



## Decolorization of AR18 dye solution by electrocoagulation: sludge production and electrode loss in different current densities

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### ABSTRACT

A lab-scale electrocoagulation batch reactor was used to remove acid red 18 as an azo dye by use of aluminum electrodes. The effects of different parameters including initial pH, current density, reaction time, initial dye concentration, distances between electrodes, and type of the electrolyte solution on the efficiency of EC process were investigated. The influence of the current densities on consumed anode and produced sludge was also studied. Optimum conditions were obtained at pH 4. Removal percentage of the dye was significantly increased with increasing current density until reaching to critical point of 26 mA/cm<sup>2</sup>. Anode mass reduction was increased from 202 mg at current densities of 26 mA/cm<sup>2</sup> to 331 mg at 42 mA/cm<sup>2</sup>. Moreover, mass of produced sludge was increased from 605.85 to 1,060 mg with a net mass of 400-mg sludge production. The shorter distances between electrodes resulted in higher decolorization. Type of the electrolyte solution was an important parameter affecting the amount of the consumed electrolyte or energy required for decolorization.

*Keywords:* Aluminum electrodes; Azo dyes; Decolorization; Electrocoagulation process; Textile wastewater

### 1. Introduction

Synthetic dyes are commonly used in many industrial processes and can be one of the major causes of water pollution worldwide. Due to their good solubility in water, they may be found in trace quantities in various industrial effluents of ink, plastic, paint, and textile manufacturing industries [1,2]. Among these, the textile industries are known to produce a wide variety of dye containing effluents. Azo dyes that

account for 50% of all commercial dyes are very toxic to human and the environment [3].

Without proper treatment, the textile dyes can remain in the environment for a longer period of time [4]. Proper removal of dyes from wastewater is a major environmental concern because they can lead to severe contamination of the surface and ground waters in the vicinity of dyeing industries. Different treatment techniques have been applied to overwhelm the problem [5]. Different biological processes [6–8], adsorption processes [5,9,10], catalytic oxidation [11],

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advanced oxidation processes [12,13], coagulation with chitosan [14], photocatalysis, and membrane processes [15] are the most widely used methods to remove dye compounds from wastewater. Due to the complexity of the dyes' aromatic molecular structure and stability against light and biological degradation, the conventional biological and physical–chemical processes cannot efficiently remove dyes from textile wastewater [16,17].

Electrocoagulation (EC) process, a simple and efficient method, has been used for the treatment of many types of dye containing effluents. Compared with traditional flocculation and coagulation, in theory, EC process has the capability of removing small colloidal particles, which have a larger probability of being coagulated because of the electrical charge. Usage of high amount of coagulants can be avoided, due to direct generation of coagulation by electro-oxidation of an anode in EC process. EC equipment is simple and easy to operate. Relatively low reaction time and small quantities of sludge production are other advantages of the process [3].

Acid red 18 (AR18), an azo dye, is widely used in textile industry. The structure and characteristics of AR18 are presented in Table 1. For the experiments, aluminum electrodes were used in a batch system. Dye removal percentages, amount of produced sludge, and lost mass of the electrode were studied.

## 2. Material and methods

### 2.1. Materials

Required concentrations of dye standards were prepared by dissolving appropriate amount of the

AR18 in 1,000-ml double-distilled water. The pH of the solution was adjusted to a desirable value using 1 N NaOH/HCl. NaCl, KCl, Na<sub>2</sub>CO<sub>3</sub>, and KNO<sub>3</sub> were used as electrolyte solution. All chemicals and reagents used in the present study were of analytical grade and were purchased from Merck.

### 2.2. Reactor design and experimental

The electrical coagulation reactor contained a glass cell of 500 ml with two opposing aluminum plates, a DC power supply, and a diffuser for aeration. The total volume of liquid in the reactor was 250 ml. Two rectangular aluminum electrodes (65 mm × 20 mm × 2 mm) with a distance of 2.5 cm were placed in parallel in the reactor working sample that was fixed at 250 ml during electro-processing. The process parameters that followed during electro-processing are presented in Table 2. For each test, electrodes were placed inside the 250 ml of the dye solution and a desired current density was applied. After passing the reaction time, samples were filtered through a Whatman filter prior to color measurements. Before each test, the electrodes were washed thoroughly with diluted acid and then rinsed with distilled water to remove any solid residues on the surfaces, dried in an oven at 60°C, and weighted using a digital scale.

