



Synthesis and Chemical Stability Studies of Sodium Ferrate(VI) Solution

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Sodium ferrate solution was prepared by the wet oxidation process using liquid FeCl_3 with NaOCl in a strong alkaline solution. The reference and the product were characterized using UV-visible, XRD and FTIR. The maximum concentration of $\text{Na}_2\text{FeO}_4(\text{VI})$ solution was 12.4 g/L (as FeO_4^{2-}). Its stability was studied by recording the absorbances of several ferrate solutions at 505 nm on daily basis for a period of one month. The average rates at pH 12 were $3.19 \times 10^{-11} \text{ M s}^{-1}$ for K_2FeO_4 and $1.09 \times 10^{-11} \text{ M s}^{-1}$ for Na_2FeO_4 . These average rates of degradation indicate that ferrate can last longer without conserving the ferrate solutions using different buffer solutions or phosphates. The peaks obtained around 700, 769 and 879 cm^{-1} using FTIR confirmed the presence of Fe-O bond in the crystals, which is $\text{Na}_2\text{FeO}_4(\text{VI})$. XRD patterns showed an isomorphism between the diffractograms of Na_2FeO_4 and K_2FeO_4 .

Keywords: Sodium ferrate(VI), Stability, Absorbance.

INTRODUCTION

Oxidation and coagulation are known as fundamental unit processes for drinking water and wastewater treatments [1]. The coagulation can destabilize suspended particulate matter and transfers it into large aggregates and adsorbs some dissolved organic materials onto the aggregates which can then be eliminated by filtration or flocculation. Several oxidants and coagulants can be applied for water and wastewater treatment [2]. The most useful coagulants consist of ferric chloride and aluminium/ferric sulphates while the oxidants/disinfectants are $\text{Cl}_2(\text{g})$, $\text{NaOCl}(\text{l})$, $\text{ClO}_2(\text{g})$, $\text{O}_2(\text{g})$ and $\text{O}_3(\text{g})$ [1,3]. Pozo-Antonio *et al.* [4] showed that when these oxidants such as oxygen are used in the wastewater treatment, they require a huge amount of energy during stirring process. Treatment also involves chlorinated or brominated products can mostly generate hazardous by-products such as bromates and trihalomethanes [5,6]. However, more effective water treatment chemicals such as oxidants and disinfectants need to be developed to meet higher treated water qualities using low energy. Such chemicals should preferably be able to disinfect water from pathogenic microorganisms, partially or totally eliminate and oxidize the inorganic and organic impurities and remove heavy metals (manganese,

zinc, *etc.*) and suspended particulate materials in a single dosing and mixing unit [2,3]. A potential chemical reagent, which meets these criteria is a ferrate(VI) salt [7,8]. The redox potential of ferrate (FeO_4^{2-}) ions (+ 2.20 V) under acidic conditions is greater than that of ozone (+ 2.08 V) and permanganate (+ 1.52 V) and is the strongest of all the oxidants/disinfectants practically being experimented on for wastewater and drinking water treatment. Furthermore, during the oxidation process, ferrate(VI) ions are normally reduced to Fe(III) ions (eco-friendly coagulant resource) [1,7-9].

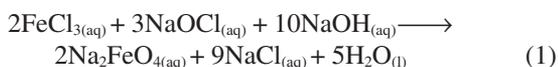
However, there are three universal methods used to produce metal ferrate(VI) salts. These include wet oxidation, electrochemical and dry oxidation methods [7,8,10]. Electrochemical and dry oxidation methods are the most applied for possible ferrate generation for drinking water treatment. In the current study, liquid sodium ferrate(VI) was synthesized using wet oxidation method as other methods showed some drawbacks in the previous studies due to safety concerns and complex reaction steps involved (time consuming), since the processes could cause detonation (dry oxidation method) while electrochemical method requires high concentrated electrolytes (*e.g.* 14 M NaOH even higher) [11,12]. Hence, this study was aimed at preparing a stable liquid sodium ferrate(VI), which could

be applied as an oxidant and coagulant for acid mine drainage treatment in our future study, characterizing and quantifying it using XRD, UV-visible and FTIR, monitoring its stability and to give some recommendations for future work.

