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Decolorization kinetics and characteristics of the azo dye acid red 18 in MSBR system at various HRTs and SRTs

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Abstract. The present work aimed to study the decolorization kinetics and characteristics of a selected azo dye under the influence of two key operational parameters including hydraulic retention time (HRT) and solid retention time (SRT). The decolorization efficiency and the two important criteria of *k* and normalized *k* (k/MLSS) were evaluated in lab-scale membrane sequencing batch reactors (MSBRs) at various HRTs of 48, 24 and 16 h (with constant SRT) and in addition, at various SRTs of infinity, 40 and 10 d (with constant HRT). According to the obtained results, both zero and first-order kinetics were properly fitted the decolorization profiles of the selected azo dye in all of the applied HRTs and SRTs. Increase of both HRT and SRT positively affected the decolorization efficiency. More MLSS concentrations corresponded to the lower HRTs and the higher SRTs resulted in higher decolorization rate constants (*k*). However, the effect of reducing the HRT was not compensated by increase of the MLSS concentration in order to reach higher decolorization efficiency. In addition, increase of the decolorization efficiency, as a consequence of the higher MLSS concentrations at longer SRTs, was restrained by decrease of the time-limited decolorization capability of biomass (represented by normalized *k*). Evaluation of both *k* and normalized *k* is suggested in order to have a more precise study on the decolorization kinetics and characteristics.

Keywords: decolorization kinetics and characteristics; hydraulic retention time; solid retention time; decolorization rate constant (*k*); normalized *k*

1. Introduction

Azo dyes are known as one of the most important groups of synthetic dye compounds applied in many industrial sectors. They account for more than 50% of all dyes used in textile, food, cosmetics and paper printing manufactories (Stolz 2001, van der Zee and Villaverde 2005, Saratale *et al*. 2011). Not only for the aesthetics problems and affecting the water transparency, but also due to the toxicity, carcinogenicity and mutagenicity of many azo dyes and their degradation products, removal of these pollutants from the effluents before release into the receiving bodies is a major environmental concern (van der Zee and Villaverde 2005, Atacag Erkurt 2010, Firmino *et al*. 2010, Saratale *et al*. 2011).

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As it is reported by many researchers, amongst the different physical, chemical and biological treatment methods, biological processes have been recognized as eco-friendly and cost-competitive options for treatment of azo dye-containing wastewaters (Farabegoli *et al*. 2010, Firmino *et al*. 2010, Spagni *et al*. 2010, Saratale *et al*. 2011, da Silva *et al*. 2012). According to van der Zee and Villaverde (2005), bacterial decolorization and degradation of the azo dye molecules generally arises through two successive stages including the reductive cleavage of the –N = N– bond/bonds of the dye molecule under anaerobic condition (as stage 1) and the aerobic degradation of the anaerobicaly produced aromatic compounds (as stage 2).

Due to complex structure of the dye molecules, the reductive cleavage reaction generally is known as a time-consuming process in the overall mineralization of an azo dye (dos Santos 2005). To gain a better insight into the mentioned problem, several studies have been performed on the anaerobic decolorization kinetics and characteristics of the azo dyes in biological treatment systems (Yu *et al*. 2001, dos Santos 2005, Lourenco *et al*. 2006, Dafale *et al*. 2008, Hakimelahi *et al*. 2012, Hosseini Koupaie *et al*. 2012, 2013). However, there is no or limited numbers of previous reports that have considered the effect of important operational parameters on the anaerobic decolorization kinetics and characteristics particularly from the viewpoint of decolorization rate constant (k) and its normalized value (normalized k = k/MLSS). Hydraulic retention time (HRT) and sludge retention time (SRT) are two key operational parameters in biological wastewater treatment systems since they can potentially change the sludge properties and affect the degree by which the pollution parameters are decreased (Ke and Junxin 2009, Pajoum Shariati *et al*. 2011).

In the present study, the main objective was to evaluate the influence of the two main operational parameters including HRT (in the range of 16-48 h) and SRT (in the range of 10 days to infinity) on the decolorization kinetics and characteristics of the selected azo dye C.I. Acid Red 18 (AR18). For this reason, three parallel membrane sequencing batch reactors (MSBRs) were run for two separate periods. In the first operational period (about 80 days), the effect of HRT and in second one (about 85 days), the effect of SRT on the decolorization kinetics and characteristics of the selected dye was studied. The color removal efficiency, the MLSS concentration, decolorization kinetics and the two important criteria, k and normalized k, were evaluated under the influence of different HRTs and SRTs.

