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Aqueous Phase Reduction of Mn(VII) using Sodium Metabisulphite: Kinetic, Thermodynamic and Stoichiometric Studies.

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Abstract

In an aqueous phase reduction of Mn(VII) using $Na_2S_2O_5$, the absorbance of Mn(VII) was monitored at 349 nm using a Unicom UV/Vis Spectrophotometer at different temperatures (303, 308, 313, and 323 K) to study the effect of pH on the reductant at pH 2, 4,6, 7, 8,10,12 and 14 while, the effect of concentration was also considered by varying the concentrations of the reductant (i.e. 4, 6, 8, 10 and 12 mg/L) over a pH of 5,9, 298 K and 5 minutes with concentration of Mn(VII) set at 10mg/L. The kinetic results showed that the reaction was first order with respect to Mn(VII) with the rate equation of $-\frac{d[Mn(VII)]}{dt} = k'[Mn(VII)]$. *The activation entropies obtained from plots of* $ln\left(\frac{k_{obs}}{T}\right)\ vs. \frac{1}{T}$ *were positive with slight variation with pH and temperature (1.05 -1.8 $JK^{-1}mol^{-1}$) suggesting that the reduction was driven by solution dynamics. The activation enthalpies varied from (-7.32 - 70 $kJmol^{-1}$) and were negative showing that the reactions were rather exothermic with the reduction of Mn(VII) by $Na_2S_2O_5$ was exothermic and entropic at an alkaline environment. The activation energies obtained were positive indicating that the products molecules are bonded by strong bonds. The stoichiometry suggests that the Mn(VII):$Na_2S_2O_5$ ratio of 0.66:1 was found to be in agreement with the theoretically obtained result for both acidic and alkaline regimes. The findings from the research show that $Na_2S_2O_5$ could be used for the reductive detoxification of Mn(VII) in polluted areas.*

Background: *Manganese containing products during process and after disposal release various manganese and ions in the surrounding atmosphere, soil and water which severely affect the quality of atmosphere, soil and water (Oves et al., 2016). Manganese is of economic significance in industrial use and the most important pollutants in the environment. Environmental pollution by manganese has become a serious threat to living organisms in surrounding atmosphere, soil and water (Siddiquee et al., 2015). Manganese toxicity is of great environmental concern because of its bioaccumulation and non-biodegradability in nature. Manganese is a vital element needed in small quantity for metabolic and redox functions (Gautam et al., 2014).*

Manganese exists in both inorganic and organic forms. An essential ingredient in steel, inorganic manganese is also used in the production of dry-cell batteries, glass and fireworks, in chemical manufacturing, in the leather and textile industries and as a fertilizer. The inorganic pigment known as manganese violet (manganese ammonium pyrophosphate complex) has nearly ubiquitous use in cosmetics and is also found in certain paints. Organic forms of manganese are used as fungicides, fuel-oil additives, smoke inhibitors, an anti-knock additive in gasoline, and a medical imaging agent. Due to these industries and other anthropogenic activities, huge amounts of manganese compounds are discharged into the environment, including air, soil and water (Liu, et al, 2012).

Materials and Methods: *The reduction of Mn(VII) by $Na_2S_2O_5$ was conducted in accordance with the method described by Iorungwa et al. (2014). A 10 mL portion of 2.0 mg/L Mn(VII) was measured into a 250mL beaker. Next, a 20 mL aliquot of 20.0 mg/L $Na_2S_2O_5$ was added and the mixture shaken thoroughly. The pH of the solution was adjusted adding a drop of 1.0 M H_2SO_4 in the acidic region and 1.0 M NaOH in the alkaline region before they were made to come in contact. The UV/Visible spectrophotometer (Unicom UV/Vis Spectrophotometer) was set at 349 nm and the absorbance of Mn(VII) was read at that wavelength. The* *absorbance of $Na_2S_2O_5$ was found to be weak at this wavelength. The set up was done in triplicate at temperatures of 298, 308,313, 318 and 323 K. In order to study the effect of reaction time on Mn(VII) reduction, separate set ups of the above mixture was made and each monitored for different time intervals of 0, 30, 60, 90, 120 seconds to minimize interference due to withdrawal of samples. The reaction was done for the initial pH values of 2, 4, 6, 7, 8, 10, 12 and 14. The stoichiometric study was conducted using five concentrations of the reductants: 4, 6, 8, 10 and 12 mg/L to assess the concentration – dependence of the reduction. The reaction was at 298 K and a moderately acidic pH of 5. The initial concentration of Mn(VII) was set at about 10.0 mg/L and the reaction time was 5 minutes. Equal volumes of 10mL each of the reductant and Mn(VII) was used for the mixture.*

Results: *The rate of reduction was basically increasing with decrease in pH. The rate of reduction was also slightly affected by increase in temperature with all reaction reaching 50 % of their completion at 4620 seconds when the initial temperature was increased to 308 K. The rate in basic solution was faster than in acidic and neutral solutions. The findings of the present study are consistent with those of Iorungwa et al., (2014).*

***Conclusion**: The study showed that the reduction of Mn(VII) by $Na_2S_2O_5$ in aqueous phase was fast in an alkaline pH with negative activation enthalpies, negative activation energies and positive activation entropy with slight variation in temperature which shows the reaction was driven by solution dynamics.*

Keywords: *Reduction, Sodium Metabisulphite, kinetic, Thermodynamic, Exothermic, Temperature, pH.*

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I. Introduction

Manganese is a member of the first row transition series of elements, consisting of Sc, Ti, V, Cr, Mn, Fe, Co, Ni, Cu and Zn, and belongs to group 7 of the periodic table, along with Tc and Re. The element has an atomic number of 25, an atomic mass of 54, several oxidation states (+2, +3, +4, +6 and +7) but the stable state is the +2 and one naturally occurring isotope (^{55}Mn) (Bouchard et *al*, 2011).