EXPERIMENTAL

The following liquid chemicals (Analytical reagent grades); ferric chloride (43 % m/m), sodium hypochlorite (15 % m/v as Cl_2) and NaOH (47 % m/m) were used without further purification. These chemicals were purchased from NCP Chlorchem Pvt. Ltd., South Africa. Ferrate salt standard (containing FeO_4^{2-} ions) was ordered from American Elements Company, U.S.A.

Preparation of liquid Na_2FeO_4 : Liquid Na_2FeO_4 was synthesized using wet oxidation method developed by Ockerman & Schreyer [13] and Thompson *et al.* [14] with some modifications. Hence, these modifications were performed to see if sodium ferrate could be synthesized using only liquid reagents, to enhance the yield and stability of the product. Some optimum conditions developed by Sun *et al.* [10] for preparing liquid sodium ferrate(VI) were revised and taken into consideration during *in situ* synthesis. The mixing proportions of the reagents were 10:5:1 (v:v:v) for NaOCl(aq), NaOH(aq) and FeCl_3 (aq), respectively. An amount of 60 mL of NaOCl(aq) and 30 mL of NaOH(aq) were mixed together into a 100 mL beaker placed in the ice bath and 6 mL of liquid FeCl_3 (aq) was slowly added into the mixture with mild stirring. The ice bath was meant to quench the excess heat generated from an exothermic reaction, although the product is not stable at high temperatures. The mixture was gently heated at 25 °C for 10 min to speed up the reaction of the production of sodium ferrate(VI). Low temperature was applied to avoid degradation of both hypochlorite ions and ferrate ions which were being produced. The basic chemical reaction for the preparation of sodium ferrate [15,16] is shown as follow:



The product obtained was exhibiting reddish-purple colour, which is very similar to the one obtained in the literature [10]. The solution was centrifuged to remove impurities and the supernatant liquid was collected for further analysis.

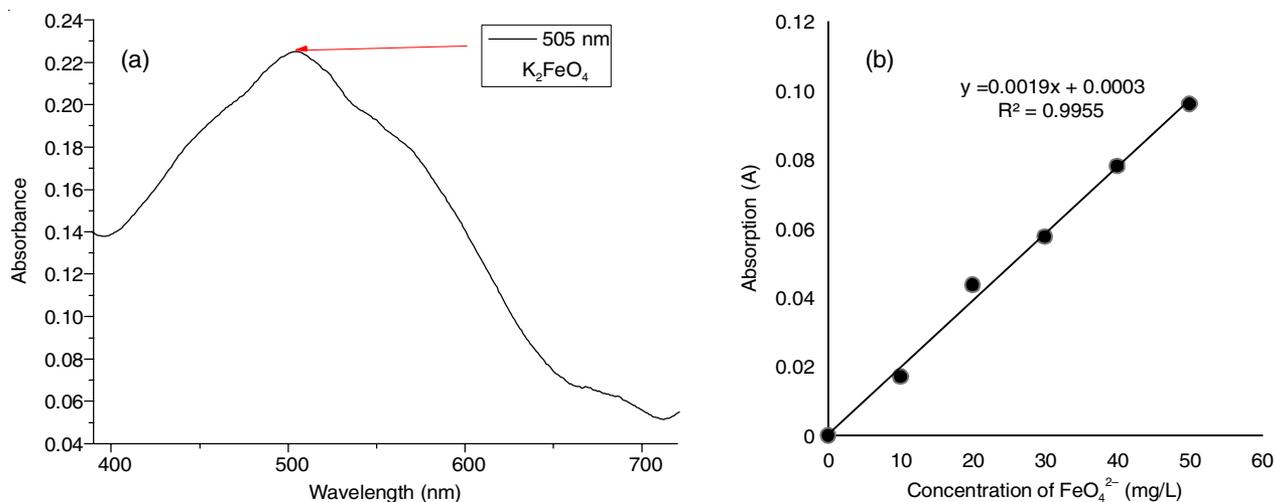


Fig. 1. (a) Absorption peak of K_2FeO_4 (FeO_4^{2-}) on UV-visible and (b) Calibration curve

RESULTS AND DISCUSSION

Liquid sodium ferrate(VI) solution produced was characterized and quantified using UV-visible spectroscopy. By using the freeze-drying process, liquid sodium ferrate was frozen at -70 °C and dried overnight, a solid Na_2FeO_4 was then obtained and characterized using X-ray diffraction and Fourier-transform infrared spectroscopy.

