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Performance evaluation of column-SBR in paper and pulp wastewater treatment: optimization and bio-kinetics

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ABSTRACT

Pulp and paper industry generates effluent containing harmful compounds like chlorophenols which are difficult to biodegrade. It requires an appropriate treatment in order to meet the stringent discharge standards. In this work, a bench scale column type sequential batch reactor (SBR) was employed for treating pulp and paper wastewater. The performance of SBR, seeded with acclimatized sludge was optimized and analysed for maximizing COD and AOX removal. The process parameters viz; pH, initial COD, cycle time and MLSS were optimized and their effects on response variables: COD removal efficiency, AOX removal efficiency and SVI were investigated. The optimum conditions were determined to be: initial COD 1200 mg/l, pH 7.5, MLSS 2100 mg/L and cycle time 15 h, for 73.2% COD removal, 57.6% AOX removal and 122.8 mL/g SVI. The complex compounds were broken down into numerous intermediate compounds thus enhancing COD and AOX removal with low SVI. The bio-kinetics of the optimized system was also analyzed in order to understand the bacterial nature towards substrate utilization. Two kinetic models namely Grau second-order model and Stover–Kincannon model were found to be fitwell with high correlation coefficients ($R^2 = 0.99$) for COD as well as AOX.

Keywords: Paper and pulp; SBR; RSM; CCD; Bio-kinetics; COD; AOX removal

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1. Introduction

The enormous rising population and speedy industrialization, has resulted in reckless discharge of effluents garnering attention all over the world [1]. Moreover, excessive demand of water by pulp and paper industries has depleted this priceless commodity, thus raising concerns of water shortage all over the globe. Due to huge water intake in manufacturing processes, the paper and pulp industry discharges large quantity of effluents with potential adverse effects on our environment. Due to excessive use of

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chlorine in the manufacturing process, various chlorophenolic compounds are formed which need appropriate treatment before discharge. These compounds discharged in the effluent stream are not characterized specifically as phenolic compounds and are generally measured as“adsorbable organichalides” (AOX). These compounds along with several other contaminants have been reported for their ill health effects, being carcinogenic to mutagenic [2–4].

Conventional treatment processes employed for treating AOX are not highly efficient as they do not remove phenolic compounds completely. Employing activated sludge process in treating pulp and paper wastewater shows bulking, resulting in poor settling properties of sludge and effluent having high suspended solids concentration [5,6]. Chemical methods using chlorine, hydrogen peroxide and metal ions improves the settling properties of sludge but they are costlier [7,8]. Adsorption based techniques, using a granular activated carbon (GAC) post-treatment with long Hydraulic retention time (HRT) has been extensively used in the past for removal of AOX, but these adsorbents need to be regularly regenerated [9]. Using conventional process involving sedimentation and flotation have been employed in past, but their inefficiency in meeting the stringent effluent discharge requirement has limited their use [6]. Settleability inside sedimentation tank was also investigated by adding coagulants like aluminium chlorides as natural polymers, thereby increasing floc size to achieve high efficiency at optimum dosage, thus attaining removal of lignin, turbidity and COD as 83.4%, 95.7% and 90% respectively and water recovery of 72.7% [10,11].

Various operations are involved at different stages of pulp and paper manufacturing, necessitating the use of chemicals. This ultimately results in effluents having mixed and high concentrations of pollutants, thereby demanding the adoption of suitable treatment techniques. The variation in AOX concentration of effluent is also attributed to the use of these chemicals at different stages of manufacturing process before the discharge of final effluent [12]. It is not possible to reach the desired level of treatment with a single process, instead combination of multiple processes in sequences is required to achieve the desired goal and efficiency. In a lab-scale investigation, activated carbon based sequencing batch biofilm reactor (GAC-SBBR) was optimized for achieving high removal efficiency of 97.2% and 99.4% w.r.t. COD and NH_3-N respectively [13]. Few studies have been carried out in past exploring the feasibility of different types of treatments facilities for AOX removal in different regions and conditions [15,16]. Among them, some have also probed the toxicity of AOX, having standard discharge limit of 1 kg/ton in paper production as per central pollution guidelines and other contaminants for their effect on environment [14,15].

Sequential batch reactor (SBR) is an improvised activated sludge process used to treat a variety of wastewaters e.g. domestic wastewater, landfill leachate, industrial wastewater, biological phosphorus and nitrogen removal. It has several advantages like flexibility in operation, and low construction and maintenance costs. Introduction of column type sequential batch reactors (SBR) with modifications in conventional ASP, having less area requirement, proper fill-draw mechanism, effective size granules formation and ability to absorb shock loading may prove to be a promising technology in this field. Granules formed in biological reactor are due to self-accumulation type of microorganisms, without attaching to any medium [17]. The agglomeration of microbial cells into granules enhances toxicity toleration limits, structural stability and biodegradation properties. In the past, many laboratory and full-scale studies employing SBR treatment have been successfully carried out; reporting a higher efficiency of pollutant removal [18–20].

Anaerobic treatment process has proven to be comparatively economical, having better sludge condition with lesser sludge production and sensitivity towards shock loading. Treatment of organics at higher temperatures in an MBR (membrane bioreactor) is quite efficient and this may be further improvised and used for commercial applications. Several studies have focused on simultaneous removal of COD and AOX from effluent of paper and pulp industries using aerobic treatment [17,18]. A study on the use of Al_2O_3 and a polymer to treat wastewater in sedimentation tank showed that at optimum dosages of 871 mg/L and 22.3 mg/L at pH 8.35, signified removal efficiency of 95.7% and 83.4% for turbidity and lignin respectively as obtained [10]. In another study, it was observed that high molecular weight polymer of Chloride for Paper industry wastewater treatment is better in the overall performance of physical treatment as compared to low molecular weight polymers with COD removal of more than 90% [21]. Another study showed that coagulation-flocculation, though effective to some extent, do not mineralize organic pollutant and adsorption process COD removal efficiency of more than 80% was obtained by adding chitosan [15].

The objective of this work is to assess and optimize the overall performance of sequencing batch reactor (SBR) in treating the paper and pulp industry wastewater so as to meet the permissible effluent discharge limits laid by regulatory authorities. This study was conducted using Design Expert software, a four-factor and five-level central composite design was employed for response surface modeling. The percentage COD removal AOX removal and SVI were selected as the response variables whereas initial COD, MLSS, cycle time and initial pH were chosen to be the process variables (independent variables). RSM has been widely used for optimizing process conditions in treating various effluents like leachate, palm oil mill effluent (POME) and in several other fields like treatment of heavy metals [22–24]. Optimization is necessary because treatment procedures for paper industry are costly and involve recalcitrant organics such as lignin, AOX and other toxic compounds generated at different stages. Moreover, it is also observed that BOD removal is difficult because some compounds with high molecular weight are not removed effectively [25]. Further it is reported by several researchers that these effluents from paper and pulp mills and other industries sometimes are carcinogenic causing toxicity even at reproduction level [26–29] which although gets reduced by technological improvements but still exist in trace amounts after completion of treatment processes. That is why the choice of adopting a relevant treatment process and its optimization for successful application depends upon the influent type and its concentration as well [30,31].

2. Material and methods

Wastewater for this experiment was collected from Naini Pulp and Paper Industries, Kashipur, UP India. Lab-scale sequencing batch reactor (SBR) was fabricated with prespex. The reactor working volume was 3.46 L and its diameter and height were 7 cm and 90 cm respectively (Fig. 1).

An air supply system (model EK8000, 6.0 W) was connected to a diffuser attached to the bottom of the reactor. The entire setup was operated using Solenoid and Gate valves connected to an automatic on-off system with different cycle time ranging from 24 h at the beginning and reduced to 6 h at the end. The solenoid valve was connected to an influent feed tank of 12 L capacity for the flow regulation. Samples were withdrawn for analysis at height of 45 cm from the base, thereby keeping volumetric exchange ratio of 50%. Another port at 30 cm height was provided for MLSS collection and periodical sludge wasting. The reactor was operated at room temperature.