Manganese toxicity is of great environmental concern because of its bioaccumulation and non-biodegradability in nature. (Gautam *et al*., 2014).

Although manganese is an essential nutrient at low doses, chronic exposure to high doses may be harmful. Nowadays, various usages of manganese in industry, agriculture, medicine and technology have led to a widespread distribution in nature raising concerns about its effects on human health and environment. Manganese ions may interact with cellular components such as DNA and nuclear proteins leading to various health problems from DNA damage and structural changes. As a result, exposure to manganese through ingestion, inhalation and dermal contact causes several health problems such as, kidney failure, hallucinations and diseases of the central nervous system. Due to extensive damage caused by manganese poisoning on various organs of the body, the investigation and identification of Inorganic redundant for the reduction of manganese toxicity is very important (Mehrandish *et al*., 2019).

II. Materials And Methods

Apparatus/Instruments, Reagents and Chemicals

The instruments and materials that were used during the analysis are as follow;

***Laboratory materials*:** Beakers, measuring cylinders, spatula, volumetric flasks, stop watch, what-man No 1 filter paper, potassium permanganate ($KMnO_4$), sodium metabisulphite ($Na_2S_2O_5$), sulfuric acid (H_2SO_4), nitric acid (HNO_3), sodium hydroxide (NaOH), biphenyl carbizide, Unicom UV/Vis Spectrophotometer, Oakton pH/Con 510 series pH meter, Adams PW 184 series electronic weighing balance, Thermostatic watch bath, Manestay Water distiller.

All the reagents, obtained were of analytical grade with very high purity level.

Preparation of Solutions

All solutions were prepared using distilled water. All glass wares and plastics were washed with distilled water, rinsed with (1:1) HNO_3 and finally with distilled water. Manganese (VII) stock solution was prepared from $KMnO_4$ (BDH) by dissolving 1.58g g of the salt in 1 litre of distilled water. A 2 mgL^{-1} Mn(VII) solution was prepared measuring exactly 10 mL of the stock solution in 20 mL of distilled water. $Na_2S_2O_5$ stock solution was prepared by dissolving 1.0g of the salt in a litre of distilled water. Interferences were avoided by adding biphenyl carbazide to Mn(VII) before adjusting the pH of the solution.

Reduction of Manganese(VII) by Sodium Metabisulphide

The reduction of Mn(VII) by $Na_2S_2O_5$ was conducted in accordance with the method described by Iorungwa *et al*. (2014). A 10 mL portion of 2.0 mg/L Mn(VII) was measured into a 250mL beaker. Next, a 20 mL aliquot of 20.0 mg/L $Na_2S_2O_5$ was added and the mixture shaken thoroughly. The pH of the solution was adjusted adding a drop of 1.0 M H_2SO_4 in the acidic region and 1.0 M NaOH in the alkaline region before they were made to come in contact. The UV/Visible spectrophotometer (Unicom UV/Vis Spectrophotometer) was set at 349 nm and the absorbance of Mn(VII) was read at that wavelength. The set up was done in triplicate at temperatures of 298, 308,313, 318 and 323 K. In order to study the effect of reaction time on Mn(VII) reduction, separate set ups of the above mixture was made and each monitored for different time intervals of 0, 30, 60, 90, 120 seconds to minimize interference due to withdrawal of samples. The reaction was done for the initial pH values of 2, 4, 6, 7, 8, 10, 12 and 14

Determination of Kinetic and Thermodynamic Parameters

The thermodynamic parameters were determined by adopting the method described by Iorungwa *et al*., (2014) with slight modifications. In this experiment, a 10mL portion of the Mn(VII) was measured into a 250mL beaker and 20ml of the reductant added. The concentration of Mn(VII) was 2.0 mgL^{-1} while the concentration of the reductant was exactly 10 times the concentration of Mn(VII) and the temperature dependence of Mn(VII) reduction by reductant was studied at the temperature; 303, 308,313, 318 and 323 K and at the initial pH values of 2, 4, 6, 7, 8, 10, 12 and 14. For each temperature and pH, the total manganese was determined at 0, 30, 60, 90, 120 seconds of reaction. A plot of the natural logarithm of concentration of Mn(VII) against time was made to obtained K_{obs}(the observed rate constant) from the slopes. The activation enthalpies and entropies were obtained from the slopes and intercepts of another plot of ln(K_{obs}/T) against 1/T. This in accordance with the equation below:

$$\ln\left(\frac{Kobs}{T}\right)=\ln\left(\frac{Kb}{h}\right)+\frac{\Delta S^0}{R}-\frac{\Delta H^0}{RT} \qquad (1)$$

