

## Removal Efficiency of Acid Red 18 Dye from Aqueous Solution Using Different Aluminium-Based Electrode Materials by Electrocoagulation Process

Nurulhuda Amri<sup>1,2</sup>, Ahmad Zuhairi Abdullah<sup>1,\*</sup>, and Suzylawati Ismail<sup>1</sup>

<sup>1</sup>School of Chemical Engineering, Universiti Sains Malaysia, Engineering Campus, 14300 Nibong Tebal, Penang, Malaysia

<sup>2</sup>Faculty of Chemical Engineering, Universiti Teknologi MARA (UiTM), Cawangan Pulau Pinang, 13500 Permatang Pauh, Penang, Malaysia

\* Corresponding author:

tel: +604-5996411

email: chzuhairi@usm.my

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**Abstract:** This work compares commercial aluminium electrode for use in the treatment of wastewater by electrocoagulation process against waste aluminium cans electrode. The applicability of the waste aluminium cans electrode was tested for decolorization of Acid Red 18 dye as a model pollutant. The batch electrocoagulation process using both types of electrode was conducted at a current density of 25 mA/cm<sup>2</sup>, a pH of 3, an initial concentration of 100 mg/L and 25 min of reaction time. The elemental composition and surface morphology of both electrode materials and the sludge produced were analyzed using SEM-EDX to establish the correlation between the properties and characteristics of both electrode materials with their dye removal performance. The results demonstrated that waste aluminium cans performed better than commercial aluminium electrode with a removal efficiency of 100% in 25 min of reaction time. This was due to the higher Al dissolution of waste aluminium cans electrode that contributed to the larger amount of Al<sup>3+</sup> released into the solution to consequently form more flocs to remove the dye molecules. In conclusion, the proposed waste aluminium electrode was considered as efficient and cost-effective and had the potential to replace the conventional ones in treating colored industrial wastewater using electrocoagulation process.

**Keywords:** waste aluminium cans; commercial aluminium electrode; electrocoagulation process; Acid Red 18; removal efficiency

### ■ INTRODUCTION

Large amounts of disposed aluminium can contribute to solid wastes either in the form of industrial or domestic waste; they originate from products such as cans, food trays, pie plates, frozen foods containers, wrapping foil, etc. [1]. Annually, 1.5 million tons of waste is created worldwide by throwing away aluminium cans that not only fill up the landfills, but also cause wastage in energy and extensive environmental damage in creating new cans [2]. Recently, the attention of many researchers has shifted towards utilization of waste aluminium cans as a raw material in the preparation of catalysts [3] and nanomaterials [4]. The exploitation of this waste as a raw material in new research fields will present several environmental and economic benefits over a long-term

period that will reduce the impact of aluminium wastes by converting them into valuable materials [5].

Aluminium (Al) is one of the electrode materials that has been extensively used, as this type of material presents high electro-dissolution rate and is effective in treating various pollutants using electrocoagulation (EC) process [6-8]. EC process is known as a potential technique for treating industrial effluent due to its simplicity in the design and operation, high efficiency, cost-effective treatment system, and environmental compatibility [9-10]. This process takes the benefits of the binding effect of charge neutralization/surface complexation/adsorption of pollutant onto the in-situ formation of metal hydroxide from the oxidation of sacrificial anode [11]. There have been numerous studies reported on the efficient performance of aluminium

electrodes used in the EC process for treatment of wastewater containing textile dyes [12-13].

Azo dyes are the largest group of dyes used in the textile industry that represent about 50% of all the commercial dyes available [10]. They generally have an azo group band (-N=N-) together with other chromophores which are responsible for their intense color, high water solubility, and resistance to degradation under common biological treatment [9]. Acid Red 18 (AR18) dye is one of the dyes under the azo group that is commonly used during the dyeing process. The major environmental problem associated with the use of these dyes is their loss in the dyeing process. It is estimated that around 10–50% of these dyes are usually lost in the effluent [14]. Wastewaters containing dyes can cause aesthetic problems due to the color even at low concentration, and their intermediate products could be either toxic, carcinogenic, or mutagenic to aquatic life [14-15]. Therefore, the treatment of effluents containing azo dyes is crucial to minimize their harmful impacts on human beings and aquatic life.

