Degradation of eriochrome black T by potassium ferrate (VI)

칼륨 페레이트에 의한 Eriochrome Black T 분해 연구

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ABSTRACT

The degradation of EBT (Eriochrome Black T) in an aqueous solution was investigated at various values of pH, Ferrate (VI) dosage, initial concentration, aqueous solution temperature. The maximum degradation efficiency was 95.42% at pH 7 and in that experimental condition, the kapp value was 872.87 M-1s-1. The degradation efficiency was proportional to the dosage of Ferrate (VI). Also, the initial rate constant of EBT degradation increased with decreasing of the EBT initial concentration. In addition, the degradation rate of EBT was increased from 74.04% to 95.42% when the temperature in the aqueous solution was increased from 10°C to 45°C. The activation energy value was 11.9 kJ/mol for EBT degradation. Overall, the results of the degradation experiment showed that Ferrate (VI) could effectively oxidize the EBT in the aqueous phase.

수용액에서 EBT의 분해는 pH, Ferrate (VI) 투입량, 초기 농도, 수용액 온도 등 다양한 변수의 조건에서 연구되었다. 최대 분해 효율은 pH 7.0에서 95.42%가 달성되었으며, 이 실험 조건에서 얻은 k_{app} 값은 872.87 $M^{-1}s^{-1}$ 이었다. EBT 분해율은 Ferrate (VI)의 투입량이 증가함에 따라 증가하였으며 EBT 초기 농도가 감소함에 따라 EBT 분해의 초기 속도 상수가 증가하였다. 또한 EBT의 분해율은 온도가 10°C에서 45°C에 도달할 때까지 수용액의 온도에 따라 증가하였으며 이 실험조건에서 활성화 에너지 값은 EBT 분해에 대해 11.9 kJ/mol의 값이 도출되었다. 따라서 분해 실험의 결과는 Ferrate (VI)가 수용액상에서 EBT를 효과적으로 분해시킬 수 있음을 보여주고 있다.

Key words: Ferrate (VI), EBT, Degradation, Removal rate

주제어: 페레이트(VI), EBT, 분해, 제거율

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1. Introduction

Environmental contamination of synthetic dyes in water is an important environmental concern. Dye-containing waste effluents are generated by several sources such as textile, paper, printing, pulp mills, food, cosmetics, and leather industries (Zolinger, 1987; Carneiro et al., 2007). It is estimated that over 10,000 different dyes and pigments are used industrially and over 1.5×10^8 thousand tons of synthetic dyes are produced annually worldwide (Robinson et al., 2001). In the textile industry, up to 10 – 15% of these dyes are lost to effluents every year during dyeing and finishing operations as a result of inefficiency in the dyeing process (Zollinger et al., 1987; Ji et al., 2008). Dyes are colored substances of synthetic origin, which are resistant to fading upon exposure to light, chemicals and water (Moore et al., 2004). In addition, dyes have high organic content, non-biodegradable and complex aromatic structure (Suteu et al., 2009; Zaharia et al., 2009). The presence of dyes in water bodies causes environmental damage to aquatic organisms by blocking sunlight, retarding photosynthetic activity, and disturbing the re-oxygenation capacity, which creates an anaerobic condition that limits aquatic plant growth (Mittal, 2010; Dave et al., 2011). In addition, contaminated surface water and groundwater would make the water bodies unsuitable for other use (Paixão et al., 2019) and can cause carcinogenic and mutagenic effects (Chung et al., 1993). Among the synthetic dyes, about 70% of the worldwide market used by dyeing industries is composed of azo dyes. A reactive azo dye contains one or more azo bonds (-N = N-) that act as a chromophore in the molecular structure (Ahmad and Hameed, 2010). On top of that, it is the largest group of organic dyes that are considered to be difficult to degrade even at low concentrations due to its high resistance to light, heat, water, chemical, and microbial attack (Gercel et al., 2008; Lunaet et al., 2013). Hence, it is very important to remove azo dyes from wastewater effluents before their discharge into water bodies.

