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ORIGINAL RESEARCH ARTICLE

KINETICS AND THERMODYNAMICS OF BLEACHING PROCESS IN AQUEOUS SOLUTION USING SODIUM HYPOCHLORITE

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ABSTRACT

The kinetics of bleaching process of oxidation of typical dye with a bleaching agent - sodium hypochlorite in aqueous medium was studied. The kinetic runs were executed using spectrophotometer to changes in concentration under pseudo first order whereby concentration of NaOCl was in large excess compared with the concentration of dye. The factors affecting the reaction rate that were studied include the concentration of dye and oxidant, temperature, ionic strength and pH of the bleaching reaction medium and the presence of a variable valence metal ion The result shows that the rate of oxidation increases with increasing in temperature, ionic strength and pH. Increasing in substrate and oxidant concentrations also increase the rate of oxidation. Higher observed rate constant k1 was obtained in the presence of Fe(III)ion. The Arrhenius activation energy for the oxidation in the absence and presence of Fe(III)ion are 56.21 kJmol^{-1} and 51.21 kJmol^{-1} respectively. The result of thermodynamic parameters such as the lowering of activation energy (Ea) and the higher value of second rate constants k_2 in the presence of Fe (III) ion provide further support for Fe (III) ion enhancement of rate of oxidation.

1.0 Introduction

Bleaching is a chemical process of decolourising a material. Bleaching thus is a chemical transformation of a coloured component to a colourless in the material or within the medium. The bleach is the generic name for such chemical product for the transformation commonly and erroneously known as cleaning and colour or stains removal. Bleaching process could either be an oxidation or reduction when the bleach is oxidant or reductant respectively. Most bleaches are of course oxidants (Abdul and Narenda, 2013; Aqeel *et al.*, 2020). Bleaching action is utilized both domestically and industrially in various applications. It is used in textile industry as in bleaching of cotton and linen. Similarly, in order to impact whiteness to textile fabrics, bleaching of textile grey fabrics is generally carried out by removing natural colouring matter. Interestingly, hair color turns lighter mostly through (bleaching) rather than through coloration. Bleaching is also utilized in leather as in tanning industry as well as in paper production (Hassan *et al.*, 2017).

Although dyes beautify the world, effluents from dye sources and application industries distort and disturb the environment and its living contents. The presence of dye in effluent is easily perceptible even in a low concentration. Aside visual aspect, the colouration of the water cans impedes the photosynthesis as the coloured materials strongly absorb sunlight. This situation affects the balance of the aquatic ecosystem (Abo Farha,2010).

Effort to decolourised the coloured effluents need to be intensified because of the increase in production of dyes and pigments. It is estimated that about 10,000 different types of dyes and pigments are produced worldwide annually. In addition, over 7 x 10^5 tons of these different dyes and pigments of dyes and pigments are produced worldwide annually (Vinod *et al.*, 2011). Out of these dyes 10% to15%

is estimated to be in effluent during the dying process. In addition, almost every industry uses dyestuff (Mohammed and Ali, 2020). Bleaching is reported to involve the use of the four main bleaching agents sodium hypochlorite, calcium hypochlorite, sodium chlorite and hydrogen peroxide. In this research work the oxidant used is a chlorine – based bleach, sodium hypochlorite (NaClO) a common household “bleach” product (Mafzal *et al*., 2019).

Bleaching is important in controlling the adverse effect of the colouration. The study of chemical kinetics of the bleaching reaction is very important. In the later stage of twentieth century the time required for bleaching is said to drop from month to days to hours. Today bleaching works faster. Chemical kinetics although studies rate (time) in a reaction Chemical kinetics necessarily studies effect and control of various factors in chemical reactions (Aicha *et al.,* 2017). This work studied the kinetics and thermodynamics of bleaching process in aqueous solution using sodium hypochlorite. The knowledge could therefore be of benefit not only to bleaching workers but also dye handlers in general.

2. Materials and Methods

2.1 Materials

The chemical reagents used in the research work include sodium hypochlorite (BDH), sodium chloride salt (MERCK), hydrochloric acid, iron (III) chloride, tartrazine (the dye), sodium hydroxide and bicarbonate (BDH). They are analytical grade, although further purified as necessary. The instruments were thermostat water bath, weighing balance, (Helmreasin) pH meter, Jenway spectrophotometer 7305, (Jenway Cole Palmer, Shanghai Co Ltd. China). Deionised water was used to prepare all the solutions.

