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Submission date: 02-Aug-2021 11:07AM (UTC+0700)
Submission ID: 1626795848
File name: Gunawan_2021_J._Phys.__Conf._Ser._1943_012181.pdf (1.26M)
Word count: 4615
Character count: 22845

Synthesis of ferrate using NaOCl and $Fe(OH)_3$ from electrolysis of used iron, and its application for metanil yellow degradation

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Abstract. Synthesis of ferrate using NaOCl and $Fe(OH)_3$ from electrolysis of used iron, and its application for metanil yellow degradation had been done. The electrolysis was conducted using used iron and zink plates at anode and cathode electrodes, respectively, Na_2SO_4 electrolyte. The dissolved iron ion from the electrolysis then was made alkaline using NaOH pellet and NaOCl was soaked to the solution to form ferrate (FeO_4^{2-}). The ferrate was used for metanil yellow degradation and it was compared with other oxidators such as permananate, hydrogen peroxide and dichromate. The ferrate solution formed was indicated by a change from brown to dark purple which was indicated the oxidation of Fe(III) to Fe(VI) with a maximum wavelength of 510 nm. The optimum degradation of metanil yellow by ferrate was obtained at pH 8, molar ratio of 2:1, and for 25 min. Ferrate was the most effective oxidator compared with permananate, hydrogen peroxide and dichromate.

1. Introduction

Clean water is one of the most vital needs because all community activities require clean water for life, including drinking, washing, cooking and others. However, nowadays water pollution has become a very serious problem in both rural and urban areas. Water pollution can occur due to chemical compounds whose levels exceed the threshold due to human activities, one of which is dye waste in textile industries.

Several methods both biological, physical, and chemical to remove dyes have been widely used. Biological decolorization methods using a mixture of fungi have been carried out, but the results obtained are less effective and require a very high cost [1]. Physical decolorization of dyes has also been widely carried out, namely by means of adsorption using zeolite and chitosan. But this physical method will produce a number of adsorbent solid waste which is filled with pollutants [2]. The chemical method for dye decolorization can be done by photodegradation using the help of photon energy or UV radiation [3]. However, the photocatalytic method requires a large amount of money due to the high cost of the photocatalyst material. Chemical decolorization, namely using coagulants, is known to have a fairly good efficiency, but this decolorization will cause new problems, which will produce sludge and also new waste, namely a coagulant that can no longer be used. Decolorization using the coagulation method will also require large costs because the sludge produced from the textile industry waste treatment requires further processing [4].

One of the methods used to reduce water pollution is the degradation method of wastewater using oxidizers. Oxidizing agents are considered effective in degrading organic and nonorganic compound

wastes [5]. Oxidizers can be used in the waste treatment process by degrading a compound into a new compound that is relatively harmless. Examples of commonly used oxidizers include dichromate ($Cr_2O_7^{2-}$), permanganate (MnO_4^-), and peroxide (H_2O_2). However, these oxidizers are not environmentally friendly because they are toxic [6]. So an oxidizer that is safer for environment and effective in oxidizing pollutants is needed, namely K_2FeO_4 or in the form of the ion as ferrate (FeO_4^{2-}) [7-10] .

Ferrate (FeO_4^{2-}) is one of the strong oxidizing agents in aqueous media. In acidic condition, the redox potential of ferrate ion (FeO_4^{2-}) is the highest of other oxidizers commonly used in wastewater treatment processes [11]. The standard half cell potential of ferrate (FeO_4^{2-}) has a value of +2.20 V to +0.72 V in acidic and alkaline solutions, respectively. Ferrate (FeO_4^{2-}) has many advantages, namely as a disinfectant, flocculant, and coagulant based on its higher reactivity and selectivity than other oxidizing agents [12]. As an oxidizer in an aqueous solution, Fe(VI) will be reduced to Fe(III) where the final product is insoluble in water and formed into $Fe(OH)_3$. The desired advantage of this process is that in treating water and wastewater, it can produce harmless products (clean water) with better quality (the risk of forming hazardous byproducts is smaller) and only requires lower operational costs[12].