Where T is the temperature in Kelvin,
k_b is Boltzmann's constant (1.38×10^{-23} J/K),
h is Planck's constant (6.63×10^{-34} Js), and R is the gas constant (8.314 J/K/mol).
The activation energies was therefore computed from the relationship:
$Ea = \Delta H - T\Delta S$ --------- (2)

Determination of Stoichiometric Parameters

The study was conducted using five concentrations of the reductant; 4, 6, 8, 10 and 12 mgL^{-1} to assess the concentration-dependence of the reduction. The reaction was done at 298 K and moderately acidic pH of 5 and 9 for the alkaline pH. The initial concentration of Mn(VII) was at 10 mgL^{-1} and the reaction in 120 seconds. Equal volume of 10mL of the reductant and Mn(VII) was used for the mixture.

III. Results

Kinetic, Thermodynamic and Stoichiometric Parameters for the Reduction Process

Table 1: The Rate Constant Observed at Various Temperature for the reduction of Mn(VII) by $Na_2S_2O_5$ at various pH

Ph	T(K)	k_{obs}	k_{obs}/T	lnk_{obs}	lnk_{obs}/T	1/T (K^{-1})
2	303	4.0×10^{-3}	1.32×10^{-5}	-5.52	-11.24	0.00330
	308	1.0×10^{-2}	3.20×10^{-5}	-4.61	-10.34	0.00325
	313	1.5×10^{-2}	4.80×10^{-5}	-4.20	-09.94	0.00320
	318	2.0×10^{-2}	6.30×10^{-5}	-3.91	-09.67	0.00315
	323	2.5×10^{-2}	7.70×10^{-5}	-3.69	-09.47	0.00310
4	303	1.0×10^{-3}	3.30×10^{-6}	-6.91	-12.62	0.00330
	308	1.5×10^{-3}	4.90×10^{-6}	-6.50	-12.23	0.00325
	313	2.0×10^{-3}	6.40×10^{-6}	-6.22	-11.96	0.00320
	318	2.5×10^{-3}	7.90×10^{-6}	-5.99	-11.75	0.00315
	323	3.0×10^{-3}	9.30×10^{-6}	-5.81	-11.59	0.00310
6	303	1.2×10^{-2}	4.00×10^{-5}	-4.42	-10.14	0.00330
	308	1.6×10^{-2}	5.10×10^{-5}	-4.14	-09.87	0.00325
	313	2.2×10^{-2}	7.00×10^{-5}	-3.82	-09.56	0.00320
	318	2.5×10^{-2}	7.90×10^{-5}	-3.69	-09.45	0.00315
	323	3.0×10^{-2}	9.30×10^{-5}	-3.51	-09.28	0.00310
7	303	3.0×10^{-3}	9.90×10^{-6}	-5.81	-11.52	0.00330
	308	5.0×10^{-3}	1.62×10^{-5}	-5.30	-11.03	0.00325
	313	1.1×10^{-2}	3.51×10^{-5}	-4.51	-10.26	0.00320
	318	1.5×10^{-2}	4.72×10^{-5}	-4.20	-09.96	0.00315
	323	1.8×10^{-2}	5.57×10^{-5}	-4.02	-09.79	0.00310
8	303	2.0×10^{-3}	6.60×10^{-6}	-6.22	-11.93	0.00330
	308	4.0×10^{-3}	1.30×10^{-5}	-5.52	-11.25	0.00325
	313	4.5×10^{-3}	1.44×10^{-5}	-5.40	-11.15	0.00320
	318	4.7×10^{-3}	1.48×10^{-5}	-5.36	-11.12	0.00315
	323	5.0×10^{-3}	1.55×10^{-5}	-5.30	-11.08	0.00310
10	303	3.0×10^{-3}	9.90×10^{-6}	-5.81	-11.52	0.00330
	308	3.5×10^{-3}	1.14×10^{-5}	-5.66	-11.39	0.00325
	313	3.9×10^{-3}	1.25×10^{-5}	-5.55	-11.29	0.00320

	318	4.0×10^{-3}	1.26×10^{-5}	-5.52	-11.28	0.00315
	323	4.3×10^{-3}	1.33×10^{-5}	-5.45	-11.23	0.00310
12	303	3.0×10^{-3}	9.90×10^{-6}	-5.81	-11.52	0.00330
	308	6.0×10^{-3}	1.95×10^{-5}	-5.12	-10.85	0.00325
	313	6.5×10^{-3}	2.08×10^{-5}	-5.04	-10.78	0.00320
	318	7.5×10^{-3}	2.35×10^{-5}	-4.89	-10.66	0.00315
	323	9.0×10^{-3}	2.79×10^{-5}	-4.71	-10.49	0.00310
14	303	1.1×10^{-2}	3.63×10^{-5}	-4.51	-10.22	0.00330
	308	1.3×10^{-2}	4.22×10^{-5}	-4.34	-10.07	0.00325
	313	1.4×10^{-2}	4.47×10^{-5}	-4.27	-10.02	0.00320
	318	1.7×10^{-2}	5.35×10^{-5}	-4.08	-09.84	0.00315
	323	1.9×10^{-2}	5.88×10^{-5}	-3.96	-09.74	0.00310