2.2 Methods

For a particular substance the stock solution of a particular molar concentration was prepared by: -

(i) measure the equivalent mass in gram (i.e. for a solid) or volume in ml (i.e. for a liquid)

(ii) dissolve this in deionized water in 250 ml standard flask, making up to mark with the deionized water, mix thoroughly. Bottle and keep

(iii) Experimental (working solution) was prepared from the respective stock solution by dilution of the stock solution, applying dilution equation ($C_1V_1 = C_2V_1$). [where C_1 is the initial concentration, C_2 is the final concentration, V_1 is the initial volume, V_2 is the final volume.]

Thus solutions of oxidant - sodium hypochlorite and substrate – tartrazine were prepared. Other solutions prepared and added to the reaction mixture were sodium chloride to monitor the effect of the ionic strength of the solution; hydrochloric acid and sodium hydroxide to study the effect of pH of the reaction medium.

Kinetics profiles were studied under pseudo first order conditions as all the reactions were conducted with the concentrations of the sodium hypochlorite (the oxidant) in large excess compared to that of tartrazine (the dye and substrate) (Bahl *et al.,* 2014). As kinetics measures changes in concentrations with time, the concentration was monitored with spectrophotometer. Therefore, the wavelength correspond to the maximum absorbance was determined by measuring the absorbance of 0.001moldm^{-3} solution of dye at different wavelength and making a plot of absorbance against wavelength of a proposed and predetermined ranges of 410 – 510nm.This produced the absorption spectrum from which the wavelength corresponding to the maximum absorbance (λ max) was noted. The proof of Beer-Lambert’s law was carried between 2×10^{-2} to 10×10^{-2} mol dm^{-3} of solution of dye at (λ max) earlier ascertained and a plot of absorbance against concentrations was made. The molar absorptivity was determined from the slope of the graph obtained. For the kinetic follow up the reaction rates was measured by setting up different flasks containing appropriate quantity of solution to constitute the reaction mixture arranged in a themostarted water bath to attain a constant temperature. The reaction was initiated by rapid introduction of sodium hypochlorite solution into the mixture. The progress of the reaction was followed by taking the following changes in absorbance of decolourising dye in the mixture as a function of time.

2.2.1 Kinetic measurement

Kinetic analysis of the bleaching process was based on the general chemical equation of a reaction between the dye and the oxidant represented by Equation 1. The rate of bleaching reaction was expressed by Equation 2. Since the concentration is directly related to the absorbance, the change in absorbance of the oxidant was used to monitor rates of reaction. The large excess of oxidant as displayed in equation 3 has made the changes in concentration of the oxidant in the reaction insignificant. This thus make the rate of reaction as first order with respect to the concentration of dye. Both equation 2 and 3 therefore relate specific rate equation k_1 and k_2 as shown in equation 5. Integrated form of equation 4 gives equation 6. The pseudo - first order rate constant were obtained from the kinetic expression (equation 6).A plot of ln (A_t) against time (t) was used to determine the value of rate constant k_1, obtained from the slope of equation 6 The rate constants were averages of at least three measurements. The second order rate (k_2) was thus deducted from equation 5

$$\text{Oxidant} + \text{dye} \longrightarrow \text{product(s)} \qquad (1)$$

$$Rate = k_2 \ [dye]\,[Oxidant] \qquad (2)$$

$$\text{But} \ \ [Oxidant] \ >> \ [dye\,] \qquad (3)$$

$$Rate = k_1[dy\,e] \qquad (4)$$

where k_2 was the rate constant for the bimolecular reaction of dye and the oxidant and k_1 is the observed pseudo- first order rate constant

$$k_1 = k_2\,[Oxidant] \qquad (5)$$

$$\ln\,(A_t) = ln(A_O) - k_1\,t \qquad (6)$$

where A_o and A_t stands for absorbance at the beginning t = 0 and at time t.

t: time in s, k_1: first order rate constant s^{-1}, k_2: second order rate constant $mol^{-1}dm^3\ s^{-1}$

A absorbance for concentration in $moldm^{-3}$

Factor affecting the rate of bleaching process were verified by carry out the kinetics measurement while varying the quantity of the particular factor and maintaining constant the value and condition of other factors. The contribution of presence of variable valence metal ions in the reaction medium on rate of beaching rate was also determined.