2. Materials and methods

2.1 Synthetic wastewater composition

The synthetic dye-containing wastewater was prepared using tap water and composed of glucose (750 mg/L, corresponded to about 750 mg/L chemical oxygen demand (COD)), urea (80 mg/L), KH_2PO_4 (33.5 mg/L) and azo dye, AR18 at concentration of 100 mg/L (corresponded to about 60 mg/L COD). The commercial grade azo dye (AR18) was purchased from Alvan Sabet Company (Iran). The main characteristics of the dye AR18 are presented in Table 1.

2.2 Operating conditions

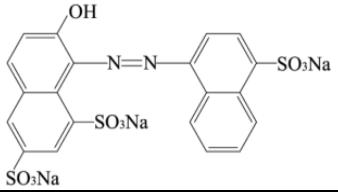
2.2.1 Reactors configuration

Three lab-scale membrane sequencing batch reactors (MSBR) were made up of Plexiglas, each

with an effective working volume of about 14 L. A 0.4 μm polyethylene Kubota flat sheet membrane with area of 0.11 m^2 was located in the lower part of each reactor. Fig. 1 shows a photograph of the MSBRs set-up.

The reactors were run according to a time-schedule consisted of four phases including feeding (the first 20 minutes of the treatment cycle), reaction (the subsequent alternating anaerobic and aerobic stages), drawing (the last 120 minutes of the aerobic stage) and idle (the last 10 minutes of the treatment cycle). For each reactor, a magnetic stirrer (rotation speed of 100 rpm) and two air pumps (total flow rate of 10 L/min) provided the anaerobic and the aerobic conditions,

Table 1 Chemical structure and general characteristics of the azo dye AR18

Parameter	Value
Color index	16255
CAS number	2611-82-7
Chemical formula	$C_{20}H_{11}N_2Na_3O_{10}S_3$
Purity	More than 98%
Molecular weight (g/mol)	604.5
λ_{max} (nm)	507
Molecular structure	

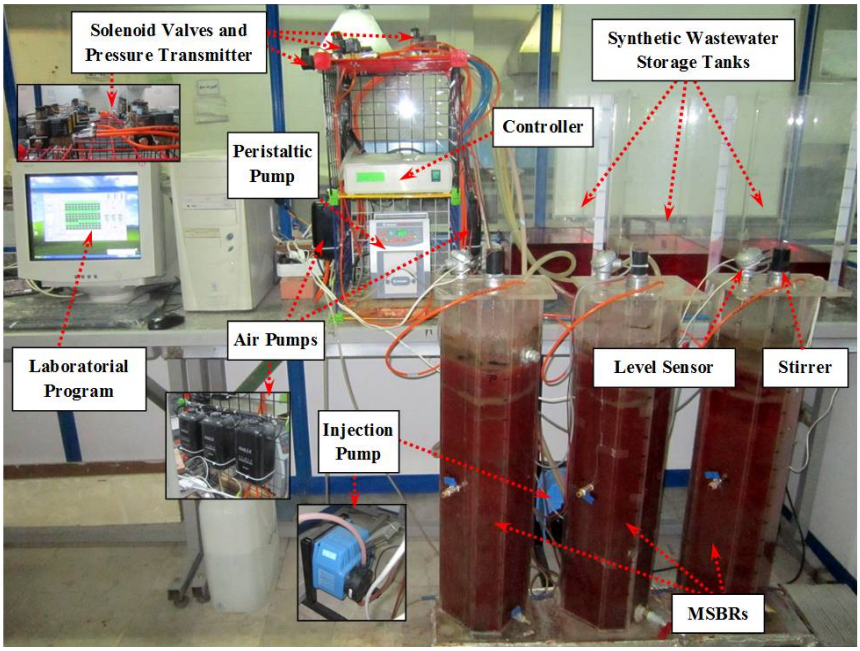


Fig. 1 Photograph of the used MSBR lab-scale set-up

respectively. Feeding of the reactors and the permeate withdrawal was done by the use of an injection pump (Etatron, DLS-MA, Italy) and a peristaltic pump (Heidolph, PD 5201, Germany), correspondingly. A flux of 36.4 L/m^{2}h with an intermittent suction mode of 260s on/40s off was kept constant during the extraction times.

For the first operational period, different HRTs were applied in three MSBRs by use of the varying numbers of the treatment cycles per day. In addition, for the second period, different SRTs were imposed to the reactors by daily purging of certain volumes of mixed liquor before permeate extraction through the membranes. Key operating parameters of the three MSBRs set-up are listed in Table 2. Operation of the cycles in three MSBRs was controlled using a laboratorial program and a controller which linked between the program and the set-up elements including solenoid valves, peristaltic and injection pumps, level sensors (for announcing in the case of any malfunction), air pumps and stirrers.

