Synthesis of ferrate from ferrous sulfate using wet chemical approach and its application for remazol black B degradation: optimization and kinetic studies

by Nor Basid Adiwibawa Prasetya

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Synthesis of ferrate from ferrous sulfate using wet chemical approach and its application for remazol black B degradation: optimization and kinetic studies

N B A Prasetya[1], S J Suripto[1], A Haris[1], Ngadiwiyana[1], N S Mohammed[2]

[1]Department of Chemistry, Faculty of Sciences and Mathematics, Diponegoro University, Semarang, Indonesia
[2]College of Education, Kirkuk University, Kirkuk, Iraq

Corresponding author: nor.basid.prasetya@live.undip.ac.id

Abstract. Chemical oxidation is an effective method for treating wastewater that contains dyes. Ferrate is known as the strongest and environmentally friendly oxidizer in aqueous media. This study aims to degrade remazol black B dyes using ferrate synthesized by the wet chemical method from Ferrous ion of $FeSO_4.7H_2O$. This research studies the optimum conditions of degradation including pH, molar ratio and optimum time, degradation kinetics and the comparison of the effectiveness in degrading Remazol Black B dyes among ferrate to permanganate, peroxide and dichromate. The results show that ferrate can oxidize remazol black B effectively at an optimum pH 8, with a molar ratio of ferrates:dyes of 5:1 and the optimum time of degradation of 120 minutes, with the degradation percentage reaches up to 95%. The degradation kinetics of dyes follows first-order kinetics, and ferrate has the highest degradation effectiveness compared to permanganate, peroxide and dichromate.

1. Introduction

The textile industry is a huge industry that uses massive amount of water in the production process [1]. Wastewater released contains parameters BOD, COD, suspended solid and relatively high dyes [2]. The dyes are mostly aromatic complex compounds that are usually difficult to degrade [3]. Therefore, the disposal of textile waste containing dyes requires an effective handling study so to avoid environmental pollution.

Remazol Black B is one of the organic dyes mostly found in textile waste. This colorant has a chromophore from the azo group (-N=N-) that binds with aromatic groups and has ease of application, low fading, and bright color but has resistance to biodegradation [4,5]. Several methods of processing dyestuff waste have been carried out, such as coagulation, adsorption, and membrane filtration, which effectively removes the color in wastewater [5,6,7]. However, these methods require further processing, for example, separation of the adsorbent from the dye. Therefore, chemical oxidation method has attracted a lot of attention in recent years. Studies on the degradation of azo dyes by different oxidation methods have been carried out, such as conventional oxidation, Fenton process, ozonation, ultraviolet (UV)-H_2O_2 rays, sonication, and continued oxidation via ferrate because of the strong oxidation ability [6,8].

Ferrate, a hexavalent form of iron ions (Fe^{6+}), is known as an effective reagent in water and sewage treatment. Ferrate has several beneficial characteristics from its chemical properties, such as high oxidation-reduction potential of ferrates under acidic conditions (+2.2 V) compared to some other oxidizing agents such as chlorine, permanganate, ozone, and hydrogen peroxide [9]. The redox potential of ferrate in the basic condition is relatively high at 0.7 V. Ferrate is an environmentally friendly oxidizer with non-toxic by-products (Fe(III)) or insoluble end products ($Fe(OH)_3$). Besides, ferrates can act dual function as an oxidant and coagulant [10].

Ferrate synthesis is known as a method that involves the chemical or electrochemical oxidation of iron (II) or iron (III) from aqueous solutions under high alkaline concentration. These methods generally employ $FeCl_3$, $Fe(NO_3)_3$, and Fe_2O_3 as the main source of iron [9, 11-14]. The use of Fe^{2+} as the main source of iron is still rarely conducted. Therefore, in this study the synthesis of ferrates using Fe^{2+} from $FeSO_4.7H_2O$ as the main source of iron. The obtained ferrate was used to degrade Remazol Black B dyes and find out the optimum reaction conditions, kinetic and degradation comparison with other oxidizers as well as Chemical Oxygen Demand (COD) analysis to the before and after degradation.