3. Results and Discussion

Spectrum and calibration of spectrophotometer

The absorption spectrum is displayed in Figure1. The wavelength corresponding to the maximum absorbance was found to be 460nm. Beer – Lambert Law was ascertained from graph of absorbance against concentration of the dye solution in the range of 2.0×10^{-2} - 10×10^{-2}M Figure 2. The slope of the of a straight line plot in Figure 2 gives the molar absorptivity as 3.157 $dm^3mol^{-1}cm^{-1}$

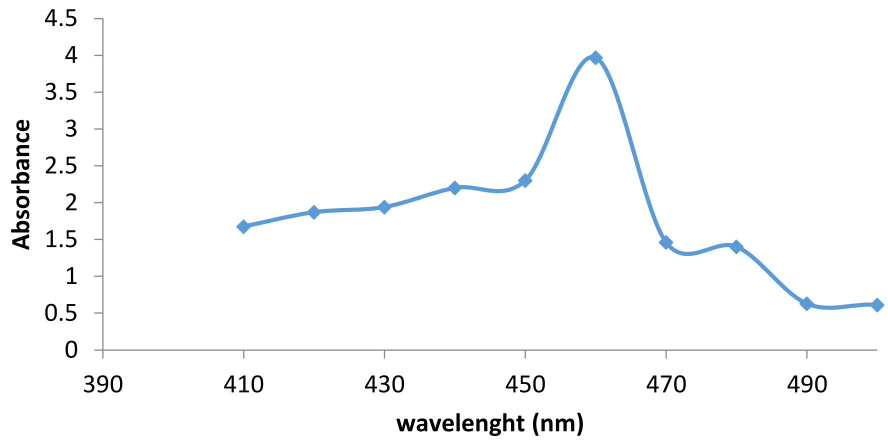

Figure 1: Calibration curve of log of Absorbance against wavelength

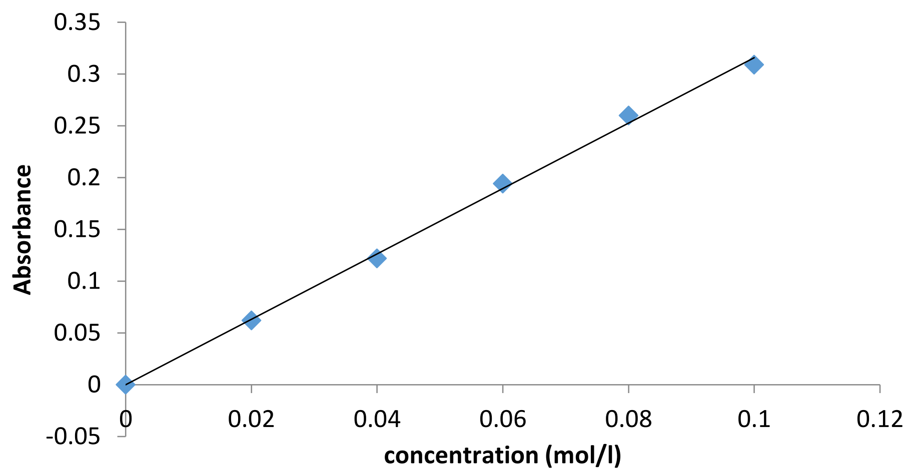

Figure 2: Plot of absorbance versus concentration for dye solution

3.1 Dependency of rate of bleaching reaction on concentration of oxidant

The effect of oxidant concentration on the bleaching rate was determined at different initial concentration of oxidant (sodium hypochlorite) while maintaining the quantity of each of other bleaching factors such as strength, temperature and the concentration of dye solution. The value of k_l for each of the oxidation reaction at each initial concentration was calculated from the slope of each plot of log of absorbance against time (Figure. 3). The values of the k_l presented in Table 1 show that the observed rate constants increased with increasing in initial concentration of sodium hypochlorite. This could be due to the increase in the oxidative power of the increasing ion hypochlorite (Olajire and Olajide 2014).