2.2.2 Inocula for the MSBRs

For each of the separate periods of the study, the MSBRs were inoculated with activated sludge taken from a local municipal wastewater treatment plant. After seeding, the sludge was allowed to acclimate to the synthetic wastewater by gradual increasing of the COD and dye concentrations until reaching the final desired values after about one month.

2.3 Analytical method

Mixed liquor suspended solids (MLSS) was measured according to the procedure outlined in Standard Methods (APHA *et al*. 1998). The maximum absorbance wavelength of the dye AR18

Table 2 Key operating parameters of MSBR systems

Operational period	Parameter	Reactors		
		MSBR1	MSBR2	MSBR3
Period 1 (Variable: HRT)	HRT (h)	48	24	16
	Treatment cycles per day	1	2	3
	Cycle time	24	12	8
	Time of anaerobic and aerobic stages	12/12	6/6	4/4
	OLR (mg COD/L.d)*	810	1620	2430
	SRT (d)	23	23	23
	Volume exchange ratio (VER)	0.5	0.5	0.5
Period 2 (Variable: SRT)	SRT (d)	infinity	40	10
	HRT (h)	24	24	24
	Treatment cycles per day	2	2	2
	Cycle time	12	12	12
	Time of anaerobic and aerobic stages	6/6	6/6	6/6
	OLR (mg COD/L.d)*	1620	1620	1620
	Volume exchange ratio (VER)	0.5	0.5	0.5

* Including the COD content of 750 mg glucose/L and100 mg dye/L per cycle

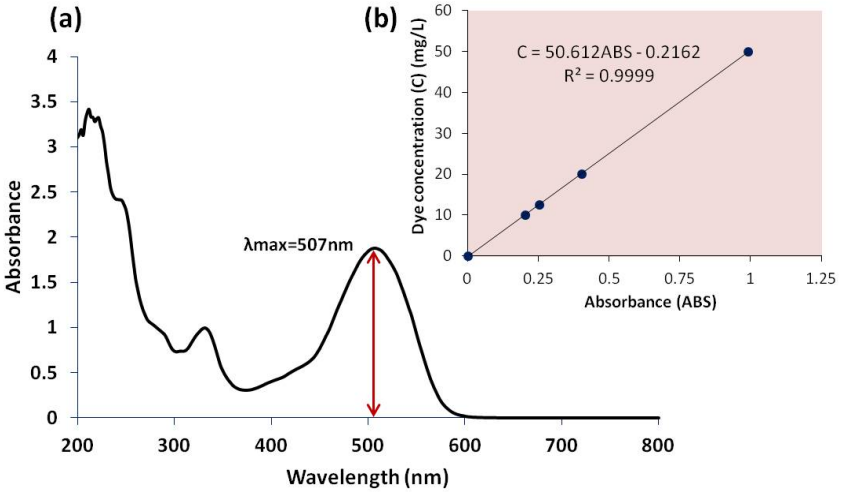


Fig. 2 (a) Determination of the maximum absorbance wavelength of the dye AR18 using UV-Vis spectrum analysis, (b) Absorbance-Concentration calibration curve of the dye

($\lambda_{max} = 507$), was determined according to the scanning pattern performed on HACH spectrophotometer DR/4000 (USA), with the background of tap water. The dye concentration of the samples and the decolorization efficiencies were determined by using the previously developed absorbance-concentration curve of the dye (Fig. 2). Before the analysis, the samples withdrawn from inside the reactors were centrifuged at 6000 rpm for 10 min.

2.4 Kinetic analysis of data

In order to analyze the AR18 decolorization profiles in three MSBRs either under the influence of HRT or SRT, a general kinetic model of the dye decolorization represented by Eq. (1) (Yu *et al*. 2001), was used

$$\frac{dC}{dt} = -kC^n \Rightarrow \left(\frac{C}{C_0}\right)^{1-n} = 1 - \frac{(1-n)k}{C_0^{1-n}} t \quad (n \neq 1) \tag{1}$$

where, C and n are the dye concentration (mg/L) and its partial reaction order, t is the time (h) and k is the decolorization rate constant (unit of k depends on the value of n).

