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Electrically Conductive Carbon-Ceramic Composite as Electrode on Indirect Electrochemical Oxidation Reactor for Remazol Black B Degradation

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Abstract

Performance of carbon-ceramic composite electrode was investigated for degradating of Remazol black B dye on indirect electrochemical oxidation reactor. Carbon-ceramic composite electrode was prepared by mixing alumina, kaolin and liquid clay with carbon addition as a conductive phase. The electrode was casted in stainless steel mold and then sintered at 900 degree Celsius in a vacuum furnace for 7 h. The surface morphology, crystalline phase and electrical conductivity of the carbon-ceramic composite electrodes were investigated. The electrode with a highest electrical conductivity was applied on indirect electrochemical oxidation reactor for degradating Remazol black B. The operational parameters investigated included the influence of initial pH, electrolyte concentration and dye concentration on color removal efficiency. The research revealed that the electrical conductivity of carbon-ceramic composite electrode with addition of 30% carbon was 5.04 S/cm. The color removal efficiency of Remazol black B on the reactor using this electrode was 87.75% for 60 min. of electrolysis time with an applied voltage of 12 V, at pH 6, with the addition of 3 g table salt per liter of waste and dye concentration of 100 mg/L. This study indicates that carbon-ceramic composite electrode is prospective to be used as an electrode on electrochemical cells for wastewater treatment.

Keywords: Carbon-ceramic composite electrode; Electrical conductivity; Indirect electrochemical oxidation; Color removal efficiency; Remazol black B textile dye

1. Introduction

The wastewater from dyeing industry contains high organic and inorganic pollutants with extreme color intensity. These effluents have a great potential to cause serious environmental problems including humans health if improperly disposed (Lellis *at al.,* 2019). For this reason, the textile wastewater should be treated before being discharged into the environment. A number of physical techniques through ultrafiltration (Zulaikha *et al.,*2014), electron beam irradiation (Duy *et al.,* 2019), adsorption by using charcoal (Patel, 2018), biological techniques by aquatic plants (Roy *et al.,* 2018; Aragaw *et al.,* 2014; Tusief *et al.,* 2019), solid state fermentation with peach-palm residue (Chicatto *et al.,* 2018) and Ganoderma fungi (Sudiana *et al.,* 2018) and chemical techniques such as ozonation (Bilinska *et al.,* 2020), electrochemical cell (Najafpoor *et al.,* 2017), and Fenton oxidation (Sharma *et al.,* 2017) have been developed, but not yet fully implemented in field scale due to high operational costs. By comparison, the electrochemical oxidation method is an eco-friendly technology with several advantages such as being easy to operate, low cost and high degradation efficiency.

Electrochemical oxidation method has been considered a promising alternative technology for organic pollutants in wastewater treatment. This technology is also classified as an eco-friendly technology because it generally requires no external chemical in degradation process (Dominguez *et al.,* 2018; Xiong *et al.,* 2019). The degradation of organic contaminants by electrochemical oxidation can be performed via direct and indirect mechanisms. In direct electrochemical mechanism, the oxidation of organic pollutants takes place after the pollutants are adsorbed onto the anode surface. Whereas, the indirect electrochemical oxidation mechanisms are mediated by electrogenerated strong oxidants including ozone, active chlorine, and persulfate at the anode surface which subsequently oxidize the organic compounds in the bulk solution. In both electrochemical oxidation mechanisms, electrocatalytic properties of electrode surface significantly affect the degradation efficiency of organic contaminants. Reactions involving the supply and reception of electrons occur at the electrode surface. Therefore, the properties of electrode materials such as electrical conductivity and porosity have a significant effect on the number of electrons participating in redox reactions. Besides having high electrocatalytic activity, electrodes in an electrochemical cell for wastewater treatment should have good physical and chemical stability, good resistance against corrosion, and low cost (Anglada *et al.,* 2009). A number of electrode materials have been used for electrochemical wastewater treatment including TiO_2-NTs/Sb-SnO_2/PbO_2, boron-doped diamond (Patel *et al.,* 2013), graphite (Bhatnagar *et al.,* 2014), graphite-PVC composite (Musa *et al.,* 2015), Ti/IrO_2, Ti/RuO_2, Ti/PbO_2 (Yao *et al.,* 2019) and carbon pasta bulk modified with carbon nanotubes (Ashrafi *et al.,* 2019).

