30]. It was transferred into the reactor and aerated again for 3−5 d for acclimatization of the sludge with the wastewater.

Table 1. Studies on the treatment of phenolic compounds in sequential batch reactor

Wastewater	**Reactor**	**Dimension**	**Initial Conc. (mg/L)**	**SRT (d)**	**HRT (h)**	**Cycle Time (h)**	**% Pollutant Removal**	**% COD Removal**	**Reference**
Phenol	SBR	Vol: 5 L	< 400	10	-	-	-	95	[15]
Phenolic	SBR	Vol: 1.65 L; I.D.×H: 6.5×60 cm	1050	-.	-	6-12	80	80	[16]
2,4-di chloro phenol	SBR	Vol: 4 L; I.D.×H: 8×100 cm	50-100	-	8	-	94	95	[17]
Phenol	SBR	Vol: 5 L; L×W×H: 20×15×25 cm	10-100	-	-	12.	99	-	[18]
4-Nitrophenol	SBR	Vol: 5 L	320-400	16	16	8	98		[19]
Polycyclic aromatic hydrocarbons	SBR	Vol: 5 L	70	-	44-77 day	168	80		[20]
4-Chloro phenol	SBR	Vol: 2.5 L	Phenol: 525 4-CP: 105 to 2100	-		12	Phenol-41		[21]
Phenol and cyanides	SBR and CSTR	Vol: 5 L	Phenol: 1400 Cyanide: 100	26	30	12	93	90	[22]
Saline	GSBR	Vol: 4 L I.D.×H: 15×40 cm	Phenol: 100-2000	-	-	17	93-99	92-99	[23]
Phenolic	SBR	Vol: 5 L	4NP: 40-60	16	16	8			[24]
Phenol and m-Cresol	SBR	Vol: 1.4 L; I.D.×H: 5×100 cm	Phenol: 200-1000	-	12	6	90-95		[25]
Phenolic	SBR	Vol: 5 L	Phenol: 3.12	4	10	4		97	[26]
Phenolic	ASP	Vol: 11 L	Phenol: 300	-	72	-	99.3	98	[27]

GSBR: Granular SBR; Vol.: Volume; I.D.×H: Internal diameter × Height; H: Height; L×W×H: Length × Width × Height.

Experimental Set-Up

The reactor was composed of Plexi glass, with the dimension of 7.37 × 40.64 cm (radius × height) and having 5 litre working volume. Aeration in the SBR was achieved by an aquarium-type air pump with sintered-sand diffusers at the bottom of the reactor. Dissolved oxygen (DO) concentration inside the reactor was maintained between 2–4 mg/L during the experiments by controlling the flow of air through air rota meter. The addition of feed and the wasting of sludge were done with peristaltic pumps. Mixed liquor suspended solid (MLSS) concentration was controlled between 1200 and 2200 mg/L with a sludge age of approximately 20 d. Excess sludge, which grew during the aeration stage, was drawn out at the end of every operating cycle, in order to maintain proper MLSS concentration. The reactors were operated in an isothermal chamber with temperature at 30±1°C.

Experimental Procedure

The reactor was operated on a fill-and-draw basis at instantaneous fill mode, with a total cycle time of 12 h which consisted of 10 h react phase, 1 h settling phase, 0.5 h decant phase and 0.5 h idle phase. The medium was aerated and agitated (300 rpm) vigorously during the oxic (aerobic) operation. Agitation provided optimal mixing and improved the overall organic matter consumption rates [31]. Samples were withdrawn from the reactor at the beginning and at the end of each phase for analysis. Samples were filtered with filter paper (Whatman filter paper no. 42 of pore size ca. 2.5 μm) and then examined for concentration. At the end of react phase, the biomass was settled for 1 h and the amount of the treated wastewater was removed. Settled organisms were used for the next treatment operation. Sludge wasting was done from the reactor daily before settling to adjust the sludge age.