2.1. Acclimatization phase and operational parameters

The laboratory scale reactor was seeded with aerobic sludge obtained from aeration tank of the paper and pulp industry effluent treatment plant. The reactor was then fed with the equalization tank wastewater and aeration arrangement was made with help of blowers such that proper aeration at the bottom of the reactor was maintained without plugging of the diffuser due to sludge during operation. At the start, the cycle of 24 h was maintained and the data was collected only after the reactor achieved steady-state.

Initially, the reactor was fed with diluted (1 in 10) paper and pulp industry wastewater. Thereafter, the feed concentration was raised to 1 in 6, 1 in 4 and 1 in 2 successfully. Once the bacteria had acclimatized to the reactor environment, it was then fed with the wastewater. The initial characteristics of the wastewater have been tabulated in Table 1. The operational cycle was then gradually reduced from 24 h to 6 h as shown in Table 2. The duration of different phases of SBR operation i.e. fill, react, settle and decant have been mentioned in Table 3.

2.2. Analytical methods

The analyses of pH, MLSS, BOD, Sludge volume index-(SVI) and COD were conducted according to the Standard Method [32]. COD was measured using the close reflux method. Dissolved oxygen was measured using HQ30d portable meter fitted with LBOD10101 probe pH measure-

Table 1
Characteristics of the paper and pulp wastewater

Parameters	Range
Dissolved COD, mg/L	600–2100
BOD, mg/L	560–1250
AOX, mg/L	11–25
Turbidity, NTU	460–550
Total suspended solids (TSS), mg/L	2450–3900
Alkalinity, mg/L of $CaCO_3$	760–1230
pH	6.1–7.8

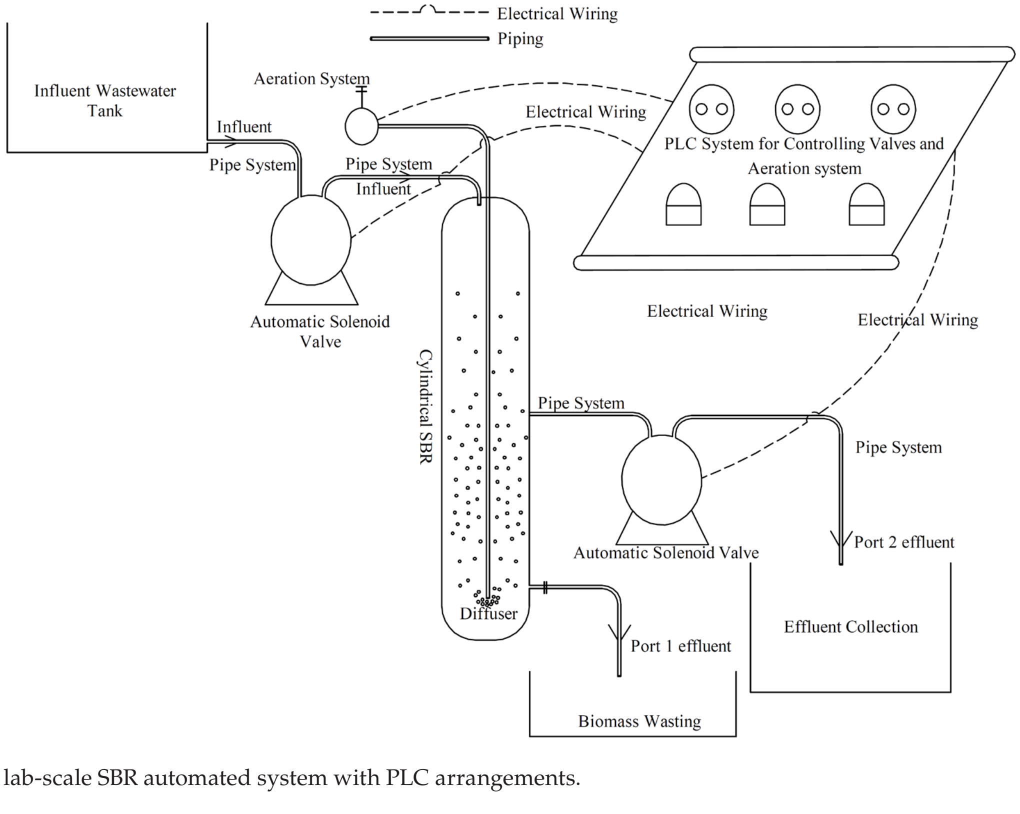


Fig. 1. Setup of lab-scale SBR automated system with PLC arrangements.

Table 2
Operational parameters of SBR

S. No.	Period (d) Parameters	0–160	160–320	320–490	490–830	830–900	900–1020
1.	Cycle/HRT (h)	24	20	18	12	8	6
2.	COD loading rate (kg COD/m^3/d)	0.29	1.26	2.26	3.46	3.86	4.6
3.	COD removal %	82	65	74	80	84	88
4.	AOX removal %	40	43	45	55	58	62
5.	SVI (ml/ gMLSS)	115–125	102	105	75–85	55–88	65–75
6.	MLSS (g/L)	2–3	3–4.1	4–6.4	6.4–7.4	5.5–7	6–8
7.	Granule size (mm)	–	0.4–1	1.2–2.2	2–3	3.5–3.8	3–5
8.	Integrity coeff. (%)	–	–	–	90	92	96

Table 3
Time of each phase in SBR operation

S. No.	Cycle	Fill	React	Settle	Decant
1.	6	0.26	5.5	0.083	0.167
2.	8	0.26	7.5	0.083	0.167
3.	12	0.26	11.5	0.083	0.167
4.	18	0.26	17.1	0.44	0.20
5.	20	0.26	18.9	0.61	0.23
6.	24	0.26	23	0.5	0.25

Table 4
Input parameters for modelling in CCD

Coded values	A: COD initial (mg/L)	B: MLSS (mg/L)	C: Cycle time (h)	D: pH
–2.0	400	1000	6	3
–1.0	800	1600	12	5
0.0	1200	2000	18	7
1.0	1600	2800	20	9
2	2000	3200	24	11

ments were done by using Hach portable meter HQ30d attached with its pH probe. Mixed liquor suspended solids (MLSS), Volatile suspended solids (VSS) were analysed on weekly basis. The aerobic granulation development was analysed using the Cenisco binocular petrological microscope. The pictures at different stages were analysed using image analysis technique and the aspect ratio was considered with the help of Averz Software.

2.3. Adsorbable organic halide analysis

Adsorbableorganic halide (AOX) measurement was made using Multi X 2500 halide analyser Jena, Germany, using the method specified as per the previous studies [33,34]. After collecting the effluent from SBR port, it was passed through activated carbon followed by sodium nitrate wash so as to purge it of inorganic chlorine. Finally, the column was burnt in the furnace at 920°C and content was analysed using column titration methods [33,34].

2.4. Analysis of intermediates using GC-MS

Intermediates were analyzed using GC/MS (Perkin Elmer 600T). The samples were prepared from the effluent acidified top H 2.0 using 1 N H_2SO_4. The extraction was done in a separating funnel three times using dichloromethane (99.5%) in 1:1 ratio. A sample of 1 µL was injected for chemical identification.

The mass spectrometric analysis was done in electron impact (EI) mode at 70 eV and helium was employed as a carrier gas with a flow rate of 1 mL/min. PE-5MS capillary column of length 30 m, diameter (i.d.) 250 µm was used in the study. The GC column and injector temperatures were kept at 250°C and 200°C during the spitless mode injections. The oven was initially kept at 50°C for 5 min and then ramped up to 250°C at 10°C/min. The MS was operated in total ion current (TIC) mode thereby scanning all the range of m/z from 30 to 500. Their retention time was measured in minutes and mass spectra were matched from the library available in the National Institute of Standard and Technology (NIST).