Various treatment technologies, including physical, chemical, and biological methods, have been used for the treatment of textile effluents [16]. However, these treatment methods still have certain limitations such as high capital and operation cost, generation of a large amount of sludge and may not be effective for all types of dyes. In Malaysia, conventional wastewater treatment systems that are most commonly applied in textile industries are biological treatment alone or physicochemical treatments followed by a biological treatment [17]. Single conventional biological treatment does not always treat the textile effluent satisfactory as most of the dyes, especially azo dyes [18] are hardly biodegradable at high concentration [13]. Thus, certain pre-treatment, such as physical or chemical process is

necessary. In this respect, the EC process can be a good alternative pre-treatment method for biological treatment system due to its advantages.

In this study, waste aluminium cans (WAC) were utilized as a potential electrode for the removal of AR18 dye using an EC process. To the best of our knowledge, no study has been reported on the utilization of this waste material as an electrode in the EC process for the treatment of water or wastewater. Most of the studies available in literature utilized directly commercial electrode materials to evaluate their performance in the EC process [8-10].

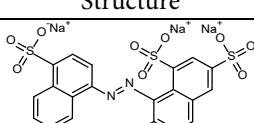
Thus, this study aimed to evaluate the applicability of WAC as an electrode and compare the removal performance between the WAC and commercial aluminium (COM) electrode in the decolorization of AR18 dye via the EC process. The Al ion ( $\text{Al}^{3+}$ ) contents remaining in the solution after the EC process using both types of electrode material were also analyzed to ensure that the quality of treated water could achieve the permissible limit for industrial effluent discharged [19]. The characteristics of both electrodes in terms of elemental composition, the surface morphology of the electrodes before and after the EC process as well as the sludge produced were also examined to establish the correlation with the dye removal efficiency.

## ■ EXPERIMENTAL SECTION

### Materials

All chemicals used in this study such as AR18 dye (> 99% purity), hydrochloric acid (HCl), sodium hydroxide (NaOH) and sodium chloride (NaCl) were of analytical grade and purchased from Merck. Table 1 shows the general properties of the AR18 dye. A stock solution of AR18 dye was prepared by dissolving 1 g of dye

**Table 1.** Properties of AR18 dye

Dye	Structure	Molecular formula	Molecular weight (g/mol)	$\lambda_{\max}^*$ (nm)
Acid Red 18		$\text{C}_{20}\text{H}_{11}\text{N}_2\text{Na}_3\text{O}_{10}\text{S}_3$	604.48	507

\* $\lambda_{\max}$  is the maximum absorbance of AR18 dye

in 1 L of deionized water. Then, the solution was diluted to give the initial concentration of 100 mg/L. The electrical conductivity of the solution was increased by the addition of 2 g/L of NaCl into the solution. Meanwhile, the pH of the solution was adjusted from the original pH of 6.8 to 3.0 using 0.1 M HCl solution.

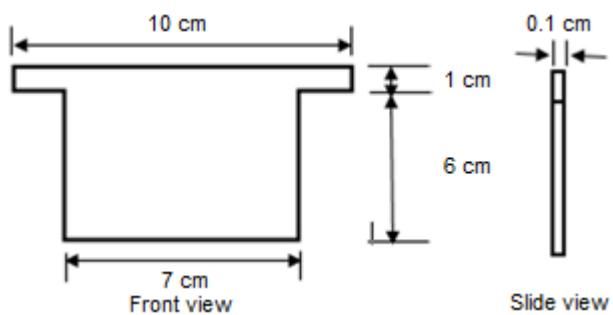
The COM and WAC were used as materials for electrodes. The COM electrodes were purchased from Neilement Engineering Resources & Services. The dimensions of the COM electrode are illustrated in Fig. 1. Meanwhile, the WAC was collected from cafeterias in Universiti Sains Malaysia. The WAC was cut into dimensions similar to those of COM electrode but with a different thickness of 0.01 cm (original thickness of WAC). They were then subjected to a pre-treatment with sandpaper to remove the paint and epoxy coating on the outer and inner walls of the can, and rinsed with deionized water. Finally, the WAC was dried in an oven at 60 °C and ready to be used in the batch EC experiment.