2. Experimental section

2.1. Materials

Ferrous sulfate pentahydrate ($FeSO_4.7H_2O$), potassium hydroxide (KOH), sodium hypochlorite (NaOCl), hydrogen peroxide (H_2O_2), potassium permanganate ($KMnO_4$), potassium dichromate ($K_2Cr_2O_7$), disodium phosphate (Na_2HPO_4), monosodium phosphate (NaH_2PO_4), and glass wool were obtained from Merck. Remazol Black B Dye content ≥50% was obtained from Sigma Aldrich, while aquadest was purchased from Diponegoro University Integrated Laboratory. All chemicals were used without further purification.

2.2. Equipments

Standard glasswares, magnetic stirrers (Venjoyit), analytical balance (O'haus PA214), pH meter, UV-Vis spectrophotometer (T60UV-Visible Spectrophotometer) and Chemical Oxygen Demand (Hach 2125925).

2.3. Synthesis of ferrates from $FeSO_4.7H_2O$

The synthesis of Ferrate (FeO_4^{2-}) was carried out by reacting KOH (31 g) with NaOCl solution (60 mL). Then, the mixture was stirred until homogeneous. Afterward, $FeSO_4.7H_2O$ (4 g) was added to the solution, and then it was continuously stirred for 30 minutes until the solution turns into a deep purple. The synthesis result was allowed to stand for one day, then it was filtered using glass wool. The solution was then characterized its maximum wavelength using a UV-Vis spectrophotometer with a range of 400-700 nm. The maximum wavelengths was also used to determine the concentrations of the synthesized ferrate.

2.4. Ferrate solution stability test

The ferrate solution was measured its absorbance using a UV-Vis spectrophotometer at the maximum wavelength of ferrate for 10 days. The absorbance value obtained was converted to a concentration value using Lambert-Beer Law.

2.5. Determination of optimum pH degradation

The first step to determine the optimum pH of degradation was by making a ratio of ferrate:Remazol Black B = 5:1. Each solution pH was adjusted from 7–11 by using some buffer solutions, which were sodium hydrogen orthophosphate and sodium dihydrogen orthophosphate for pH 7 and 8, as well as sodium hydrogen orthophosphate, sodium tetraborate, and sodium hydroxide for pH 9-11. Then 4 mL was taken from each solution, reacted for 60 minutes, and the absorbance of the sample was measured at the maximum wavelength of Remazol Black B.

2.6. Determination of the optimum molar ratio of degradation

Determination of the molar ratio was carried out by making molar ratio ferrate:Remazol Black B of 1:1; 2:1; 3:1; 4:1 and 5:1. Each sample with a different ratio was conditioned in the optimum pH with the addition of a buffer. A total of 4 mL of ferrate solution and dyes were then mixed and stirred for 60 minutes. The absorbance of the solution was analyzed using a UV-Vis spectrophotometer at the maximum wavelength of Remazol Black B.

2.7. Determination of the optimum degradation time

Determination of the optimum degradation time was done by making a solution of ferrate and Remazol Black B dyes under optimum pH and molar ratios. Each solution was taken as much as 4 mL, and the solution was stirred. The absorbance was measured every 10 minutes using a UV-Vis spectrophotometer at the maximum wavelength of remazol black B until reaching 180 minutes.

2.8. Degradation of Remazol Black B dyes and degradation kinetics

The degradation solution was measured its absorbance using UV-Vis spectrophotometer at the maximum wavelength of the dyes. The absorbance value of the sample was plotted in the equation obtained by a standard curve to calculate the value of the remazol Black B concentration from degradation. The degradation solution was also scanned for its wavelengths in the range of 200-800 nm using a UV-Vis spectrophotometer. Determination of the percentage of degradation of Remazol Black B dyes was calculated using the following formula:

$$\%\ \text{Degraded Dyes} = \frac{[\text{RBB}]_i - [\text{RBB}]_f}{[\text{RBB}]_i} \times 100\%$$

where $[\text{RBB}]_i$ is the initial concentration of remazol black B, while $[\text{RBB}]_f$ is the concentration of remazol black B after the reaction.