2.5. Experimental design with RSM using CCD

For optimizing the experimental conditions, Design Expert (version 11.0) was used. RSM using central composite design (CCD) was subjected to the sequential sum of squares test and lack of fit test. CCD model was used for understanding the relationship of 4 process variables: initial COD conc., MLSS, cycle time and pH with the response in terms of COD and AOX removal percentages and SVI.

In the optimization process through Design Expert software, a four-factor and five-level CCD was employed. A set of 30 experiments with 16 factorial points and 8 axial points and 6 central points was conducted to obtain the response surface model. The said parameters were encoded as shown in Table 4. Using CCD method, a model is generated reminiscent of the response. In case of the current study SBR process, a quadratic equation was observed to be appropriate for analysis.

2.6. Kinetic modelling

The kinetics of removal of COD and AOX from the column type SBR reactor was also studied using different kinetic models: first order model, Grau second-order model, modified Stover–Kincannon model and Monod model [28,29]. All these are well-established models for kinetic analysis for treatment of wastewater using biological systems. The analysis was done when the steady state was achieved in the reactor after acclimatization. Different parameters were estimated as described in the above sections.

3. Results and discussion

Model satisfactoriness is adjudged from the diagnostics (predicted versus actual value plots). Adequate agreement between real data and that obtained from models is evident from the diagnostic plots shown in Fig. 2.

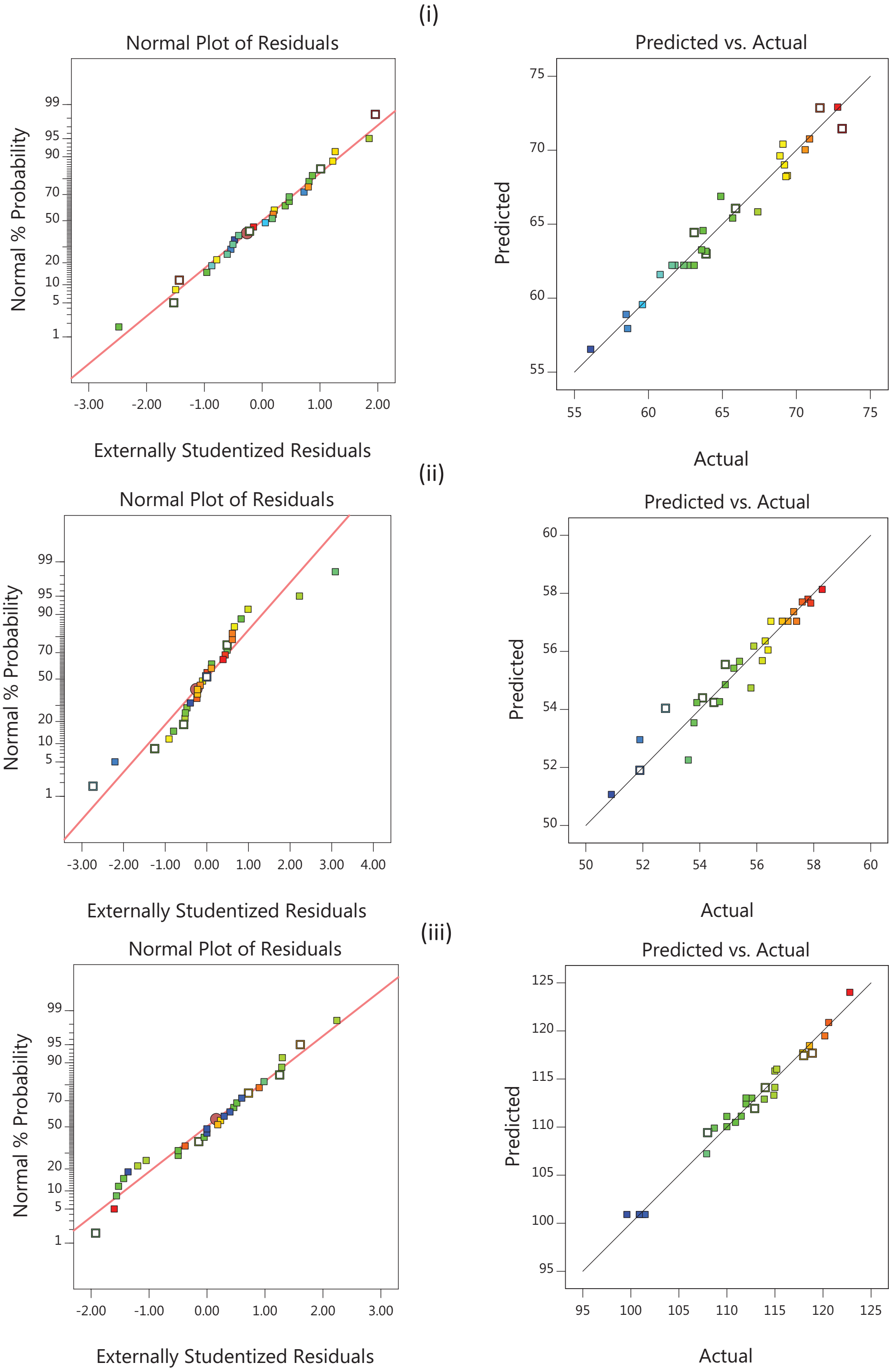


Fig. 2. Normal probability versus studentized residuals and predicted versus actual plots for (i) COD removal (ii) AOX removal and (iii) SVI.

The following polynomial regression model equations were obtained:

$$\%\text{COD Removal} = 62.21 - 2.78A + 1.875B - 0.7C + 2.8D + 0.65AB + 0.5125AD + 0.5604A^2 + 1.735B^2 + 0.6104C^2 + 0.735D^2 \quad (1)$$

$$\text{AOX Removal } \% = 57.03 + 0.08A - 0.97B - 0.20C + 0.14D - 0.72AB + 0.86CD + 0.12A^2 - 0.21B^2 - 1.39C^2 - 0.47D^2 \quad (2)$$

$$\text{SVI} = 100.9 - 0.60A - 0.275B - 1.016C - 2.2D + 1.125AB + 1.425AD + 0.862BD + 2.275CD + 4.691A^2 + 3.166B^2 + 2.79C^2 + 2.679D^2 \quad (3)$$

where A is initial COD (mg/L), B is MLSS conc. (mg/L), C is cycle time (h) and D is pH at which the study was carried out.

3.1. ANOVA of quadratic models for COD removal, AOX removal and SVI

In order to estimate the accuracy of models developed from experimental data, ANOVA (analysis of variance) can be used. ANOVA shows the statistical indices like, F-value and P-value. If F has large value with small P-value (0.05), the model can be considered as statistically significant. Also, large value of correlation coefficient shows the accuracy of the proposed model. The lack of fit should not be significant for the model to fit.

Tables 5, 6 and 7 show the ANOVA tables for the models in terms of COD removal, AOX removal and SVI respectively. For the designed system with selected variables and desired response outputs, the quadratic polynomial models were found to fit well, which is in accordance with previous studies [35]. ANOVA tables for all the three models have been presents in Tables 5, 6 and 7, the F-value of 47.24, 25.07 and 89.42 for COD, AOX removal and SVI, in the same order, have p-values < 0.0001 for all three models. No significant lack of fit was found and its values for different parameters were 0.06, 0.50 and 0.10 respectively. High values for R^2 = 0.96, 0.92 and 0.98 were obtained for COD removal, AOX removal and SVI respectively. These are also closed to the respective adj. R^2 values. This shows close agreement of the experimental and actual values.