Carbon materials such as activated carbon, mesoporous carbon, carbon nanofiber, carbon nanotubes, and graphene are widely used as electrodes due to their high electrical conductivity, relatively low cost and ease to obtain, good chemical inertness, and wide potential range (McCreery, 2008; Festinger *et al.,* 2020). In addition, carbon rods from batteries are also effectively used as electrodes in indirect electrochemical oxidation reactor for color removal from textile wastewater (Sastrawidana *et al.,* 2018) and COD degradation of restaurant wastewater (Sastrawidana *et al.,* 2018). One of the limitations of carbon electrode is easy to erode especially when the electrochemical cell is applied at higher cell voltages.

The research on modifying carbon-based electrodes especially carbon-ceramic electrodes is interesting to be done because these electrodes are porous, allowing for an increase in the active surface and high electrical conductivity. Besides, it is easy to prepare, robust and high stability (Kutner *et al.,* 1998). Carbon-ceramic electrodes are a new class of electrodes first introduced in 1994 by Lev *et al.,* 1994). These electrodes consist of a silica ceramic matrix with carbonaceous materials as a conductive phase (Corb *et al.,* 2007). To increase their electrocatalytic property, the preparation of carbon-ceramic electrodes has been modified using metal or metal oxides composite such as Ferrocenecarboxylic acid (Skeika *et al.,* 2011), nickel-catechol complex (Jalali *et al.,* 2014), gold nanoparticles (Shamsipur *et al.,* 2017), SiO_2/SnO_2 (Ciorcero *et al.,* 2018), Cu-nanoparticles and multiwall carbon nanotubes (Jafari et al., 2020) and Au-Copper oxide nanocomposite (Mohammad-Rezaei and Golmohammadpour, 2020). Modification of carbon-ceramic electrodes using metal or metal oxides has an impact on increasing the price of these electrodes. For this reason, it is necessary to conduct research on the modification of carbon-ceramic electrodes using cheaper materials. A number of non-metal materials such as organoclay (Pramono *et al.,* 2019), silicon carbide (Solovei *et al.,* 2019) were used as a ceramic matrix in preparation of carbon-ceramic electrodes for electroanalysis application.

The aim of the present study was to prepare carbon-ceramic composite electrodes that have good electrical conductivity, mechanically stable, and inexpensive. The carbon-ceramic composite electrode was prepared using alumina, kaolin, and liquid clay as a ceramics matrix with the addition of different carbon content which was used

to provide the conductive phase. Kaolin and clay consist of silicates which were used to maintain the plasticity property of ceramics, while alumina was used to improve the mechanical stability such as hardness and corrosion resistance of ceramics. This electrode was investigated in terms of their electrical conductivity, surface morphology, and the crystalline phase. The electrode with the highest electrical conductivity value was then tested for its electrocatalytic ability to degrade Remazol black B dye as one of textile dyes frequently used in dyeing fabrics in Bali, Indonesia in indirect electrochemical oxidation reactor.

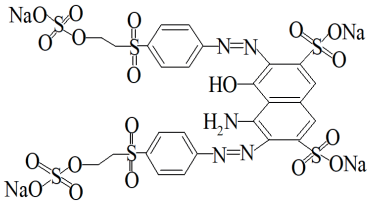
2. Materials and Methods

2.1 Materials for carbon-ceramic composite electrode preparation

In this study, the main chemical used for preparation of electrically carbon-ceramic composite electrodes were alumina, kaolin, liquid clay and carbon from coconut shell. The size of the raw conductive ceramic composite materials passed in sieve was 500 mesh. The metal elements content of carbon from coconut shell and liquid clay were analyzed by X-ray Fluorescence and the analysis result is listed in Table 1.