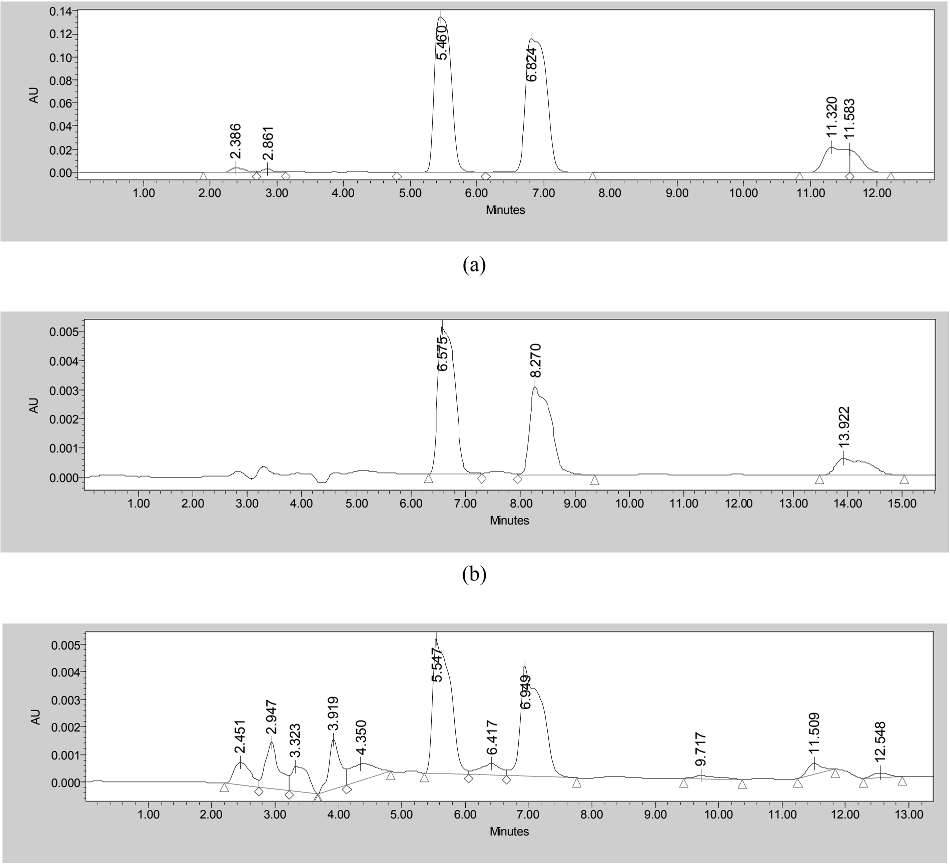
Analytical Measurements

The synthetic substrate was prepared fresh each day at a volume of 3 litres with distilled water and the substrate sample was taken initially and after the completion of the 12 h cycle. The samples obtained during the experimental run were filtered and analysed for the phenol catechol and resorcinol concentrations. COD, MLSS and mixed liquor volatile suspended solid (MLVSS) were also determined in accordance to standard methods [32].

The concentration of resorcinol, catechol and phenol in the aqueous solution were determined at 260 nm wavelength using high performance liquid chromatograph (HPLC) supplied by Waters (India) Pvt. Ltd., Bangalore (Fig. 1). A mixture of 40% methanol, 1% acetic acid and rest millipore water obtained from Milli-Q purification system was used as a solvent in a symmetry C18 column having size of 4.6 mm × 150 mm. Flow rate of solvent in the column was 0.5 ml/min.

The proximate analysis of the wasted AS was done as per Indian Standards (IS-1350). The heating value of the sludge was estimated by using a standard adiabatic bomb calorimeter equipped with a digital firing unit (Toshniwal, Bombay).

To understand the morphology of the AS before and after the treatment of refinery wastewater, and to study the distribution of the elements in AS, a scanning electron microscope (SEM) QUANTA, Model 200 FEG, USA was used. Samples were first gold coated using Sputter Coater, Edwards S150, which provided conductivity to the samples, and then the SEMs and energy dispersive X-ray (EDX) spectra of the dried AS were taken.



(a)

(b)

(c)

Fig. 1. Peaks corresponding to phenol, catechol and resorcinol present in wastewater before and after treatment in SBR, (a) before treatment, (b) after treatment, 1.25 day HRT and instanteneous fill, (c) after treatment, 1.25 day HRT and 1.5 h fill time

Thermal analysis of raw and used AS was carried out using a thermal analysis (TA) instrument (Perkin-Elmer Pyris Diamond). Thermogravimetric analysis (TGA), differential thermogravimetric (DTG) and the derivative thermal (DTA) analyses were carried out from the data and plots obtained from the instrument. This instrument operated with the following specifications: weight of the sample, 10–15 mg (max. 100 mg); temperature range, ambient to 1000°C. The thermo-analytical curves of the solid samples were obtained from this instrument under air atmospheres with a flow rate of 0.2 L/min.