3.2. Effect of variables on process responses

The 3-D surface plots, plotted using design Expert Software, were obtained by plotting the response against two variables and keeping others constant. The % removal efficiencies lie between 56.1 and 73.1% for COD, 50.9 and 58.9% for AOX, and the SVI values were obtained in the range 99.6–122.8 mL/g as per the 3-D surface plots shown in Figs. 3, 4 and 5 respectively.

3.2.1. COD removal

Fig. 3(i) shows the effect of MLSS and the initial COD conc. on COD removal percentage at a cycle time of 18 h. COD removal percentage of 73.1% was achieved at MLSS of 3200 mg/L keeping the initial COD at 400 mg/L. On further increasing the initial COD, organic overloading occurred resulting in the slowing down of the consumption of organic compounds by microorganisms [36]. Also, it can be seen from Fig. 3(iii), the removal efficiency shows an increasing trend as with increase in MLSS, which is in accordance with earlier research [7,37]. Higher concentra-

Table 5
ANOVA for selected quadratic model for % COD removal

Source	Sum of squares	df	Mean square	F-value	p-value	
Model	574.40	10	57.44	47.24	< 0.0001	Significant
A-COD	185.93	1	185.93	152.91	< 0.0001	
B-MLSS	84.37	1	84.37	69.39	< 0.0001	
C-cycle	11.76	1	11.76	9.67	0.0058	
D-pH	188.16	1	188.16	154.75	< 0.0001	
AB	6.76	1	6.76	5.56	0.0293	
AD	4.20	1	4.20	3.46	0.0786	
A^2	8.61	1	8.61	7.08	0.0154	
B^2	82.61	1	82.61	67.94	< 0.0001	
C^2	10.22	1	10.22	8.41	0.0092	
D^2	14.83	1	14.83	12.20	0.0024	
Residual	23.10	19	1.22			
Lack of fit	21.23	14	1.52	4.06	0.0652	Not significant
Pure error	1.87	5	0.3737			
Cor total	597.50	29				
R^2	0.96					
Adjusted R^2	0.9410					
Predicted R^2	0.9112					

Table 6
ANOVA for selected quadratic model for % AOX removal

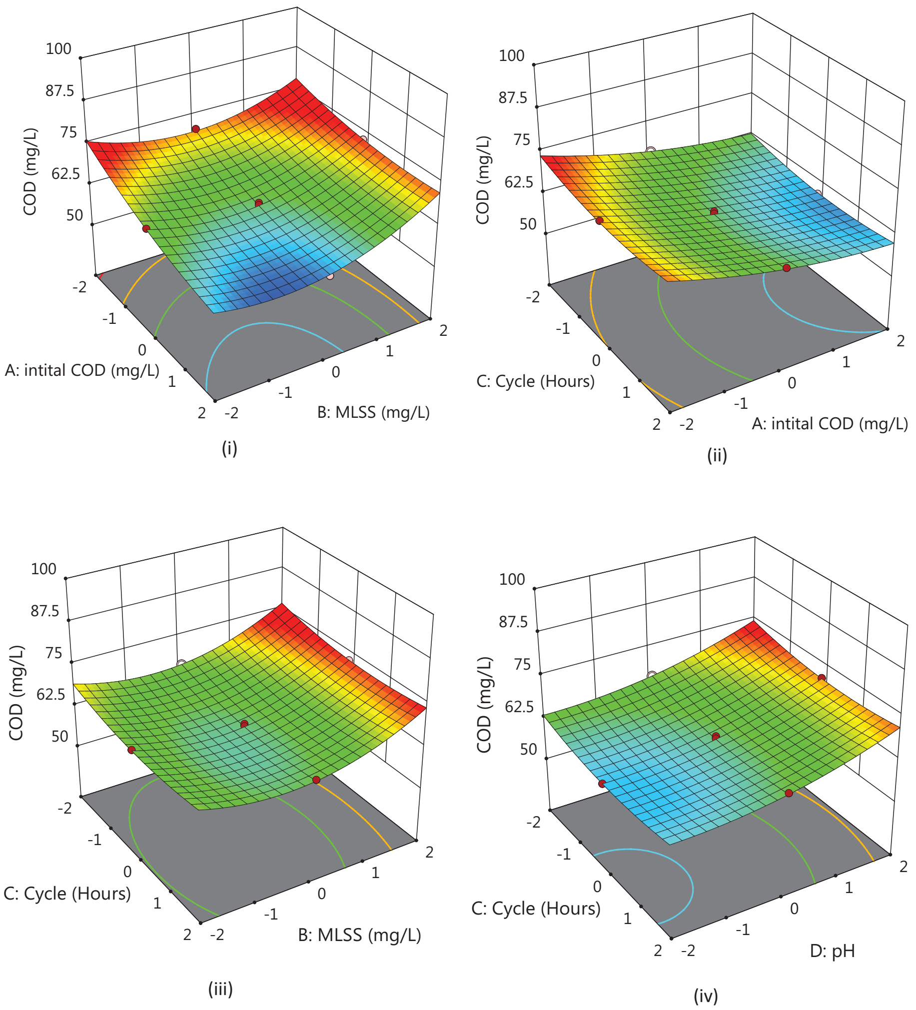
Source	Sum of squares	df	Mean square	F-value	p-value	
Model	103.42	10	10.34	25.07	< 0.0001	Significant
A-COD	0.1667	1	0.1667	0.4040	0.5326	
B-MLSS	22.82	1	22.82	55.30	< 0.0001	
C-Cycle	1.04	1	1.04	2.52	0.1286	
D-pH	0.4817	1	0.4817	1.17	0.2935	
AB	8.41	1	8.41	20.38	0.0002	
CD	11.90	1	11.90	28.85	< 0.0001	
A^2	0.4286	1	0.4286	1.04	0.3209	
B^2	1.24	1	1.24	3.00	0.0994	
C^2	52.80	1	52.80	127.98	< 0.0001	
D^2	6.19	1	6.19	15.00	0.0010	
Residual	7.84	19	0.4126			
Lack of fit	7.25	14	0.5176	4.36	0.0565	Not significant
Pure error	0.5933	5	0.1187			
R^2	0.92					
Cor total	111.26	29				
Adjusted R^2	0.8925					
Predicted R^2	0.8385					

Table 7
ANOVA for selectedquadratic model for SVI

Source	Sum of squares	df	Mean square	F-value	p-value	
Model	1234.69	12	102.89	89.42	< 0.0001	Significant
A-COD	8.64	1	8.64	7.51	0.0140	
B-MLSS	1.82	1	1.82	1.58	0.2261	
C-Cycle	24.81	1	24.81	21.56	0.0002	
D-pH	116.16	1	116.16	100.95	< 0.0001	
AB	20.25	1	20.25	17.60	0.0006	
AD	32.49	1	32.49	28.24	< 0.0001	
BD	11.90	1	11.90	10.34	0.0051	
CD	82.81	1	82.81	71.97	< 0.0001	
A^2	603.75	1	603.75	524.69	< 0.0001	
B^2	275.05	1	275.05	239.03	< 0.0001	
C^2	213.76	1	213.76	185.77	< 0.0001	
D^2	196.88	1	196.88	171.10	< 0.0001	
Residual	19.56	17	1.15			
Lack of fit	17.26	12	1.44	3.13	0.1084	Not significant
Pure error	2.30	5	0.4600			
Cor total	1254.25			29		
R^2	0.98					
Adjusted R^2	0.9734					
Predicted R^2	0.9447					

tion of MLSS provided more contact time with biomass in the reactor and the wastewater, thus resulting in better degradation.