Characterization and quantification of sodium ferrate (VI) using UV-visible: A stock solution of 100 mg/L (as FeO_4^{2-}) was prepared by dissolving 16.52 mg of K_2FeO_4 as a standard in 100 mL of ultrapure water. Working solutions were prepared (from 0 to 50 mg/L), and their absorbances were measured at $\lambda_{\text{max}} = 505$ nm [17] on UV-vis spectroscopy Cary 60 (Agilent Technologies) as shown in Fig. 1a-b. The correlation coefficient was $R^2 = 0.9955$.

Sodium ferrate(VI) with chemical formula of Na_2FeO_4 was successfully produced and quantified with the concentration of 12.4 g/L or 0.10 M (as FeO_4^{2-}) (reading on UV-vis: 0.0124 g/L multiplied by a dilution factor: 1000). The concentration was determined to evaluate the effectiveness of the method and found to be higher than some concentrations reported in the literature using other methods [15]. This confirmed that sodium hypochlorite has totally oxidized all iron sources (liquid ferric chloride). Cui *et al.* [18] used microwave method (dry oxidation method) to prepare sodium ferrate with solid Fe_3O_4 (s) as iron source in the reaction and the calculated concentration was 1.41 g/L. Batarseh *et al.* [15] also synthesized sodium ferrate using wet oxidation method and the resulting concentration was 2 g/L as Fe. Currently, Laksono, and Kim [19] synthesized sodium ferrate using wet oxidation method with purpose of removing 2-bromophenol from environmental matrices. Ferric chloride and sodium hydroxide were in solid states and the resulting concentration of ferrate was 42 g/L as Fe. However, quantifying sodium ferrate in this study was the main purpose of next project, which is treating acid mine drainage using sodium ferrate(VI) with a known concentration and to check the effectiveness of the method.

Characterization of sodium ferrate(VI) using FTIR: To characterize sodium ferrate(VI) produced using FTIR technique, a portion of liquid sodium ferrate(VI) synthesized was

frozen under -70°C and dried using a vacuum dryer overnight. The FTIR spectrum indicating IR absorption peaks (functional groups) of NaFeO_4 is presented in Fig. 2. For similarity reason, the literature was checked and the results showed that the formation or chemical shifts of peaks can depend on many factors or parameters such as drying temperatures, reaction time, composition, concentration and states of the reactants. This means that if ferric/ferrous sulphate or nitrate compounds are used as iron source, these sulphate and nitrate ions coexist with the ferrate and cause the interference, which impacts on chemical shifts of functional groups. However, the peaks of sodium ferrate obtained between 900 and 600 cm^{-1} in present study were similar to the peaks as reported by other researchers [20-22].

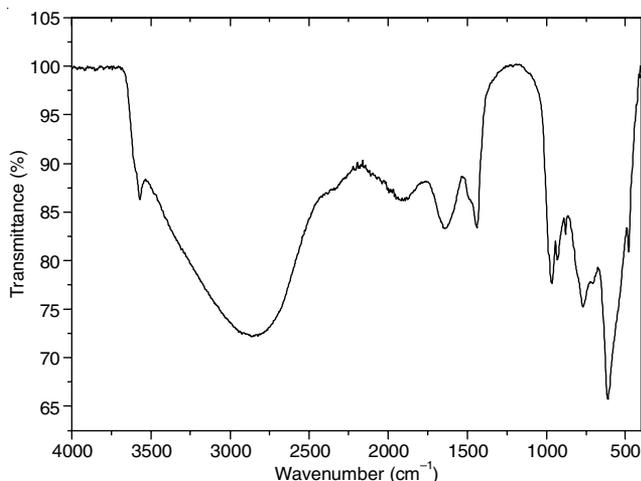


Fig. 2. FTIR spectrum of sodium ferrate(VI)

The stretching vibration characteristic peaks of Fe-O bond in ferrate obtained at around $879, 769, 700, 769\text{ cm}^{-1}$ confirmed the presence of Fe-O bond in crystals, which is sodium ferrate(VI) salt. The small difference in chemical shifts of functional groups of the product of this study comparing with the literature could be caused by the conditions of production and crystallization. Moreover, the peaks observed between 1920 and 900 cm^{-1} should be assigned to the characteristic peaks of the C-O bond stretching vibration, which could be caused by CO_2 in air [23]. The peaks obtained in $4000-2400\text{ cm}^{-1}$ region are ascribed to H-O bond from water [21,24]. Additionally, the visibility and the sharpness of peaks also confirmed the high purity of the product.