Zero and first order reaction kinetic models (Eqs. (2)-(3)), derived from Eq. (1), were investigated to find out the most appropriate kinetic model for decolorization of AR18 in three reactors at different HRTs and SRTs.

$$C = -k_0 t + C_0 \ (zero\,order, n = 0) \tag{2}$$

$$C = C_0 e^{(-k_1 t)} \ (first\ order, n = 1) \tag{3}$$

As a supplementary measure, normalized k (Eq. (4)) was evaluated to provide a better understanding of the decolorization kinetics and characteristics. dos Santos (2005) and Dafale *et al*. (2008) have also utilized a similar criterion in their works.

$$\text{Normalized } k = \frac{k}{MLSS} \tag{4}$$

3. Results and discussion

3.1 Decolorization performance and the change of biomass concentration

As an example of the UV-Vis spectrum change through the reaction cycles, the absorbance spectrums of the samples taken from MSBR2 operating at steady state condition, HRT of 24 h and SRT of 23 d (first period of the study) are illustrated in Fig. 3. According to this figure, the main drop of the influent absorbance peak at 507 nm occurred at the end of the anaerobic stage. Moreover, the absorbance spectrums corresponded to the samples taken from inside the reactor at the end of the anaerobic and aerobic stages were closely matched with each other especially in the visible region. There was also a small decrease in the absorbance of the samples extracted from the membrane at the end of the aerobic stage, in comparison with those of the samples taken from inside the reactor at the end of the anaerobic and aerobic stages. This decrease could be attributed to some reasons including slight absorption of the dye molecules into the compact sludge cake attached over the membranes' surface and the dye degradation by the interior anoxic and anaerobic layers of the sludge cake. The absorbance spectrums of the other MSBRs at various HRTs and SRTs followed almost similar trends to what is shown in Fig. 3 (data not presented). As a result, it can be confirmed that in all applied HRTs and SRTs, most color removal occurred in the anaerobic stage and the contribution of aerobic stage to decolorization of the dye AR18 was insignificant.

Variation of the color removal efficiency and change of the biomass concentration during the first operational period in three MSBRs with different HRTs are illustrated in Fig. 4. According to Fig. 4(a), after about 35 days of the operation under the influence of different HRTs (with constant SRT of about 23 d) the MLSS concentrations in three reactors became relatively constant. The mean steady state MLSS concentrations at HRTs of 48, 24 and 16 h in reactors 1, 2 and 3 were

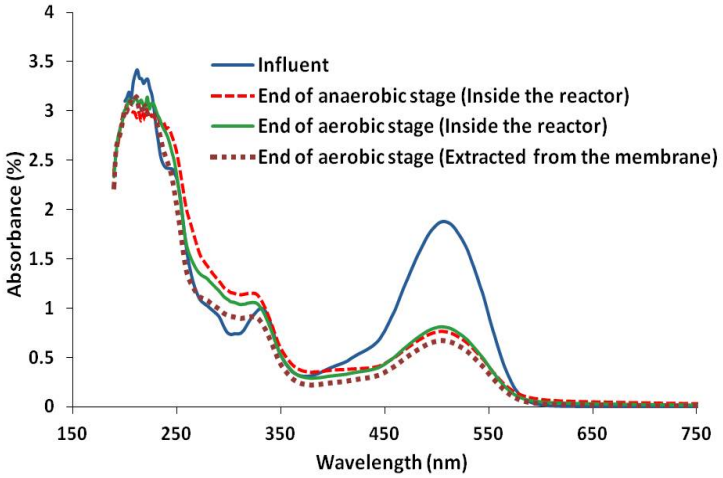
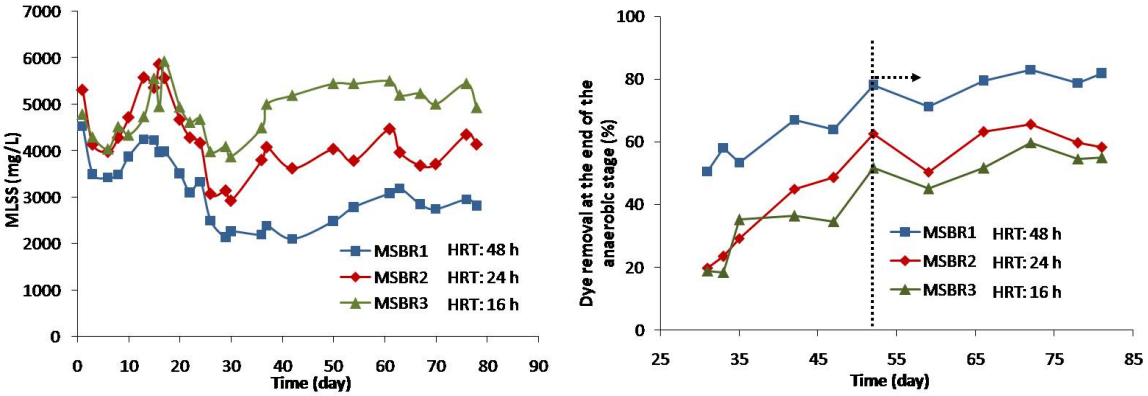


Fig. 3 UV–Vis spectrum of the influent and the samples taken through one anaerobic-aerobic reaction cycle in MSBR2 (HRT: 24 h, SRT: 23 d)