2.2 Textile Dye Characterization

The textile dye used as a treated sample in an indirect electrochemical oxidation reactor using carbon-ceramic composite electrode was Remazol black B from Sigma-Aldrich with CAS number of 17095-24-8. The IUPAC name for remazol black B is tetrasodium 4-amino-5-hydroxy-3, 6-bis[[4-[[2-(sulphonatooxy)ethyl] sulphonyl] phenyl] azo] naphthalene-2, 7-disulphonate with their chemical structure is presented as follows:



Remazol black B textile dye

2.3 Preparation of carbon from coconut shell

The carbon powder was prepared by burning the coconut shell in a slightly closed container for 2 h. Then, the carbon was crushed into powder using a grinding machine. Metal oxides contained in the carbon powder were removed by dissolving it in 0.5 M HCl solution while stirring it with a magnetic stirrer for 24 h.

Table1. Metal elements content of natural clay and coconut shell carbon

Liquid clay	Al	Si	K	Ti	Fe	Ca	Cu
	19.4	59.1	9.0	3.9	5.98	2.17	0.12
Carbon from coconut shell	Cr	Mn	K	Ti	Fe	Ba	Cu
	0.63	0.2	68.0	0.55	12.8	11.00	1.1

Table 2. Material compositions for carbon-ceramic composite electrode preparation

Electrodes	Materials proportion				
	Carbon (%)	Kaolin (%)	liquid clay (%)	Alumina (%)	PVA (%)
1	0.0	46.5	46.5	5.0	2.0
2	5.0	44.0	44.0	5.0	2.0
3	10.0	41.5	41.5	5.0	2.0
4	15.0	39.0	39.0	5.0	2.0
5	20.0	36.5	36.5	5.0	2.0
6	25.0	34.0	34.0	5.0	2.0
7	30.0	31.5	31.5	5.0	2.0

Furthermore, the carbon powder was filtered with Whatman No.1 filter paper, neutralized with NaOH solution, and then heated at 105°C for 3 h. until the constant weight was obtained. The dried carbon was crushed into a fine carbon powder using a ball milling machine for 24 h, and then filtered with sieve size of 500 mesh.

2.4 Preparation of carbon-ceramic composite electrode

A total of seven formulations of carbon-ceramic composite electrodes were prepared with different carbon contents. The summary of the material composition of carbon-ceramic composite electrodes materials are listed in Table 2.

The material mixtures were heated in oven at 80°C for 3 h. while they were homogenized by manual stirring. The solid mixture was casted in stainless steel mold with diameters of 0.5 cm and length of 10.0 cm and then pressed at load of 5 tons using mechanical pressing machine. The final product of carbon-ceramic composite electrode for electrical conductivity test had a diameter of 0.5 cm and length of 1.0 cm, while for textile dye degradation test was prepared the electrode with diameter of 0.5 cm and length of 8 cm. Each electrode was sintered at temperature 900°C for 6 h in vacuum furnace. The physical performance of carbon-ceramic composite electrodes with different carbon content is shown in Figure 1.

2.5 Characterization of carbon-ceramic composite electrode

2.5.1 Electrical conductivity measurement

Electrical conductivity of carbon-ceramic composite electrode was determined by measuring their resistivity according to ASTM D257 standard method. Specimen was placed between two electrodes for sixty second, and then the voltage was applied and resistance measured. The resistance of ceramic electrode was determined by the formula:

$$\rho = \frac{R.A}{l}$$

ρ = resistivity (Ωm)
R= resistance (Ω)
A = cross sectional area (m2)
l = length of specimen (m)

The electrical conductivity was calculated using the equation

$$\sigma = \frac{1}{\rho}$$

σ = electrical conductivity (S/m).
ρ = resistivity (Ωm)

2.5.2 Physical characterization

Scanning electron microscope (SEM, Phenom) was used to analyze the surface morphology of carbon-ceramic composite electrodes while the diffraction pattern was identified using X-ray diffractometer (XRD) merk PanAnalytical, type E'xpert Pro with a generator settings of 35 mA,40 kV and scan angle range of 20-90o. From diffractogram was calculated the percentage of crystalline phase of electrode using Origin Pro 8.5 software.

2.6 Experimental procedure

Batch experiments for Remazol black B degradation was conducted in an electrochemical oxidation reactor. Carbon-ceramic composite electrode with diameters of 0.5 cm and length of 8.0 cm was used as an anode and cathode. A DC power supply was used as an electric source. The experimental setup is shown in Figure 2.