It can be seen from Fig. 3(ii) that as we go on reducing the cycle time reduces from 24 to 18 h, the COD removal decreases due to less contact of organic matter with biomass, but as the cycle time increases, the efficiency increases. The 3D surface plot in Fig 3(iii) clearly shows that increasing MLSS upto 3200 mg/L maximizes COD removal to 73.1% keeping initial COD constant. This may be attributed to a



(i) (ii) (iii) (iv)

Fig. 3. 3D surface plot of COD removal % (i) MLSS vs COD initial (ii) cycle time vs COD initial (iii) cycle time vs MLSS (iv) cycle time vs pH from SBR treatment system.

higher population of microorganisms available to consume the organics. pH is a key parameter in the development of the microbial community in aerobic reactor. The granule formation is closely linked to the pH of the reactor. The 3D surface plot in Fig. 3(iv) shows that COD removal efficiency increases as pH moves from acidic to basic range. The basic pH range is optimum for the growth of bacteria, resulting in granule formation, while in the acidic environment mass growth of fungi takes place as has been reported by several researchers [38,39].

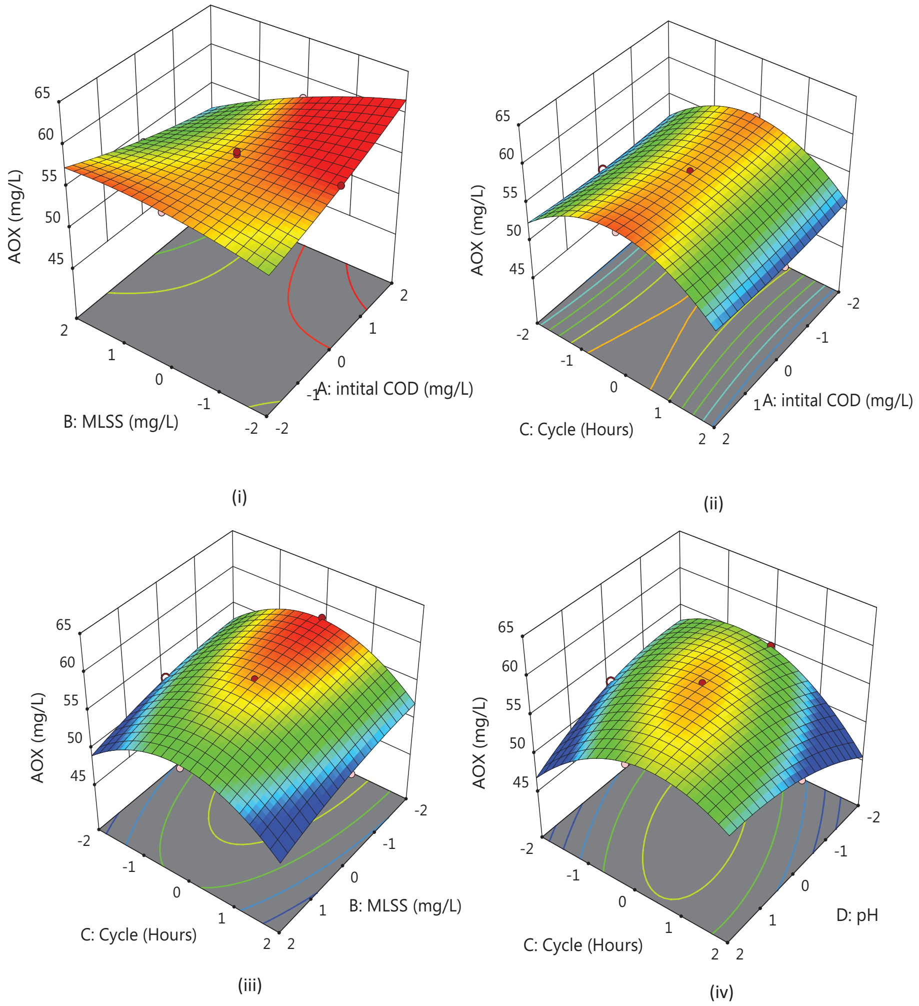
3.2.2. AOX removal

Figs. 4(i), (ii), (iii) and (iv) show the3-D surface plots for AOX removal from the Naini paper and pulp industry wastewater. The upper and lower range of AOX removal obtained in the model were 58.3% at cycle time of 12 h, initial COD of 800 mg/L, MLSS of 1600 mg/L and initial pH of 5 and 50.9% at cycle time of 24 h, initial COD of 2000 mg/L, MLSS of 3200 mg/L and initial pH of 11, respectively. The increase in both MLSS and initial COD improves AOX removal for the cycle time of 18 h and the removal only deteriorates for either increase or decrease in the cycle time. During the cycle of 12–18 h contact of biomass with lignin was good and due to increase in cycle time up to 24 h the removal percentage of AOX decreased drastically because of decrease in the F/M ratio in the SBR. AOX may be consumed by microbes as co-substrate.

From the 3D surface plots (Figs. 4 ii and iv), with increase in cycle time up to 18 h, improvement in AOX removal is evident but later it decreases with increase in cycle time up to 24 h, which has also been reported earlier as well [40,41]. With increase in initial COD concentration the efficiency of AOX removal reduced. The removal was 58.9% at HRT of 12 h with initial COD of 800 mg/L.

3.2.3. Sludge volume index (SVI)

Sludge Volume index is a commonly used index to judge the settling properties of any wastewater during different stages of treatment. Normal sludge has SVI of the order of 100 mL/g and for bulking sludge it can be as high as 200 mL/g or more [35]. Figs. 5(i), (ii), (iii) and



(i) (ii) (iii) (iv)

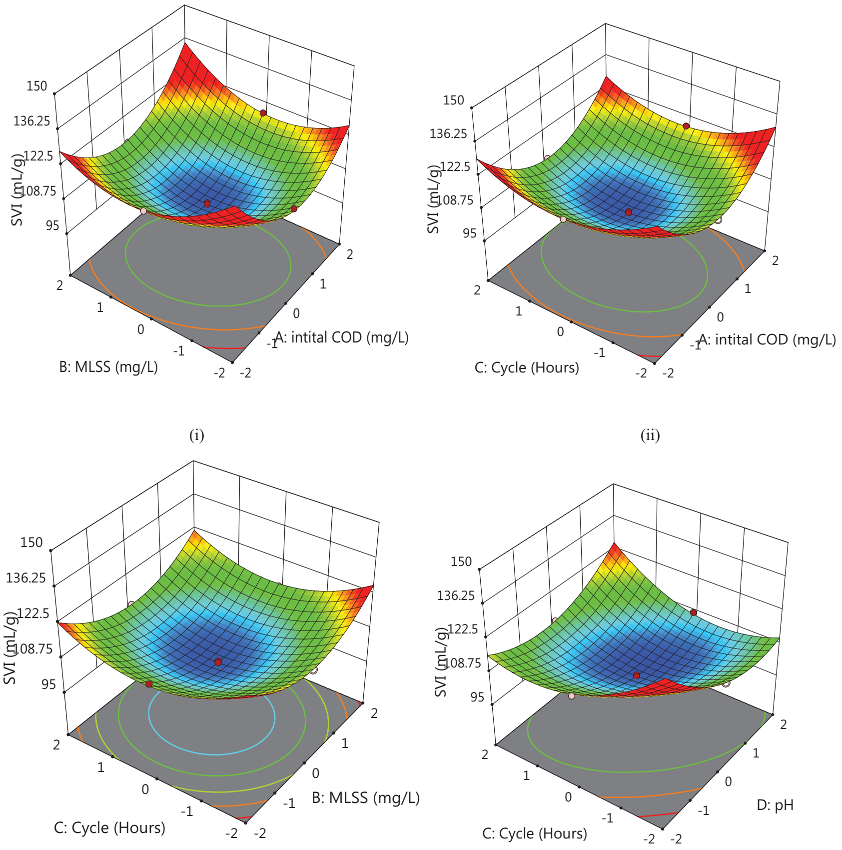
Fig. 4. 3D surface plot of AOX removal % (i) MLSS vs COD initial (ii)cycle time vs COD initial (iii) cycle time vs MLSS (iv) cycle time vs pH from SBR treatment system.