Theory

A cycle in SBR comprises of fill, react, settle, decant and idle phases. The total cycle time (t_C) is the sum of all these phases.

$$t_C = t_F + t_R + t_S + t_D + t_I \tag{1}$$

Where, t_F is the fill time (h), t_R is the react time (h), t_S is the settle time (h), t_D is the decant time (h), and t_I is the idle time (h). HRT for the SBR systems is given as:

$$HRT = \frac{(t_C)}{V_F / V_T} \frac{1}{24} \tag{2}$$

Where, V_F is the filled volume of wastewater and decanted effluent for a cycle, and V_T is the total working volume of the reactor.

RESULTS AND DISCUSSION

Effect of HRT

The HRT represents the average period of time wastewater remained in the system. The effect of HRT on the biodegradation of phenol, resorcinol and catechol was studied with HRTs of 0.625, 0.714, 0.833, 1, 1.25, 1.667, 2.5 d at instantaneous fill condition. The COD and phenol, resorcinol and catechol removal efficiencies during the treatment in the SBR with varying HRT are shown in Fig. 2. Variation in biomass concentration with the HRT is shown in Fig. 3.

The results obtained from the experiment showed that an increase in HRT from 0.625 d to 1.25 d increases the COD and phenol, resorcinol and catechol removal efficiencies. This may be due to the fact that higher HRT gives a longer contact time between biomass in the reactor and the wastewater, and thus better degradation rates. It may be seen in Fig. 3 that the MLSS and MLVSS concentration is highest at HRT = 1.25 d. Thus, a higher growth rate of micro-organism during 0.625–1.25 d increases the removal efficiencies.

Further increase in HRT from 1.25 d to 2.5 d decreased COD and phenol, resorcinol and catechol removal efficiencies (Fig. 2). It may also be seen in Fig. 3 that the MLSS and MLVSS concentration decreased with an increase in HRT from 1.25 to 2.5 d. The

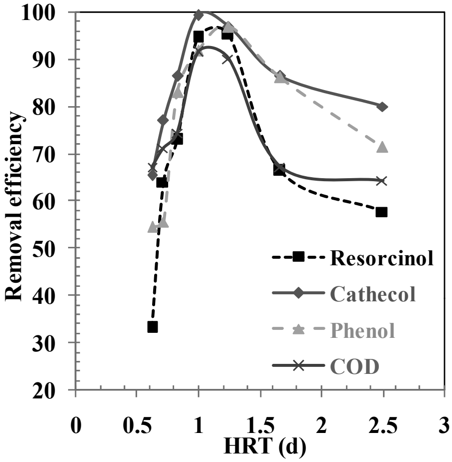


Fig. 2. Effect of hydraulic retention time (HRT) on the removal of resorcinol, catechol, phenol and COD at SRT = 20 d, instantaneous filling

decrease of COD and phenol, resorcinol and catechol removal efficiencies at HRT, thus, may be due to lower growth rate of micro-organism and accumulation of older cells.

Effect of Fill Time

In this part of the study, HRT was kept constant at 1.25 whereas fill time was varied in the range of 0.5–2 h. The removal efficiencies with varying fill time are shown in Fig. 4. Variation of biomass concentration with the fill time is depicted in Fig. 5. The results showed that an increase in fill time from 0.5 h to 1.5 h increased the MLSS concentration from 1900 mg/L to 2100 mg/L, however, a further increase in fill time to 2 h decreased the MLSS concentration to 1780 mg/L. The results obtained from the experiment showed that an increase in fill time from 1.5–2 h decreased the removal efficiency of substrates

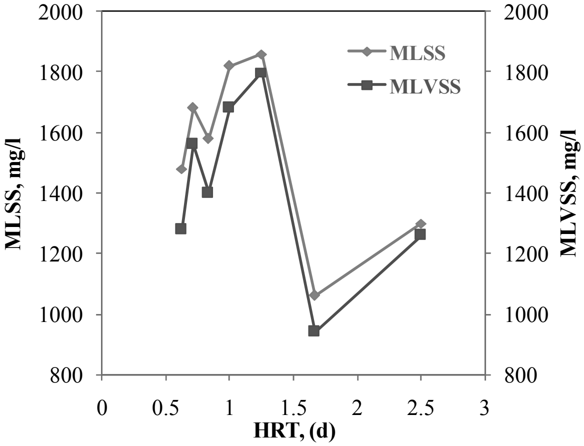


Fig. 3. Effect of hydraulic retention time (HRT) on the final MLSS and MLVSS concentration at SRT = 20 d, instantaneous filling