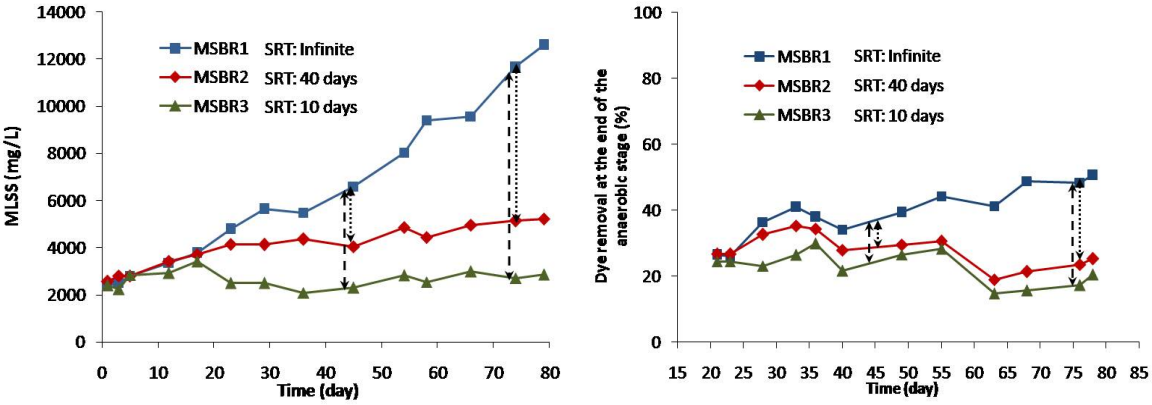


(a) MLSS concentration (b) Color removal efficiency

Fig. 4 Variation of MLSS concentration and color removal efficiency at different HRTs (SRT: 23 d)

about 2694 ± 354, 3966 ± 278 and 5176 ± 301 mg/L, respectively. Moreover, as seen in Fig. 4(b), the relative steady color removal was achieved in about 20 days after the first dye insertion into the reactors (showed with dashed arrow). The mean values of the color removal efficiency for the last 30 days of the operation were 78.7 ± 4.9%, 59.9 ± 5.3% and 52.9 ± 4.1% for HRTs of 48, 24 and 16 h, respectively. To sum up, it can be said that reducing the HRT, which was accompanied by considerable increase of the MLSS concentration, resulted in noticeable decrease of the AR18 decolorization efficiency. Therefore, it can be realized that the effect of reducing HRT was not compensated by increase of the MLSS concentration in order to reach higher decolorization efficiency. Furthermore, incomplete decolorization of AR18 (about 78%) even during the longest anaerobic stage in MSBR1 suggests that a longer treatment period is required to achieve almost hundred percent decolorization efficiency.

The decolorization efficiencies and the MLSS concentrations of three MSBRs during the second operational period (various SRTs and constant HRT of 24 h) are indicated in Fig. 5. As



(a) MLSS concentration (b) Color removal efficiency

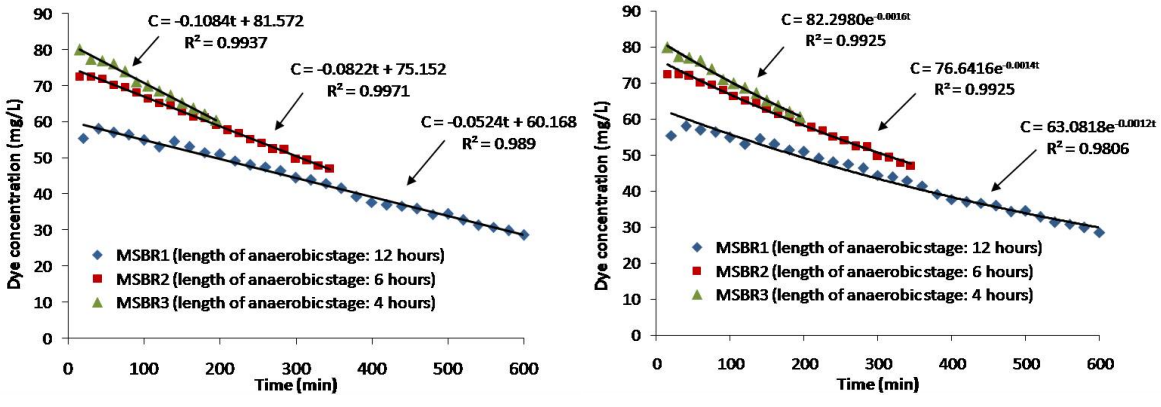
Fig. 5 Variation of MLSS concentration and color removal efficiency at different SRTs (HRT: 24 h)