(iv) shows the surface plot obtained using Design Expert software. The SVI initially reduced with increase in MLSS to 1600 mg/L and then increased as the MLSS increased to 3200 mg/L. This shows the low food availability for the microorganism. It can be easily seen that upper limit of SVI is 122.8 mL/g at MLSS of 3200 mg/L for the cycle time of 12 h with the initial COD conc. of about 1600 mg/L. This is because of the reduction in DO and F/M ratio caused due to filamentous bacteria. Such bacteria increases at low DO which allows oxygen to enter into the flocs [36]. Higher cycle time shows a negative effect on size. As shown in Fig. 5(iii), increasing the value of cycle time at initial COD of 400 mg/L decreases the SVI up to a certain stage and then it increases with further increase in the Cycle time. The SVI increases with increasing the cycle time primarily due to decrease in F/M ratio. The somewhat similar effect is being observed by varying the cycle time with pH value. These results show good setting properties of the sludge with typical effect of no bulking, implying that SBR treatment process can successfully be employed for pulp and paper wastewater treatment [42,43].

3.3. Optimization of the modeled parameters

Design expert explores the design space, to optimize the process. With the addition of optimization function of the software, the most suitable values can be judged and applied to practical systems as well. In the present case, the input variables were optimized with a view to maximize COD and AOX removals and minimize the SVI. The results revealed that the COD removal and AOX removal efficiency were 73.1% and 58.3% respectively and the SVI after optimization was obtained as 122.8 mL/g. The average experimental values were obtained for validation after carrying out the studies as per optimum values suggested by software optimization procedure and the same has been tabulated in Table 8. A close agreement between the theoretical and experimental values shows that RSM maybe truly applied in optimizing the SBR process for COD and AOX removal.

The formation of granules during different phases of SBR operation is shown in Fig. 6 small aerobic granules were observed at 320 d with the non-granulating biomass washed out of the reactor. The concentration



(i) (ii) (iii) (iv)

Fig. 5. 3D surface plot of SVI mL/g (i) MLSS vs initial COD (ii) cycle time vs initial COD (iii) cycle time vs MLSS. (iv) cycle time vs pH from SBR treatment system.

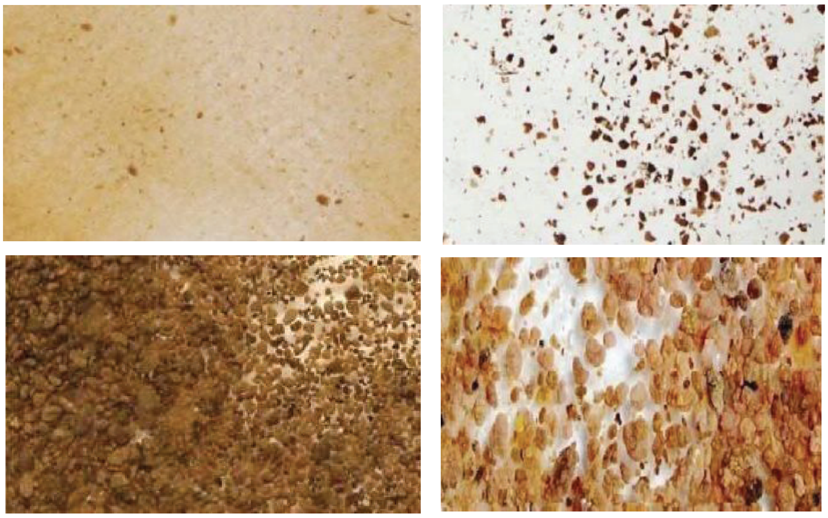


Fig. 6. Images showing granulation process after acclimatization. (i) Seeded sludge initially. (ii) Aerobic granules after 320 days. (iii) Aerobic Granules after 830 days. (iv) Aerobic granules 1020 days.

of granular sludge increased markedly up to 830 d. The granules after 1020 days were significantly larger. Table 8: Predicated and experimental values of responses at optimum conditions (initial COD 1200 mg/L, cycle time 15 h, pH 7.5 and MLSS 2100 mg/L theoretical and 2250 mg/L actual).

Table 9 presents a summary of some studies conducted on the treatment of Paper and pulp wastewater. Results

Table 8
Predicted experimental values of response at optimized conditions*

Parameter	Optimum value	Avg. exp. values
COD removal efficiency (%)	73.2	70.5
AOX removal efficiency (%)	57.6	51.2
SVI	122.8	128

*Initial COD = 1200 mg/L, cycle time = 15 h, pH = 7.5 and MLSS = 2100 mg/L theoretical and 2250 mg/L actual.

from the current work were found to be comparable; differences can be attributed to the type of process, reactor type and operating conditions.

3.4. Intermediates formed during SBR treatment post optimization

The treated wastewater samples were qualitatively analyzed with GC-MS and the organic compounds detected during the analysis were tabulated in Table 10, these have been reported in the past studies as well [4,50]. Some of the important by-products obtained after treatment are shown in Fig. 7. These also correspond with pre-

Table 9
Studies based on aerobic/SBR treatment depicting various parameters

S. No.	Type of effluent	Type of operation	Operating condition of treatment process	Removal percentage (%)	Ref. No.
1.	Paper and pulp	Biological (cylindrical SBR)	pH = 7.5, cycle time = 15 h, MLSS = 2100 mg/L, COD initial = 1200 mg/L	COD = 70.5%, AOX = 51.2% SVI = 128 mL/g	Present study
2.	Kraft paper and pulp	Biological (cylindrical SBR)	Temp: 30±2°C, pH = 5, DO: 2 mg/L HRT: 6 and 12 h, SRT: 5–10 d, MLSS: 4500–5000 mg/L, aver. SVI = 94.4 mL/g, AOX = 4–25 mg/L	COD = 90%, BOD = 92% AOX = 60%	[4]
3.	Paper and pulp	Biological (cylindrical SBR)	Temp: 30±2°C, pH = 6,DO: 2–3 mg/L HRT: 6 and 24 h, MLSS: 2000–3400 mg/L, COD initial = 1700–2700 mg/L, BOD = 680–1100 mg/L, AOX = 13–20 mg/L	COD = 87%, BOD = 90% AOX = 55%	[44]
4.	Paper and pulp	Anaerobic process	Temp: 30±2°C, pH = 6.1–7, HRT: 48 h, MLSS: 2000–3400 mg/L, COD initial = 4100 mg/L, TDS = 2500 mg/L, OLR = 3000 mg/L	COD = 95% at pH = 7	[45]
5.	Paper and pulp	Combination (biological + Fenton)	pH = 7–8, SRT = 24–72 h TSS: 443 mg/L, VSS = 233 mg/L BOD = 917 mL/g, COD = 1800 mg/L (BOD/COD ratio = 0.51)	COD = 68% DOC = 90%	[46]
6.	Recycled paper mill effluent	Anaerobic baffled reactor	Temp: 25±2°C, pH = 7–4, HRT: 04 d , TSS: 645 mg/L, COD initial = 4328 mg/L, TDS = 3345 mg/L, DO = 1.5 mg/L	COD = 85% at Anaerobic mesophilic conditions	[47]
7.	Wood and pulp	Coagulation with aluminium chloride and adsorption on tuff, then nanofiltration membrane	Total carbon (TC) = 569.0 mg dm^{-3}, inorganic carbon (IC) = 209.8 mdm^{-3}, total organic carbon (TOC) = 359.2 mg dm^{-3}; chemical oxygen demand (COD) = 422.0 mg dm^{-3}; All tests were performed without adjusting pH.	Total carbon: 67% TOC: 77% Inorganic C: 49%	[48]
8.	Recycled paper	Aerated granulated carbon sequencing batch biofilm reactor	pH = 6.4, COD = 746 mg/L, Suspended solids = 267 mg/L, NH_3-N = 4.1 mg/L PO_4^{-3} = 0.03 mg/L	COD: 97% Cl-phenols: 81% Nitrogen (NH_3) = 100%	[13]
9.	Recycled. paper	Granulated activated carbon sequencing batch biofilm reactor	Average COD = 1152 mg/L, Average AOX = 249 mg/L (2-CP), 98 mg/L (2,4-DiCP) and 4 mg/L (2, 3, 4, 5-TeCP).	COD: 53–92% AOX: 26–99%	[21]
10.	Synthetic wastewater	Hybrid growth sequencing batch reactor (HG-SBR)	MLSS = 8300 mg/L, sucrose = 563 mg/L), NH_4Cl = 172 mg/L), $FeCl_3$ = 12 mg/L), K_2HPO_4 = 180 mg/L, KH_2PO_4 = 35 mg/L and $NaHCO_3$ = 100 mg/L), pH = 6.8	Higher phenols degradation 93.3% was recorded for 539 mg/L initial COD, while, 87.9% and 64.6% of COD removal 540 was recorded for 1935 mg/L and 2300 mg/L initial COD respectively.	[49]