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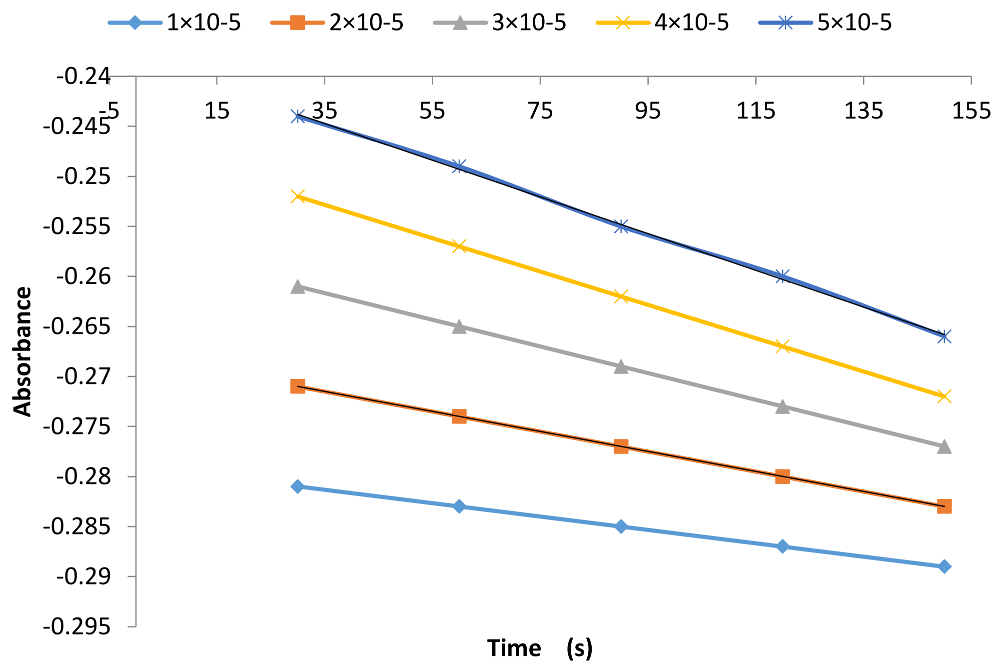

Figure3 The Plot Log Abs vs t for the effect of oxidant conc. on bleaching rate in the absence of Fe(III)ion

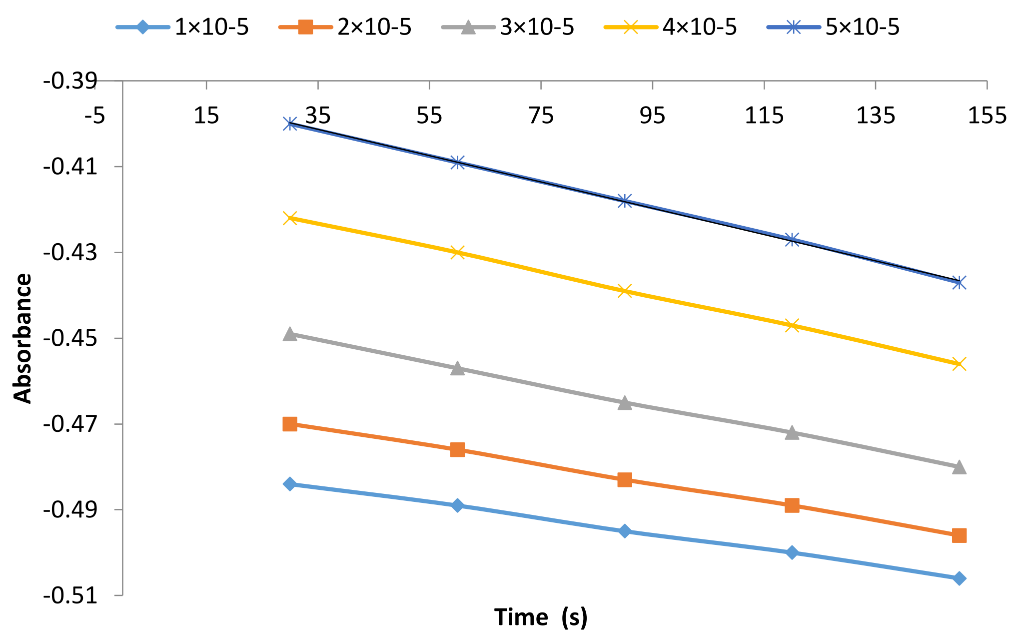

Figure 4 The Plot Log Abs against t for the effect of oxidant conc. on bleaching rate in the presence of Fe(III)ion

