

1 **Performances of Anoxic- Aerobic Membrane Bioreactors for The**  
2 **Treatment of Real Textile Wastewater**  
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10 **ABSTRACT**

11 Wastewater from textile industry is considered one of the major environmental challenges due to the  
12 large volume of highly colored, polluted and toxic effluent. This study investigated the treatability of  
13 real textile wastewater by pilot-scale anoxic-aerobic Membrane Bioreactor (MBR) system without  
14 sludge wasting for an operation period of 100 days. The proposed system was investigated under  
15 different Internal Recycle (IR) ratios and the impact of IR ratio on Total Organic Carbon (TOC), Total  
16 Nitrogen (TN) and Color removals were examined. Under IR ratios between anoxic and aerobic tanks  
17 of 0.0, 0.5 and 2.0, the respective average removal efficiency of TN was 20.9%, 53.4% and 71.7%,  
18 whereas average color removal of 81%, 85% and 88%, respectively was noted. The results indicated  
19 that increase of recycle ratio from 0.5 to 2.0 enhanced TN removal to about 71% and color removal to  
20 above 85%. The IR between anoxic and aerobic tanks has a significant role in TN and color removal  
21 due to its effect on the development of bacterial communities. On the other hand, the results indicate  
22 over 93% TOC removal, which was independent of IR ratio.

23

24

25 **Keywords:** Anoxic-Aerobic Bioreactor, de-colorization, Internal Recycle, Textile Wastewater.

## 26 1 INTRODUCTION

27 Besides the massive water consumption by the textile industry, the wastewater from this industry has  
28 become a major environmental issue due to its highly polluted and toxic properties (Majewska-Nowak,  
29 2010; Makertihartha *et al.*, 2017). De-sizing, printing, dyeing, sieving scouring, washing, rinsing,  
30 bleaching, mercerizing, carbonization, finishing and dyeing are the main processes in textile industry.  
31 The water usage depends on fabric type and generated waste streams are loaded with high  
32 concentration of residual dye from the dyeing processes (Dilaver *et al.*, 2018). As a consequence of  
33 progressively strict restriction on the pollutant content of industrial wastewater, it is urgent to remove  
34 hazardous materials from industrial effluents before discharge it to ecosystems (Yin *et al.*, 2018).  
35 Severe environmental problems are presented by colored textile effluents as many kinds of synthetic  
36 dyes have been used in textile industry. Theses effluents contain various chemical compositions with  
37 high concentration of inorganic and organic compounds. It is reported that more than 100,000 usable  
38 textile dyes are available in market, and about 700,000-1,000,000 tons of pigment are manufactured  
39 annually. While 280,000 tons of dye are released from textile manufactory to the environment every  
40 year via effluents produced (Samaei, Gato-Trinidad and Altaee, 2018).  
41 The highly colored wastewater has an impact on photosynthetic function in plant. In addition, colored  
42 wastewater harmfully affects aquatic life because of low sun light penetration and oxygen utilization.  
43 Furthermore, textile wastewater contains metals and chloride that may be harmful to particular aquatic  
44 species (Holkar *et al.*, 2016). Also, dye degradation products such as aromatic amines have a toxic and  
45 carcinogenic effects on the ecosystem (Alventosa-Delara *et al.*, 2014). Precisely, discharge of highly  
46 polluted effluents without decent and sufficient treatment has adversely affected the water bodies, soil  
47 and ecosystems (Oliveira *et al.*, 2018).  
48 Different physicochemical treatment methods have been proposed and applied for the treatment of  
49 textile wastewater. These include; ion exchange, adsorption, oxidation, coagulation, and membrane  
50 separation. Nonetheless, these options are obstructed by technical and economic barriers such as

51 production of harmful byproduct, formation of massive amount of sludge (disposal and handling  
52 problems) and high operating/energy costs. It should be noted that most of conventional treatment  
53 methods are greatly expensive and need huge amount of energy and dealing with chemicals (Siddique  
54 *et al.*, 2017). Among textile wastewater treatment options, biological methods are considered a  
55 promising technology to deal with colored wastewater. The biological treatment has many advantages  
56 such as eco-friendly, less sludge production, full mineralization, cost effective and less consumption of  
57 water (Holkar *et al.*, 2016).