### Instrumentation

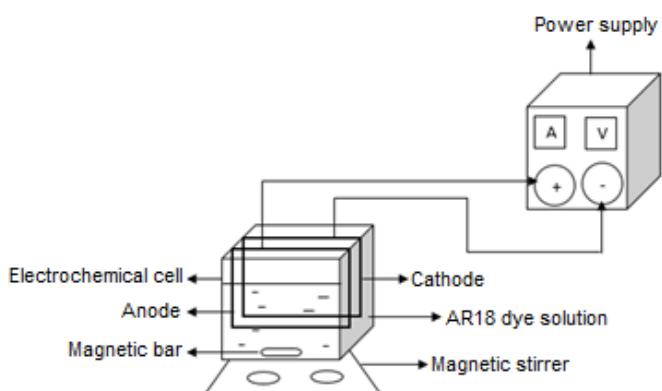
The solution pH was measured using a pH meter (Eutech pH2700, Thermo Scientific, Singapore). The concentration of AR18 dye solution was determined using a double beam UV-visible spectrophotometer (Shimadzu 1800, Japan) at a wavelength of 507 nm. The surface morphology and the elemental composition of both electrode materials and sludge produced after the EC process were examined using a scanning electron microscope (SEM) equipped with an energy-dispersive X-ray spectroscopy (EDX) facility (Quanta 450 FEG, FEI, Netherlands) operated at an accelerating voltage of 5 kV. The concentrations of Al<sup>3+</sup> in the treated water were examined using an inductively coupled plasma-optical emission spectrometer (ICP-OES) (ICAP 7600, Thermo Scientific, USA).

### Procedure

Batch monopolar EC experiments were carried out in a 1.1 L rectangular reactor, as shown in Fig. 2. Two pieces of flat WAC and COM plates were used as electrodes, and the distance between the electrodes was set at 0.5 cm. The area of each electrode dipped into the solution for both



**Fig 1.** The specific dimensions of a COM electrode



**Fig 2.** Schematic diagram of the experimental set-up for the EC system

WAC and COM was 6 cm × 7 cm. The electrodes were connected to a DC Power supply (Dazheng PS-305D, 0–5 A, 0–30 V) to supply and control the required current during the experiment. The water sample (800 mL) was initially fed into the EC cell, and the current density was set at 25 mA/cm<sup>2</sup> for 25 min. The solution was continuously stirred at 250 rpm during the experiment in order to get a homogeneous solution. All EC experiments for WAC and COM were operated at the best-operating conditions (current density of 25 mA/cm<sup>2</sup>, pH of 3, initial concentration of 100 mg/L, electrode distance of 0.5 cm, NaCl concentration of 2 g/L and reaction time of 25 min) as obtained previously. In the preliminary work, a series of one-factor-at-a-time experiments for the effects of current density (10–30 mA/cm<sup>2</sup>), pH (3–9), contact time (0–60 min), NaCl concentration (2–5 g/L) and inter-electrode distance (0.5–2 cm) were conducted using the same batch EC system. However, the effect of operating parameters to obtain the best set of operating conditions is not reported in this paper.

Samples of the treated wastewater were taken at different times and filtered through a Whatman filter paper no. 1 prior to its color measurement. Each analysis was conducted in triplicate. The AR18 dye removal efficiency is calculated using Eq. (1):

$$\text{Removal Efficiency (\%)} = \frac{C_0 - C_t}{C_0} \times 100\% \quad (1)$$

where,  $C_0$  is the initial dye concentration (mg/L) and  $C_t$  is dye concentration at a time,  $t$  (mg/L).

## ■ RESULTS AND DISCUSSION

### Elemental Composition

The elemental composition of WAC and COM were determined using EDX analysis, and the results are presented in Table 2. From the results, both WAC and COM electrodes were classified as aluminium alloys. Al was the main metal element in both electrode materials that represented about 85.67% and 79.05% by weight respectively for WAC and COM. The composition of Al was found to be higher in WAC than that of COM by about 6.62% with additional elements of Mg and Mn. Meanwhile, COM had Fe elemental composition of 0.41% by weight. The difference in the composition and alloying elements of these materials was believed to be one of the factors that contributed to the difference in the performance of AR18 dye removal in the EC process. Dura and Breslin [8] reported the performance of Al-Zn-In and Al-Mg alloy electrodes for the removal of phosphate,  $Zn^{2+}$ , and Orange II dye. It was observed that both types of Al alloy performed well in removing all the pollutants as compared to pure Al electrode due to their low overpotential values as obtained from the polarization and cyclic polarization experiment. These low overpotential

values indicated a higher dissolution behavior of Al alloy than the pure Al electrode, which was more passive. However, the performance of Al-Zn-In was slightly better than Al-Mg, attributed to the instability of the passive film that was formed on the Al-Zn-In electrode to significantly enhance the dissolution of the electrode and the production of  $Al^{3+}$  ion.