### 2.3. Analysis

Temperature, EC, pH, and AR18 at both before and after EC process were measured in the filtered samples. The pH and temperature were determined using a pH-meter (Knick 765 model) and electrical

Table 1  
Chemical structure and molecular weight of acid red 18

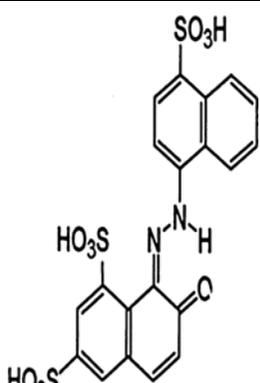
Dye	Structure	Purity (%)	Commercial name	CAS no.	Molecular formula	FW (g/mole)	$\lambda_{\max}$ (nm)
AR18		75	New cocchine	2611-82-7	C <sub>20</sub> H <sub>11</sub> N <sub>2</sub> Na <sub>3</sub> O <sub>10</sub> S <sub>3</sub>	604.47	506

Table 2  
The runs and operational conditions of electrocoagulation experiments

Run	Experiment	Operational conditions					
		Anode–cathode	pH	Current density (mA/cm <sup>2</sup> )	Reaction time (min)	Dye concentration (mg/l)	Electrode distance (cm)
1	Effects of initial pH and the dye removal mechanism	Al–Al	4–11	10.5	30	100	1
2	Effect of current density	Al–Al	4	3.5–42	30	100	1
3	Effect of reaction time	Al–Al	4	29	2–80	100	1
4	Effect of initial concentration	Al–Al	4	29	40	25–400	1
5	Effect of electrode distance	Al–Al	4	29	40	100	1–4
6	Effect of supporting electrolyte	Al–Al	4	29	40	100	1

conductivity was measured using Inolab instrument. A colorimetric method was used to quantify AR18 concentration in the samples. AR18 concentration was measured at a wavelength of 518 nm using a UV–vis spectrophotometer according to the Standard Methods for the Examination of Water and Wastewater.

### 3. Results and discussion

#### 3.1. Effect of initial solution pH

Solution pH is one of the most important variables affecting the efficiency of electrochemical process [18]. The effect of initial pH in the range of 4–11 was investigated as a critical parameter in the removal of AR18 by EC. The results of the experiments are shown in Fig. 1. The highest removal efficiency (51%) was occurred at pH 4. By increasing pH, removal percent-

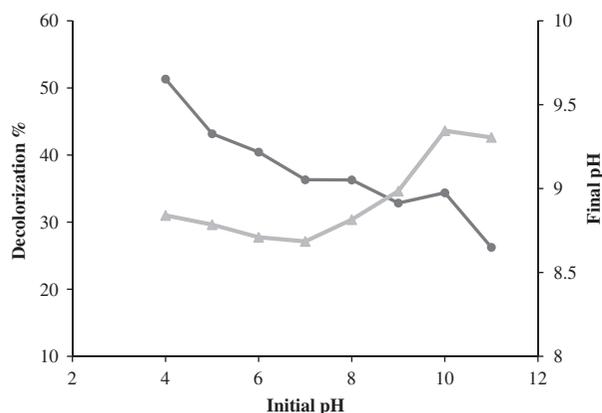


Fig. 1. Effect of initial pH (4–11) on decolorization efficiency by EC process.