Currently, several treatment technologies are used to remove dyes from waste effluents such as electrochemical treatment, coagulation, precipitation, solvent extraction, and membrane filtration. However, these methods have several disadvantages such as high energy consumption, incomplete ion removal, and production of toxic sludge and other waste products that require further treatment and disposal (Eccels, 1999). That is the main reason why this article chooses a powerful oxidant like K_2FeO_4 to remove the EBT in an aqueous solution. A few decades ago, Iron (Fe) was found at an oxidation state of +6, called Ferrate (VI), which has the potential to oxidize in an environmentally friendly manner in water and wastewater treatment (Ivanenko, 2020; Li et al., 2020). It has a relatively high potential for the treatment of water and wastewater because Ferrate (VI) has a unique ability as an oxidant at the beginning of the reaction, and then becomes coagulant because Ferrate (VI) turns into Fe(III), which will increase the purification efficiency (Zhang et al., 2020). This paper presents a review of Ferrate (VI) properties and its use in the environmental and industrial removal of EBT dye. Ferrate (VI) is best known for its stability and ease to make (Thompson et al., 1951). It is a black-purple compound that remains stable in moisture excluded air for a long period. In aqueous solution, the Ferrate (VI) ion (FeO_4^{2-}) is monomeric with a high degree of four 'Covalent character" equivalent oxygen atoms (Goff and Murmann, 1971; Hoppe et al., 1982). The properties of high stability and low solubility of Potassium Ferrate (VI) in a saturated KOH solution helped prepare this compound (Moeset, 1897). Because of its relatively high stability, Potassium Ferrate (VI) (K_2FeO_4) was used to prepare other alkaline earth Ferrates such as $SrFeO_4$ and $BaFeO_4$ (Losana, 1925; Gump et al.,1954; Scholder, 1962).
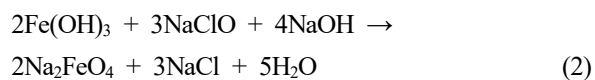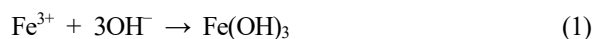
2. Materials and Methods

2.1 Materials

Iron(III) Nitrate $Fe(NO_3)_3 \cdot 9H_2O$ was purchased from KATAYAMA CHEMICAL (Japan), sodium hypochlorite solution (NaOCl) and Potassium hydroxide (KOH) as primary materials were all obtained from JUNSEI (Chuo-ku, Tokyo). Eriochrome Black T (EBT) also originated from JUNSEI (Chuo-ku, Tokyo). Buffer solutions for pH adjustment (pH 4, 7, and 10) were all purchased from Samchun Chemicals Company (Seoul, Korea).

2.2 Synthesis of Ferrate (VI)

Ferrate (VI) can be synthesized in various techniques (Thompson et al., 1951). This study used the wet oxidation technique. The preparation of Potassium Ferrate (VI) used in this study was described in (Li et al., 2005). Administering 30 g of KOH to 100 mL of refrigerated NaOCl. When the KOH was completely dissolved by stirring, cooled the solution and then filtered it with GF/C filter paper to remove NaCl in the water. Repeated this process 3-4 times and then kept the filtrate refrigerated to maintain the temperature low. After that, 11.1 g of $Fe(NO_3)_3 \cdot 9H_2O$ was administered, followed by stirring for about 40 minutes to synthesize FeO_4^{2-}. To make it into a solid-state, 16.7 g of KOH was additionally administered and continued stirred for about 40 minutes, and then refrigerated for about 30 minutes. After storage, the solution was filtered with a G4 glass filter, and the filtrate was immediately administered to a saturated KOH solution (11M) and refrigerated again. After filtration with GF/C filter paper, Ferrate (VI) crystals can be obtained. The associated chemical reactions are described below. (Talaiekhozani et al., 2016):

$$Fe^{3+} + 3OH^- \rightarrow Fe(OH)_3 \tag{1}$$

$$2Fe(OH)_3 + 3NaClO + 4NaOH \rightarrow$$
$$2Na_2FeO_4 + 3NaCl + 5H_2O \tag{2}$$

$$Na_2FeO_4 + 2KOH \rightarrow K_2FeO_4 + 2NaOH \tag{3}$$

To obtain higher purity, Ferrate (VI) crystals were dissolved in 3 M KOH, mixed with saturated KOH solution, stored in the refrigerator, and filtered with GF/C filter paper. Repeated two to three times. Finally, the material remaining on the filter paper was washed sequentially with 25 mL n-hexane, 10 mL methanol, and 10 mL diethyl ether 4 times, then dried in a vacuum, and stored. The prepared Ferrate (VI) was measured with a DR 5000 UV-spectrometer (HACH) using the ABTS measurement method (Lee et al., 2005), and the purity was about 93%.