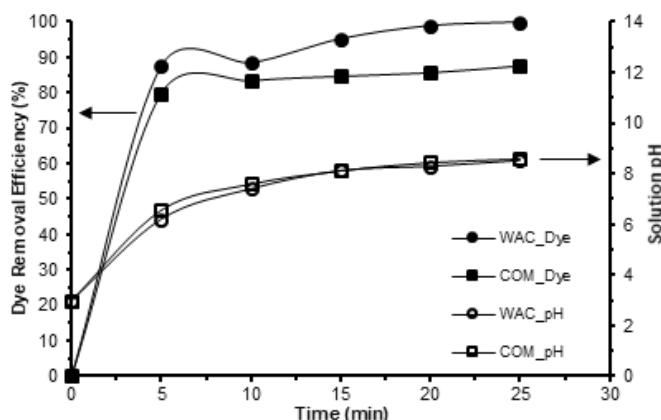
### **Removal efficiency of AR18 dye using different electrode materials**

In order to examine the potential of this waste electrode as a green alternative to the commercial Al electrode, the performance of WAC and COM in the decolorization of AR18 dye was investigated. The effect of different electrode materials on AR18 removal was conducted at the best-operating conditions as mentioned earlier. Fig. 3 illustrates the removal efficiency of AR18 dye and solution pH for WAC and COM electrodes with time. As can be observed, the removal trend was similar for both electrodes in which the decolorization efficiency increased drastically in the first 5 min of the reaction with up to 80% removals owing to higher precipitation rates under the acidic condition. After this point, the removal rates gradually increased, reaching a plateau in the next 20 min due to the transition of the dye removal mechanism from precipitation to the adsorption process [13]. However, it was found that the WAC performed better as compared to the COM with removal efficiencies of 100.00% and 87.56%, respectively after 25 min.

This finding was in agreement with the elemental analysis results (Table 2) as one of the Al alloying elements in WAC was Mg that could activate and enhance the Al dissolution through anode oxidation. Higher dissolution of WAC electrode produced a larger amount of  $Al^{3+}$  in the solution and subsequently proceeded through a hydrolysis reaction. This reaction generated various cationic monomeric and polymeric aluminium hydrolysis species. These cationic species were attracted to the negative charge of AR18 dye molecules, to result in the removal by either precipitation or adsorption mechanism. The result was consistent with Dura and Breslin [8]. They observed that the Al-Mg alloy electrode exhibited higher dissolution in

**Table 2.** Elemental compositions of WAC and COM electrodes

Element	WAC	COM
	Weight (%)	Weight (%)
Al	85.67	79.05
C	9.60	16.54
O	2.55	3.99
Mg	1.16	-
Mn	1.02	-
Fe	-	0.41

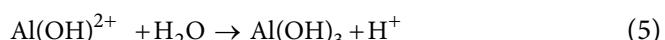
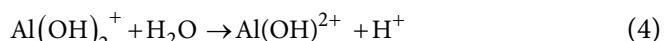


**Fig 3.** The removal efficiency of AR18 dye and the solution pH by WAC and COM electrodes

the presence of NaCl with more than 90% removal efficiencies of phosphate ions by the EC process.