Table 1 k_I as oxidant concentration varied

[Oxidant]×10^{-3}M	k_I ×$10^{-4}s^{-1}$
1.0	4.61
2.0	6.14
3.0	6.90
4.0	7.00
5.0	8.20

3.2 Dependency of bleaching rate on concentration of dye

The effect of concentration dye on the rate of bleaching reaction was carried out with different initial concentration of dye while keeping the values of other influencing factors constant. The pseudo first order rate constant (k_I) at each initial concentration of dye was evaluated from the slope of plot of log of absorbance against time. The result presented in table 2 show that there was increasing in k_I with increasing in the initial concentration of solution of dye, which shows that the reaction rate depends on initial concentration of dye. This suggests that more dye molecules are available for the reaction as availability of molecule of a reactant raise the rate of such reaction (Manivannan *et al*., 2015).

Table 2 k_I as dye concentration varied

[dye]×10^{2}M	k_I ×$10^{-4}s^{-1}$
1.0	1.53
2.0	2.30
3.0	3.07
4.0	3.83
5.0	4.61

3.3 Dependency of bleaching rate on ionic strength of the medium

The effect of ionic strength of the medium on bleaching process carried out at different ionic strength of the medium shows the value of k_I for the bleaching rate as recorded in table 3 increase with increasing in the ionic strength of the medium within the range of experiment. The rate of reaction was first order with respect to sodium chloride concentration thus, affect the reaction rate (Sania *et al*., 2012; Okeola *et al.,* 2020)

Table 3 k_I at variation of concentration of salt

[NaCl] ×10^{-3}M	k_I ×$10^{-4}s^{-1}$
1.0	5.37
2.0	6.14
3.0	6.90
4.0	7.05
5.0	8.44

3.4 Dependency of bleaching rate on pH of the medium

The effect of pH on the rate of bleaching was studied in alkaline medium. Table 4 indicates a rise in the observed rate constants k_I within the pH of the medium between 8.70 and 11.20. The observation shows that controlling the pH of the bleaching medium can regulate rate of bleaching process (Olajire and Olajide 2014).

Table 4 k_I at variation of pH

pH	$k_I \times 10^{-4}s^{-1}$
8.70	1.20
9.10	1.53
9.80	2.30
10.60	3.07
11.20	4.61

3.5 Effect of presence of transition metal ion on the bleaching rate

The effect of presence of transition metal ion on the bleaching rate was studied by repeating the dependency of the reaction rate on dye and oxidant concentration in the presence of Fe (III) ion in the reaction mixture. The value of k_I was calculated from the slope of each plot of log absorbance against time (Figure 4). The rate constants k_I observed, also increased with increasing in concentration of dye and oxidant respectively, but the values of k_I were relative greater the value of k_I obtained in the absence of Fe (III) ion in the reaction mixture (Tables 5a and 5b). The second order rate constant k_2 was determined from the slope of the linear plot of k_I against respective dye concentration (equation 5) both in the absence and presence of Fe (III) ion. The value of k_2 was found to be 1.39 $M^{-1}S^{-1}$ and 1.59 $M^{-1}S^{-1}$ in the absence and presence of Fe (III) ion respectively. The higher value of k_2 in the presence of Fe (III) ion shows that the presence of the transition metal ion enhances rate of oxidation according to literature report (Gamal *et al.*, 2017). The variable oxidation state of transition metal ion facilitates the redox reaction oxidation. Increase in the rate constant could be due to activation of sodium hypochlorite (Olajire and Olajide 2014; Ogori *et al.,* 2018).