2.3 Experimental Procedures

As mentioned in the abstract, all the batch experiments for EBT degradation using Ferrate (VI) (K_2FeO_4) were conducted as followed: EBT and Ferrate (VI) solutions were prepared in distilled water. For Ferrate (VI), the solution should be spined around 1500 to 2000 rpm to completely distill the Ferrate (VI). EBT degradation by Ferrate (VI) was investigated for the effect of pH (4, 7 and 10), different Ferrate (VI) dosages (5, 8, 10 mg/L), and temperature (10℃ to 55℃). This paper has also investigated the effect of Ferrate (VI) when treated with different initial concentrations of EBT. The initial pH was adjusted using acid or base and using buffer solution to keep it constant. The temperature was controlled using a temperature control bath. The degradation experiment was started by injecting a specific Ferrate (VI) dose into the reactor with stirring. At a specific time (0.5, 1, 2, 5, 10 min), a sample was taken using a water sampler, and then the sample was quenched using the 0.05 mM of $Na_2S_2O_3.5H_2O$ to stop further reaction (1ml of $Na_2S_2O_3.5H_2O$ for 5 mL of the sample). After that, the EBT absorption in the sample was measured by DR 5000 UV-spectrometer (HACH) (λ=530 nm).

2.4 Analytical methods

In this study, the concentration of EBT was determined using the EBT calibration curve. Once the measurement of EBT absorption (abs) in the sample was completed, the value of abs was applied to the calibration curve of EBT to calculate the left-over concentration of EBT (C). The EBT concentration measurement results will be used to calculate the % degradation efficiency using the following equation (4), where C_o is the initial concentration and C is the final concentration:

$$Degradation\ Efficiency\ (\%) = \frac{Co - C}{Co} \times 100\% \tag{4}$$

3. Results and discussion

3.1 Synthesis of Potassium Ferrate (VI)

Making Potassium Ferrate (VI) using the wet oxidation method proved to be successful with the final purity of Ferrate (VI) being 93% and due to the usage of n-benzene, methanol, diethyl ether, and KOH, Ferrate (VI)'s purity

(>90%) can stay for 20 days (in 35-40% relative humidity (RH) environment), while the decay rate was considerably enhanced at higher humidity such as 90–95% RH (Wei et al., 2015). However, the self-decay condition of Potassium Ferrate (VI) in this study needs further investigation.

3.2 Effect of initial pH on degradation of EBT

As the pH conditions are very influential on Ferrate (VI) degradation efficiency, finding out the amount of removal rate of EBT by using Ferrate (VI) at different pH was necessary. The experiments conducted to measure the remaining concentration (mg/L) of EBT (100 mL of 100 mg/L EBT) after reacting with Ferrate (VI) ([Ferrate (VI)] = 10 mg/L) as a function of time under different pH condition (4, 7, 10). Acid or base was used to adjust pH values and buffer solution was used to keep it constant. Finally, the sample was taken at these time marks: 30 seconds, 1, 2, 4, 6, 8, 10 minutes.

Fig. 1 showed the neutral pH condition of EBT solution had the highest degradation rate by Ferrate (VI) (94.56%), followed by pH 4 (85.436%) and the least efficient condition was pH 10 (79.75%). This result was predictable and the reasons for that are at a low pH (<5), Ferrate (VI) is very unstable and decreases rapidly (within minutes). The decomposition rate of Ferrate (VI) with water is far greater
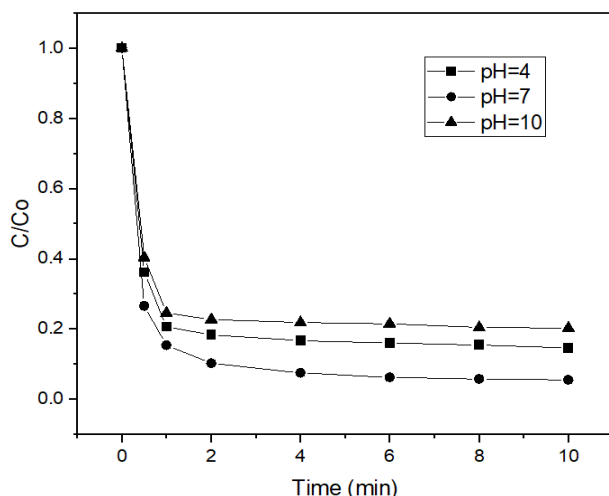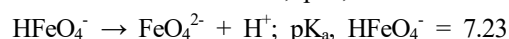