XRD analysis: Prior to XRD analysis, both the reference and the prepared samples were grounded and sieved to make a fine powder. The powder was then packed in the sample holders and loaded in the sample rack for analysis. The XRD patterns or diffractograms of potassium ferrate and sodium ferrate are shown in Figs. 3 and 4. The XRD patterns were analyzed by scanning from 2θ ranging between 4.00 and 80.00° . Sharp peaks of crystalline K_2FeO_4 at 2θ values are $30.40^{\circ}, 31.29^{\circ}, 32.54^{\circ}, 39.36^{\circ}, 40.51^{\circ}$ and 57.30° .

Sharp peaks of crystalline of Na_2FeO_4 at 2θ values are $27.27^{\circ}, 29.23^{\circ}, 31.56^{\circ}, 45.35^{\circ}$ and 56.35° and were more visible compared to reference (K_2FeO_4). Figs. 3 and 4 show strong similarity and prove the crystal structure of both ferrates and demonstrate the similarities with Na_2FeO_4 [20] and BaFeO_4

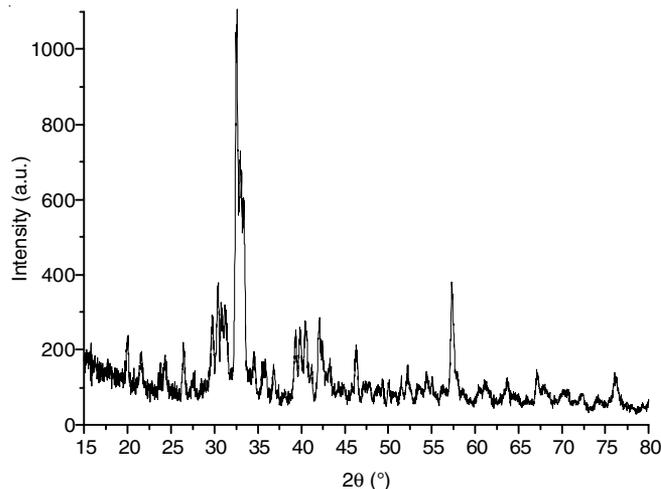


Fig. 3. XRD patterns of potassium ferrate

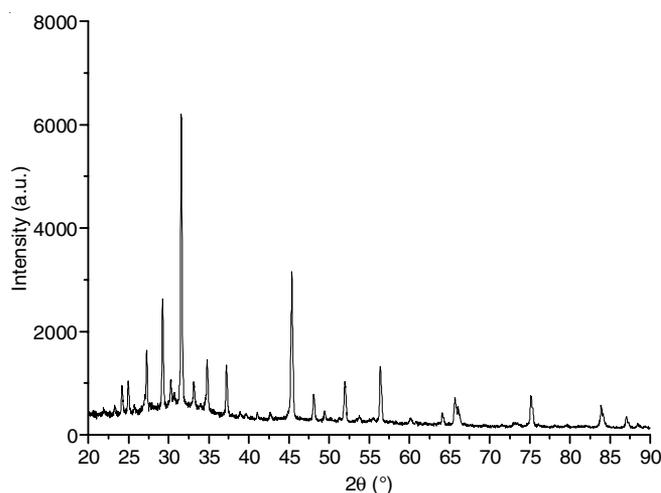


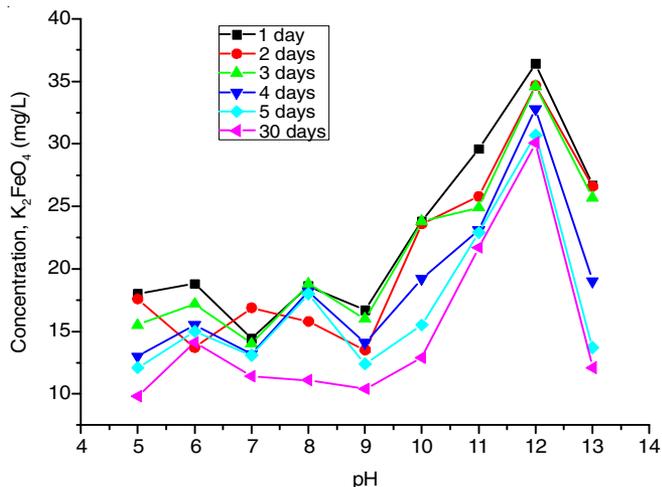