Table 2: Activation data for Mn(VII) reduction by $Na_2S_2O_5$ at 25 °C

pH	-ΔH° (KJ/mol)	ΔS° (J/K/mol)
2	70.00	1.24
4	42.24	1.14
6	35.58	1.14
7	75.33	1.26
8	30.43	1.11
10	11.47	1.05
12	37.41	1.13
14	19.79	1.10

Table 3: Activation Energy for the Reduction Mn(VII) using Sodium metabisulphite.

pH	$Na_2S_2O_5$
2	72.48
4	45.03
6	37.88
7	77.85
8	33.19
10	14.20
12	40.22
14	22.65

Table 4: Amount of Mn(VII) consumed as the concentration of $Na_2S_2O_5$ is varied

$Na_2S_2O_5$ (mg/L)	Consumed Mn(VII) (mg/L)
4	2.644
6	3.966
8	5.288
10	6.611
12	7.933

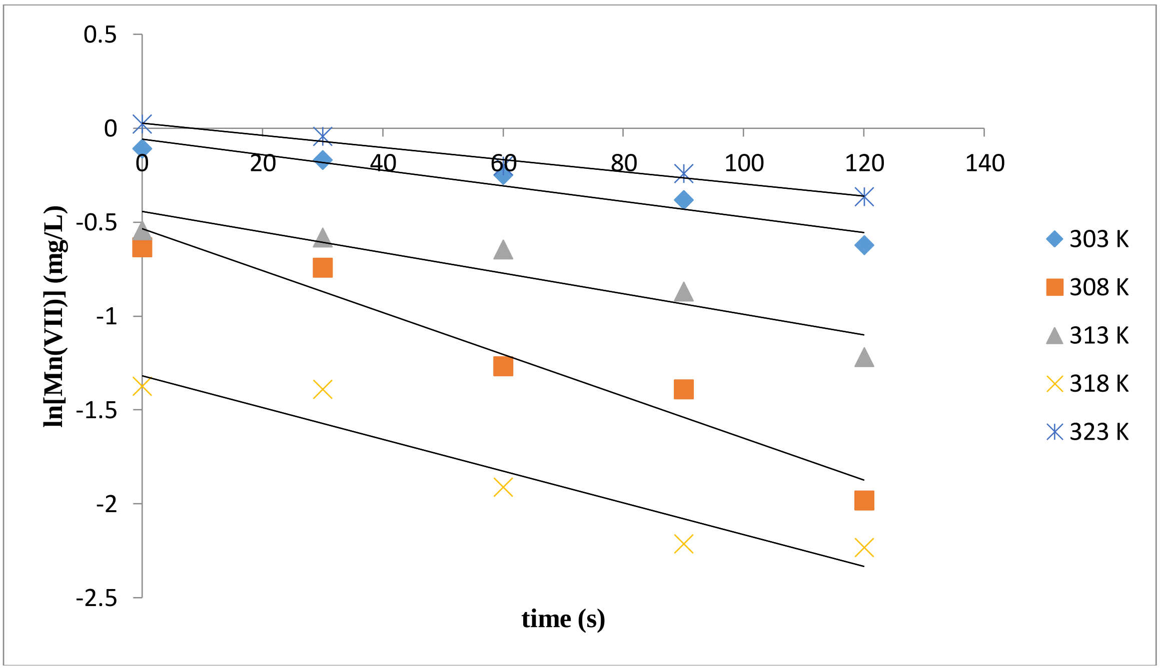

Fig 1: A plot of natural logarithm of [Mn(VII)] using $Na_2S_2O_5$ against time (s) at pH 2