seen in Fig. 5(a), after about 20 days of the operation, the MLSS concentration reached steady state condition in MSBRs 2 (4586 ± 462 mg/L) and MSBR3 (2585 ± 284 mg/L) with SRTs of 40 days and 10 days, respectively. However, in MSBR1, due to the SRT of infinity and no sludge withdrawal from the reactor, the biomass concentration increased continuously during the entire experimental period. In addition, according to Fig. 5(b), the average values of the color removal efficiency in MSBRs 1, 2 and 3 with SRTs of infinity, 40 days and 10 days, were 39.5 ± 8.0%, 27.7 ± 5.0% and 22.7 ± 4.9%, respectively. The trends of the MLSS concentration and the decolorization efficiency in three reactors reveals that the difference between decolorization efficiencies of the MSBR1 and the MSBR2 or the MSBR3 was increased over the time as well as the difference between their MLSS concentrations (shown with dashed arrows at the days 45 and 75, for example). Nevertheless, the decolorization efficiency in MSBR1 with the MLSS concentration of more than 9500 mg/L after the 65th day did not exceed 50%. This shows that in comparison with the MLSS concentration, the decolorization efficiency was increased with a comparatively lower rate in MSBR1.

Based on the statistical analysis using one-way ANOVA (95% confidence interval), the obtained *p* values for the mentioned responses (MLSS and decolorization efficiency) were less than 0.001, which are much smaller than 0.05. This indicates that both HRT and SRT significantly affected the biomass concentrations and the decolorization efficiencies of the MSBRs. Increasing the SRT from 10 days to infinity (with constant HRT of 24 h) resulted in increase of the MLSS concentration and consequently in increase of the color removal efficiency. On the other hand, increasing the HRT from 16 h to 48 h (with constant SRT of 23 days) enhanced the decolorization efficiencies, while the MLSS concentration was indirectly influenced by the HRT. The relation between the MLSS concentrations and the decolorization characteristics is discussed more in Section 3.3.

3.2 Regression analysis of the kinetic models for AR18 decolorization

Zero- and first-order kinetic models were examined to find out the most appropriate reaction order for the decolorization of AR18 under the influence of HRT and SRT as two main operational

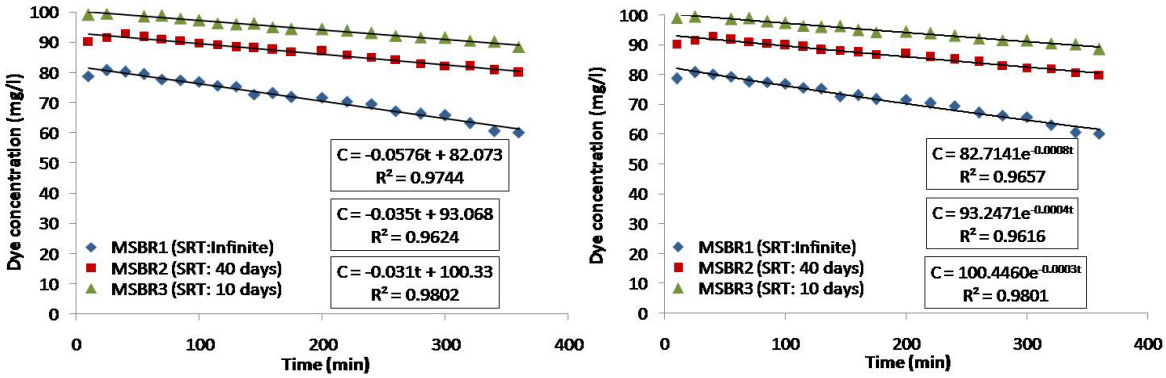


(a) Linear (b) Exponential

Fig. 6 Residual color profiles and the fitted functions for the decolorization of AR18 in three reactors at various HRTs (SRT: 23 d)

factors. The decolorization profiles of the monoazo dye AR18 for three HRTs of 48 h, 24 h and 16 *h* (with constant SRT of about 23 days) are illustrated in Fig. 6. The color removal profiles of three reactors with different SRTs of infinity, 40 days and, 10 days (with constant HRT of 24 h) are also presented in Fig. 7.