2.9. Comparison of the dye degradation effectiveness by ferrate with $KMnO_4$, H_2O_2, and $K_2Cr_2O_7$

$KMnO_4$, H_2O_2, and $K_2Cr_2O_7$ solutions were prepared at the optimum pH and molar ratio of degradation as ferrate. Then, each solution was taken as much as 4 mL and was reacted with 4 mL remazol black B solution during the optimum time of degradation of ferrate. The absorbance was determined using a UV-Vis spectrophotometer on a maximum wavelength of Remazol Black B.

2.10. COD analysis

The first step of COD determination was the homogeneity of the sample. Then some samples were pipetted and added with a digestion solution. Furthermore, the solution was carried out to closed reflux for 2 hours at a temperature of 150°C ± 2°C. After the solution had cooled, then absorbance measurements were carried out with wavelengths of 600 nm or 420 nm. Then, the COD content was calculated based on the linear regression equation formed on the calibration curve (SNI 6989.2: 2009).

3. Result and discussion

3.1. Synthesis of ferrate

In this study, ferrate solution was synthesized using the wet method by reacting $FeSO_4.7H_2O$ and NaOCl under strong base conditions. To form the solution to be alkaline, KOH was added because ferrate has better stability under alkaline conditions than under acidic conditions. Ferrate species are formed in an alkaline atmosphere, while in the acidic atmosphere, the dominant species is $HFeO_4^-$ ion [13]. This synthesis uses $FeSO_4.7H_2O$ as a source of iron (II) and NaOCl as an oxidizer that will oxidize Fe(II) to Fe(VI). The synthesized ferrate has a specific color as a blackish-purple color. This color change indicates that the ferrate was successfully synthesized, with the overall reaction of ferrate synthesis shown in the following equation:

$$FeSO_4.7H_2O + 2OCl^- + 4OH^- \rightarrow FeO_4^{2-} + 2Cl^- + SO_4^{2-} + 9H_2O$$

3.2. Determination of maximum ferrate wavelength

The ferrate solution from the synthesis is blackish purple. So, this solution can be characterized using a UV-Vis spectrophotometer to obtain the maximum wavelength of 510 nm. The results are convenient with references that ferrate has a wavelength absorption between 505-510 nm [15]. This maximum wavelength is used to obtain the absorbance value of the ferrate solution which will be used to calculate the concentration of ferrate solution. The molar absorption of ferrate at a maximum wavelength of 510 nm in solution is 1150±25 $M^{-1}cm^{-1}$ [16]. This value is used to calculate the concentration of ferrates using the Lambert-Beer Law equation.

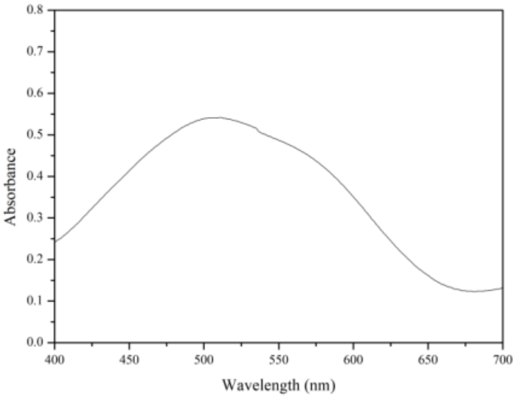

Figure 1. The absorbance of ferrate solution at a wavelength of 400-700 nm

3.3. Ferrate stability test

Ferrate stability test was determined by measuring the absorbance of ferrate solution for 10 days using a UV-Vis spectrophotometer. This stability test is carried out to determine the stability and effectiveness of ferrate as an oxidizer after being stored for a certain period of time. The results indicate a decrease in the concentration of ferrate from the first day to the 10th day shown in Figure 3. Besides, there is a decomposition of ferrates which is visually characterized by changes in the color of the solution. Initially, the color of solution is blackish purple and then changes rapidly to a brownish color which indicates that the ferrate has decomposed until the solution becomes colorless with the deposition at the bottom of the glass.

These results indicate that ferrate has low oxidative stability after being stored for a certain time. The low stability of ferrate due to its strong oxidizing properties makes it easy to oxidize other chemical species in the surrounding environment. Ferrate is unstable in the solution and will quickly be reduced to Fe(III) or the insoluble end product ($Fe(OH)_3$) [17]. A positive E°cell value proves that the decomposition of ferrate in water occur spontaneously, so that makes the ferrate in the solution unstable.