Fig. 1. Effect of different initial pH on the removal of Co [EBT]=100 mg/L by Ferrate (VI).
(Experiment condition: Ferrate (VI) dosage=10 mg/L; Temperature = 25°C; reactor volume =120 mL)

than the reaction rate with pollutants (Huang, 2021), whereas, at a high pH (>7), Ferrate (VI) is more chemically stable and lasts longer in the experimental reactor. Consequently, the experimental results demonstrated the highest EBT degradation was achieved in neutral conditions (pH 7). The result also confirmed that the initial pH had a great impact on Ferrate (VI) oxidizing power because the decolorization rate at pH 7 was greater than at pH 10. It is also worth mentioning that the initial pH of EBT kept constant by buffer in all cases had slightly changed during the reaction and therefore the final solution's pH was slightly different than the initial pH (about ± 0.1).

In addition, these experiments demonstrated the strong oxidant power of Ferrate (VI) and can degrade azo dye Eriochrome black T (EBT) efficiently, because almost total decolorization was obtained. The pH 7 was the optimal condition for the degradation in this paper because of the combined effect between Ferrate (VI) speciation and halogenated phenol speciation. Ferrate (VI) is a stronger oxidant upon protonation and speciation. Thus, the reaction rate to remove contaminants is expected to increase (Sharma et al., 2008). The Ferrate (VI) speciation compounds were H_2FeO_4 (Rush et al., 1996) and $HFeO^{4}$ (Sharma et al., 2008). These two compounds have great oxidation power and exist in pH acid and neutral.

$$H_2FeO_4 \rightarrow HFeO_4^- + H^+; \ pK_a, \ H_2FeO_4 = 3.5$$
$$HFeO_4^- \rightarrow FeO_4^{2-} + H^+; \ pK_a, \ HFeO_4^- = 7.23$$

However, acidic conditions led to the self-decomposition of Ferrate (VI) into Fe (III) and oxygen. While under basic conditions, Ferrate (VI) is very stable due to the oxygen ligand of Ferrate (VI) exchange which is very slow with water (Sharma et al., 2002; Sharma, 2010). Therefore, a decrease in the removal efficiency has been observed in the acidic and basic conditions. As a result, the highest degradation efficiency has been achieved in the neutral condition.

Kinetic studies in the degradation of EBT by Ferrate (VI) at various pH were also carried out. The kinetics for the Ferrate (VI) reaction and other, different compounds have been investigated and are very well determined by the law of second-order reaction rates (Yang et al., 2011; Sharma, 2013; Liu, 2019; Wang, 2019; Acosta et al., 2020).

Second-order reaction rate law following (Equation (5)) is rearranged and $d\dfrac{[EBT]}{dt}$ is the integrated to become (Equation (6)):

$$-d\frac{[EBT]}{dt}=k_{app}[Fe(VI)]_{(tot)}[EBT]_{(tot)} \tag{5}$$

$$ln\frac{[EBT]t}{[EBT]0}=k_{app}\int_0^t [Fe(VI)]dt \tag{6}$$

Where $[EBT]_t$ and $[EBT]_0$ is concentration of EBT after treatment at t time and initial concentration of EBT respectively, values of k_{app} is the apparent of second-order rate constant. Fe(VI) is Ferrate (VI). is Ferrate exposure. The value of k_{app} ($R^2 = 0.9\sim$) for the reaction of Ferrate (VI) with Eriochrome Black T degradation in this study is shown in Fig. 2. The values of rate constant (k) for the reaction of Ferrate (VI) with EBT under many different conditions could be determined and calculated graphically. The k values were obtained from the slope of the plots of Ferrate exposure and C/C₀. And from k_{app} value, we can know the reaction speed of every experiment in the study.

The value of the rate constant (k) increased from 480.24 $M^{-1}s^{-1}$ to 563.16 $M^{-1}s^{-1}$ as the pH 10 and 4 respectively. K_{app} value reached its peak at pH 7 (811.65 $M^{-1}s^{-1}$) indicating that the highest k for EBT removal was achieved at neutral pH condition.