Table 5 Effect of presence of Transition metal ion (Fe (III) ion) in the reaction medium

Table 5a k_I at variation of dye concentration in the absence and the presence of Fe (III) ion

	absence of Fe (III) ion	presence of Fe (III) ion
$[dye] \times 10^{-2}M$	$k_I \times 10^{-4}s^{-1}$	$k_I \times 10^{-4}s^{-1}$
1.0	1.53	3.84
2.0	2.30	4.61
3.0	3.07	5.41
4.0	3.83	6.14
5.0	4.61	7.67

Table 5b k_I at variation of oxidant concentration in the absence and the presence of Fe (III) ion

	absence of Fe (III) ion	presence of Fe (III) ion
$[Oxidant] \times 10^{-3}M$	$k_I \times 10^{-4}s^{-1}$	$k_I \times 10^{-4}s^{-1}$
1.0	4.61	6.14
2.0	6.14	6.90
3.0	6.90	7.68
4.0	7.00	7.90
5.0	8.20	9.97

3.6 Effect of variation in temperature on the bleaching rate

The oxidation reactions were carried in the presence and absence of Fe (III) ion at different temperature from (333 - 373) K while maintaining constant all other factors in the reaction. The k_1 increased with increasing in temperature while with a relative higher value in the in the presence of Fe (III) ion (Table 6). Thermodynamic properties associated with the bleaching process were determined. These include the Activation energy (Ea) determined from the slope of the linear plot obtained from natural logarithm k_2 against the reciprocal of the temperature in Kelvin 1/T(°K) based on Arrhenius equation 5 for the reaction in the absence (Figure 5) and presence of Fe(III) ion (Figure 6)

Table 6 k_1 at variation of temperature ion

	absence of Fe (III) ion		Presence of Fe (III) ion	
Temperature (K)	$k_1 \times 10^{-4} s^{-1}$	k_1 ($mol^{-1}s^{-1}$)	$k_1 \times 10^{-4} s^{-1}$	k_2 ($mol^{-1}s^{-1}$)
333	1.20	0.024	2.14	0.032
343	1.54	0.04	2.93	0.051
353	2.31	0.06	3.42	0.073
363	3.01	0.07	3.92	0.084
373	4.03	0.09	4.51	0.096

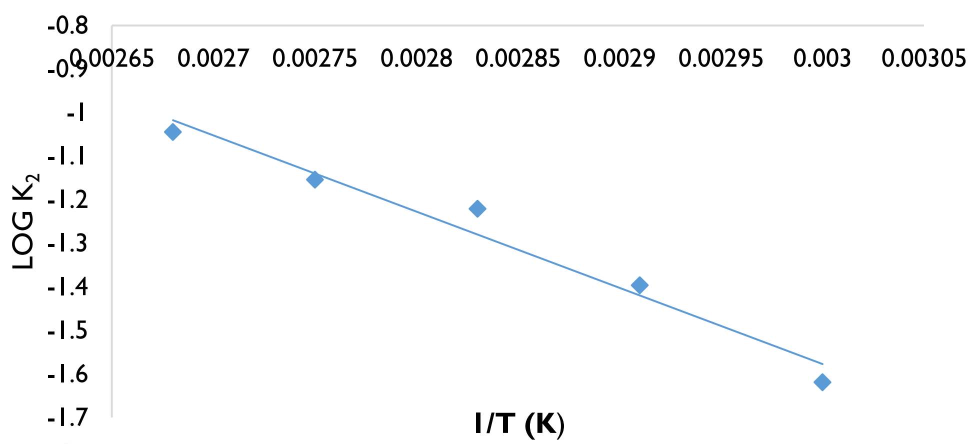

Figure 5 A plot of log of k_2 against 1/T(K) for bleaching process in the absence of Fe(III) ion