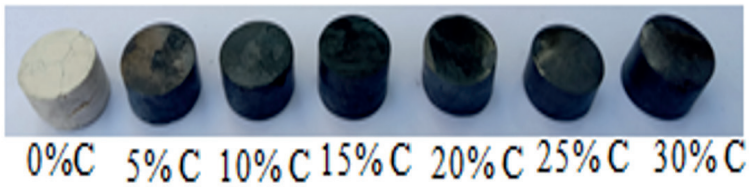


Figure 1. Photograph of carbon-ceramic composite electrode prepared from different carbon content

A total of 500 mL of Remazol black B dye solution with a concentration of 100 mg/L was placed in an electrochemical reactor while stirring using magnetic stirrer to homogenize electrolyte distribution. Operating variables which investigated were the effect of initial pH, table salt concentration, and dye concentration.

2.7 Analytical procedure

A double beam Shimadzu-1700 UV-Visible spectrophotometer was used to measure the absorbance of dye at 600.5 nm at before and after treatment. The color removal efficiency was calculated by the following formula:

$$Color\ removal\ efficiency(\%) = \frac{A_o - A_i}{A_o} x100\%$$

Where Ao and Ai are the absorbance of dye at before and after treatment, respectively.

3. Results and Discussion

3.1 Characteristic of Carbon-Ceramic Composite Electrode

3.1.1 Electrical Conductivity

In this research, carbon-ceramic composite electrodes were prepared using alumina, kaolin and liquid clay as ceramic matrix with carbon from coconut shell as conductive phase. The effect of the carbon content on the electrical conductivity of carbon-ceramic composite electrode is presented in Figure 3.

It can be clearly observed in Figure 3, that the electrical conductivity of the carbon-ceramic composite electrode increases with the increase in carbon content. The addition of 5% carbon has an electrical conductivity value of 0.35 S/cm, and when the carbon is increased to 30% inside ceramic composite, the electrical conductivity increases to 5.04 S/cm.

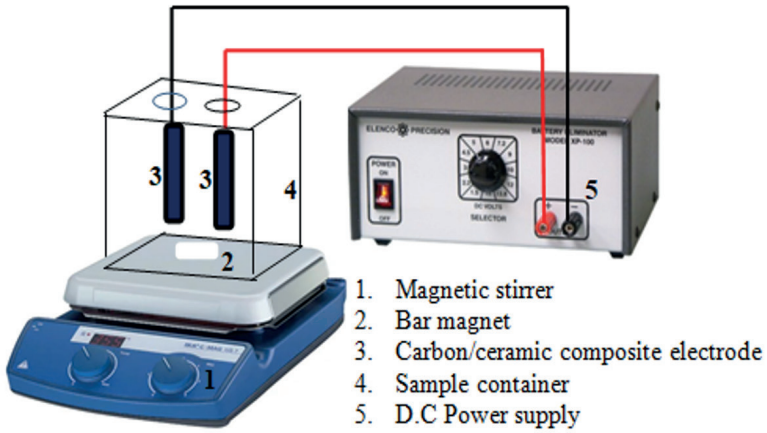


Figure 2. Schematic diagram of experimental set up

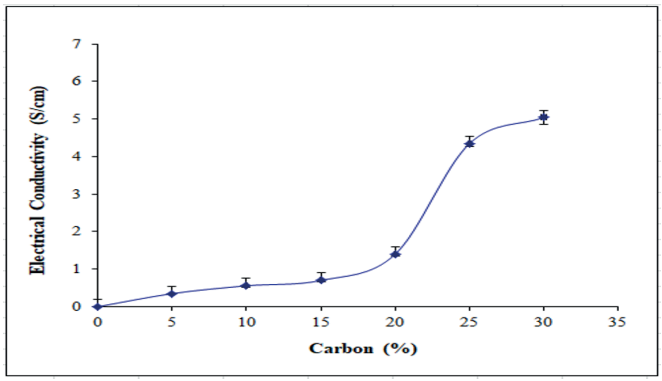


Figure 3. Electrically conductive carbon-ceramic composite electrodes with different carbon content

This is mainly attributed to the electrical conductivity of carbon black of 20 S/cm (McCreery, 2008). In addition, the increase of the amount of carbon in the ceramic composite causes a conductive pathway to form which makes the free electrons in the composite to travel easily, and eventually the electrical conductivity will increase. This finding is in line with Pramono *et al.* (2019), who reported that the higher carbon powder content increases the electrical conductivity of carbon ceramic composite. In comparison, the study conducted by Liu *et al.* (2012), also found that adding carbon black to rubber was able to increase the electrical conductivity, which was indicated by a decrease in resistivity from 587 Ω cm to 165Ω cm.