age significantly decreased and reached to 26.52% at pH 11. The results of the study revealed the pH as an important operating factor influencing the performance of EC process. Maximum dye removal attained at the optimum pH. Precipitation of the dye started at pH 4 and any change in the value of optimum pH reduced the amount of the percent removal [19]. Initial pH of the solution was increased from 4 to 8.8 after 30-min electro-processing (Fig. 1). By increasing values of initial pH from 4 to 8, the value of final pH remained almost unchanged, but the removal efficiency dropped from 50.25 to 36.29%. There is the possibility that reaching final pH at lower initial pH, takes a longer reaction time, but at pH above 8, the value of final pH can even exceed 9. In the case of low pH (<4), monomeric cations of  $\text{Al}^{3+}$  and  $\text{Al}(\text{OH})^{2+}$  become predominant in the solution and are not important in the removal performance. When pH of the solution varies from 4 to 9,  $\text{Al}^{3+}$  and  $\text{OH}^-$  ions generated by electrodes react with each other and generate polymeric hydroxides species such as  $\text{Al}_6(\text{OH})^{153+}$ ,  $\text{Al}_7(\text{OH})^{174+}$ , and  $\text{Al}_{13}(\text{OH})^{345+}$  which transform finally into insoluble  $\text{Al}(\text{OH})_3$  by complex precipitation/polymerization reactions. At high pH values,  $\text{pH} > 10$ , monomeric form of  $\text{Al}(\text{OH})^{4-}$  increases [20,21]. Aluminum hydroxide is tended insoluble; therefore, it has no capability to produce flocs [22]. It can be observed from Fig. 1 that the final pH increases due to the formation of  $\text{OH}^-$ . The efficiency decrement by pH increase can be explained in two ranges of pH. In the pH between 4 and 9, as initial pH increases, the amount of soluble  $\text{Al}(\text{OH})^{4-}$  increases at a shorter reaction time against the amount of insoluble cationic species. In other words, after 30-min reaction time and in initial pH of 4, the concentration of  $\text{Al}(\text{OH})^{4-}$  is lower than that of pH 8. This is due to the fact that initial pH of 8 is reached in a short period of time,

and anions easily become predominant in the solution. In the pH range between 9 and 11,  $\text{Al}(\text{OH})_4^-$  becomes dominant in the solution and consequently decreases the dye removal percentage. Similar results were obtained by Daneshvar et al. [22,23]. With respect to final pH, at low initial pH (from 8 to 9), the final pH of the solution is increased. Inversely, at high initial pH (from 9 to 11), final pH of the solution is increased. Indeed, EC process acts as a pH neutralizer [23].

### 3.2. Effect of current density

Current density is another important parameter affecting the rate of removal efficiencies in the reactor. Applied current density directly affects the amount of floc formation [24]. In order to investigate the effects of applied voltage on the performance of EC process in the removal of AR18 in aqueous solutions, current in the range of 3.5–42  $\text{mA}/\text{cm}^2$  were applied. The obtained removal efficiencies are given in Table 2 and Fig. 2. The dye removal percentage increased from 47 to 89.5% by increasing current density from 3.5 to 26  $\text{mA}/\text{cm}^2$ . Increased current density produces more coagulants and consequently causes more floc formation. In other words, at higher current densities, the produced flocs can absorb dye molecules and consequently increases EC performance [25]. As current density increases, more aluminum dissolves from anode electrode into the solution and also small bubbles of hydrogen are generated according to Faraday's laws which overall result in more availability of aluminum hydroxyl polymers for the adsorption of the dye molecules [21]. By increasing current densities from 26 to 42  $\text{mA}/\text{cm}^2$ , only a negligible change in the removal percentage was observed (Fig. 2) which reveals the attaining maximal removal efficiency [18].

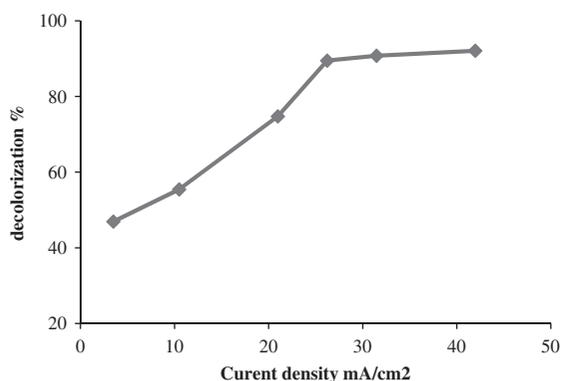


Fig. 2. Effect of current density (3.5–42  $\text{mA}/\text{cm}^2$ ) on decolorization efficiency of EC process.