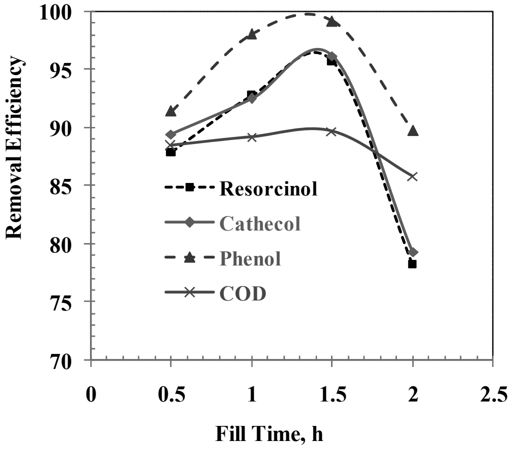


Fig. 4. Effect of fill time on the on the removal of resorcinol, catechol, phenol and COD at SRT = 20 d and HRT = 1.25 d

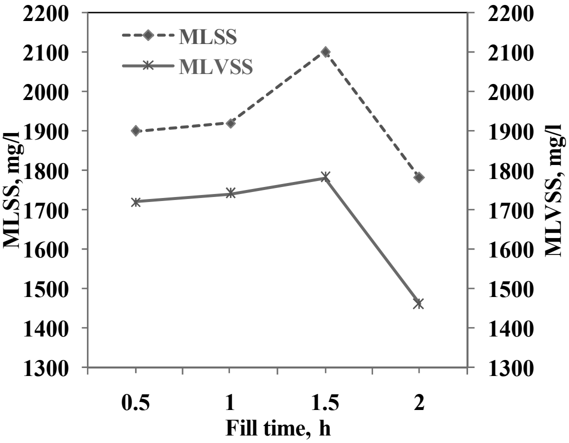


Fig. 5. Effect of fill time on the final MLSS and MLVSS concentration at SRT = 20 d and HRT = 1.25 d

(Fig. 4). This may be due to the fact that lower fill time gives short contact time between biomass in the reactor and the waste water, and thus reduced degradation rates.

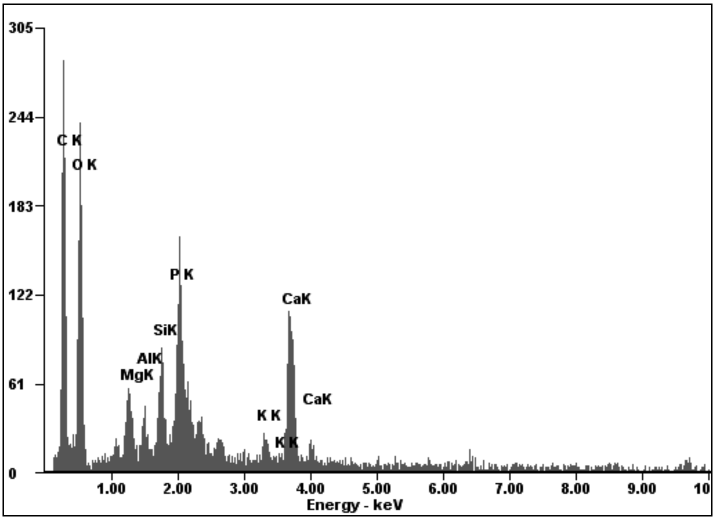
Characterization of Sludge

To maintain an appropriate level of MLSS concentration within the reactor, some amount of sludge is wasted after every cycle which prevents the problem of sludge bulking, excessive growth of filamentous bacteria and maintains the efficiency of SBR. The amount of sludge wasted depends on hydraulic retention time and solid retention time.