Initial pH of the dye solution is also an important parameter affecting the predominance of coagulant species produced during the EC process. Generally, the pH of the treated water changes during the EC process. As can be seen in Fig. 3, the similar trends of increasing solution pH throughout the EC treatment were observed for both types of electrode material to indicate that the metal hydroxide species that formed in the solution were identical due to the constant initial solution pH of 3.0 set for WAC and COM electrodes. Overall, the solution pH increased rapidly to a maximum of 8.6 in 25 min. During the first 5 min, the initial solution pH increased drastically from 3.0 to 6.2 and 6.5 for WAC and COM electrodes, respectively. This was attributed to the hydroxyl ion release from water reduction occurring at the cathode during the EC treatment. Meanwhile, the dissolution of anode produced  $\text{Al}^{3+}$ , as expressed by Eq. (2). In acidic condition,  $\text{Al}^{3+}$  can directly undergo spontaneous hydrolysis reactions producing various monomeric species such as  $\text{Al(OH)}_2^+$  and  $\text{Al(OH)}^{2+}$  which then transform into  $\text{Al(OH)}_3$  as shown in Eq. (3) to (5). However, the other ionic species ( $\text{Al(OH)}_4^-$ ,  $\text{Al(OH)}^{2+}$ ,  $\text{Al}_2(\text{OH})_2^{4+}$ ) and polymeric species ( $\text{Al}_6(\text{OH})_{15}^{3+}$ ,  $\text{Al}_7(\text{OH})_7^{4+}$ ,  $\text{Al}_8(\text{OH})_{20}^{4+}$ , etc.) may also present in the system depending on the pH of the solution to be finally converted to  $\text{Al(OH)}_3$  flocs. These  $\text{Al(OH)}_3$  flocs are beneficial for the adsorption of soluble organic pollutants

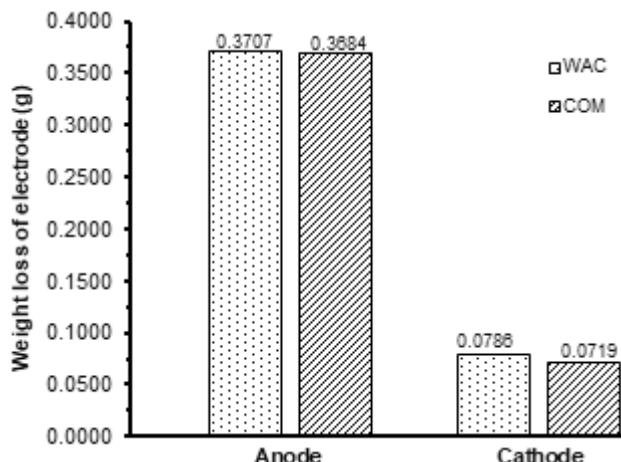
due to their large surface area. The overall process of flocs formation can be described as [13]:



The high decolorization efficiencies as demonstrated by both types of electrodes in the first 5 min were mainly due to the dye precipitation process by  $\text{Al}^{3+}$  as well as monomeric and polymeric species that formed in the solution at  $\text{pH} < 6.5$ . However, as the pH was increased exceeding 6.5, most of the aluminium hydroxide precipitated as Al coagulant, thus responsible for the adsorption of the remaining dye molecules. The finding in this work was in good agreement that of Khorram et al. [13]. They reported that dye precipitation process was the main mechanism that was responsible for the high removal efficiency followed by the adsorption of the dye polymeric colloidal species by  $\text{Al(OH)}_3$  flocs which had a lesser effect. Thus, it can be summarized that the dye removal mechanisms for both WAC and COM electrodes were identical as the solution pH for both types of electrode material were almost the same throughout the EC process.

On the other hand, the finding was also consistent with that reported by Elabbas et al. [20]. They observed that changes in the solution pH were greatly influenced by the nature of the electrode materials. They found that the increase in the solution pH with time for aluminium alloy was slightly higher than that of pure aluminium leading to higher removal efficiency of chromium and COD. Since both types of electrode used in this study were categorized as the same nature of electrode material, which was aluminium alloys, they possessed rather similar behavior towards the pH change during the EC process.

Besides, the excellent removal performance of WAC could also be explained by the higher dissolution of WAC electrodes as compared to COM that was represented by the weight loss of electrodes, as shown in Fig. 4. Higher weight loss of WAC anode electrode



**Fig 4.** Weight loss of anodes and cathodes after the EC process

contributed to the higher amount of  $\text{Al}^{3+}$  released into the solution to destabilize the colloidal particles and form more flocs to remove the dye molecules. Results obtained in this work revealed that WAC was more superior to those reported in the literature. Khosravi et al. [10] reported that the highest AR18 removal of 92.3% was obtained by Al electrode at an optimum reaction time of 40 min, pH 4 and a current density of  $26 \text{ mA/cm}^2$ . In addition, Azarian et al. [15] observed that the highest AR18 removal of 95.0% was obtained by Al electrode under an optimum current density of  $1.2 \text{ mA/cm}^2$ , pH 7 and reaction time of 45 min.