Previously, [13] conducted research on metanil yellow dye degradation using ferrate. It was produced by an electrochemical method producing in-situ ferrate ion electrogeneration under acidic condition and metanil yellow dye degradation was 94% with 12 mM ferrate. Iron (Fe) which is often used for ferrate synthesis generally comes from $Fe(NO_3)_2$ or electrolysis of iron as anode to produce iron ion [14-16].

In this research, a degradation study of methanil yellow dye was performed using ferrate by reaction of NaOCl and $Fe(OH)_3$. Iron ion was obtained from electrolysis of used iron. To date, the synthesis of ferrate by this way has never been carried out. Effects of pH, molar ratio and time on the degradation of the dye metanil yellow and the effectiveness comparation of ferrate in degrading methanyl yellow dye with other oxidizing agents ($KMnO_4$, H_2O_2, and $K_2Cr_2O_7$) were also studied.

2. Experimental

2.1. Instruments

Instruments used: analytical balance of Ohaus (Pioneer), UV-Vis Spectrophotometer (LW V-200 ES), Digital DC power supply (Aditeg APS 3005), glassware, aluminum foil, fine filter paper, magnetic stirrer.

2.2. Materials

The materials used in this study include hydrochloric acid (HCl 37%, Merck pa), iron(III) chloride hexahydrate ($FeCl_3.6H_2O$, Merck pa), sodium hydroxide (NaOH, Merck pa), sodium tetraborate ($Na_2B_4O_7\ 10H_2O$, Merck pa), metanil yellow ($C_{18}H_{14}N_3NaO_3S$, Sigma Aldrich pa), potassium hydroxide (KOH, Merck pa), sodium hypochlorite (NaOCl, technical reagents), Glasswool (Merck), potassium permanganate ($KMnO_4$, BHD Chemical ltd pa), hydrogen peroxide (H_2O_2, Merck pa), potassium dichromate ($K_2Cr_2O_7$, Merck pa), iron plate from unused transformator, zink plate.

2.3. Producing Fe^{3+} from iron plate by electrolysis

Zink and iron plates from unused transformator 1.5 cm x 5 cm were cleaned using acetone in sonificator. After dried the 4 iron plate were used as anodes and zink plate was used as a cathode in the electrolysis system. The electrolysis was commenced by immersing both electrodes (the area immersed were 4 cm x1.5 cm and 4.5 x 2 cm for iron and zink plates, respectively) into the beaker containing 50 mL Na_2SO_4 1M with applied potential of 2.3 V for 1 h.

2.4. Synthesis of ferrate (FeO_4^{2-}) using $Fe(OH)_3$ and the oxidizer NaOCl

The solution after electrolysis was added with NaOH 14 M, then reacted with 50 mL of NaOCl solution. The mixture was stirred until the KOH dissolved completely. The solution was closed tightly and

allowed to stand for 1 day and then filtered using glasswool. The maximum wavelength of the ferrate was determined by a UV-Vis spectrophotometer.

2.5. Determination of ferrate solution stability

A total of 0.5 mL of ferrate solution was diluted into a 25 mL, then measured the absorbance of the ferrate using a UV-Vis spectrophotometer at the maximum wavelength of the ferrate, namely 510 nm. To see the level of ferrate stability, the absorbance of the ferrate was measured for 10 consecutive days.

2.6. Determination of the maximum wavelength of metanil yellow at pH 1-14

A series of 2.5 mL of 100 mg/L metanil yellow dye solutions was prepared at pH 1 until 14 using HCl and NaOH solution, then they were measured using a UV-Vis spectrophotometer at visible wavelength.