58 The anoxic-aerobic system is considered a proper selection for obtaining nitrogen removals by  
59 denitrification and nitrification processes. Integrated anoxic-aerobic treatment systems with membrane  
60 processes enhance development of slow-growing bacteria, such as ammonia oxidizing bacteria with  
61 capacity to degrade refractory compounds and other organic micropollutants (OPMs) (Su *et al.*, 2014;  
62 Ma *et al.*, 2018). The combination of anoxic-aerobic and the Internal recycle (IR) from aerobic to  
63 anoxic leads to following: 1)enhanced mixing/mass transfer, 2)dilution of media, 3)transport of  
64 dissolved oxygen from aerobic tank and, 4) supply of nitrate (Li *et al.*, 2012; Phan *et al.*, 2016).  
65 Moreover, IR between anoxic and aerobic tanks may form conditions that promote development of  
66 specific bacterial community distributed between two redox environments, which serve the core  
67 function of the integrated anoxic-aerobic MBR systems (Luo *et al.*, 2015; Zhang *et al.*, 2018; Xue *et*  
68 *al.*, 2019).

69 Membrane bioreactor (MBR) are the combination of membrane technology and biological treatment.  
70 MBR has been applied in many industrial wastewater treatment systems (Arslan *et al.*, 2016) The main  
71 benefits of using MBR for textile effluent treatments are high degree of carbon, nitrogen and color  
72 removal, and complete solid removal which results into highly quality of treated wastewater for reuse  
73 application (Luong *et al.*, 2016).

74 Removal of azo dye from wastewater with biological method is a complicated process, including de-  
75 colorization and mineralization. However, there is little understanding of the functional microbial

76 community involved in the whole dye degradation process. Studies on the combination of different  
77 redox potentials in hybrid systems for color removal are still scarce (Zhu *et al.*, 2018).  
78 In this study, dying process wastewaters from woolen textile sector, which is an important waste source  
79 was treated by anoxic-aerobic membrane bioreactor. In line with the aforementioned research gaps, this  
80 this study aimed to determine the extent of color removal under various redox conditions common to  
81 textile wastewater treatment processes. Integrated anoxic-aerobic MBR was operated to treat real  
82 textile wastewater under different redox conditions. Carbon, nitrogen and color removal was assessed  
83 in each reactor to determine the effect of different redox regimes. The results of this study point out the  
84 importance of redox environment on color removal.

## 85 2 MATERIALS AND METHODS

### 86 2.1 Material

87  
88 The textile wastewater was collected from woolen textile dying factory located in Istanbul-Turkey. The  
89 collected wastewater was kept at 4<sup>0</sup> C to eliminate biological growth. A summary of the influent  
90 characteristics is shown in table 1.

91 **Table 1.** Characteristics of the Textiles wastewater used in this study

Parameter	value
COD (mg/l)	750
BOD (mg/l)	350
TOC (mg/l)	200
TN (mg/l)	47
PH	7.82
Color (ADMI unit) *	480
Conductivity (ms)	3680
Alkalinity (mg/l)	435
Chloride (mg/l)	89.84
Nitrate -N (mg/l)	8
Sulfate (mg/l)	394.6
Ammonia-N (mg/l)	35
TKN (mg/l)	39
Organic N (mg/L)	4
TP (mg/l)	0.161

92 \* American Dye Manufacturer's Institute (ADMI) color unit.

93

94 2.2 *Experimental setup and procedure*

95 A batch mode anoxic-aerobic ceramic MBR of 15 L working volume in each reactor was used in this

96 study (Figure 1). The membrane was composed of flat sheet ceramic membrane module (Chembrane -

97 Denmark) with a nominal pore size of 0.1  $\mu\text{m}$  and an effective membrane surface area of 0.528  $\text{m}^2$

98 submerged in an installed aside stream tank. A shift vacuumed/ pressured pump (BEM20, Priom

99 Teknik, Turkey) was operated using cycles of 10 min of suction followed by 2 min of backwash to

100 prevent clogging on the membrane module. A designated volume of water was recycled from aerobic

101 tank to anoxic tank. Subsequently, the internal recirculation (IR) was calculated as the ratio of volume