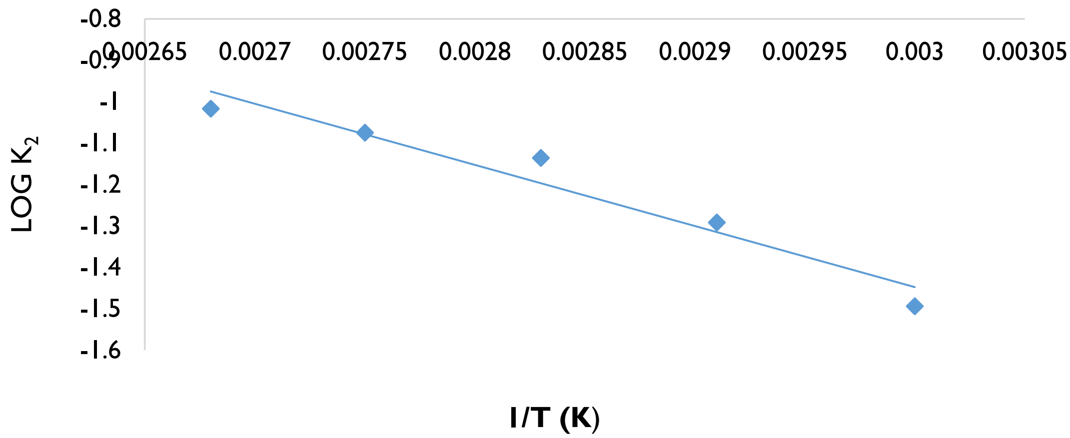

Fig. 6 A plot of k_2 against 1/T(K) for bleaching process in the presence of Fe(III) ion

The values of Enthalpy of activation of the reaction ($\Delta H^{\#}$), Gibbs free energy of activation ($\Delta G^{\#}$) and the entropy of activation ($\Delta S^{\#}$) are determined on the ground of respective equations 6,7 and 8 (Mortimer *et., al.* 2002).

$$\ln k_2 = lnAe^{-Ea/RT} \qquad (6)$$

$$\Delta H^{\neq} = Ea - RT \qquad (7)$$

$$\Delta G^{\neq} = \Delta H^{\neq} + T\Delta S \qquad (8)$$

$$logA = \frac{RT}{H} + \frac{S^{\neq}}{RT} \qquad (9)$$

$$10S^{\#} = 2.303R (Log_{10} A - Log_{10} (KT/h) \qquad (10)$$

The result of the Arrhenius activation energy Ea obtained from the slope of the linear plot is 56.21 kJ mol^{-1} (in the absence of Fe (III) ion), and 51.21 kJ mol^{-1} (i.e. presence of Fe (III) ion).

The values of the thermodynamic properties are presented in table 7 for the bleaching reaction in the absence and presence of Fe (III) ion respectively. The lower value of Arrhenius energy of activation (Ea) when Fe (III) ion in reaction mixture indicate a lower energy reaction pathway showed. This is an indication of Fe (III) ion enhancement in bleaching reaction. IFawzy *et al*, 2016) The fairly higher negative value of entropy of activation ($\Delta S^{\#}$) and free energy of activation ($\Delta G^{\#}$) and lower Enthalpy of activation of the reaction ($\Delta H^{\#}$) also indicate this positive role of Fe (III) ion in accelerating the bleaching reaction according to literature report (Prem *et al*, 2017)

Table 7 The thermodynamic activation parameters for bleaching process by NaOCl in the absence and presence of Fe(III) ion

Condition	Ea (kJ/mol	$\Delta H^{\#}$ (kJ/mol)	$\Delta S^{\#}$ (kJ/mol)	$\Delta G^{\#}$ (kJ/mol)	A(L/mol/s)
Absence of Fe(III)ion	56.21	52.41	-32.3	62.6	142.7
Presence of Fe(III)ion	51.21	48.32	-41.4	56.3	128.7

4. Conclusion

The kinetics of bleaching process with a bleaching agent - sodium hypochlorite in aqueous medium was studied using spectrophotometer. The effect of the following factors that influence the rate of chemical kinetics were studied the concentration of dye and oxidant, temperature, ionic strength and pH of the bleaching reaction medium and the presence of a variable valence metal ion. The study was carried out under pseudo first order where the concentration of one of the two reactants would be prepared in large excess to make the amount reacting insignificant so that change in concentration would be based on would be based on other reactant. The values of factor to be studied were varied while the values of other factors were made constant. In each experiment the rate constant was determined for each of the different values rate constant. The study of these observed rate constants generated the results and basis of discussion. The observation shows that the rate of bleaching can be controlled by monitoring pH, ionic strength as well as the temperature of the bleaching medium. The concentration of the bleach and the dye are can also be controlled to monitor bleaching rate. The result shows that increasing in factors such as concentration of oxidant and the dye within the range of the exercise improve the rate of oxidation reaction, the bleaching process. Increasing in temperature, ionic strength and pH of the reaction medium within the range in this study also increased the rate of the oxidation reaction. The lower value of Ea among other thermodynamic results and higher k1 in the presence of variable valence metal ion, Fe (III) ion, demonstrated the enhancement and catalytic role of the metal ion.

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