3.3 Effect of initial dosage of Potassium Ferrate (VI) on Degradation of EBT

The degradation of EBT by Ferrate (VI) was examined at different dosages of Ferrate (VI) to study the effect of oxidant dosages. As shown in Fig. 3, the removal rate of EBT increased significantly with increasing dosages of Ferrate (VI). At a higher ratio, nearly complete removal of EBT was quickly achieved in the 30s. This was not unexpected and has similar findings to other pollutants (Jiang, 2007) because an increased amount of Ferrate (VI) was available to oxidize EBT when the initial oxidant concentration was high in the reaction solution. Moreover, we can see that the EBT degradation mainly occurred in the first 120 seconds, and the concentration changed slightly in subsequent reaction stages. The simultaneous auto-degradation of Ferrate (VI) could explain this phenomenon, which was also found in previous research.

As the Fig. 3 indicated, unsurprisingly, the highest removal rate was C₀ [Ferrate (VI)] = 10 mg/L (94.56%), followed by C₀ [Ferrate (VI)] = 8 mg/L (85.83%) and the lowest removal rate was C₀ [Ferrate (VI)] = 5 mg/L (70.09%). These results strongly suggested that the higher Ferrate (VI) concentration, the more EBT can be removed and it dues to Ferrate (VI) by-products can lead to the formation of Fe(III), which has abilities as a natural coagulant against pollutants (Zheng
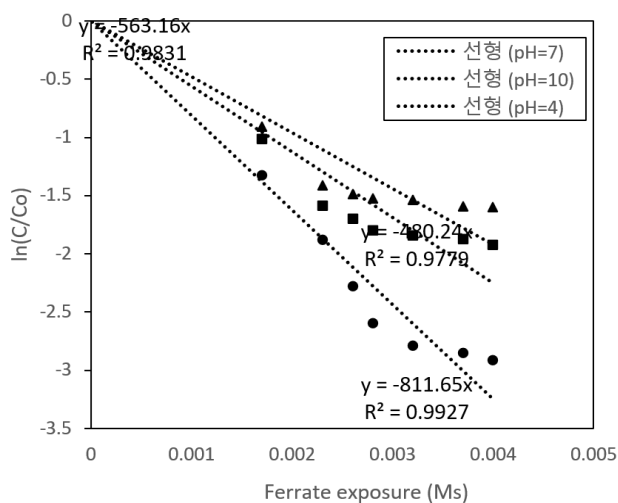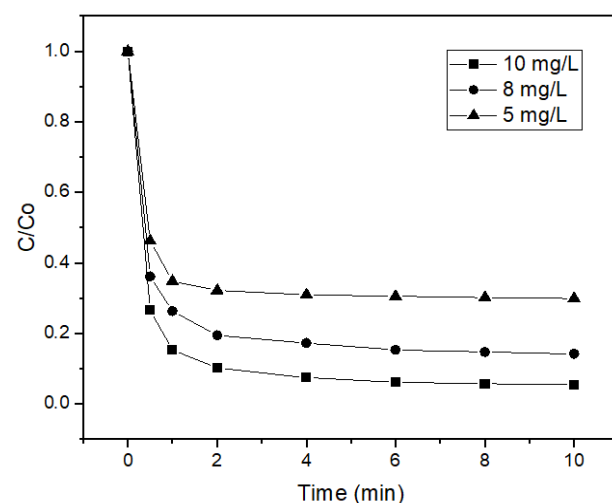

Fig. 2. Removal rate of EBT by Ferrate at various pH levels. (Experiment condition: Ferrate (VI) dosage=10 mg/L; Temperature=25°C; reactor volume =120 mL).