For a better comparison between the linear and exponential functions fitted to the AR18 decolorization profiles, the values of the obtained decolorization rates (k_0 and k_1) and the regression coefficients (R^2) are listed in Tables 3 and 4. As seen in Fig. 6 and Table 3, for MSBRs



(a) Linear (b) Exponential

Fig. 7 Residual color profiles and the fitted functions for the decolorization of AR18 in three reactors at various SRTs (HRT: 24 h)

Table 3 Kinetic constants obtained for anaerobic decolorization of the dye at different HRTs and constant SRT of 23 d

Kinetic order	Reactors		
	MSBR1 HRT = 48 h	MSBR2 HRT = 24 h	MSBR3 HRT = 16 h
Zero-order kinetic	k_0 = 0.052 R^2 = 0.989	k_0 = 0.082 R^2 = 0.997	k_0 = 0.108 R^2 = 0.993
First-order kinetic	k_1 = 0.0012 R^2 = 0.980	k_1 = 0.0014 R^2 = 0.992	k_1 = 0.0016 R^2 = 0.992

Table 4 Kinetic constants obtained for anaerobic decolorization of the dye at different SRTs and constant HRT of 24 h

Kinetic order	Reactors		
	MSBR1 SRT = Infinite	MSBR2 SRT = 40 d	MSBR3 SRT = 10 d
Zero-order kinetic	k_0 = 0.057 R^2 = 0.974	k_0 = 0.035 R^2 = 0.962	k_0 = 0.031 R^2 = 0.980
First-order kinetic	k_1 = 0.00081 R^2 = 0.966	k_1 = 0.00041 R^2 = 0.961	k_1 = 0.00033 R^2 = 0.980

with different HRTs, the obtained values of R^2 for the zero and first-order models were in the range of 0.980-0.997. Also according to Fig. 7 and Table 4, the R^2 values of the linear and exponential fitted functions to decolorization profiles of the MSBRs with different SRTs, were between 0.961-0.980. These results confirm that both zero and first-order kinetic models were appropriately fitted the color removal data of three reactors in all of the applied HRTs and SRTs. However, with regard to the very small values of the decolorization rate constant in first-order models (k_1), it can be said that the zero-order kinetic is sufficient for showing the trend of the color removal change by time, in all of the applied conditions.

Hosseini Koupaie *et al*. (2013) obtained different results for decolorization kinetics of the same dye (AR18) with the same concentration (100 mg/L) in anaerobic/aerobic fixed-bed sequencing batch biofilm reactor. They reported that the first-order kinetic was the best decolorization model for their treatment system. Difference between the results obtained in the present study and reported by the study group of Hosseini Koupaei *et al*. (2013) is most likely due to the different biological treatment procedures and experimental conditions applied in the two studies (values of HRT and SRT, influent COD concentration, introduction of packing media or microfiltration membrane, etc).

According to the literature information described by Lourenco *et al*. (2006), different kinetic orders including zero-, first- and even half-order kinetic have been previously reported for the decolorization of mono-azo dyes. They attributed these contradictory kinetic results to the unlike experimental, operational and environmental conditions applied in different research works (Lourenco *et al*. 2006).

3.3 Decolorization characteristics from the viewpoint of k and normalized k values

In order to study the decolorization characteristics of the dye AR18 from the viewpoint of k and normalized k values under the influence of various HRTs and SRTs, the zero-order decolorization rate constants (k_0) were utilized in this section.

Data of the values of k_0 (zero-order decolorization rate constants) and the MLSS concentrations of three MSBRs with different HRTs are presented in Fig. 8. As can be realized from this figure, by decreasing the HRT from 48 h to 16 h, the decolorization rate constants (values of k_0) were increased. Decreasing the HRT was also accompanied by increase of the MLSS concentration. Therefore, it can be stated that higher MLSS concentrations corresponded to lower HRTs resulted in faster dye concentration descending rates. For a more precise evaluation of the decolorization behaviors in three MSBRs, the k_0 values were normalized by the corresponding MLSS concentrations (normalized $k_0 = k_0$/MLSS) and represented in Fig. 8. Based on the obtained results, the difference between the values of the normalized k_0 in three MSBRs (0.193×10^{-4}, 0.207×10^{-4} and 0.209×10^{-4} mg dye/mg MLSS.min for MSBRs 1, 2 and 3, respectively) was unremarkable. Despite the difference between the values of k_0, after considering the relative similar values of the normalized k_0, it can be concluded that the time-limited decolorization ability of biomass (represented by normalized k_0) in three MSBRs with different HRTs of 48 h, 24 h and 16 h was almost the same.