102 of the recycled water to the volume of feed. The aerobic reactor and MBR tanks were continuously

103 aerated via air nozzles connected to an air pump. The mixed liquor of the anoxic tank was stirred by an

104 overhead lab mixer (Mtops MS 5010, Kore) to ensure homogeneous distribution in tank. The system

105 was operated at constant flux of 25 LMH and total hydraulic retention time (HRT) of 24 h (12 h for

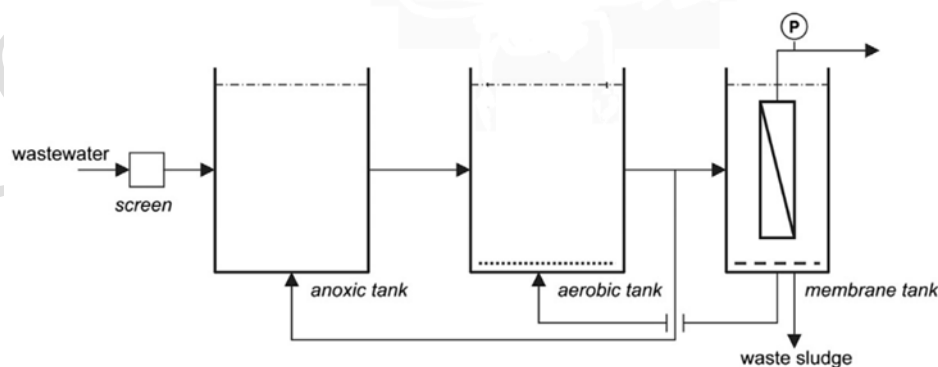
106 anoxic and 12 h aerobic), details of the operating cycles are specified in **Table 2**. Aeration in each

107 reactor was controlled to achieve the desired redox conditions. The dissolved oxygen concentration

108 (DO) in aerobic reactor was maintained at 3-5  $\text{mg L}^{-1}$  while DO for anoxic reactor was kept at around

109 0.12  $\text{mg L}^{-1}$ . The Oxidation-reduction potential (ORP) for the anoxic and aerobic reactors were -

110 115 $\pm$ 25 mV and 130 $\pm$ 16 mV, respectively.



111

112

**Figure 1.** Schematic Diagram of the Anoxic-Aerobic MBR system.

113 *2.3 MBR operation condition*

114 The anoxic-aerobic system was initially seeded with activated sludge from a biological nutrient  
115 industrial wastewater treatment plant at Dilavos-Turkey. It was operated for a total 100 days, for the  
116 initial 33 days the system was operated without internal recycle (IR=0.0) and no sludge withdrawal.  
117 This condition allowed sludge acclimatization and stabilization for TOC, color and TN removal.  
118 Subsequent to this, the system was operated with IR = 0.5 for 35 days to investigate the relation  
119 between IR, carbon, nitrogen and color removal. In the last 35 days, the system was operated with  
120 IR=2.0.

121 **Table 2.** Operating cycles for anoxic-aerobic MBR.

Activity	Anoxic	Aerobic
Cycle duration (h)	12	12
Filling (min)	0.5	7
Reaction time (h)	11.125	11
Settling time (min)	45	45
Emptying time (min)	7	7

122

123 *2.3 Analytical methods*

124 Samples for analyses were collected from influent, anoxic, aerobic and MBR tanks. Chemical oxygen  
125 demand (COD) was measured according to Standard methods(American Public Health Association  
126 (APHA), 1997). The TOC and TN were analyzed using (IL550 and IL530 TOC-TN Analyze, Hach,  
127 Germany), Ammonia was measured using Ammonia-Selective Electrode (Standard method: 4500-NH<sub>3</sub>  
128 D), Thermo Orion model 710 A+ meter and Orion 9512 electrode (Thermo Fisher Scientific, USA).  
129 Nitrate was measured using Nitrate Electrode (Standard method: 4500-NO<sub>3</sub><sup>-</sup> D), the 9707BNWP  
130 Nitrate Combination Electrode was used combatable with Thermo Orion 710 A+ meter (Federation,  
131 1999). Color was measured according to Standard method: 2120 F by using DR 5000,

132 Spectrophotometer, Germany. The analytical procedure was repeated three times and the average data  
133 was reported. All chemicals used in the experiments were of analytical grade.