The amount of anodic corrosion and floc formation can be obtained by measuring anode mass abatement and the quantity of sludge produced. Increase in current density causes more loss in anode mass, leading to an increase in the amount of sludge formed. This means that at current densities from 26 to 42  $\text{mA}/\text{cm}^2$ , loss of anode mass increased from 202 to 331 mg (a net mass of 129 mg). These correspond to respective increase in the removal efficiencies from 89.5 to 92.1%. Fig. 3 also depicts that the amount of sludge production increases from 605.85 to 1,060 mg, causing difficulties associated with sludge disposal and consequently reducing the performance of the process. Therefore, current increment acts as an important parameter in the dye removal until reaching to the critical point of 26  $\text{mA}/\text{cm}^2$ , at which the optimum removal of AR18 is occurred by EC process.

In the present study, the energy consumption of 7  $\text{kW h}/\text{m}^3$  at current density of 3.5  $\text{mA}/\text{cm}^2$  increased to 22  $\text{kW h}/\text{m}^3$  at current density of 26  $\text{mA}/\text{cm}^2$ . The three-time increase in energy consumption enhanced the removal efficiency from 47 to 89.5%. But, energy consumption reached to 51.6%  $\text{kW h}/\text{m}^3$  by increasing current density to 42  $\text{mA}/\text{cm}^2$ , which is not cost effective.

### 3.3. Effect of reaction time

Another influential factor in EC performance is reaction time [19]. The effect of reaction time on the removal efficiency of the dye is shown in Fig. 4. It can be noticed from the figure that the decolorization efficiency increases by increasing reaction time. AR18 removal efficiency reached from 41.5 to 92.3% at reaction time of 2 and 40 min, respectively. Higher

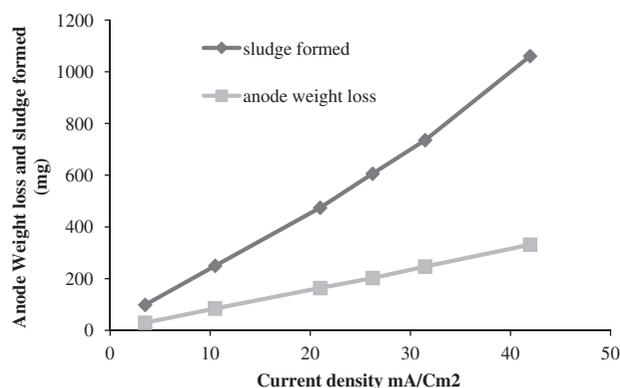


Fig. 3. Anode mass reduction and sludge production in EC process against current density (3.5–42  $\text{mA}/\text{cm}^2$ ).

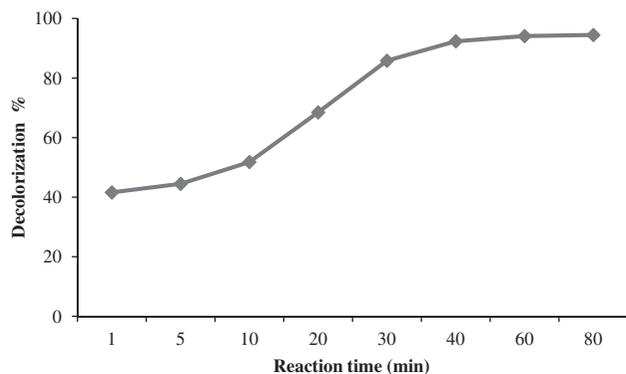


Fig. 4. Effect of reaction time (2–80 min) on decolorization efficiency of EC process.

reaction time leads to the formation of more hydroxyl ions and aluminum cations which consequently result in more and stronger floc production. But, this trend continues until reaching to optimum reaction time. Similar results were obtained by other researchers [26,27]. From Fig. 4, it is apparent that increasing the reaction time from 40 to 80 min, increases the percentage of the dye removal from 92.3 to 94.5%, respectively. Optimum reaction time for the removal of AR18 was 40 min. Therefore, it can be concluded from calculated energy consumption that increasing reaction time to more than 40 min is not cost effective. In fact, while amount of energy consumption is doubled by doubling the reaction time, negligible increase in removal efficiency is observed.