Reduksi : $FeO_4^{2-} + 4H_2O + 3e^- \rightarrow Fe(OH)_3 + 5OH^-$ $E^o = 0,72$ V
Oksidasi : $OH^- \rightarrow O_2 + 2H_2O + 4e^-$ $E^o = -0,401$ V
$4FeO_4^{2-} + 10H_2O \rightarrow 4Fe(OH)_3 + 8OH^- + 3O_2$ $E^o = 0,319$ V

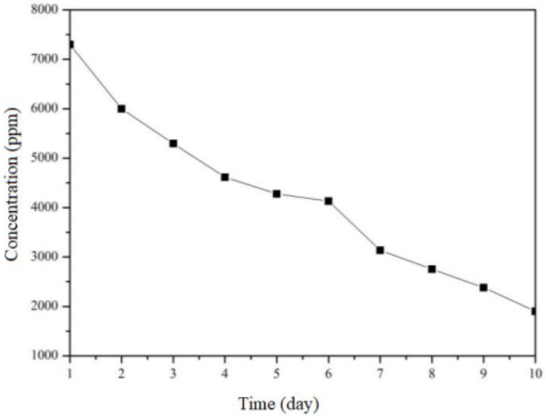

Figure 2. Graph of decrease in the concentration of ferrate solution

The kinetics that occurs at the rate of stability of the ferrate solution is measured as a function of time, with the parameter used in the reaction kinetics is the order of reaction. Determination of reaction kinetics by making plot graphs ranging from zero-order to third order. From the graph, R^2 values for the zero-order to third-order are obtained, 0.964; 0.980; 0.910, and 0.789, respectively, as seen in Figure 3.

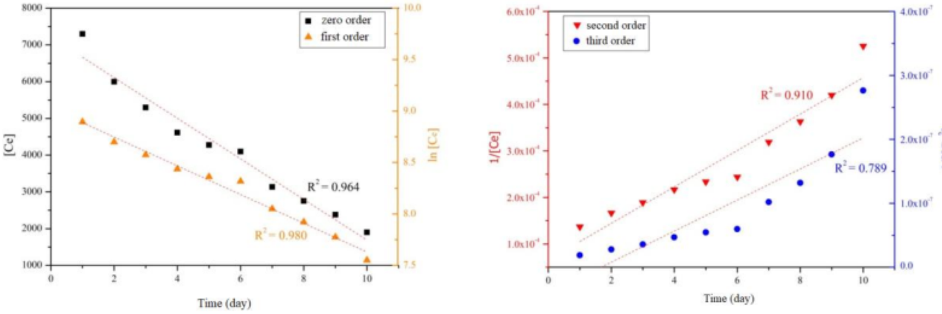

Figure 3. Graph of zero- and first-order (left) and Second- and third-order (right) stability of ferrate solution

R^2 value obtained from first-order has the highest and the closest value to one. So, the rate of ferrate solution stability is following first-order. This data is consistent with the reference that the rate of decomposition of ferrates follows first order on ferrates that have a pH> 10 [18].

The reaction order shows the effect of concentration on the reaction rate. The reaction follows first-order if the reaction rate is directly proportional to first of the reactant concentrations. This indicates that the stability of the ferrate solution is influenced by the ferrate itself.

3.4. Determination of optimum pH degradation

Figure 4 shows the highest percent degradation occurred at pH 8 with a percentage of 61.6%. Meanwhile, pH 7 and 9-11 showed a lower percentage of degradation of 56.2%, 52.4%, 50.9%, and 49.1%, respectively. Ferrate speciation in several pH ranges plays an important role in the degradation of dyes. In the acidic atmosphere, ferrates solution has species $H_3FeO_4^+$, H_2FeO_4, and $HFeO_4^-$. In neutral conditions, the ferrates species involved are $HFeO_4^-$ and FeO_4^{2-} with $HFeO_4^-$ species, which are more dominant, whereas in the alkaline solution conditions, the ferrate has FeO_4^{2-} species. $HFeO4^-$, is a type of iron monoprotonated species with reaction rates 3-5 times faster in oxidation capacity than the dose of FeO_4^{2-} in aqueous solution [19].