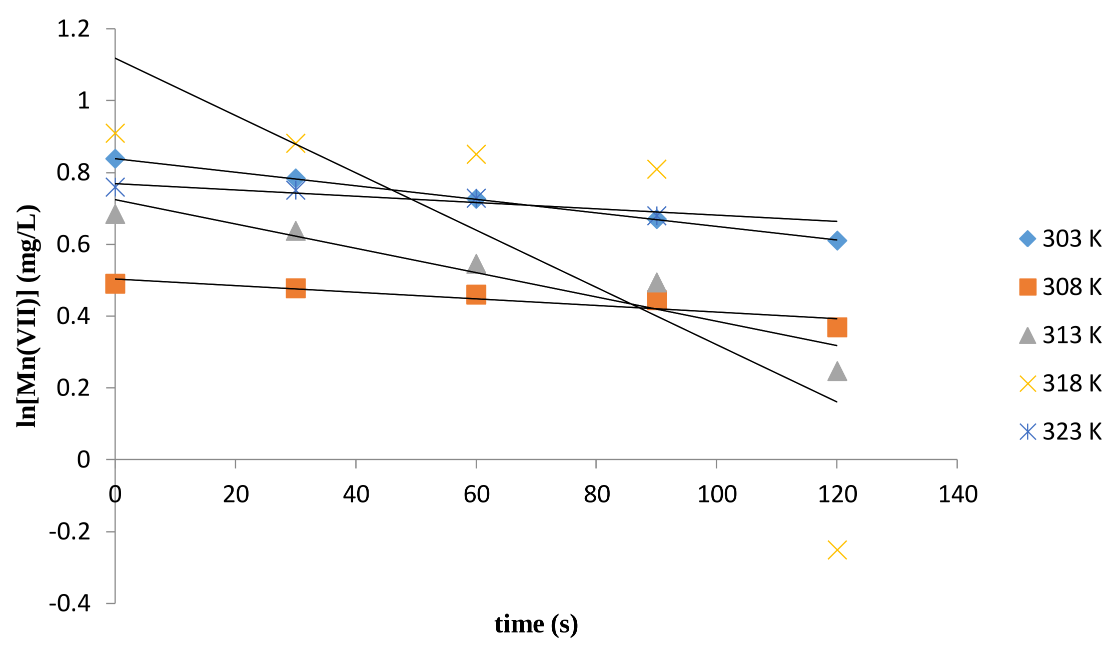

Fig. 2: A plot of natural logarithm of [Mn(VII)] using $Na_2S_2O_5$ against time (s) at pH 4

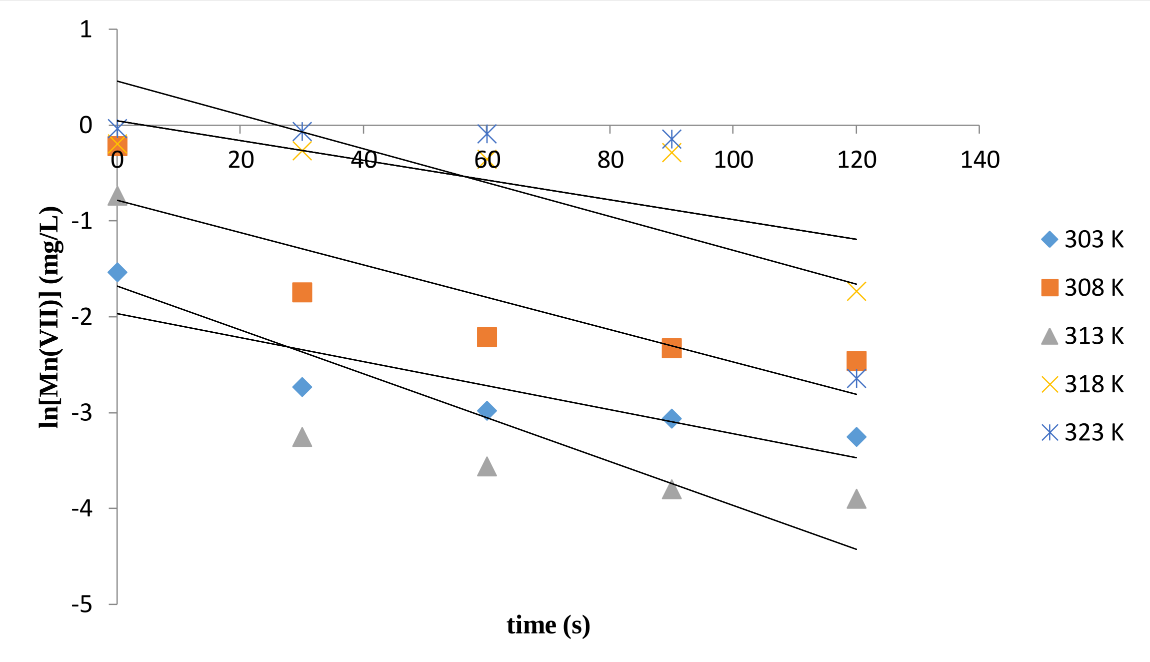

Fig. 3: A plot of natural logarithm of [Mn(VII)] using $Na_2S_2O_5$ against time (s) at pH 6