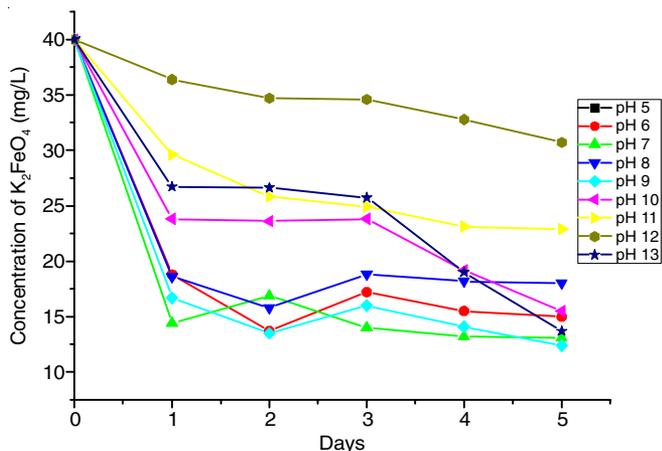
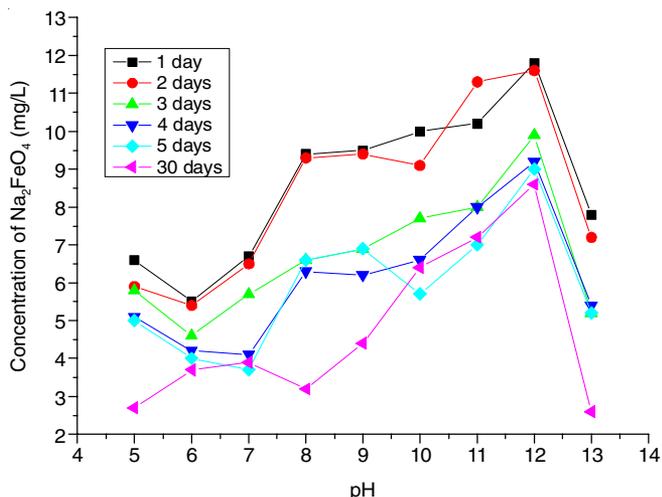
Fig. 4. XRD patterns of sodium ferrate

[25]. Both reference and product ferrates analyzed in this study had an orthorhombic shape.

Stability: To monitor the stability of both ferrate salts over time, lower concentrations of reference K_2FeO_4 (40 mg/L : $3.34 \times 10^{-4}\text{ M}$) and the product Na_2FeO_4 (12.40 mg/L : $1.03 \times 10^{-4}\text{ M}$), respectively were prepared to avoid photodegradation of concentrated solutions and their pH values were adjusted from 5 to 13. No pH buffers were used to control the pH values.

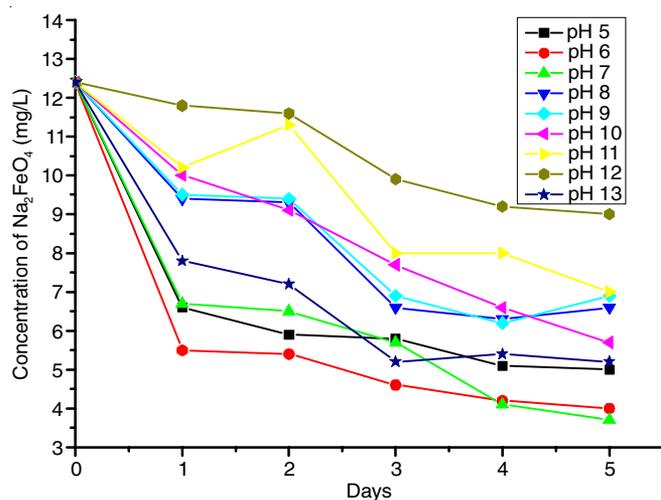
During this study, the rate of degradation of synthesized sodium ferrate(VI) was monitored in the period of one month without any addition of preservatives or buffer solutions such as disodium tetraborate or borax and disodium hydrogen phosphate anhydrous or phosphates [9,26]. The concentrations of both potassium and liquid sodium ferrate(VI) salts from each daily run on UV-visible were plotted against pH as shown in Figs. 5-8.