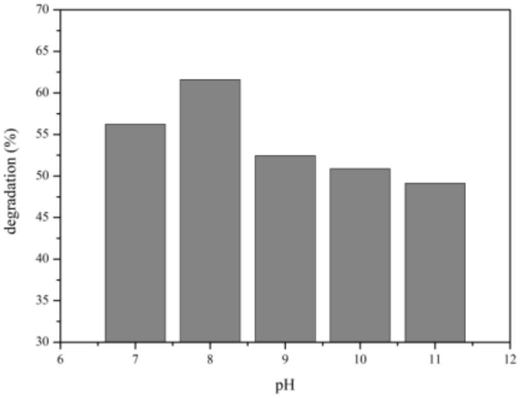

Figure 4. Graph of optimum pH for the degradation of Remazol Black B by ferrates under varying pH under conditions of 5:1 molar ratio, 60 minutes contact time, and a total volume of 8 mL

The results obtained indicate that the optimum pH is reached at pH 8. At this pH, there is $HFeO_4$ species - making it possible to play a major role in oxidizing Remazol Black B dyes, while at pH 7 a small percentage of degradation is possible due to the increasing number of ferrous monoprotonation species ($HFeO_4$-) causes the oxidation process to take place quickly but is not able to stabilize the solution so that under bases conditions ferrate easily decompose itself. The results also show that at pH 9-11, a decrease in the percentage of degradation is due to strong alkaline conditions, the oxidation ability of ferrates decreases with increasing ferric stability and the number of FeO_4^{2-} species.

3.5. Determination of the optimum molar ratio of degradation

Ferrate concentration is one of important factors affecting the degradation performance of dyes. Optimization of the concentration ratio is carried out at pH 8 using different molar ratios between ferrate:dye from 1:1 to 5:1 with a reaction time of 60 minutes. The optimum molar ratio results are shown in Figure 5. The percentage degradation of 1:1 5:1 molar ratio are 39.4%, 52.5%, 68.4%, 70.8% and 76.1%, respectively. These results indicate the efficiency of ferrate degradation increases with increasing concentration of ferrate ratio. The highest degradation percentage occurs at 5:1 molar ratio. It can be concluded that the higher the concentration of ferrate, the higher the ability of degradation. These results are consistent with the reference that stated efficiency of degradation increases with increasing molar ratio [16].

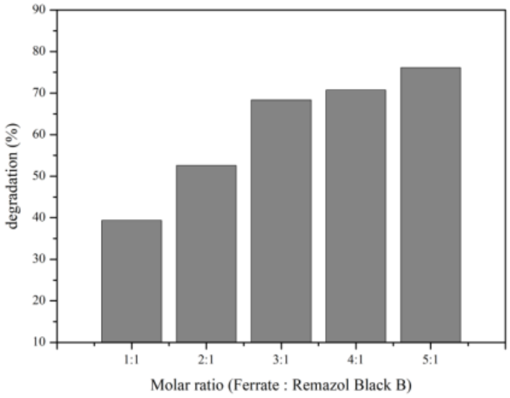

Figure 5. Graph of optimum molar ratio to degradation of Remazol Black B by ferrate with a various molar ratio condition, at pH 8, 60 minutes contact time, and a total volume of 8 mL

3.6. Determination of the optimum degradation time

The optimum time was determined by varying the time by conditioning the ferrate and Remazol Black B dyes below optimum pH and the optimum molar ratio (pH 8 and 5:1 molar ratio). Determination of the optimum time aims to find out the best time needed for ferrate to degrade Remazol Black B. The optimum degradation time results using ferrates are shown in Figure 6. These results show that the longer the degradation time, the greater the percentage of degradation. Determination of the optimum time is stopped after the 120th minutes, where the percent degradation of the dye reaches up to 95%. 120 minutes is set as the optimum time because the addition of degradation time will not give a much different. In other words, the percent degradation obtained starts constantly.