3.1.2 Surface morphology

SEM was performed to visualize the surface morphology of the control electrode (without carbon addition) and electrode with the variation of carbon addition. As shown in Figure 4a, the surface morphology of the control electrode rather heterogeneous and it appears the protuberances and hollow space.

When the carbon content within the electrode is 5% w/w (Figure 4b), the carbon particles have been linked together to form a chain, but due to the small amount of carbon the chains are separated by a cavity so theceramic electrode has low electrical conductivity. The presence of cavities in the composite will weaken the electrical conductivity of the electrodes. In contrast, when the carbon content in the carbon-ceramic electrode is 30% (Figure 4c), the surface morphology is more homogenous in which the carbon particles are evenly distributed on the surface of the ceramic and cover the pores of the ceramic particles. As a result, carbon-ceramics electrode with carbon content of 30% has higher electrical conductivity than carbon-ceramics electrode with 5 wt.% carbon content.

3.1.3 Diffraction pattern analysis

XRD was used to identify the amorphous or crystalline nature of carbon-ceramics composite electrode after sintered at 900°C. XRD diffraction test results for carbon-ceramic composite electrodes were presented in Figure 5.

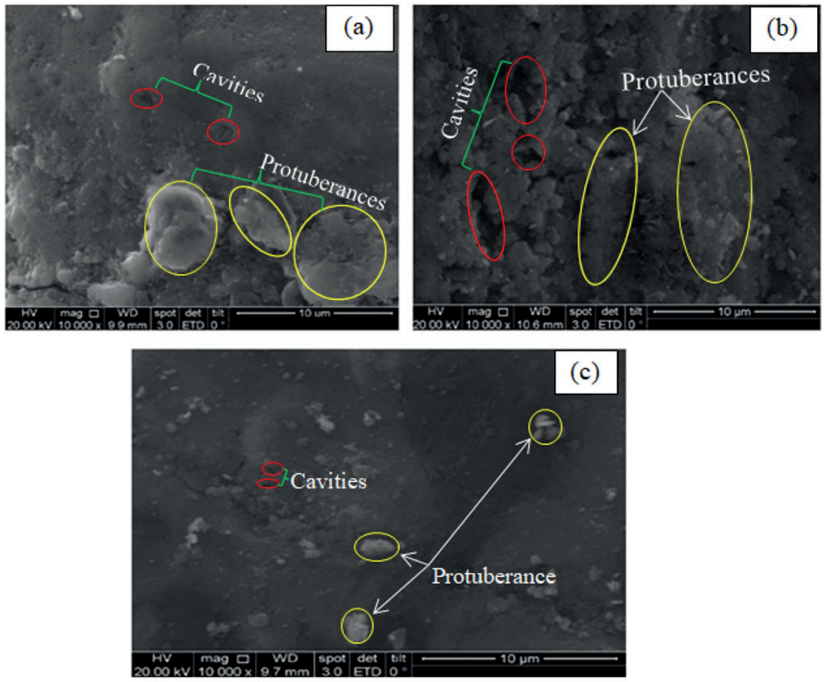


Figure 4. SEM micrographs of carbon-ceramic composite electrode after sintered at 900oC, (a) surface morphology of electrode without carbon addition, (b) surface morphology of electrode with 5 wt. % of carbon content and (c) surface morphology of electrode with 30 wt. % of carbon content.