2.7. Determination of the optimal ph of the degradation of metanil yellow by ferrate

Determination of the optimum pH of degradation was carried out by making a ferrate: metanil yellow molar ratio of 5:1. A total of 2.5 mL ferrate 100 mg/L was adjusted from 7 to 11 using an alkaline solution into a 25 mL measuring flask, as well as 0.5 mL of metanil yellow of 100 mg/L. Furthermore, the ferrate and metanil yellow solutions at fixed certain pH were mixed in 100 mL volumetric flask by dilution and stirred for 30 min. Then the solution was measured for its absorbance at the wavelength of the metanil yellow. Furthermore, the percentage of metanil yellow degradation by ferrate is calculated using the formula:

$$\% \text{ degraded dye} = ([\text{dye}]_{\text{initial}} - [\text{dye}]_{\text{residual}}) / ([\text{initial dye}]) \times 100\%$$

2.8. Determination of the optimal molar ratio of metanil yellow degradation by ferrate

The molar ratio was determined by a molar ratio of ferrate:methanil yellow, with ratios of 1:1; 2:1; 3:1; 4:1; 5:1. A total of 3 mL of the ferrate solution adjusted to the molar ratio was reacted with 3 mL of the dye solution of metanil yellow at the optimum degradation pH and homogenized with a stirrer for 30 min. The absorbance of the solution was measured using a UV-Vis spectrophotometer at the maximum wavelength of metanil yellow solution.

2.9. Determination of the optimal time for metanil yellow degradation by ferrate

A total of 3 mL of ferrate solution and 3 mL of metanil yellow dye solution with a concentration at the optimum molar ratio were added to the optimum pH, then the solution was homogenized using magnetic strirrer. Every 2 min interval of the sample solution was measured its absorbance using a UV-Vis spectrophotometer at the maximum wavelength of the metanil yellow dye.

2.10. Effectiveness comparison of ferrate in the metanil yellow degradation compared to other oxidizing agents

A total of 3 mL of each 100 mg/L solution of potassium permanganate, potassium dichromate and hydrogen peroxide and 3 mL of the dye solution of metanil yellow with a concentration at the optimum molar ratio were added to at the optimum pH, then each solution was stirred for 30 min. The absorbance of the solution was measured using a UV-Vis spectrophotometer at a wavelength of metanil yellow.

2.11. Determination of UV-Vis spectra of metanil yellow sample before and after the degradation process

Determination of the absorption spectra of metanil yellow before and after treatment were carried out at optimum pH, molar ratio, and time of degradation by mixing 15 mL ferrate and 15 mL metanil yellow dye solutions at pH 8. The solution was homogenized by stirring for 30 min. The dyes before and after treatment were scanned at wavelengths from 200 until 800 nm using UV-Vis Spectrophotometer.

3. Results and discussion

3.1. Producing Fe^{3+} from iron plate by electrolysis

Synthesis of solid $Fe(OH)_3$ was carried out by electrolysis of iron plate waste as anode to form iron ion at potential decomposition as shown in figure 1, then it was reacted with NaOH. The obtained $Fe(OH)_3$ was 8.105 g. The addition of NaOH serves to make the solution in an alkaline atmosphere, because $Fe(OH)_3$ can only be formed as a solid in an alkaline pH, which is at a pH of more than 6.5 [17]. The $Fe(OH)_3$ precipitate formed is slightly reddish brown, which can be separated by filtering it using filter paper and drying it in a desiccator.

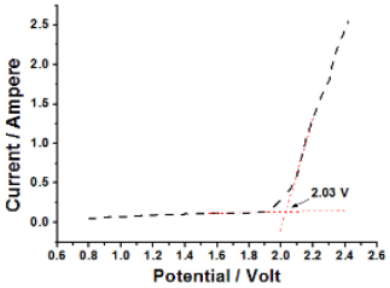

Figure 1. Electrolysis curve of iron plate as anode and zink as cathode.