The pseudo-first and pseudo-second-order models were used for the evaluation of kinetics. Pseudo-first-order equation is given as:

$$\ln C_t = \ln C_0 - k_1 t \quad (1)$$

Pseudo-second-order equation was employed as follows:

$$\frac{1}{C_t} = k_2 t + \frac{1}{C_0} \quad (2)$$

where  $C_0$  is the AR18 concentration at the beginning of the process;  $C_t$  is concentration at the time  $t$ ;  $k_1$  and  $k_2$  are the first- and second-order kinetic constants, respectively.

Values of  $K_1$  and  $K_2$  were calculated from the plots of  $1/C_t$  vs.  $t$  and from  $\ln C_t$  vs.  $t$ , respectively. Fig. 5(a) and (b) shows the pseudo-first and pseudo-second-order models. According to regression coefficients depicted in the figures, experimental data tend to fit with pseudo-second-order model.

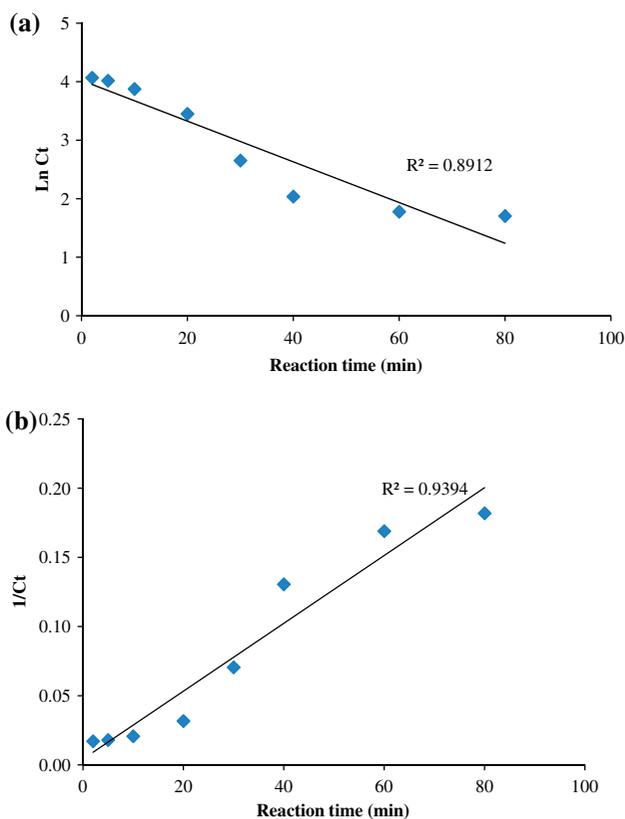


Fig. 5. Kinetic models for AR18 removal by EC process: (a) pseudo-first-order model and (b) pseudo-second-order model.

### 3.4. Effect of initial dye concentration

The experimental data for the removal of AR18 are presented in Fig. 6. Decolorization efficiency shows an increasing trend from 25 to 100 mg/l of dye concentrations, whereas it decreased at the concentration range of 100–400 mg/l. Generally, residual dye

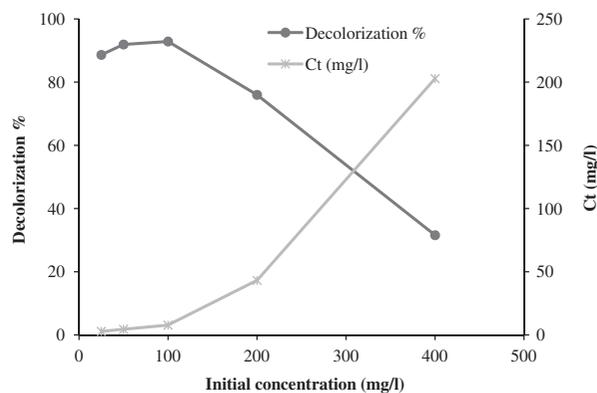


Fig. 6. Effect of initial dye concentration on EC efficiency.

concentration ( $C_t$ ) increases with increasing initial dye concentration. For example, at concentrations 25 and 100 mg/l, the  $C_t$  values were 2.79 and 7.75 mg/l, respectively. Song et al. reported that the maximum removal efficiency was obtained at low dye concentrations [28]. By increasing the initial dye concentration, the amount of required coagulant should be increased due to more surface area required for adsorption, which continuously becomes compensated by the corrosion of anodic electrode. In terms of decolorization efficiency, optimum residual dye concentration is considered as 100 mg/l.