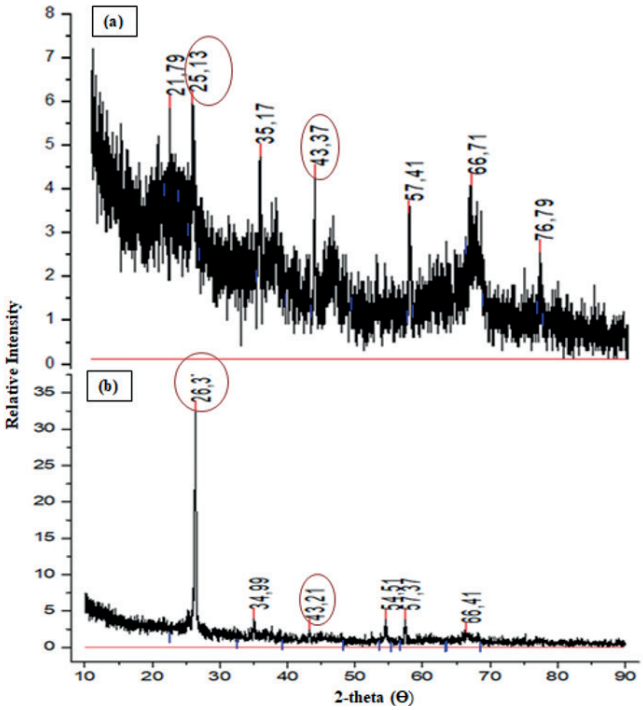


Figure 5. XRD pattern of carbon-ceramic composite electrode, (a) XRD pattern of electrode without carbon addition and (b) XRD pattern of electrode with 30 % wt. carbon content

The crystalline phase for carbon material is observed at 2θ angles of 25° and 45° corresponding to the 002 and 100/101 plane, respectively (Konwar *et al.,* 2016). As evident in Figure 5a, seven unsharpened and irregular XRD peaks were observed for the electrode without carbon addition at a 2Θ angle of 21.79°; 25.13°; 35.17°; 43.37°; 57.41°; 66.71°, and 76.79° and its indication that the electrode has a low crystallinity degree. The XRD peaks at around angle 25° and 35° represent both kaolin and alumina peaks, while the XRD peaks approximately at 2Θ = 43°; 57° and 66° correspond to the alumina peaks. By using Origin Pro 8.5 software, the crystallinity degree of the electrode without carbon content was obtained by 28%, with a crystal area of 41.478 and a profile area of 148.003. As seen in Figure 5b, the strong and sharp XRD peaks of the carbon-ceramic composite electrode with 30% carbon content were observed at 2Θ = 26.3° in which this diffraction angle (2Θ) near 26.5° corresponding to the 002 plane of crystalline graphite (Popova, 2017). The crystallinity degree of this electrode was 50.593% with the crystal and profile area of 68.538 and 134.471, respectively. The increased degree of crystallinity means that the particles are arranged more regularly, thus facilitating the electrons transfer.

3.2 Degradation Remazol Black B

3.2.1 Influence of initial pH

To investigate the effect of initial pH on color removal of Remazol black B dye, a total of 500 mL dye solution with concentration of 100 mg/L was treated in electrochemical cell for 60 min. under variation of pH (2-10) with addition 3 g of table salt and cell voltage applied of 12 V. The distance of anode to cathode is kept constant at 5 cm. Figure 6 shows the effect of initial pH on color removal efficiency of Remazol black B dye.