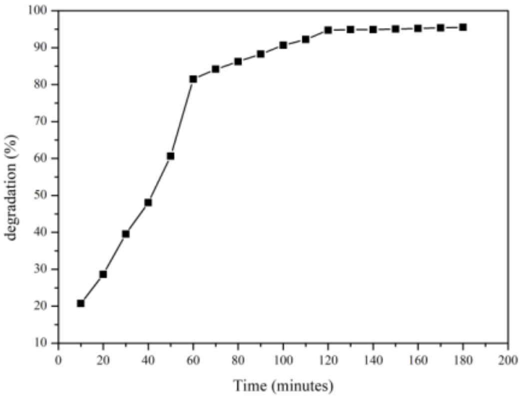

Figure 6. Graph of the optimum time to degradation of Remazol Black B dyes by ferrates under conditions at a 5:1 molar ratio and optimum pH 8 at various contact times with a volume of 8 mL

The kinetics of the degradation reaction is measured as a function of time, the parameter specified in the reaction order. Theoretically, this azo dye degradation reaction will follow first-order. The reaction order is determined by plotting a graph for reactions of zero-order, first-order, second-order, and third-order to obtain R^2 of 0.878, 0.968, 0.867 and 0.613, respectively. Thus, the azo dye degradation reaction in this study follows the order first reaction. The graph of the order of Remazol Black B degradation reaction can be seen in Figure 7.

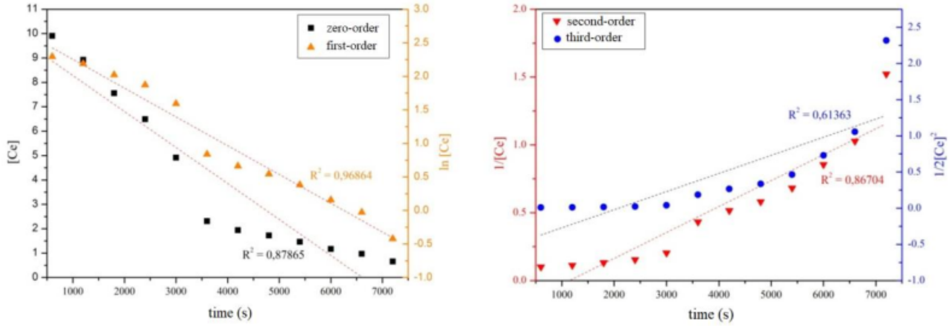

Figure 7. Graph of zero- and first-order (left) and second- and third-order (right) degradation of Remazol Black B by ferrate

3.7. *Comparison of the effectiveness of ferrates in degradation of Remazol Black B compared to other oxidizing agents*

Comparison of the effectiveness of ferrate in degrading Remazol Black B dyes is obtained by comparing it with other oxidizing agents, such as permanganate, peroxide, and dichromate. Each oxidizer chosen for degradation is used in the form of salts, $KMnO_4$, H_2O_2, and $K_2Cr_2O_7$. The results obtained can be seen in Figure 8.

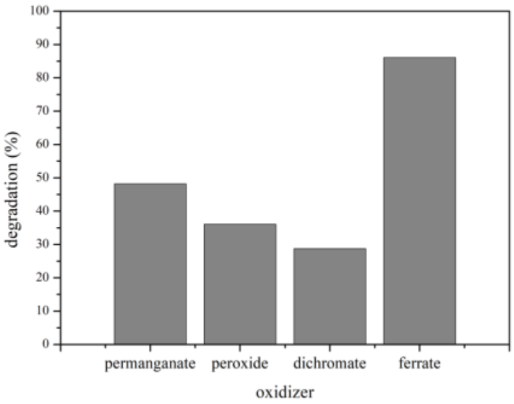

Figure 8. Comparison graph of the effectiveness of ferrate with other oxidizing conditions under pH, molar ratio, and optimum time with a sample volume of 8 mL

The percentage degradation of potassium dichromate, hydrogen peroxide, potassium permanganate, and ferrate are 28.8%, 36.1%, 48.2%, and 86.1%, respectively. The results show that ferrate has the greatest effectiveness in degrading Remazol Black B dyes compared to other oxidizing agents. This happens because the pH used is the optimum pH of the ferrate suitable for the stability of the ferrate. Ferrates are stable only under alkaline pH conditions, and when in acidic conditions ferrate oxidizes water [20].