3.2. Synthesis of ferrate (FeO_4^{2-}) from $Fe(OH)_3$ and NaOCl

$Fe(OH)_3$ obtained from the electrolysis of iron anode then was carried out using the "wet chemical" method to form ferrate, which in this method involves the oxidation of Fe^{3+} salts using NaOCl under alkaline conditions [18,19]. The synthesis of the ferrate solution was carried out by NaOCl solution addition in alkaline condition to the beaker containing $Fe(OH)_3$ solid then stirred until dissolved completely. NaOCl functions as an oxidizer that will oxidize Fe (III) ions to Fe(VI) ions [20]. The synthesized ferrate has a blackish purple color. The reactions that occur in ferrate synthesis are oxidation-reduction reactions which are shown as follows:

Reduction : $ClO^- + H_2O + 2e \leftrightarrow Cl^- + 2OH^-$ (1)

Oxidation : $Fe(OH)_3 + 5OH^- \leftrightarrow FeO_4^{2-} + 4H_2O + 5e$ (2)

Result : $5ClO^- + 2Fe(OH)_3 + 5OH^- \rightarrow 3Cl^- + 2FeO_4^{2-} + 5H_2O$ (3)

(brown) (violet)

The synthesized ferrate solution was stored in a closed and dark place for 24 h to maximize the oxidation process [21]. After that, the ferrate solution was filtered using a glasswool to remove impurities in the solution. The ferrate solution obtained was stored in a closed container and is dark in color to prevent the reduction to Fe^{3+} again as the following reaction [22]:

$$2FeO_4^{2-} + 5H_2O \longrightarrow 2Fe(OH)_3 + 3/2O_2 + 4OH^- \quad (4)$$

3.3. Determination of maximum wavelength of ferrate

The synthesized ferrate was characterized using a UV-Vis spectrophotometer. The maximum wavelength of the ferrate was carried out at visible wavelengths from 400 nm to 700 nm. The absorbance measurement for the ferrate vs wavelength is shown in Figure 2. The maximum wavelength of the ferrate is 510 nm which corresponds to the reference [23].

The actual ferrate concentration can be determined using the Lambert-Beer law equation. Since the molar absorptivity of ferrate at a maximum wavelength of 510 nm in solution is 1150 ± 25 $M^{-1}cm^{-1}$ at pH 9.5 to 10.5 [24] and pH 9.2 [25]. The concentration of the ferrate solution obtained was 8996 ppm.

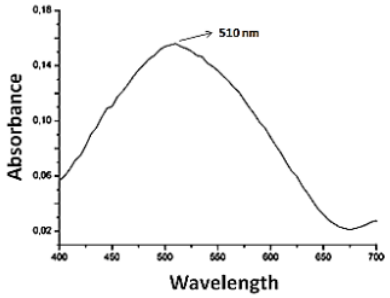

Figure 2. Absorbance of ferrate solution with 1 cm cuvet width at various wavelengths.

3.4. Determination of the stability of the ferrate solution

The stability of the ferrate solution can be analyzed by measuring the absorbance of the solution for 10 consecutive days using a UV-visible spectrophotometer shown in Figure 3. The results indicate that ferrate has unstable properties. The longer the storage, the lower the ferrate concentration. This is due to the fact that ferrate is a very strong oxidizing agent so it will be very easy to oxidize chemical species in the solution and it will be rapidly reduced from Fe (VI) to Fe (III) species as indicated by turning the solution to become brown [21].

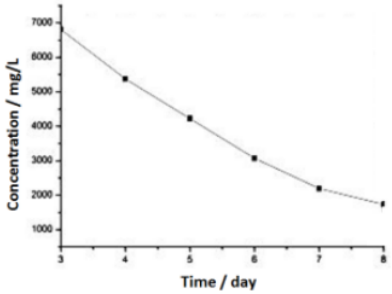

Figure 3. Stability curve of Ferrate

From Figure 3 shows that on the first day of observation, the ferrate solution has a deep purple color with a very high concentration of 8996 ppm. However, on the next day the ferrate concentration decreased, and the color of the ferrate solution had also turned brown and settled at the bottom of the solution as $Fe(OH)_3$ because the ferrate was reduced.