### 134 **3 RESULT AND DISSECTION**

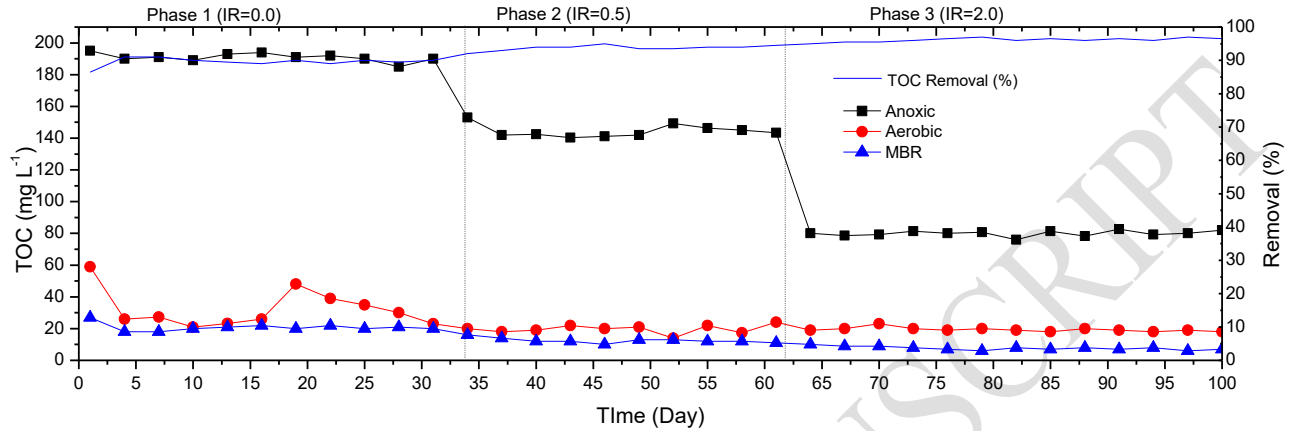
135 This research study was accomplished with real textile wastewater in a pilot-scale plant operated in a  
136 batch mode, using anoxic-aerobic membrane bioreactor at 24 h HRT. the observed concentration of  
137 mixed liquor suspended solids (MLSS) was 5.2 - 5.8 g L<sup>-1</sup> for aerobic tank and 2.5 - 2.7 g L<sup>-1</sup> for the  
138 anoxic tank. The MLVSS/MLSS ratios were stable at 0.68 ± 0.04 and 0.71 ± 0.03 for the anoxic and  
139 the aerobic tanks, respectively. The Mixed liquor pH for aerobic tank was stable at 8.15 ± 0.65 whereas  
140 that of anoxic tank was 7.25 ± 0.05.

#### 141 *3.1 Organic matter removal (TOC)*

142  
143 In this study, TOC is used as indicator of organic matter contamination. TOC analysis is specific to  
144 organic compounds and theoretically measures all the covalently bonded carbon in water. In all the  
145 countries, the analysis of TOC is recognized as the most suitable index for the control of civil and  
146 industrial wastes (Visco, Campanella and Nobili, 2005). The TOC removal of the integrated anoxic-  
147 aerobic MBR during the 100 days operation time is presented in figure 2. As can be seen, average TOC  
148 removal of 93% (6 - 22 mg/L residual TOC) was observed in the system, far below the Turkish  
149 national discharge standard of effluent from Textile industry (COD < 200 mg L<sup>-1</sup>). The results also  
150 indicate potential of wastewater treatment for reuse applications, if appropriate further refining such as  
151 nanofiltration and reverse osmosis are applied. Notably, the aerobic tank served as an efficient  
152 treatment step for TOC with a small variation in TOC removal with IR ratios due to the dilution  
153 effects. In addition, effluent TOC concentration was not affected by increase in IR ratio. This indicates  
154 stability of the system for TOC removal. The results of this study are in agreement with those reported  
155 by Sun et al, in which over 85% removals of organic matter were recorded for treatment of textile  
156 auxiliary's wastewater under IR of 0.5, 1.5 and 2.5. Moreover, in the same study, Sun et al observed



157 that effluent COD concentration was not affected when the recycle ratio was increased from about 0.5  
158 to 2.5. (Sun *et al.*, 2015).