3.8. Determination of sample absorption spectra before & after the degradation process

Determination of sample absorption spectra before and after the degradation process was carried out to determine the absorption at wavelengths of 200-800 nm before and after the degradation process. The measurement results are shown in Figure 9.

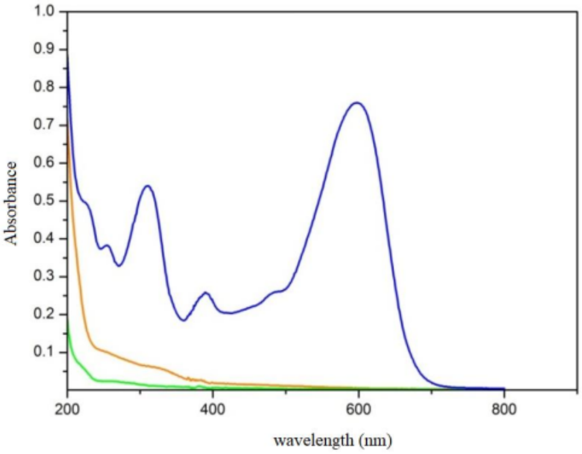

Figure 9. UV-visible spectra of buffer solution pH 8 (green line), Remazol black B before degradation (blue line), and Remazol black B after degradation (orange line) with ferrate

The absorbance spectra of Remazol Black B before degradation had several peaks at wavelengths of 255, 310, 390, and 598 nm. Based on the chemical structure of Remazol Black B, the peak at 255 is characteristic of conjugated benzene rings. The peaks at 310 and 390 nm support the conjugation of the benzene system, while the peak seen at the wavelength of 598 nm is caused by the azo group (-N=N-), which forms a chromogenic conjugation system.

Spectra after degradation shows the loss of the peak in the 590-610 nm wavelength range, which is probably due to the process of breaking of the nitrogen double bonds of the azo dyes. In addition, the absence of peaks at wavelengths of 200-800 nm indicates that the Remazol Black B sample has been degraded into short-chain carbon compounds and does not have chromophore groups (color giving). Degradation of dyes is characterized by the occurrence of decolorization or a decrease in color intensity [20]. Decolorization of the Remazol Black B makes the initial solution turns form dark blue to colorless, as shown in figure 10.

Aromatic degradation of dyes takes place more slowly due to the complexity of aromatic compounds. Also, the breakdown of complex color molecular structures causes a decrease in absorbance peaks in the range 300-320 nm. A relatively slow breakdown lasts longer so that at wavelengths <250, the spectra still provide a fairly high absorbance absorption [21].

(a) (b)

Figure 10. Remazol Black B sample: (a) before degradation (b) after degradation

3.9. *Characterization of samples from degradation results with COD*

Sample of Remazol Black B before and after degradation process was analyzed by COD. The purpose of this analysis aimed to determine the water quality after the degradation process to determine the success of the degradation process. The existing organic compounds are chemically decomposed by using strong oxidizing agents. COD represents the total amount of oxygen needed to oxidize organic material chemically (biodegradable) to carbon dioxide and water. The results of the analysis showed a decrease in COD levels in which COD before degradation of 71.8 ppm and after the degradation process of 8.6 ppm. In other words, ferrate can reduce the COD by up to 88%.

4. Conclusion

Ferrate was successfully synthesized from ferrous chloride by wet chemical approach to produce a blackish-purple ferrate solution. Ferrate effectively degrades remazol black B at optimum pH 8 with a molar ratio of ferrate: remazol black B of 5: 1 and the optimum degradation time of 120 minutes with a degradation percentage up to 95%. The degradation kinetics of Remazol Black B dyes with ferrates following the first order of reaction and ferrates have the highest degradation effectiveness when compared to oxidizers $KMnO_4$, $K_2Cr_2O_7$, and H_2O_2. The COD analysis reveals that ferrate can reduce the COD value by up to 88%.

Acknowledgments

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