Fig. 3. Removal ratio of different C₀[Ferrate] in C₀[EBT]= 100 mg/L with pH 7 and experiment temperature = 25°C.
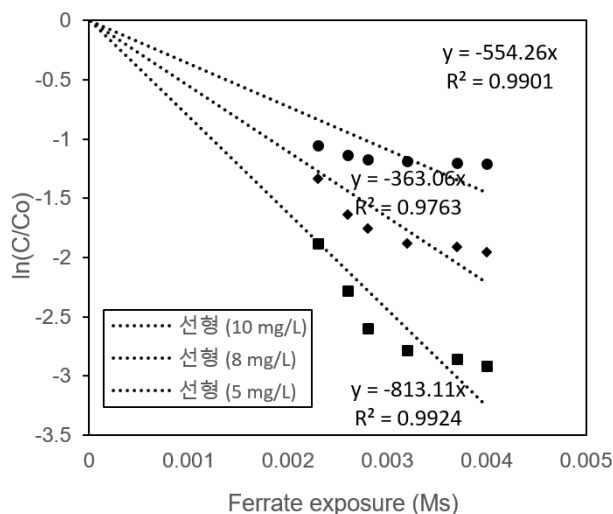
Fig. 4. Ferrate (VI) exposure and Ferrate (VI) doses plot in the determination of EBT kapp value.
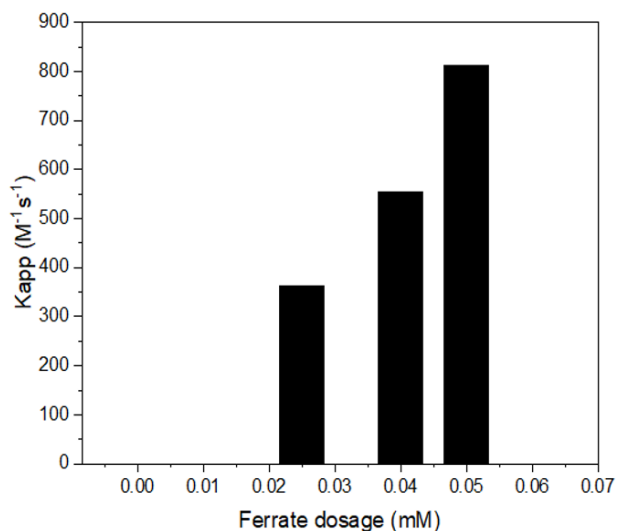

Fig. 5. Plot of Ferrate (VI) doses and kapp as bar graph.

et al., 2020; Kovalakova., 2021). However, it is worth mentioning that an excess amount of Ferrate (VI) may lead to a decrease in removal efficiency. This is due to the fact that an excess of Ferrate (VI) induced the self-decomposition of Ferrate (VI).

Similar to pH parameters, kinetic studies in the degradation of EBT by different dosages of Ferrate (VI) were conducted. Second-order reaction rate law following (Equation (5)) and the result in Fig. 4 showed the value of the rate constant (k) increased according to Ferrate dosage which was 363.06 $M^{-1}s^{-1}$, 554.26 $M^{-1}s^{-1}$ and 813.13 $M^{-1}s^{-1}$ (5 mg/L, 8 mg/L,

and 10 mg/L respectively). The plot of Ferrate (VI) dosage and k_{app} value was illustrated in Fig. 5.

3.4 Effect of initial concentration of EBT on the degradation

As the oxidation process is significantly affected by the initial concentration of the targeted compound, the degradation rate of different EBT initial concentrations (C_o [EBT]) by using Ferrate (VI) was carried out. The experiments conducted to measure the remaining concentration (mg/L) of EBT after reacting with Ferrate (VI) ([Ferrate (VI)] =10 mg/L) as a function of time under different initial concentration (50, 100, 150, 200 mg/L). As predicted, decolorization of Ferrate (VI) was getting lower when the initial concentration of [EBT] is higher. And thus, the result shown the highest removal rate by Ferrate (VI) was confirmed to be at C_o [EBT] = 50 mg/L when it reached 99.5% of removal rate when using [Ferrate] = 10 mg/L at pH 7 and temperature at 25°C, following is C_o [EBT] = 100 mg/L and C_o [EBT] = 150 mg/L with 95% and 77.8% respectively. The result also indicates that an increase in initial dye concentration not only could reduce the decolorization rate but also reduce the K_{app} value significantly. The results of the experiments were shown in Fig. 6 and the k_{app} value is shown in Table 1.
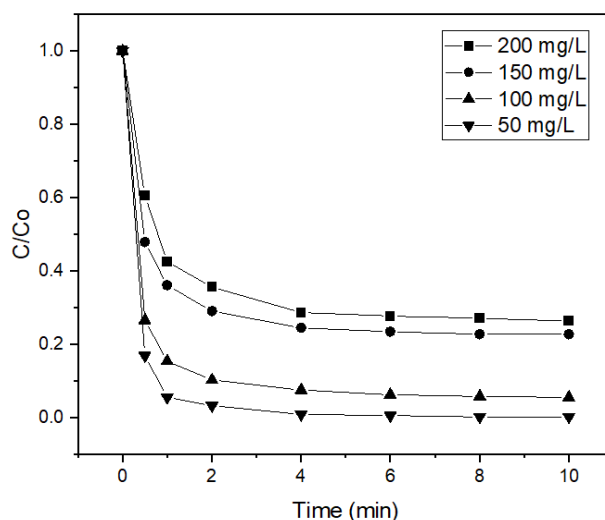

Fig. 6. Removal ratio of different C_o [EBT] after 10 minutes of removal by Ferrate (VI) 10 mg/L with pH 7, Experiment temperature = 25°C.