Initially, Fe(VI) solutions had a characteristic dark-purple colour, which changed instantaneously to yellowish colour as the self-decay of Fe(VI) into $\text{Fe}(\text{OH})_3$ occurred at some lower pH values. Figs. 5 and 6 show that potassium ferrate is highly unstable especially at lower pH values (5, 7 and 9). Ferrate ions are normally stable at $\text{pH} > 10$, but an exception was found at $\text{pH} 13$. It is normally supposed that ferrate ions show a different reduction pathway, causing anionic ion species formation such

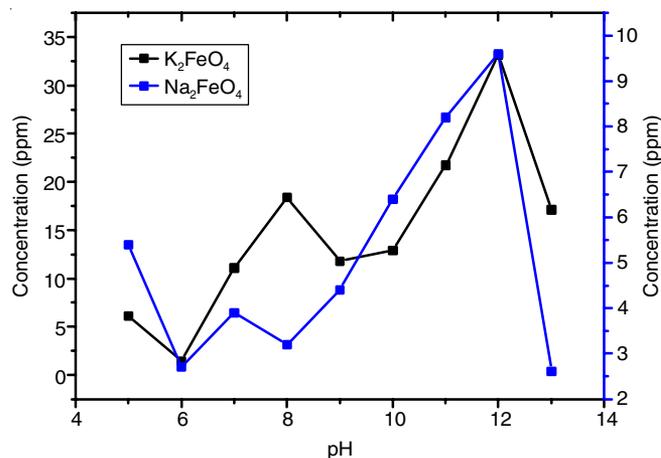
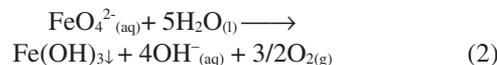
Fig. 5. Concentrations of K_2FeO_4 (mg/L) versus pH (standard) in 30 daysFig. 6. Change of concentrations of K_2FeO_4 with time at various pH valuesFig. 7. Concentrations of Na_2FeO_4 vs. pH in 30 days

as $Fe(OH)_4^-$ and $Fe(OH)_6^{3-}$ instead of $Fe(OH)_3(s)$ at pH greater than 12. In this study, ferrate solutions showed to be stable at pH 12 since no preservatives or buffers have been used during the storage, which could participate in the down-shift of pH values.

Degradation rate: Ferrate(VI) ions in aqueous solution, self-decomposes into ferric hydroxide, hydroxide ions and molecule of oxygen as shown in eqn. 2.

Fig. 8. Change of concentrations of Na_2FeO_4 with time at various pH values

Like reference potassium ferrate, synthesized sodium ferrate(IV) showed to be stable at pH 12. Both potassium ferrate and sodium ferrate(IV) stability were low at pH 7. This can be explained by the meta-stable state of FeO_4^{2-} (pH \approx 7), which signifies its rapid decay and shift to a stable state. Figs. 7 and 8 also showed that sodium ferrate was unstable at pH < 10. After 5 days, the samples were kept in a dark place at ambient temperature ($25 \pm 1^\circ C$). The samples were analyzed again to check the residual concentrations after one month (30 days) and the results are presented in Fig. 9.

Fig. 9. Concentrations vs. pH of K_2FeO_4 (standard) and Na_2FeO_4 (product) after one month

Previous studies showed that this self-decomposition depends on many factors including initial concentration of ferrate, temperature, alkalinity, pH of the solution and coexisting ions. To study the kinetics of ferrate decomposition in water, a set of both aqueous K_2FeO_4 and Na_2FeO_4 solutions with an initial concentration of $3.34 \times 10^{-4} M$ and $1.03 \times 10^{-4} M$ were prepared with different pH values from 5 ranging to 13. The temporal change in Fe(VI) concentration was measured by UV-visible every day in a period of one month. As shown in above figures, the slow self-decomposition rate was observed at pH 12 for