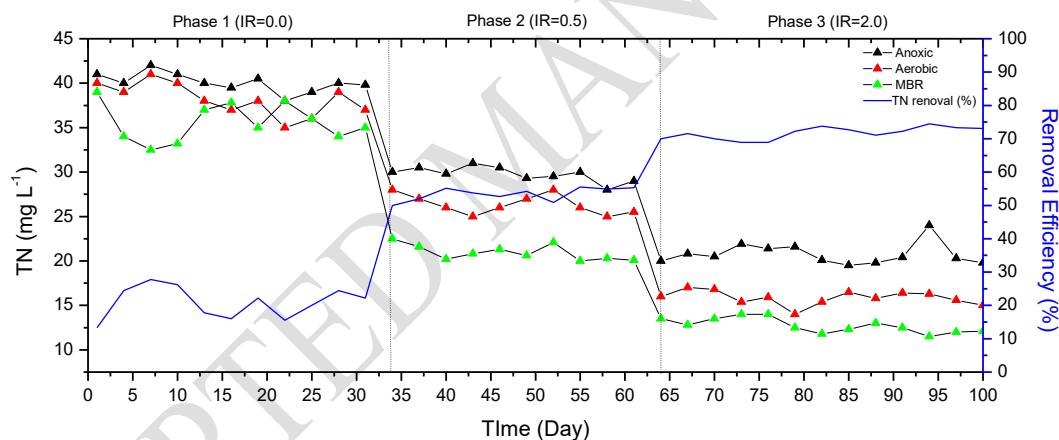
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160 **Figure 2.** TOC concentration and removal efficiency over the entire operation period of the anoxic-  
161 aerobic MBR.

### 162 3.2 Nitrogen removal

163 An overview of TN concentration and removal efficiency for the entire operation period is given in  
164 Figure 3. The 100-day operation period was divided into three phases according to the IR ratio used.  
165 Phase one lasted for 31 days with zero IR. Phase two lasted 35 days with 0.5 IR ratio and phase three  
166 lasted 35 days with 2.0 IR ratio. To gain a better insight into nitrogen removal, analyses were done for  
167 TN,  $\text{NH}_3\text{-N}$  and  $\text{NO}_3\text{-N}$  concentrations in all the three phases. Stable performance of  $\text{NH}_3\text{-N}$  and TN  
168 removal from anoxic-aerobic MBR was observed 7 days after start of operation. As shown in Figure 4,  
169 nitrification occurred in the aerobic reactor and the effluent  $\text{NH}_3\text{-N}$  concentration was  $7 \text{ mg L}^{-1}$ , which  
170 demonstrates that 94% of  $\text{NH}_3\text{-N}$  was oxidized. During the first Phase, with an average influent TN  
171 concentration of  $35 \text{ mg L}^{-1}\text{-N}$ , removal efficiency of about 20% was recorded. TN concentration in the  
172 effluent from anoxic reactor, aerobic reactor and MBR tank were  $40 \pm 2$ ,  $38 \pm 2$  and  $36 \pm 2 \text{ mg L}^{-1}$ ,  
173 respectively, indicating that TN was mainly removed in anoxic reactor as  $\text{NO}_3\text{-N}$ . To improve TN  
174 removal, IR was increased from 0.0 to 0.5 which ensured increased nitrate load to the anoxic reactor in  
175 Phase 2(32–61 days). When  $\text{NH}_3\text{-N}$  concentration of anoxic reactor was only  $23 \pm 2 \text{ mg L}^{-1}$  because of