### 3.5. Effect of inter electrodes distance

As can be seen in Fig. 7 and Table 2 (run 5), it is clear that by increasing distance between electrodes (at constant current of 29 mA/cm<sup>2</sup>), the initial voltage should also be increased significantly. For a constant percent removal of 72%, required voltages at the distances 1 and 4 cm are 10.1 and 28.2 V, respectively. As distance between electrodes increases, the resistance between electrodes also increases which requires more initial voltage in order to reach the same current density and consequently desired removal efficiency, causing more energy consumption. Therefore, shorter distance between electrodes results in higher decolorization efficiency and less energy consumption.

### 3.6. Effect of electrolyte type

The results of run 6 (Table 2) are presented in Fig. 8. In this run of the experiments, various salts at the concentration of 1 g/l were used as background electrolyte and the initial and the final voltage were measured in order to obtain optimum current

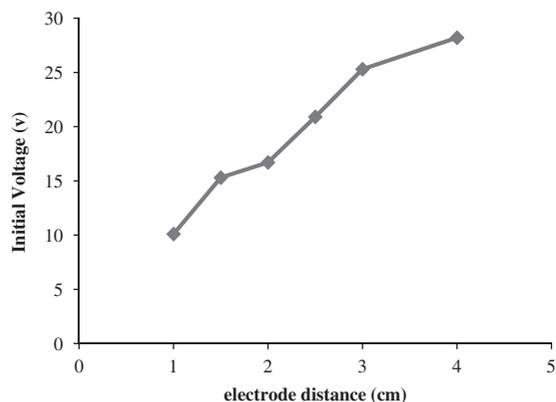


Fig. 7. Effect of electrodes distance on decolorization efficiency of EC process.

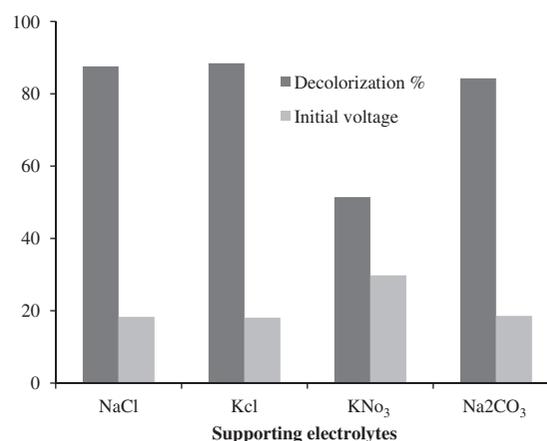
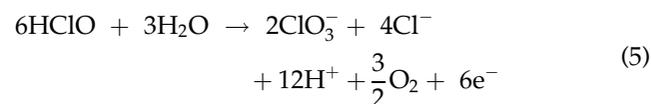


Fig. 8. Effect of different electrolytes on decolorization efficiency by EC process.

densities. These salts include sodium chloride, potassium chloride, sodium carbonate, and potassium nitrate. The maximum decolorization percentage as well as the lowest applied voltage was observed for sodium chloride, potassium chloride, and sodium carbonate.

Generally, with the addition of NaCl, internal conductivity and current density of the solution are enhanced and the necessary voltage for reaching to a certain current density is declined, so the consumption of electrical energy is reduced [29]. In the other hand, chlorinated salts as sodium and potassium are broken down to produce highly oxidative reagents such as chlorine, hypochlorous acid, hypochlorite, and hypochlorite ions (Eqs. (3)–(5)) from chloride ions at the anode surface [27,30].



Comparing the effects of four types of electrolytes on the removal efficiency revealed that sodium carbonate to have almost similar behavior as the chlorine. The removal efficiency has only declined in the presence of potassium nitrate. This can be attributed to the variation in the final pH in different electrolytes. The final pH was increased from 9 in the presence of chlorine and carbonate to 11 in the presence of potassium nitrate. The removal efficiency increases significantly with the increasing pH values.