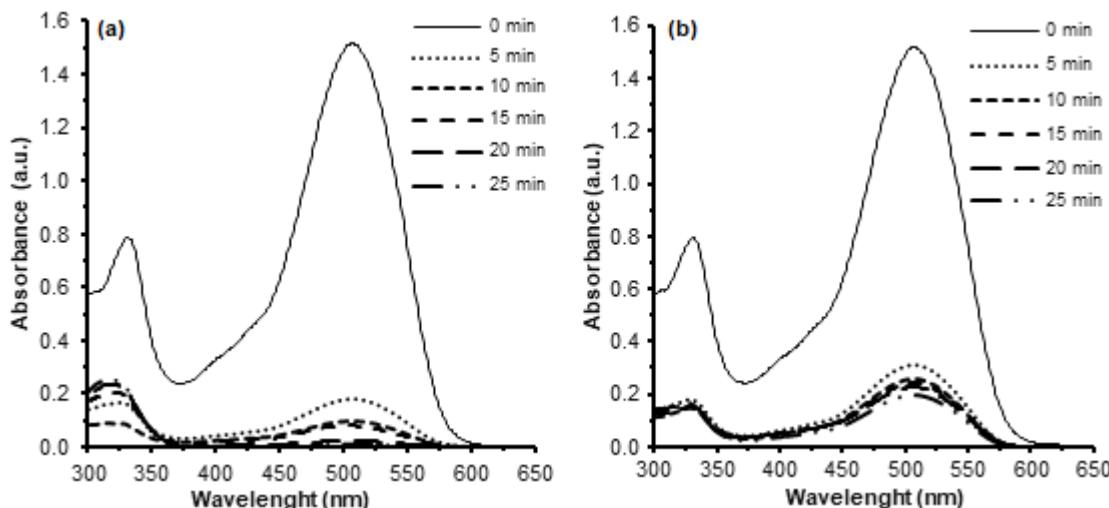
The rate of AR18 dye decolorization can also be

confirmed by the absorption spectrum, as illustrated in Fig. 5. The absorbance by AR18 dye can be characterized by two peaks at 507 nm in the visible region and 320 nm in the ultraviolet region. As can be seen clearly, the main peak at 507 nm decreased significantly with time without the appearance of new absorbance peaks for both types of electrode during the EC process. However, the peak for WAC disappears faster than that of COM within 25 min of reaction time. This suggested that the decolorization involved the decomposition of AR18 dye through the breaking of  $-\text{N}=\text{N}-$  belonging to the chromophore group. A similar observation was reported by Azarian et al. [15], but the time taken for the 507 nm peak disappearance was relatively longer, i.e., between 30 to 120 min.