176 the dilution of the raw wastewater, the effluent concentration from recycled mixed liquor from aerobic  
 177 reactor was  $3 \text{ mg L}^{-1}$ . During stable operation, the final effluent concentration of  $\text{NH}_3\text{-N}$  in the aerobic  
 178 reactor remained less than  $2 \text{ mg L}^{-1}$ , while the  $\text{NO}_3^- \text{-N}$  concentration averaged  $20 \text{ mg L}^{-1}$ . TN  
 179 concentration in the effluent from anoxic reactor, aerobic reactor and MBR tank were  $28 \pm 2$ ,  $26 \pm 3$  and  
 180  $20 \pm 4 \text{ mg L}^{-1}$ , respectively. Despite increasing IR ratio to 0.5 in phase 2, the observed TN removal  
 181 efficiency at this phase was around 53%. Finally, increasing the IR ratio to around 2.0 in phase 3 (62<sup>nd</sup>–  
 182 100<sup>th</sup> day) resulted into effluent  $\text{NH}_3\text{-N}$  concentration in the anoxic-aerobic MBR of less than  $1 \text{ mg/L}$   
 183 and the TN concentration in the final effluent of  $12 \pm 3 \text{ mg L}^{-1}$ . This suggests that TN mainly reduced in  
 184 anoxic and aerobic reactors due to the removal of recycled  $\text{NO}_3\text{-N}$ . The results indicated that increase  
 185 of recycle ratio from 0.5 to 2.0 enhanced the percentage of TN removal to about 71%.

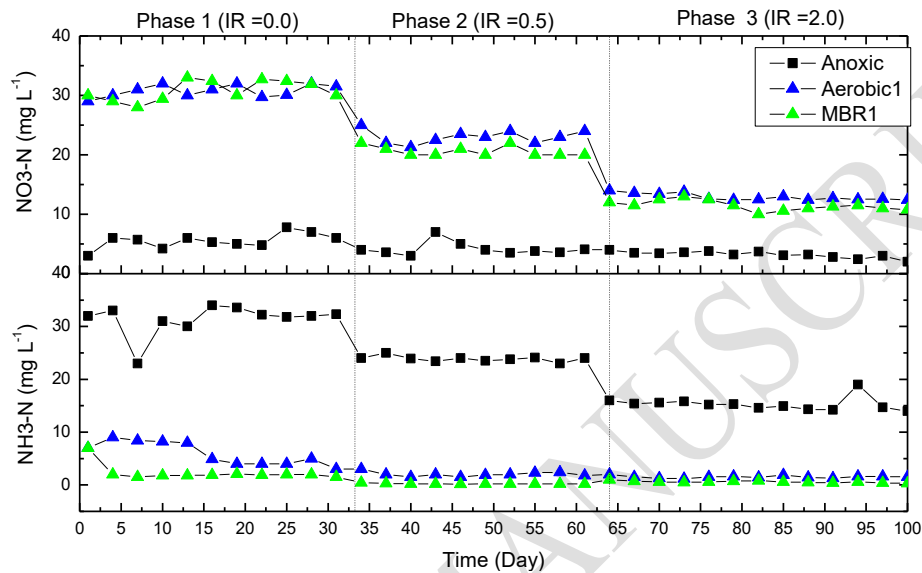


186

187 **Figure 3.** TN concentration and removal efficiency over the entire operation period of the anoxic-  
 188 aerobic MBR.

189 It is well-known that for a pre-denitrification system, the internal mixed liquor recycle flow from the  
 190 aerobic to the anoxic zones plays an important role that affects the TN removal efficiency. Tan and Ng  
 191 reported TN removal efficiencies of domestic wastewater as 57%, 74%, 72% and 61% for recycle ratio  
 192 of 1, 3, 5 and 10, respectively (Tan and Ng, 2008). Similarly, Baeza et al. reported TN removal  
 193 efficiencies of 67%, 72% and 80% for the recycle ratio of 0, 2 and 5, respectively (Baeza, Gabriel and  
 194 Lafuente, 2004). Under anoxic conditions, performance of denitrification could be enhanced by the

195 availability of easily biodegradable organic matter, enough HRT and low dissolved oxygen (DO)  
196 concentration in the recycle flow. In the present study, HRT of 12 h was maintained in the anoxic  
197 reactor for all the three phases, which is sufficient for anoxic denitrification (Kim *et al.*, 2008)