Table 3  
Constituents of electrocoagulation sludge

Constituents	L.O.I.	Na <sub>2</sub> O	MgO	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	SO <sub>3</sub>	Cl	Fe <sub>2</sub> O <sub>3</sub>	Zn
(%)	36.73	0.674	0.131	58.285	0.441	2.074	1.466	0.189	0.011

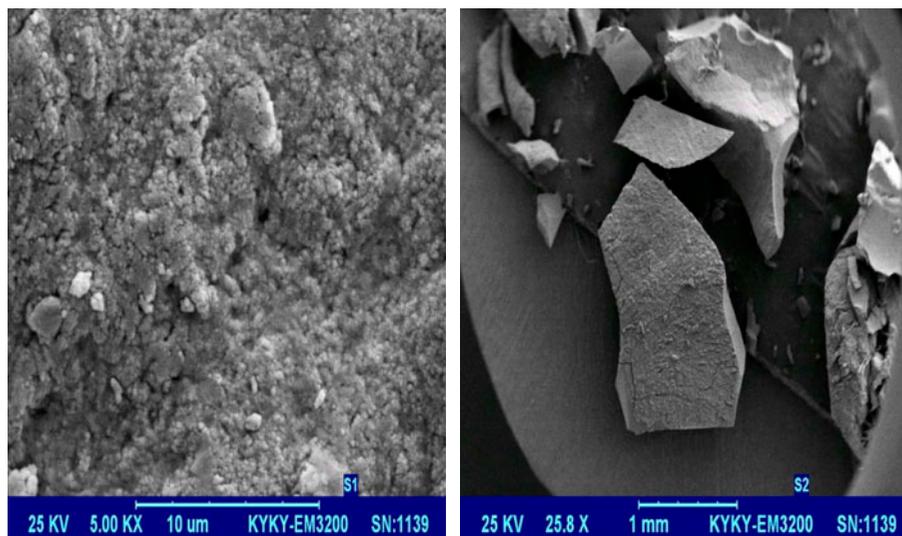


Fig. 9. SEM images of sludge flocs in EC process with different magnifications.

### 3.7. XRF and SEM analyses

To identify the constituents of electrochemical sludge, XRF analysis was used. Details of XRF analysis are presented in Table 3. L.O.I. is loss on ignition and shows the volatile portion of EC sludge after heating the sample. It is a common method for the determination of organic and carbonate content of sediments [31]. The amount of loss on ignition was determined equal to 36.73%. This portion of sludge can be related to adsorbed and precipitated dye (AR18) and also byproduct of degradation. A large portion of sludge (58.285%) is related to aluminum oxides (Al<sub>2</sub>O<sub>3</sub>). In between, iron oxide and zinc (as semi quantitative result) were detected. Presence of sulfophenyl groups may confirm the decomposition of AR18 during EC process. Also, high levels of chlorine can be appeared due to the addition of chlorinated salts.

Fig. 9 shows two different magnifications of sludge flocs in EC process. According to Fig. 9, formed flocs have sizes about 1 mm and are non-uniformly shaped which can act as good flocs to remove AR18. Fig. 9 depicts flocs with a magnification of  $5 \times 10^3$ . The figure clearly shows that the surface of flocs has many pores which can easily adsorb the dye molecules.

### 4. Conclusion

Removal of AR18 by EC process using aluminum electrodes was investigated in the present study. We found that the amount of produced sludge and decrement of anodic electrode mass in the applied current as important parameters in the system operation. Several major influential variables of EC process were investigated and the results revealed that:

(A) The initial pH of solution is one of the important variables affecting the performance of EC process. At pH about 4, the maximum decolorization percentage was observed and the percent removal of the dye decreased by increasing pH.

(B) Increase in current density improves the removal efficiency, but leads to increase in anodic corrosion and also more sludge formation. Taking into account the sludge production, optimum current density was found 29 mA/cm<sup>2</sup>.

(C) Decolorization efficiency increased by increasing reaction time and the sorption process obeyed the first-order kinetic model.

(D) As the distance between electrodes increased, more energy was consumed to reach the desired removal percentage.

(E) The type and also the nature of electrode material affect the amount of energy consumption.

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