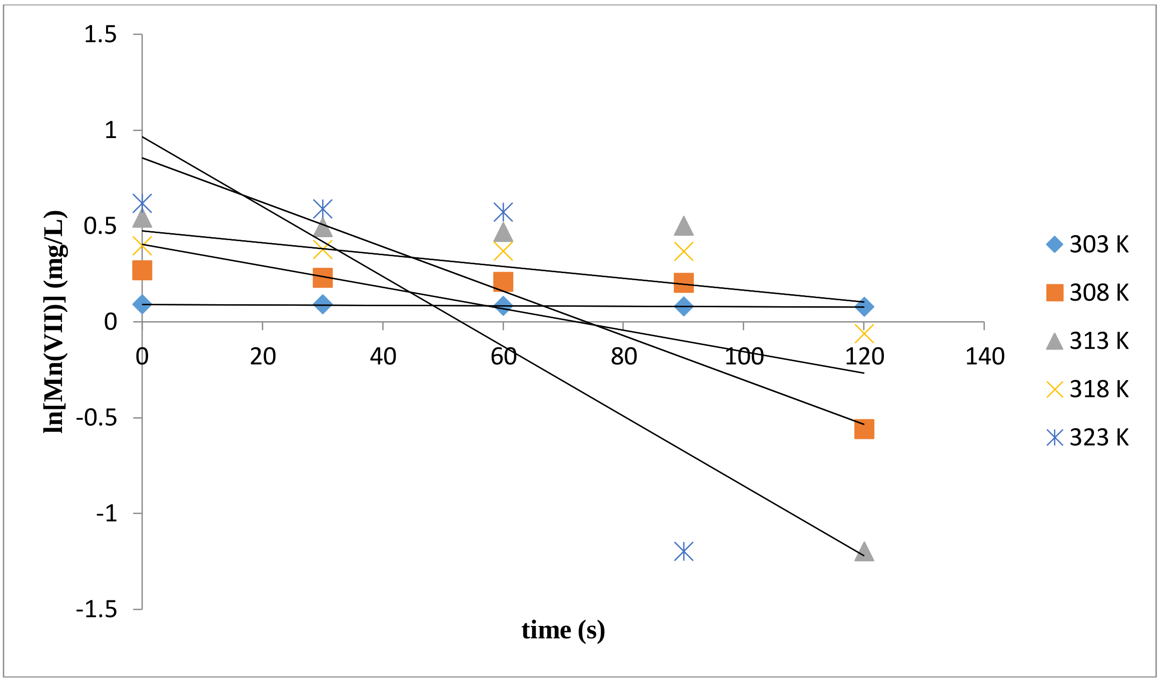

Fig. 4: A plot of natural logarithm of [Mn(VII)] using $Na_2S_2O_5$ against time (s) at pH 7

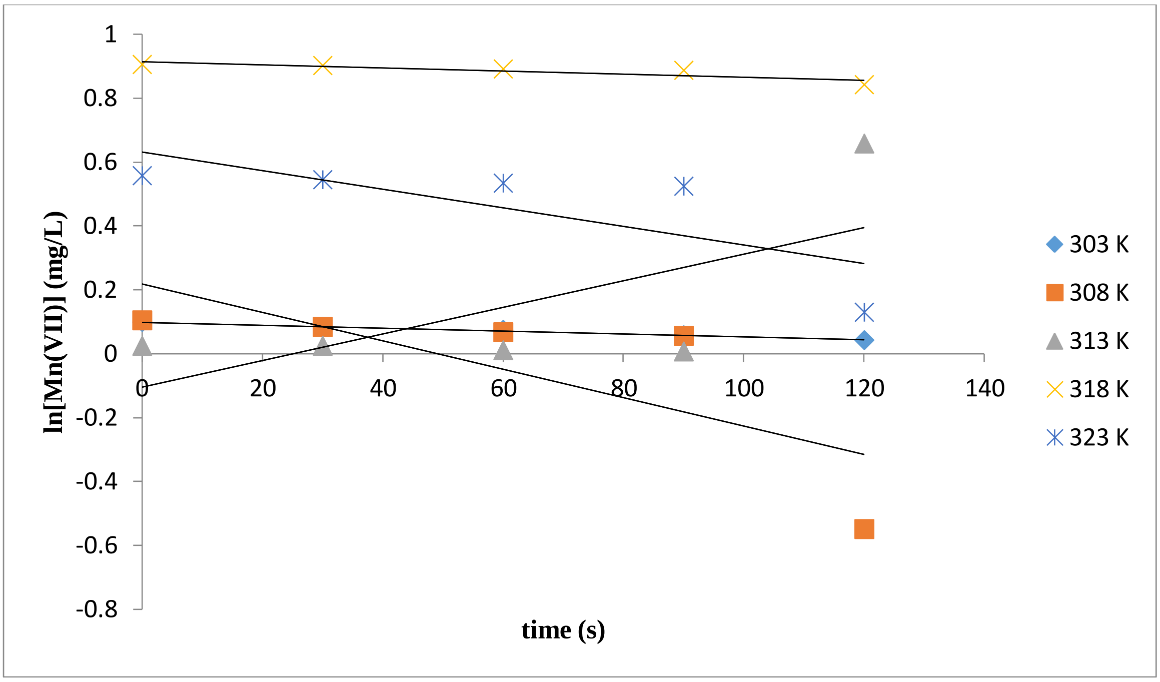

Fig. 5: A plot of natural logarithm of [Mn(VII)] using $Na_2S_2O_5$ against time (s) at pH 8