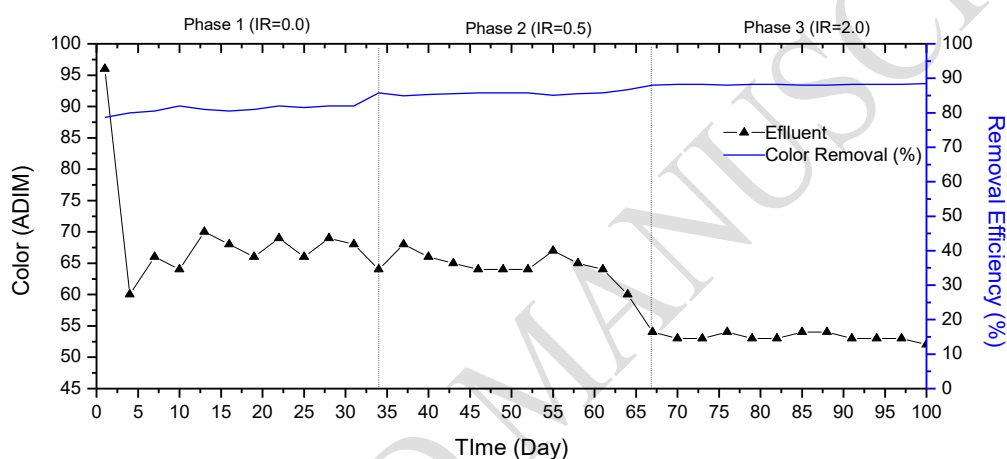
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199 **Figure 4.**  $\text{NO}_3\text{-N}$  and  $\text{NH}_3\text{-N}$  concentration over the entire operation period of the anoxic-aerobic  
200 MBR.

### 201 3.3 Color removal

202 The differences in the bacterial communities developed under conditions with/without IR not only  
203 explain the variations in the nutrient removal performance by the system (section 3.2) but also provide  
204 insights into the variation in color removal under different redox conditions as showed in figure 5. The  
205 average color removal efficiencies in the integrated anoxic-aerobic MBR system were 81%, 85%, and  
206 88% for (IR=0.0), (IR=0.5), and (IR=2.0), respectively, resulting in high average system efficiencies.  
207 The color removal ranged from 78% to 82% for IR ratio of 0.0. Increasing the IR ratio to 0.5 and 2.0  
208 resulted into 3% and 6%, respective enhancement in cooler removal. The enhancement in color  
209 removal under various IR between anoxic and aerobic reactors could be attributed to the development  
210 of an environment suitable for bacterial community shared between these two redox regimes, and  
211 possible excretion of diverse enzymes responsible for the core function of the integrated anoxic-aerobic  
212 MBR system (Phan *et al.*, 2016; Mallick and Chakraborty, 2019). In addition, in anoxic treatment, due

213 to the low level of oxygen present in the system, an alternate electron acceptor is required. The azo  
 214 bond can act as this electron acceptor, which results in decolorization owing to cleavage of the azo  
 215 bond. integrated anoxic-aerobic treatment processes can remove reactive azo dye color from  
 216 wastewater more effectively than traditional aerobic processes. A biomass can be developed that is  
 217 viable and effective in both anoxic and aerobic environments. The anoxic phase exhibits both a higher  
 218 percentage color removal and a greater rate of color removal than the aerobic phase. (Khehra *et al.*,  
 219 2006; Rass-Hansen *et al.*, 2007; Venkata Mohan, Rao and Sarma, 2007)



220

221 **Figure 5.** Color removal efficiency over the entire operation period of the anoxic-aerobic MBR

222 **4 CONCLUSION**

223

224 Simultaneous organic matter, TN and color removal in textile wastewater treatment by an integrated  
 225 anoxic-aerobic MBR was examined. The study demonstrates that a system comprising of an anoxic-  
 226 aerobic bioreactor and ceramic membrane is effective for textile wastewater treatment. Organic matter  
 227 removal was generally higher than 93 %. The use of an anoxic reactor was effective in TN and color  
 228 removals which reached values higher than 70% and 85%, respectively. The IR between anoxic and  
 229 aerobic tanks demonstrated a significant role in TN and color removal due to development of bacterial  
 230 community under various IR. Nevertheless, the TOC concentration was not affected by IR ratio.  
 231 Further research is needed to investigate the bacterial communities responsible for color degradation

232 under different IR ratios, in addition, investigation of IR effect on elimination of generated aromatic  
233 amines is required.

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