3.5. Characterization of the maximum wavelength of metanil yellow at various pH variations

The measurement results obtained that the optimum wavelength is 436 nm. The curves for determining the optimum wavelength of metanil yellow at pH 1 to 14 can be seen in Figure 4. Metanil yellow is a pH indicator that is often used in the titration process because of its clear and distinct color changes. At an acidic pH, which is around 2.9-4.0, metanil yellow has a reddish color. In acidic conditions, there will be neutralization of the metanil yellow ion ($C_{18}H_{14}N_3SO_3^-$) in the presence of protonation (H^+), while at pH above 4.0, metanil yellow has a yellow color. In alkaline conditions, there is no neutralization [26].

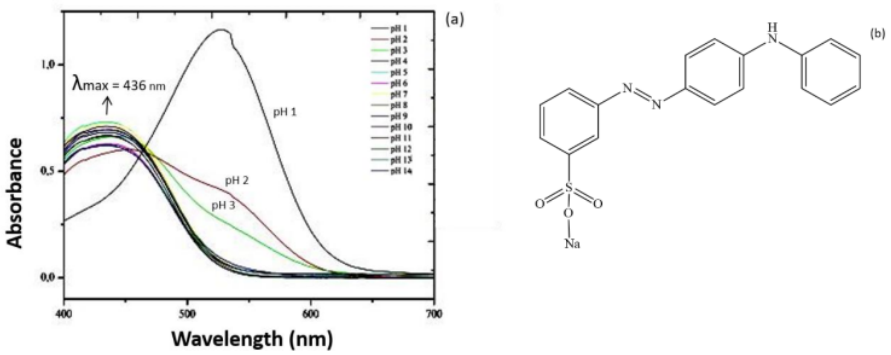

Figure 4. (a) The maximum wavelength of metanil yellow at various pH variations, the concentration of metanil yellow is 10 mg/L, (b) metanil yellow structure.

3.6. Determination of the Optimum pH for metanil yellow degradation by ferrate

The power of ferrate oxidation is very dependent on pH conditions. So it is necessary to know the optimal pH conditions for the degradation of metanil yellow. Ferrate is a strong oxidizer at alkaline pH and it will have a better stability than in acidic conditions. This is because ferrate will only form in an alkaline atmosphere while in an acidic atmosphere the dominant species is the $HFeO_4^-$ ion [19].

In determining the optimum pH of degradation was done by conditioning the ferrate and metanil yellow in alkaline pH conditions, from pH 7 to pH 11. Figure 4a shows that at pH 7 to 11 the maximum wavelength of metanil yellow does not have a shift so that the absorbance measurement of the solution is carried out at a wavelength of 436 nm. The molar ratio between ferrate and metanil yellow used was 5:1.

In acidic conditions the ferric solution has the species $H_3FeO_4^+$, H_2FeO_4, and $HFeO_4^-$. In neutral conditions the ferrate species involved are $HFeO_4^-$ and FeO_4^{2-} with the $HFeO_4^-$ species being more dominant, while in the alkaline solution conditions th4 $HFeO_4^-$ and FeO_4^{2-} species but the more dominating species are FeO_4^{2-} [27]. In Figure 2 shows that ferrate is stable only under alkaline pH conditions and will oxidize water when it is in acidic conditions. The results showed that the optimum pH for degradation of metanil yellow occurred at pH 8 with a degradation of 81%. The results are in line with [19] that the stability of ferrate in an alkaline pH atmosphere is better than an acidic one.

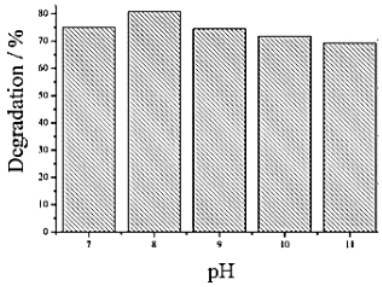

Figure 5. Degradation of metanil yellow by ferrate at various pH with a molar ratio of 5:1, contact time 30 min.