The data of the decolorization rate constants (k_0), the MLSS concentrations and the equivalent normalized k_0 values (k_0/MLSS) of three reactors with different SRTs are illustrated in Fig. 9. According to the presented data, it can be found that the reduction in the SRT (from infinity in MSBR1 to 10 days in MSBR3, caused a relatively small decrease in k_0 values. Furthermore, decreasing the SRT resulted in considerable decrease of the MLSS concentration. Therefore, the

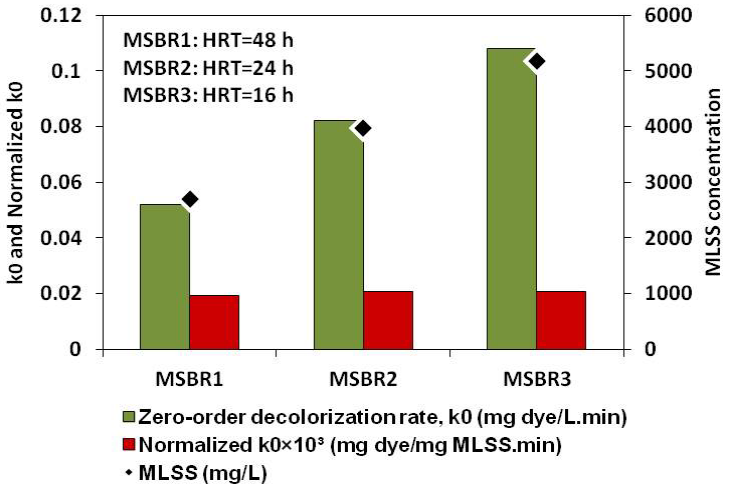


Fig. 8 Trends of the k_0, normalized k_0, and MLSS concentrations in MSBRs with different HRTs

variation of the MLSS concentrations by changing the SRT is the most probable reason for the seen trend of the k_0 values. In opposition to what was observed about the values of k_0, the normalized k_0 values were noticeably increased with decreasing the SRT (0.05×10^{-4}, 0.08×10^{-4} and 0.12×10^{-4} mg dye/mg MLSS.min for MSBRs 1, 2 and 3, respectively).

As a reason for these unlike trends of the k_0 and normalized k_0, it might be stated that employing of high sludge ages in MSBRs 1 and 2 facilitated the growth and enrichment of slow-growing microorganisms, which, according to some researchers have higher capability for degradation of organic pollutants such as dye molecules (Brik *et al*. 2006, Masse′ *et al*. 2006, Zhao *et al*. 2009). However, based on the results obtained in this study, it seems that the lower biomass growth rate of such microbial populations has resulted in slower cellular metabolic activity.

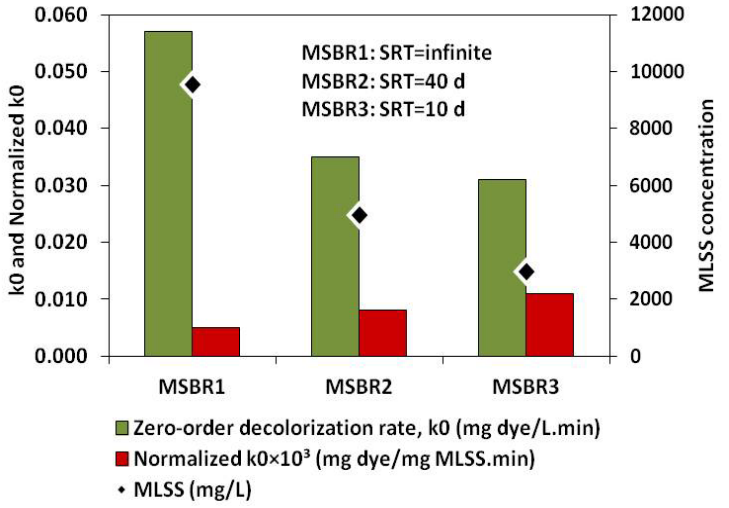


Fig. 9 Trends of the k_0, normalized k_0, and MLSS concentrations in MSBRs with different SRTs

Consequently, by increasing the SRT, the decolorization rate (k_0) slightly increased as a result of the higher MLSS concentration, while the time-limited decolorization capability of biomass (represented by normalized k_0) was significantly decreased. This considerable decrease of the normalized k values at high SRTs of 40 days and infinity, was the main reason for the relative low decolorization efficiencies obtained in MSBRs 1 and 2 (data presented in Fig. 5).

4. Conclusions

The important conclusions of this research are as follows:

- Increasing both HRT and SRT resulted in enhancement of the AR18 decolorization efficiency.
- By increase of the MLSS concentration due to decreasing the HRT and increasing the SRT, the values of the decolorization rate constants (k) were elevated.
- The values of the normalized k, as a measure of the time-limited decolorization capability of biomass, were considerably decreased by increasing the SRT, while they were not much influenced by changing the HRT.
- The effect of reducing HRT was not compensated by increase of the MLSS concentration in order to reach higher decolorization efficiency.
- The effect of higher MLSS concentrations on increase of the decolorization efficiency at longer SRTs was controlled by decrease of the normalized k.

Based on the results of the present work, both k and normalized k are suggested to be evaluated as two important criteria in color removal studies.

Acknowledgments

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