Table 10
Analytical results of metabolites obtained from treated effluent through GC/MS

	S. No.	Retention time (min)	Metabolites detected
Influent	1.	7.150	Ethyl hydrogen oxalate
	2.	8.165	2,4,4,- trimethyl-1-hexene
	3.	12.877	Cyclopropane, nonyl
	4.	12.997	Dodecane
	5.	13.196	Dodecamethylcyclohexasiloxane
	6.	15.427	Decamethylcyclopentasiloxane
	7.	15.64	2,6-Di-tert-butylphenol
	8.	15.743	Tetradecane
	9.	24.282	Nonadecene
	10.	31.047	4-nitrophenyl palmitate
	11.	33.409	Hexamethylcyclotrisiloxane
Effluent	1.	9.9	Phenol,2-methoxy-
	2.	17.05	Benzene ,1,3,5 trimethyl-2-(3-methyl 1,3,butadienyl-
	3.	15.628	2,5-Di-tert-butylhydroxybenzene
	4.	18.774	Cyanoacetamide
	5.	20.23	1,4-benzenediol
	6.	30.54	hexadecanoic acid,methyl ester

vious studies [51,52]. The numerous peaks in the influent are due to various major and minor compounds present, as mentioned in Table 10. However, the smaller and fewer peaks are detected in the effluent due to smaller number of byproducts detected after treatment. These results can be inferred as the breakdown of complex phenolic compounds including chlorophenols. The breakdown of complex compounds into simpler organic intermediate compounds is an encouraging result. This might not be reflected in the results, in terms of COD removed, but these organics can easily be removed by any subsequent biological treatment.

3.5. Kinetic models post optimization

3.5.1. First order model

The linearized first order model was applied to the data obtained. K_1 and R^2 was calculated by plotting between $(S_e–S_o)/S_e$ against HRT as shown in Fig. 8. The constant obtained was 1.403 ($R^2 = 0.861$) and 0.047($R^2 = 0.75$) for COD and AOX respectively. These results suggest that the model obtained does not fit well for either of the cases.

3.5.2. Grau second-order model

The results obtained were plotted between HRT/$((S_o–S_e)/S_o)$ and HRT as shown in Fig. 8 (ii). The constant obtained for COD was $K_s = 10^{-4}$ and for AOX was 0.061, as shown in Table 11. The finding suggests good correlation coefficient ($R^2 = 0.99$). The results obtained shows this model fits well for both COD and AOX.

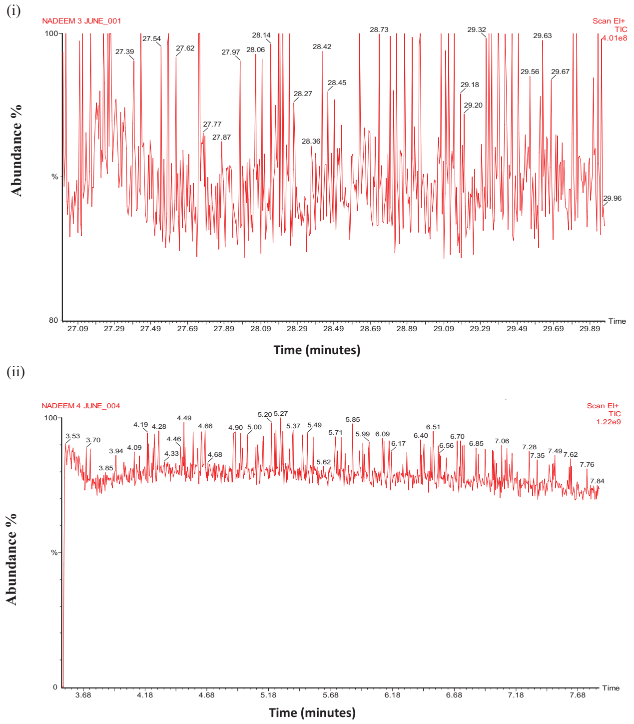


Fig. 7. (i) Chromatographic analysis of influent before SBR treatment and (ii) Chromatographic analysis of intermediate formation after SBR treatment.

3.5.3. Modified Stover–Kincannon model

The substrate utilization rate is well expressed as organic loading rate in this model and is widely used in modeling the kinetics of a biological reactor for the treatment of wastewater. This model contains parameters to estimate the efficiency and performance of a biological system. Fig. 8 (iii) shows the substrate removal plot for COD as well as AOX removal having $R^2 = 0.99$. Kinetic constants K_B and U_{max} for COD were calculated as 0.25 and 1.66 g/L/d respectively, while for AOX these were obtained as 11.6 and 99 g/L/d respectively as shown in Table 11.

3.5.3. Monod model

The plot of specific AOX and COD utilization rates is shown in Fig. 8 (iv) in which VX/Q $(S_o–S_e)$ was plotted against $1/S_e$. Slope of the line gives the value of K_s/K and the intercept gives the value of 1/K. The obtained values of K_s/K and 1/K are 1.445 and 0.1541 for COD and 17.1 and 0.987 for AOX respectively. The values of half saturation coefficient obtained for COD and AOX removal was obtained as 0.083 and 0.1567 g/L respectively. From these values it can be inferred that bacteria have high affinity towards substrate. The Monod model was not found to fit well in case of AOX ($R^2 = 0.5947$). A