3.7. Determination of optimum ferrate and dye molar ratio for metanil yellow degradation

The molar ratio is an important variable affecting the efficiency of dye degradation. The determination of the optimum molar ratio for degradation of the metanil yellow by ferrate was carried out with molar ratios of ferrate to metanil yellow of 5:1; 4:1; 3:1; 2:1; 1:1 and at pH 8 as the optimum pH.

Figure 6 shows optimum molar ratio for metanil yellow degradation was 2:1 with degradation of 89.5%. The higher molar ratio between ferrate and dye results in an increase in the amount of ferrate(VI) available for the degradation reaction. However, excessive ferrate (VI) ratios will reduce the efficiency of degradation because ferrate(VI) will undergo decomposition [28].

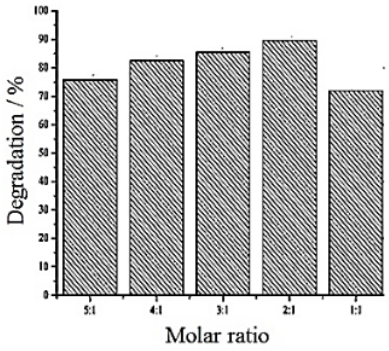

Figure 6. Metanil yellow degradation by ferrat with varied molar ratio at pH 8 for 30 min.

3.8. Determination of the optimal time of degradation of metanil yellow by ferrate

The determination of the optimum time for degradation of the metanil yellow by ferrate was carried out by conditioning the ferrate and methanyl yellow at the optimum pH (pH 8) and at the optimum molar ratio of 2:1. Figure 7 shows the optimum time of 25 min by the degradation of 93.3%.

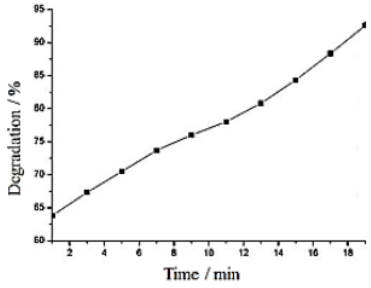

Figure 7. Degradation of metanil yellow by ferrate under optimum pH and molar ratio conditions at various contact times.

3.9. The effectiveness comparison of ferrate with potassium permanganate, potassium dichromate and hydrogen peroxide for metanil yellow degradation

The effectiveness degradation comparison was done by mixing metanil yellow with each of the oxidizers, namely ferrate, potassium permanganate, potassium dichromate and hydrogen peroxide. The works were done at optimum pH of metanil yellow degradation by ferrate, namely pH 8 and at the optimum molar ratio of 2:1 since they were the optimum condition for ferrate as an oxidizer. Although the conditions were not good for other oxidizer. Each solution containing metanil yellow and oxidizer was stirred until homogen for 30 min and then the absorbance was measured using a UV-Vis spectrophotometer at the maximum wavelength of metanil yellow. Figure 8 shows effectiveness comparison the oxidizers in degrading metanil yellow, it was found that ferrate was the strongest oxidizer then potassium permanganate, hydrogen peroxide, and potassium dichromate with the metanil yellow degradation of 92.4; 75.7; 65.6; 65.1%, respectively.

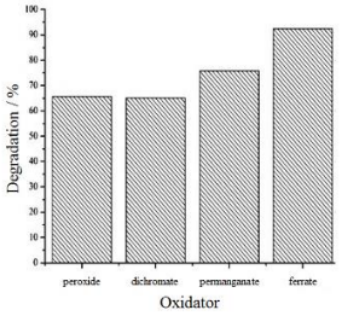

Figure 8. The effectiveness comparison of ferrate with other oxidizers at optimum pH, molar ratio and time conditions for metanil yellow degradation.

Ferrate is a strong oxidizer at alkaline pH compared to acidic conditions [19]. Ferrate has a redox potential value in alkaline conditions of 0.72V [29]. Among the transition metals, the reduction potentials for Cr(VI)/Cr(III) and Mn(VII)/Mn(IV) are significantly lower than for ferrate (VI) [28].