both ferrate salts. The speciation of Fe(VI) against pH signifies that HFeO_4^- predominates in mildly acidic conditions and FeO_4^{2-} is the dominant form in alkaline conditions. In other words, FeO_4^{2-} showed instability at acidic pH values. The literature indicated that the self-decay of Fe(VI) follows second-order kinetics at $\text{pH} < 10$ and first-order kinetics at pH greater than 10. According to this trend, it was assumed that the kinetics of both Na_2FeO_4 and K_2FeO_4 decompositions in aqueous solution follow the first-order reaction model ($\text{pH} > 10$) and the average rate can be determined using the following equation:

$$\text{Average rate} = -\frac{\Delta[\text{FeO}_4^{2-}]}{\Delta t} = -\frac{[\text{FeO}_4^{2-}]_{\text{final}} - [\text{FeO}_4^{2-}]_{\text{initial}}}{t_{\text{final}} - t_{\text{initial}}}$$

By applying this formula using the data provided in Table-1, the average rates of both the reference and the product at $\text{pH} = 12$ in one month are calculated and the average rate of K_2FeO_4 was $3.19 \times 10^{-11} \text{ M s}^{-1}$ while the average rate of Na_2FeO_4 was $1.09 \times 10^{-11} \text{ M s}^{-1}$.

TABLE-1
CONCENTRATION OF K_2FeO_4 (REFERENCE) AND
CONCENTRATION OF Na_2FeO_4 (PRODUCT)

Time (day)	Concentration (mol/L)		pH
	K_2FeO_4	Na_2FeO_4	
0	3.34×10^{-4}	1.03×10^{-4}	12
1	3.04×10^{-4}	9.85×10^{-5}	12
2	2.90×10^{-4}	9.68×10^{-5}	12
3	2.89×10^{-4}	8.26×10^{-5}	12
4	2.74×10^{-4}	7.68×10^{-5}	12
5	2.56×10^{-4}	7.51×10^{-5}	12
30	2.51×10^{-4}	7.18×10^{-5}	12

It is observed from the results that both the synthesized liquid sodium ferrate(VI) and the reference (K_2FeO_4) may be stable at ambient temperature upto one month of storage without any addition of preservatives at $\text{pH} > 9$. The rate at which ferrate (VI) salts self-decompose depends on pH of solution. Small variation of initial concentrations in both the synthesized sodium ferrate(VI) and the reference was found at $\text{pH} = 12$, which confirms high stability of ferrate (VI) ions in strong alkaline media. Lower concentrations were found at $\text{pH} < 9$ because ferrate (VI) ions can participate in water oxidation by releasing oxygen [27,28]. This indicates that ferrate(VI) salts can only be stored at high pH and last longer without adding preservatives to avoid coexistence of undesired ions in the solution.

Conclusion

Liquid sodium ferrate(VI) was quantitatively produced through wet oxidation method and characterized using UV-visible, XRD and FTIR. Serial dilutions were made, and the calculated concentration of liquid sodium ferrate(VI) was 12.4 g/L or 0.10 M as FeO_4^{2-} . The stability of reference (K_2FeO_4) as well as of the product (Na_2FeO_4) was monitored through a period of one month at various pH values ranging from 5 to 13. Results showed that ferrate(VI) salts can be synthesized and used in water treatment without using preservatives such as buffer solutions and phosphates to store the product as the later can bring coexistence ions in the solution. Significant stability of the reference and product was found at high pH ranging from 9 to 12. For the reference, at $\text{pH} = 12$, the starting concen-

tration was $3.34 \times 10^{-4} \text{ M}$ and the residual concentration was $2.51 \times 10^{-4} \text{ M}$. For the product (Na_2FeO_4) at same pH, the starting concentration was $1.03 \times 10^{-4} \text{ M}$ and the residual concentration was $7.18 \times 10^{-5} \text{ M}$ after one month. The average degradation rates observed in 30 days were 3.19×10^{-11} and $1.09 \times 10^{-11} \text{ M s}^{-1}$ for K_2FeO_4 (standard) and Na_2FeO_4 (product), respectively. The stretching vibration characteristic peaks obtained around 879, 700, 769 cm^{-1} using FTIR confirmed the presence of Fe-O in the crystals, which is Na_2FeO_4 salt. The XRD patterns of K_2FeO_4 with sharp peak at $2\theta = 32.54^\circ$ and Na_2FeO_4 with sharp peak at $2\theta = 31.56^\circ$ showed an interesting isomorphism of their diffractograms.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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