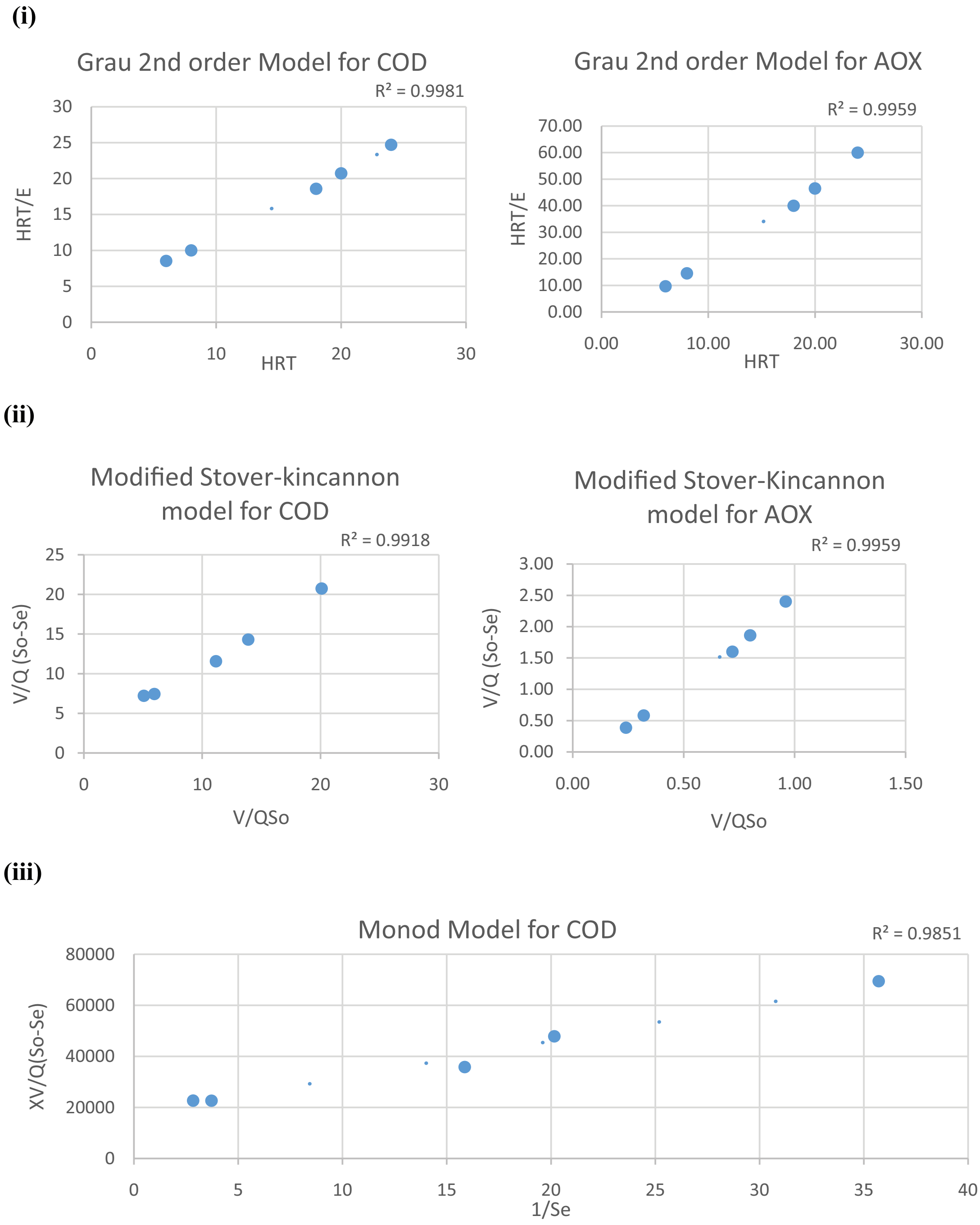


Fig. 8. Kinetic modeling plots for COD and AOX removal using (i) Grau second-order model (ii) Modified Stover–Kincannon (iii) Monod model.

good fit was obtained in case of COD with correlation coefficient (R^2) obtained as 0.98. The values of constants are shown in Table 11.

4. Conclusion

In this work, experiments were statistically designed using RSM for optimizing SBR performance. Process variables viz initial COD, pH, MLSS and cycle time were optimized for the responses; SVI, COD removal efficiency and AOX removal efficiency. A four factor, five level central composite design (CCD) was used to optimize the treatment process.

The optimum HRT for COD consumption as well as AOX removal was determined to be 15 h for the MLSS of about 2500 mg/L. This finding is rather interesting because

Table 11
Kinetic modeling details with linearized equation used for modeling purpose

Parameter	Model analyzed	Equation used	Linearized equation	Kinetic parameters obtained		R^2 value
COD	1st order	$\frac{-ds}{dt}=\frac{Q}{v}\left(s_0-s_e\right)$	$\frac{s_0}{s_e}-1=k_1 HRT$	$K_1 = 1.403$	–	0.861
	Grau 2nd order model	$\frac{-ds}{dt}=\frac{k_2 x s_e 2}{s_0^2}$	$\frac{s_0}{s_0-s_e}HRT=HRT-S_0\Big/k_2 x$	–	$K_s = 10^{-04}$	0.9926
	Modified Stover-Kincannon model	$-\frac{ds}{dt}=\frac{Q}{v}\left(\frac{s_0-s_e}{s_0}\right)=\frac{U_M\frac{Qs_0}{v}}{k_B+\frac{Qs_0}{v}}$	$\frac{v}{Q\left(s_0-s_e\right)}=\frac{k_B v}{U_M s_0 Q}+\frac{v}{U_M}$	$K_B = 0.252$	$U_M = 1.66$	0.9918
	Monod model	$-\frac{ds}{dt}=\frac{Q}{v}\left(\frac{s_0-s_e}{s_0}\right)=\frac{U_M\frac{Qs_0}{v}}{k_B+\frac{Qs_0}{v}}$	$\frac{vx}{Q\left(s_0-s_e\right)}=\frac{k_s}{ks_e}+\frac{1}{k}$	$K = 0.058$	$K_s = 0.084$	0.9851
AOX	1st order	$\frac{-ds}{dt}=\frac{Q}{v}\left(s_0-s_e\right)-k_1 s_e$	$\frac{s_0}{s_e}-1=k_1\theta$	$K_1 = 0.047$	–	0.75
	Grau 2nd order model	$\frac{-ds}{dt}=\frac{k_2 x s_e 2}{s_0^2}$	$\frac{s_0}{s_0-s_e}HRT=HRT-S_0\Big/k_2 x$	–	$K_2 = 0.061$	0.9926
	Modified Stover-Kincannon model	$-\frac{ds}{dt}=\frac{Q}{v}\left(\frac{s_0-s_e}{s_0}\right)=\frac{U_M\frac{Qs_0}{v}}{k_B+\frac{Qs_0}{v}}$	$\frac{v}{Q\left(s_0-s_e\right)}=\frac{k_B v}{U_M s_0 Q}+\frac{v}{U_M}$	$K_B = 11.67$	$U_M = 99$	0.9959
	Monod model	$-\frac{ds}{dt}=\frac{Q}{v}\left(\frac{s_0-s_e}{s_0}\right)=\frac{U_M\frac{Qs_0}{v}}{k_B+\frac{Qs_0}{v}}$	$\frac{vx}{Q\left(s_0-s_e\right)}=\frac{k_s}{ks_e}+\frac{1}{k}$	$K = 1.013$	$K_s = 0.156$	0.5997

it is counter intuitive to the fact that higher HRTs result in higher removal of COD. The depreciation in the removal efficiency post 18 h HRT was attributed to the reduction in F/M ratio which resulted in the consumption of cell mass and consequently lower removal efficiencies. A high value of R^2 (0.9613, 0.9295 and 0.9844, for SVI, COD removal efficiency and AOX removal efficiency respectively) was obtained for the 3 models implying a good fit of the experimental data using second-order regression models. The optimum values of the responses COD removal, AOX removal and SVI were 70.5%, 51.2% and 128 mL/g respectively for the initial conditions of; COD 1200 mg/L, MLSS 2250 mg/L and cycle time 15 h and pH 7.5. Kinetic studies at the optimum conditions suggested that the Grau 2nd order Model and Modified Stover-Kincannon model fitted the data well for both COD and AOX.

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Nomenclature

ds/dt — Change of substrate conc. w.r.t time
Q — Flow rate
V — Volume of reactor
S_o — Initial substrate concentration
S_e — Final substrate concentration
K_1 — First order rate constant
K_2 — Second order rate constant
K_s — Maximum substrate utilization rate
K_B — Saturation value constant
U_M — Maximum substrate utilization rate
R^2 — Correlation coefficient
F/M — Food to microorganism ratio

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