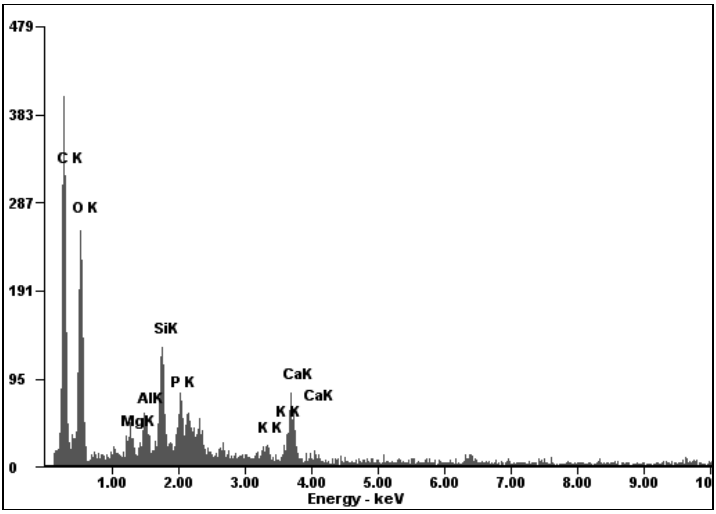
Sludge obtained at the bottom of settling tank or that obtained at the top of the filter media can further be utilized as a fuel. Physico-chemical and thermal characteristics of the dried AS were found to come across proper disposal option for dried AS. The sludge wasted during the idle phase of the cycle was collected, filtered, dried in desicators, and stored in glass containers.

The proximate analysis of wasted sludge was done as per Indian Standards. Moisture content, volatile matter, ash and fixed carbon of sludge were found to be 4.1%, 40.3%, 13.7% and 41.9%, respectively. Fig. 6 displays the EDX spectra as well the relative percentage of elements present in the raw and used AS obtained after treatment of wastewater. Raw and used AS were found to contain 35.95% and 37.54% oxygen; 44.77% and 50.19% carbon, respectively. The increase in carbon content (5.42%) was due to the presence of organics which were removed from the wastewater during the treatment process. The increased peak intensity for carbon in the AS was also due to the growth of biomass in the SBR. Relative percentage of magnesium, aluminum, phosphorus and calcium decreased in the treated AS as compared to that in raw AS due to the utilization of these elements as nutrients for the growth of microorganism.

Thermal stability of AS is directly dependent on the decomposition temperature of its various organics. The AS contains high amount of carbon and oxygen. At high temperature, the organics present in AS decompose producing CO (200−600°C), CO_2 (450−1000°C) [33] water vapor and free hydrogen (500−1000°C). The thermo-gravimetric analysis curves (TGA, DTA and DTG) of raw AS under oxidizing