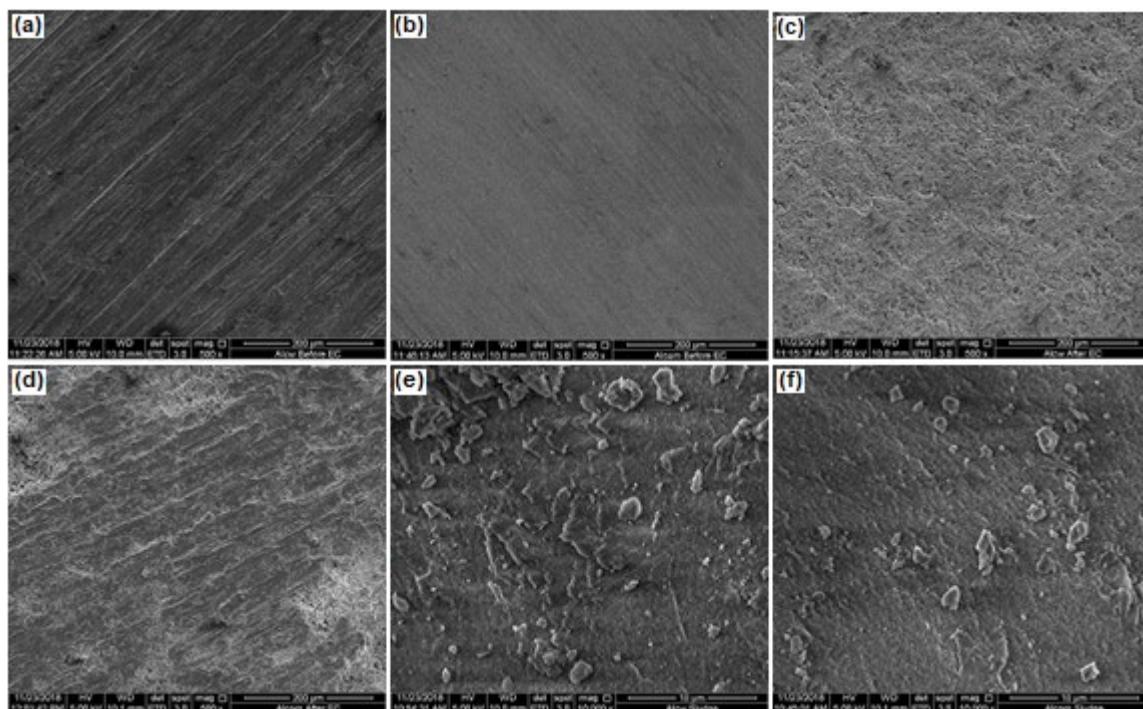
In order to ensure that the EC was a safe process and did not need any further treatment for treating excessive residual  $\text{Al}^{3+}$  ions after the process, the treated sample was then examined using ICP-OES. From the results in Table 3, it can be concluded that the residual  $\text{Al}^{3+}$

**Table 3.** The concentrations of Al ion remaining in the treated water and the acceptable limit for Al ion from industrial effluent discharge [19]

Electrode material	Treated water [Al <sup>3+</sup> ] (ppm)	Standard A [Al <sup>3+</sup> ] (ppm)	Standard B [Al <sup>3+</sup> ] (ppm)
WAC	0.6		
COM	2.7	10	15



**Fig 5.** Absorbance spectra of AR18 dye during the EC process at different reaction times using (a) WAC and (b) COM electrodes



**Fig 6.** SEM images for (a) WAC before EC (500 $\times$ ), (b) COM before EC (500 $\times$ ), (c) WAC after EC (500 $\times$ ), (d) COM after EC (500 $\times$ ), (e) WAC sludge (10k $\times$ ), (f) COM sludge (10k $\times$ )

concentrations in treated water using WAC and COM were 0.6 and 2.7 ppm, respectively. Thus, this was an additional benefit of the use of waste electrode as the concentration of  $\text{Al}^{3+}$  in the treated water was much lower compared to that of COM. This might be due to the fact that most of the  $\text{Al}^{3+}$  ions produced from the dissolution of WAC anode effectively combined with hydroxyl ions to form a higher amount of  $\text{Al(OH)}_3$  flocs that ended up in the WAC sludge as confirmed by the surface morphology and EDX analysis as shown in Fig. 6(e) and 7(a), respectively. However, both electrodes still could be considered as harmless electrodes since they showed lower residual Al concentrations as compared to the permissible limit stated in the Malaysian's Fifth Schedule of Environmental Quality (Industrial Effluent) Regulation 2009 which are 10 ppm for Standard A and 15 ppm for Standard B[19].

### Surface Characterization

Fig. 6(a-d) present the microscopic images of the surface morphology of both electrode materials while those of dried sludges produced after the EC process are

shown in Fig. 6(e-f). The images of electrodes before treatment show that the WAC electrode had a relatively rougher surface structure as compared to COM, which features a relatively smooth surface. The rough structure surface of WAC was mainly caused by the pre-treatment of the electrode with sandpaper to remove the paint and epoxy coating on the outer and inner walls of the can. However, both material surfaces changed to rather coarser structures after the EC process. WAC surface presented a well uniform distribution of visible dents generated as a result of higher electrode dissolution.