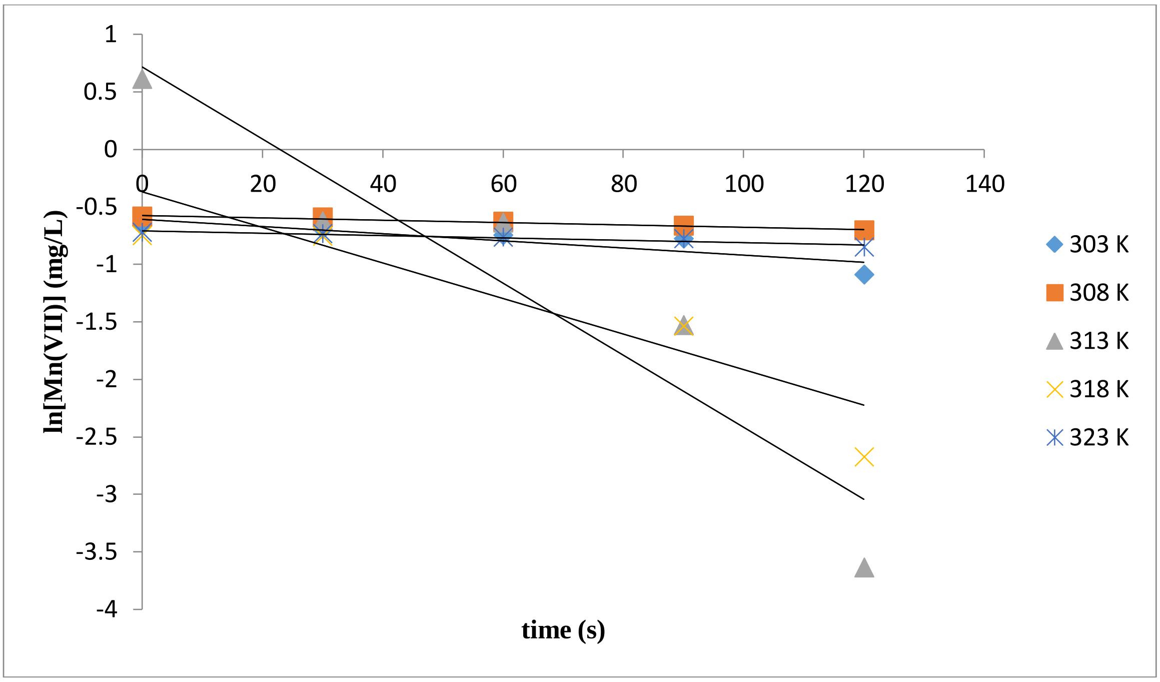

Fig. 6 A plot of natural logarithm of [Mn(VII)] using $Na_2S_2O_5$ against time (s) at pH 10

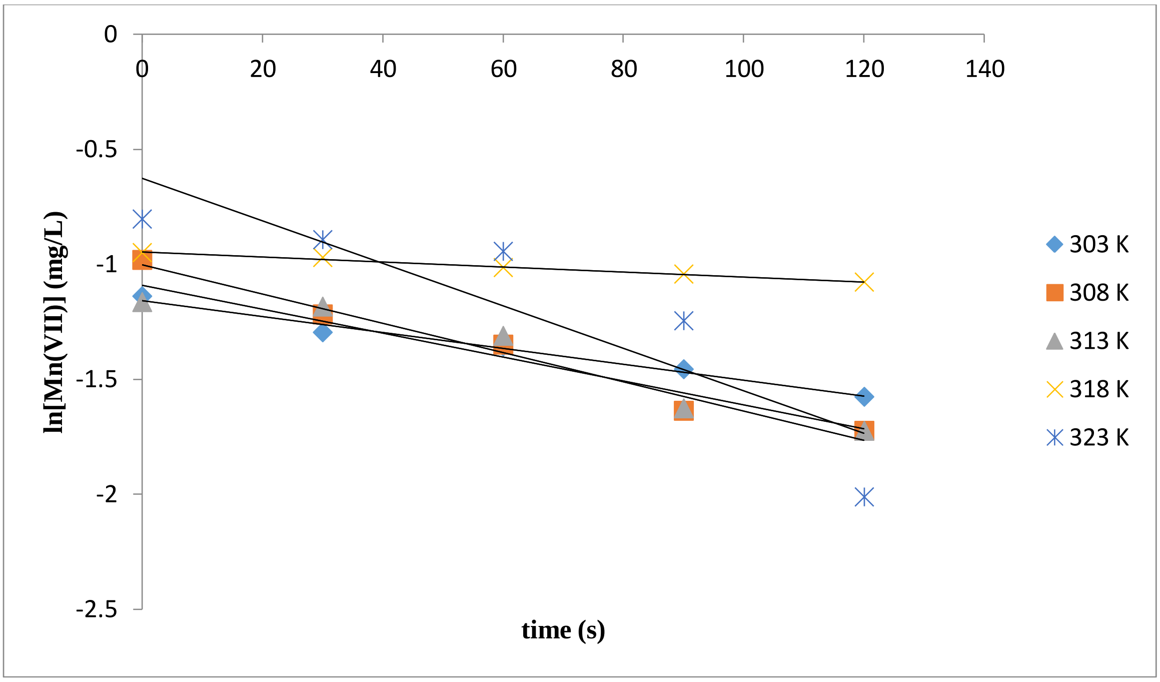

Fig. 7. A plot of natural logarithm of [Mn(VII)] using $Na_2S_2O_5$ vs time (s) at pH 12