Table 1. k_{app} value of EBT using Ferrate (VI) in various EBT initial concentration

EBT initial concentration (mg/L)	K_{app} $(M^{-1}s^{-1})$
50	1586.1
100	811.65
150	428.12
200	371.82

3.5 Effect of temperature of EBT on Degradation of EBT

The reaction temperature is also an important factor to influence the aqueous stability of Ferrate (VI) (Wagnet et al., 1952). Therefore, a set of experiments was carried out, using a hotplate stirrer and water jackets to achieve five different temperatures (10, 18, 35, 45, 55°C) to explore its effect on EBT oxidation. As shown in Fig. 7, the degradation of EBT decreased with increasing temperature. To be specific, the removal rate of EBT was 89.9%, 94.56%, 94.88%, and 95.42% at 18°C, 25°C, 35°C, and 45°C, respectively. The lowest removal percentage was at 10°C and 55°C (74.04% and 76.1%). The removal of EBT by Ferrate (VI) in this experiment was established as a second-order reaction. Fig. 9 shows the k_{app} value in the temperature range of 10°C to 55°C with the highest k_{app} value observed at a temperature of 45°C.
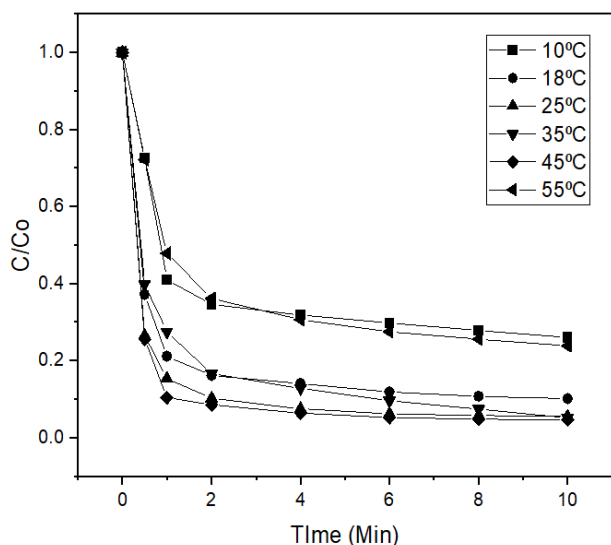
The experimental results make sense as reactants can only react when they are in contact, and molecules move faster and collide more frequently at higher temperatures (45°C). This was different from the reaction of organic chemicals with conventional oxidants (e.g., persulfate, potassium permanganate), where heating played a positive role in the efficient degradation of target compounds. In previous work, (Wagner et al., 1952) showed that Ferrate (VI) solution was much more stable at 0.5°C than at 25°C. Due to the accelerated self-decay of Ferrate (VI), high temperature was detrimental
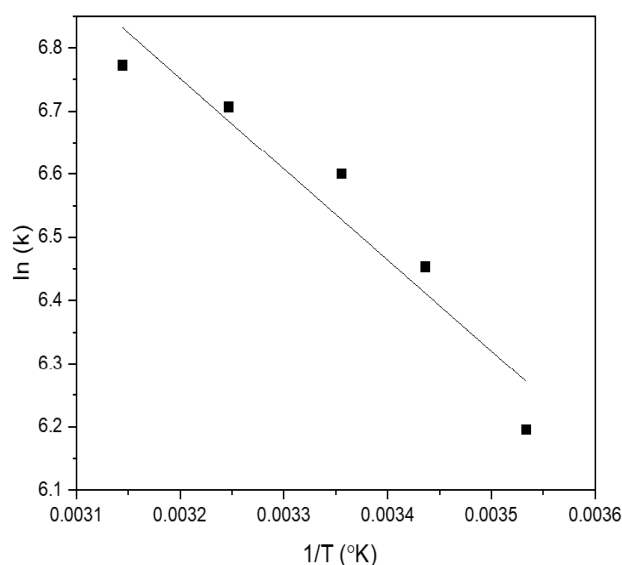