Meanwhile, a non-uniform distribution of visible dents was observed on the surface of COM. Thus, it can be summarized that the more uniform dissolution of WAC electrodes led to the higher removal of AR18 dye as compared to the non-uniform dissolution of COM electrode. The produced sludge image exhibited similar features for both electrode materials indicating the presence of identical elemental constituents such as Al, O, Na, and Cl. However, it is observed that the number and size of particles existing in WAC were larger than those of COM sludge suggesting greater formation of

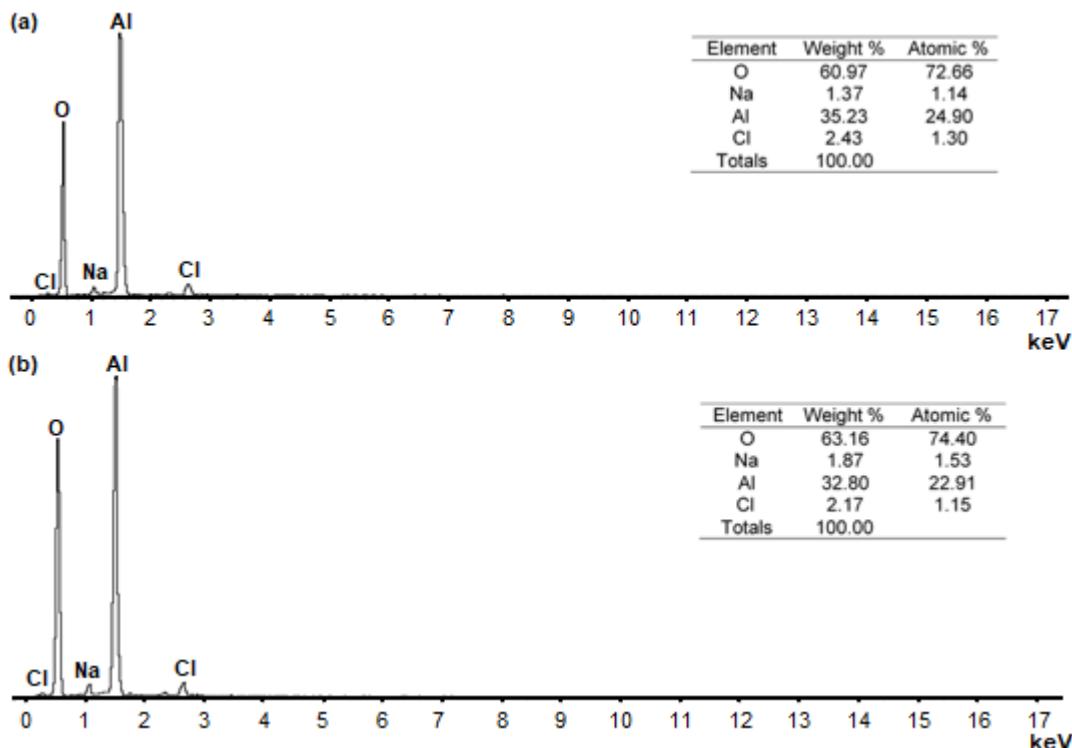


Fig 7. EDX Analysis results of sludge produced after the EC treatment using (a) WAC and (b) COM electrodes

Al(OH)<sub>3</sub> flocs in the WAC sludge. This can be confirmed by the EDX analysis results, as shown in Fig. 7(a) and 7(b) where the composition of Al element (by weight %) for WAC sludge was higher than COM with a difference of 2.43%. The presence of Na and Cl ion was also detected in both types of sludge due to the usage of NaCl as a supporting electrolyte in the EC process.

## ■ CONCLUSION

In the present work, the dye removal efficiency and changes in the solution pH with time as well as the quality of the treated water using WAC and COM electrodes via EC process were studied. The WAC electrode demonstrated better performance than COM electrode as it could decolorize the AR18 dye faster with 100% removal efficiency in 25 min and produced lower residual Al concentration of 0.6 ppm in the treated water after the EC treatment. During the EC process, it was also observed that pH of the solution increased considerably with both types of electrode materials reaching a maximum of 8.6, indicating that they performed similar dye removal mechanism due to the identical nature of the electrode

materials. In summary, the utilization of this material offered several advantages as it could reduce the cost for electrode material that needs to be replaced periodically and at the same time transforming this WAC into a valuable material.

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