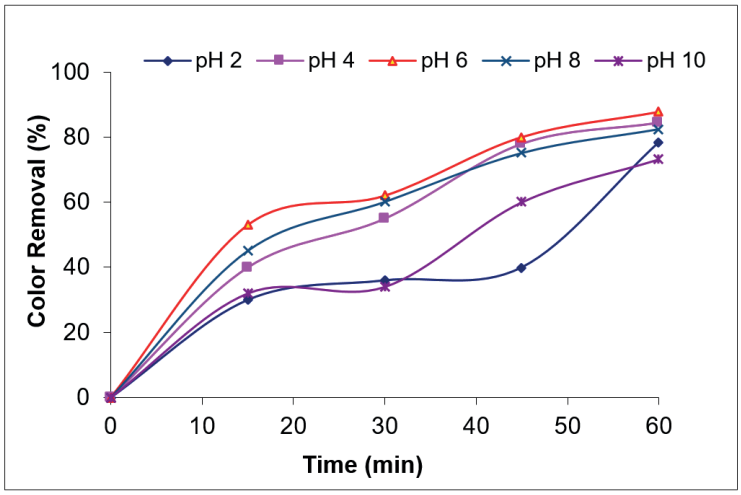


Figure 6. The influence of initial pH on color removal efficiency

It can be seen from Figure 6 the color removal rate of Remazol black B by indirect electrochemical oxidation method using carbon-ceramic composite electrodes increased with the increase in pH at range of 2 to 6, and then tends to slightly increased in pH treatment over than 8. However, the treatments at pH range of 2-10, the color removal efficiency was 73.42-87.75% after 60 min. for electrolysis time. Therefore, the pH of 6 is the considered as the suitable pH for Remazol black B degradation. In indirect electrochemical oxidation processr using sodium chloride as an electrolyte, In-situ generated active chlorine from oxidation of chloride ion at the anode plays an important role in the degradation process of the dye. The formation of active chlorine can be described by the following reaction.

$$Anode: 2Cl^- \longrightarrow Cl_2 + 2e$$
$$Chatode: 2H_2O + 2e \longrightarrow 2OH^- + H_2$$
$$Overal: 2H_2O + Cl^- \longrightarrow Cl_2 + 2OH^- + H_2$$

Chlorine gas (Cl_2) formed it tend to undergo react with the water to form hypochlorous acid (HOCl) and then it will partially dissociated to form hypochlorite ion.

$$Cl_2 + 2H_2O \rightleftharpoons HOCl \rightleftharpoons OCl^- + H^+$$

As shown in the reaction equations of 5-7, that the relative proportion of species active chlorine formed was dependent on the pH. Chlorine (Cl_2) is predominantly formed when the electrolysis conducted at very acidic conditions, HClO dominant at 3.3<pH<7.5, while OCl^- is present at the electrolysis pH running above 7.5 (Azeroual *et al.,* 2017). Active chlorine is strong oxidants by attributed with the reduction potential (E^ored) order of Cl_2, HOCl and ClO^- were 1.36 V, 1.49 V and 0.89 V vs. SHE, respectively. Based on the reduction potential value of active chlorine, HOCl is the strongest oxidant and it plays a dominant role for degradation of Remazol black B when the chemical oxidation process is carried out at pH of 5-7. This finding confirms the research result by Subramaniam *et al.,* 2016 who reported that the optimum pH for color removal efficiency of textile wastewater by an electrochemical oxidation method using graphite electrode was at pH 5 with color removal percentage of 93.89% for 120 min. at pH 5, current density of 0.28A/cm2, sodium chloride doses of 2.5 M and the distance between electrodes of 3 cm.

3.2.2 Influence of table salt concentration

Degradation process of Remazol black B in indirect electrochemical oxidation reactor was carried out at initial pH 6 and cell voltage of 12 V for 60 min. electrolysis time with different table salt dose as source chloride ion (1-5 g/L). The color removal efficiency of 100 mg/L Remazol black B at different table salt dose was presented in Figure 7

As shown in Figure 7, it is clearly observed that the electrocatalytic degradation of Remazol black B is significantly influenced by the presence of chloride ions in bulk solution. The color removal efficiency was 87.75% with applied voltage of 12 V in the presence of 3 g/L of table salt for 60 min. electrolysis time. However, the addition of table salt more than 3 g/L did not significantly affect the improvement of the color removal efficiency. The effect of electrolyte concentration on oxidation rate of dye was also investigated by Labiadh *et al.* (2017) who reported that the oxidation rate of indigo carmine dye increased with the increases of sodium chloride used in electrochemical oxidation process.

3.3.3 Influence of initial dye concentration

To investigate the effect of dye concentration on color removal efficiency, experiments were conducted at different dye concentrations (50, 100, 150 and 200 mg/L) for 60 min. with pH of 6, table salt dose of 3 g/L and an applied cell voltage of 12 V. Percentage of color removal efficiency at different Remazol black B concentration is presented in Figure 8.