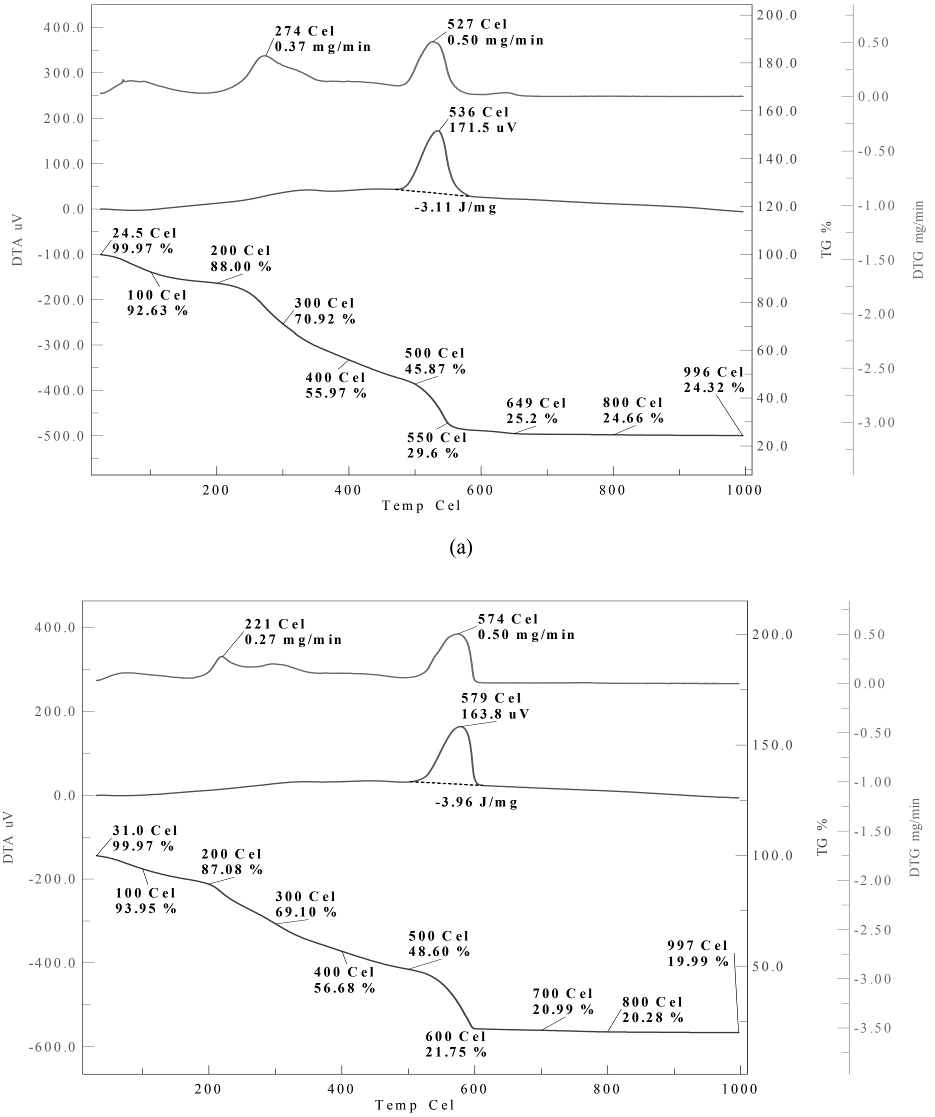
Before treatment



After treatment

Fig. 6. EDX of activated sludge before and after treatment

atmosphere are shown in Fig. 7a. Three different zones can be envisaged: from room temperature to 200°C, from 200 to 550/600°C, and from 550/600 to 1000°C. Main weight loss was recorded in the second zone, while the first and the third zones correspond to comparatively small weight losses. For raw AS, the first zone corresponds to the removal of moisture and light volatiles up to 200°C with a total loss of about 12%. This initial zone is followed by the active oxidation zone from 200 to 550°C with total degradation of 58.4%. Subsequently, the sample weight decreased very slowly with total degradation of 5.3% in the third zone up to 1000°C. The residue left at 1000°C is ash and is about 24.3% of the original sample weight. Thermal degradation characteristics of treated AS shows (Fig. 7b) removal of moisture and light volatiles of about 13% up to temperature of 200°C followed by an active oxidation zone between 200 and 600°C. Total degradation during this zone is about 65.3%. Beyond 600°C, the



(a)

(b)

Fig. 7. DTA-DTA-TG plot of activated sludge in various conditions in air atmosphere (a) before and (b) after growth

sample weight almost remained constant with total degradation of 1.8% up to 1000°C. Raw and treated AS did not show any endothermic transition between room temperature and 300°C, indicating the lack of any phase change during the heating process [34]. The strong exothermic peak centered on 530°C for raw AS and 580°C for treated AS is due to the oxidative degradation of the sample.

The heating value of the sludge was found to be 12.03 MJ/kg. This sludge can be utilized for making blended fuel briquettes with other organic fuels, which can be further used as a fuel in the furnaces. The bottom ash obtained after its combustion can be blended with the cementitious mixture. This mixture can be used in construction purposes, thus, recovering energy from the sludge.

CONCLUSIONS

In the present work, experimental study was done for optimization of SBR for the treatment of synthetic phenolic wastewater. The optimum HRT value was found to be 1.25 d whereas optimum fill time was found to be 1.5 h. Phenol, resorcinol, catechol and COD removal efficiency at optimum conditions were found to be 99%, 95%, 96% and 89%, respectively. EDAX analysis shows the increased carbon content and utilization of nutrients (like P, K, etc.) in the sludge after the treatment of wastewater in the SBR. The heating value of the activated sludge was found to be 12.09 MJ/kg. The filtered sludge can be dried and fired as fuel in the furnaces/incinerators for its heat recovery. The bottom ash can be used for blending with organic manure for its use in agriculture/horticulture or can be blended with clay/coal fly ash for use in making bricks/ceramic tiles for the building industry.

ACKNOWLEDGEMENT

Authors are thankful to the Ministry of Environment and Forest, Government of India for providing financial assistance to carry out this research work.

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