The pH value has a significant effect on the efficiency of dye degradation by potassium permanganate [30]. Potassium permanganate in alkaline conditions has a redox potential of 0.54 V and in acidic condition of 1.5 V, which means that potassium permanganate in alkaline conditions will have less effectiveness in degrading metanil yellow than in acidic conditions.

Hydrogen peroxide exhibits properties as oxidizing and reducing agents depending on pH. At acidic pH hydrogen peroxide will act as a strong oxidizer because it has a redox potential of 1.78 V, while at alkaline pH it will act as a reducing agent with a redox potential of 0.88 V [28]. The experimental results show that the degradation of metanil yellow by hydrogen peroxide in alkaline conditions is not very effective because the dominant species is HO^{2-}, this indicates that peroxide is unstable because it is easily decomposed into H_2O and O_2.

The dichromate and chromate ions contain chromium with an oxidation number of +6, which is the highest oxidation state of chromium in the compounds. Therefore, in a chemical reaction, the dichromate and chromate ions will undergo a reduction reaction. The reduction reaction of dichromate ions depends on the acidity of the solution. If the solution containing the dichromate ion is alkalized, the $Cr_2O_7^{2-}$ ion changes to the CrO_4^{2-} ion.

$$Cr_2O_7^{2-} + 2OH^- \longrightarrow 2CrO_4^{2-} + H_2O$$

Therefore, if the reaction takes place in an acidic atmosphere, it acts as an oxidizing agent, $Cr_2O_7^{2-}$ and vice versa, if the reaction is carried out in an alkaline state, the oxidizing agent is CrO_4^{2-}. The chromate ion is the predominant species in alkaline solution, while the dichromate ion is the predominant ion in acidic solution. The chromate ion (CrO_4^{2-}) is a weak oxidizer in an alkaline solution than in an acidic solution [31].

3.10. Determination of metanil yellow uv vis spectra before & after the degradation processes

Metanil yellow has two absorption peaks, namely at wavelengths of about 250 and 436 nm as shown in Figure 9. It can be seen that the sample before degradation has a maximum absorption with the highest peak at a wavelength of 436 nm. After the metanil yellow was degraded by ferrate at pH 8 for 30 min, it was showed that there were no more maximum absorption peaks in the wavelength regions of 250 nm and 435 nm. The loss of the peaks could be possible that the sample after degradation with ferrate is no longer the metanil yellow.

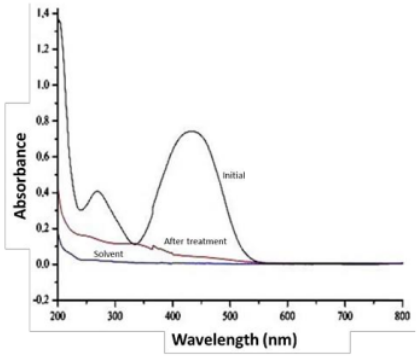

Figure 9. Metanil yellow spectra (a) before and (b) after degradation processes.

4. Conclusion

Ferrate was successfully prepared using NaOCl and $Fe(OH)_3$ from the electrolysis of used iron. The ferrate had a dark purple color and had a maximum wavelength of 510 nm. Application of the ferrate for degradation of metanil yellow was effective at pH 8. The optimum ferrate to metanil yellow molar ratio and time for degradation of metanil yellow by ferrate were 2:1 and 25 min, respectively. The effectiveness of the oxidizing agents to degrade metanil yellow were ferrate, potassium permanganate, hydrogen peroxide and potassium dichromate, with the percent degradation of 92.4; 75.7; 65.6; 65.1%, respectively.

Acknowledgment

The authors are thankful to the DIKTI (PDUPT scheme) No. 5/E1/KP.PTNBH/2020, and contract number 225-91/UN7.6.1/PP/2020.

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