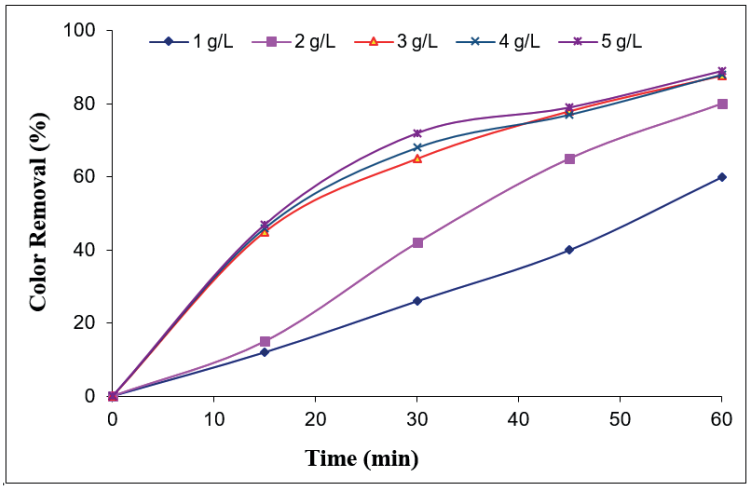


Figure 7. The influence of table salt concentration on color removal efficiency

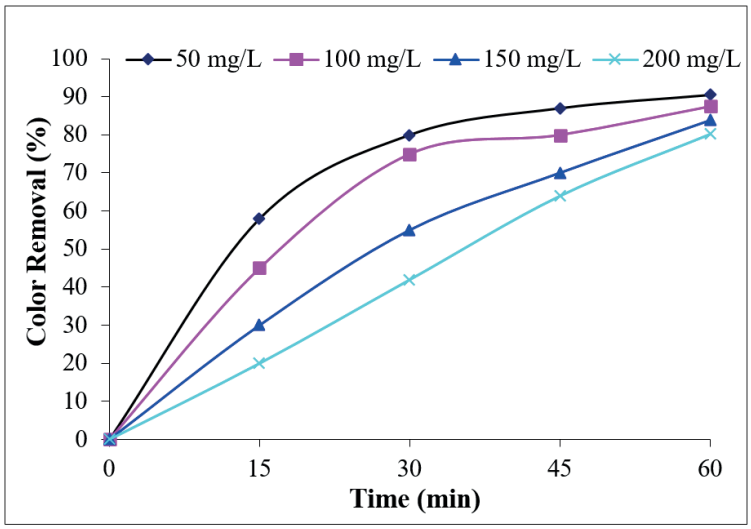


Figure 8. The influence of initial dye concentration on color removal efficiency

It is quite clear observed from Figure 8, the color removal efficiency decreases with an increase in dye concentration. The electrocatalytic process is controlled by mass transfer, in which the electrocatalytic activity is faster at low dye concentrations than the diffusion process. Color removal efficiency of dye in the concentration range of 50 - 100 mg/L was 90.52 - 87.75 %, however decrease to 83.80 - 80.25% when the dye concentration is increased at range of 150 - 200 mg/L. This finding is consistent with the research result conducted by Baddouh *et al.* (2020) who reported that an increase of dye concentration results in decrease in the electrocatalytic rate of degradation. The complete color efficiency with COD reduction was achieved of 73% within 90 min. of electrolysis for initial dye concentration of 100 mg/L. In this study, after the electrolysis running for 60 min, the color removal efficiency was achieved in the range of 80.25 - 90.52% for an initial dye concentration at range of 50 - 200 mg/L.

4. Conclusion

The electrically conductive of the carbon ceramic composite electrode is significantly affected by the amount of carbon used as the conductive phase. Carbon-ceramic composite electrodes was prepared with the composition of alumina, kaolin, liquid clay, carbon powder and PVA as a binder in successive of 5%, 31.5%, 31.5%, 30%, and 2% based total weight had electrical conductivity of 5.04 S/cm. This electrode was electrically conductive and proven to be effective as an electrode to degrade textile dyes in indirect electrochemical oxidation reactor. Color removal efficiency was obtained of 87.75% in the degradation of 100 mg /L Remazol black B textile dye for 60 min. of electrolysis time at initial pH of 6, addition of 3 g/L table salt and an applied cell voltage of 12 V. In addition, carbon-ceramic composite electrode was potential to be used as electrode in electrochemical cell for wastewater treatment.

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