Fig. 8. Arrhenius plot for the decomposition of EBT in different temperature with pH 7, C_o[EBT]=100 mg/L.
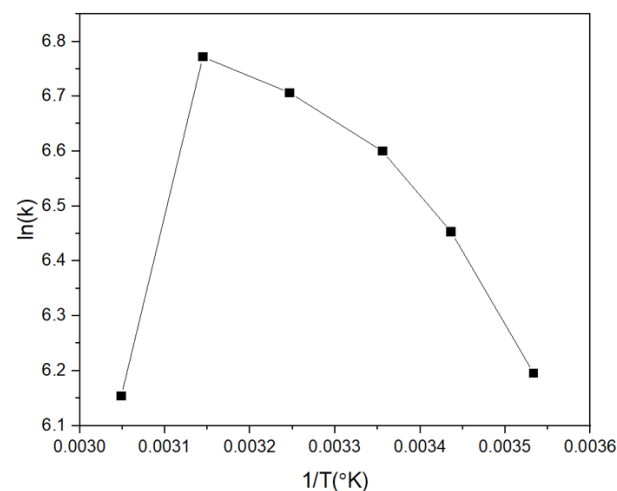

Fig. 7. The ratio of different temperatures after 10 minutes of removal by Ferrate (VI) 10 mg/L, pH 7, Experiment temperature = 25°C.

Fig. 9. k_{app} values in temperature variation with pH 7, C_o[EBT]=100 mg/L.

to the removal of EBT. At temperatures higher than 45°C (for example, 55°C), Ferrate (VI) tends to be unstable. Other researchers have also confirmed these phenomena that the Ferrate (VI) concentration decreases with an increase in temperature above 50°C (Thomas et al., 2021). The changing of k_{app} value in different temperatures can be seen in Fig. 9.

In general, the temperature is very influential in the aqueous decomposition rate of Ferrate (VI) (Machala., 2009). The Arrhenius equation (Equation (7)) is used to determine the activation energy, and the activation energy value was found to be 11.9 kJ/mol for EBT degradation.

$$\text{Ln}\left(\frac{rate1}{rate2}\right)=-\frac{Ea}{R}\times\left(\frac{1}{T1}-\frac{1}{T2}\right) \qquad (7)$$

where E_a, R and T are activation energy, universal gas constant (8.314 J K^{-1}mol^{-1}), and reaction temperature (K). Fig. 8 represents the Arhenius plot for the decomposition of EBT in different temperatures.

3.6 Application of Potassium Ferrate (VI) in industrial wastewater

As mentioned before, wastewater produced in textile industries emerges from color-containing units with a wide range of contaminants is a common problem in nowadays society. As such, the application of Potassium Ferrate (VI) in industrial wastewater has been experimented with many times by other researchers. For example, Potassium Ferrate (VI) was proved to be an effective compound for the treatment of wastewater from carpet factories (Moradnia et al., 2016), as a disinfectant and coagulant in water and wastewater treatment (Jiang et al., 2005), Potassium Ferrate (VI) can remove 50% more color (Vis$_{400}$-abs) and 30% more COD, and inactivate 3-log$_{10}$ more bacteria in comparison with aluminium sulfate (AS) and ferric sulfate (FS) at the same or even smaller doses (Jiang et al., 2005). Potassium Ferrate (VI) has also been confirmed to be an environmentally friendly oxidant for wastewater treatment (Sharma, 2002). This study is also great evidence to confirm the application of Potassium Ferrate (VI) in textile wastewater.

4. Conclusions

The performance of the Ferrate (VI) process to degrade EBT has been successfully investigated. According to this research, the reactivity of Ferrate (VI) performance depends on the pH, and a neutral pH was the optimal condition for EBT degradation. As for the effect of the Ferrate (VI) dosages, the increase in the dosages was found to be linearly related to degradation efficiency, while under some conditions (e.g., a 10 mg/L Ferrate (VI) dosage treated C_o [EBT] = 50 mg/L) can be degraded entirely in less than 10 min. For temperature, 45°C was observed as the optimal condition with an activation energy of 11,9 KJ/mol for EBT degradation. From the investigation of the degradation of EBT by Ferrate (VI) and other results from other researchers, it has been found that Ferrate (VI) can be a promising solution in industrial wastewater treatment. However, this is the first time this experiment topic being conducted so further research and experiment need to be carried out.

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