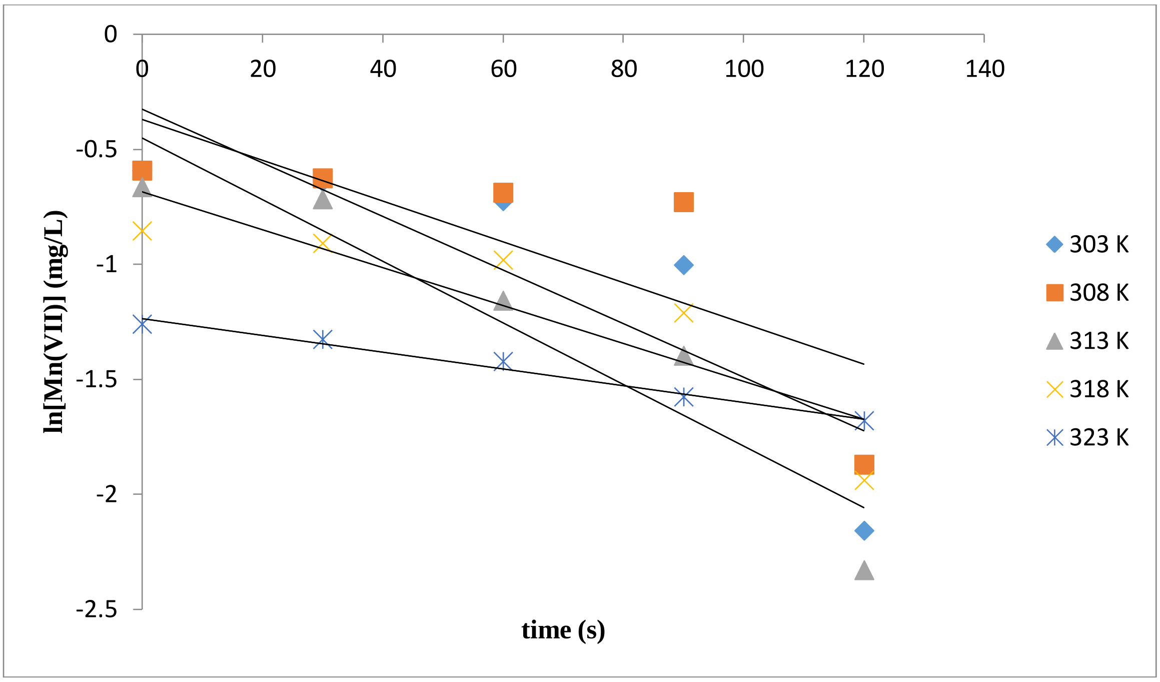

Fig. 8. A plot of natural logarithm of [Mn(VII)] using $Na_2S_2O_5$ vs time (s) at pH 14.

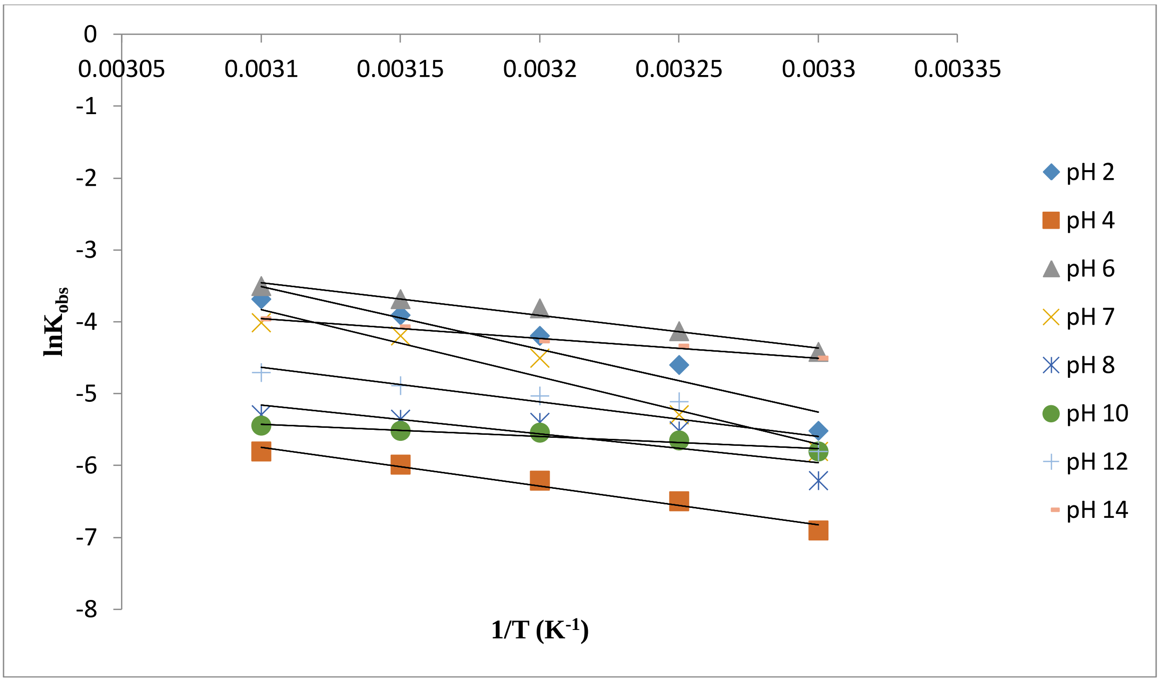

Fig. 9. A plot of $\ln k_{obs}$ vs 1/T for the reduction of Mn(VII) by $Na_2S_2O_5$

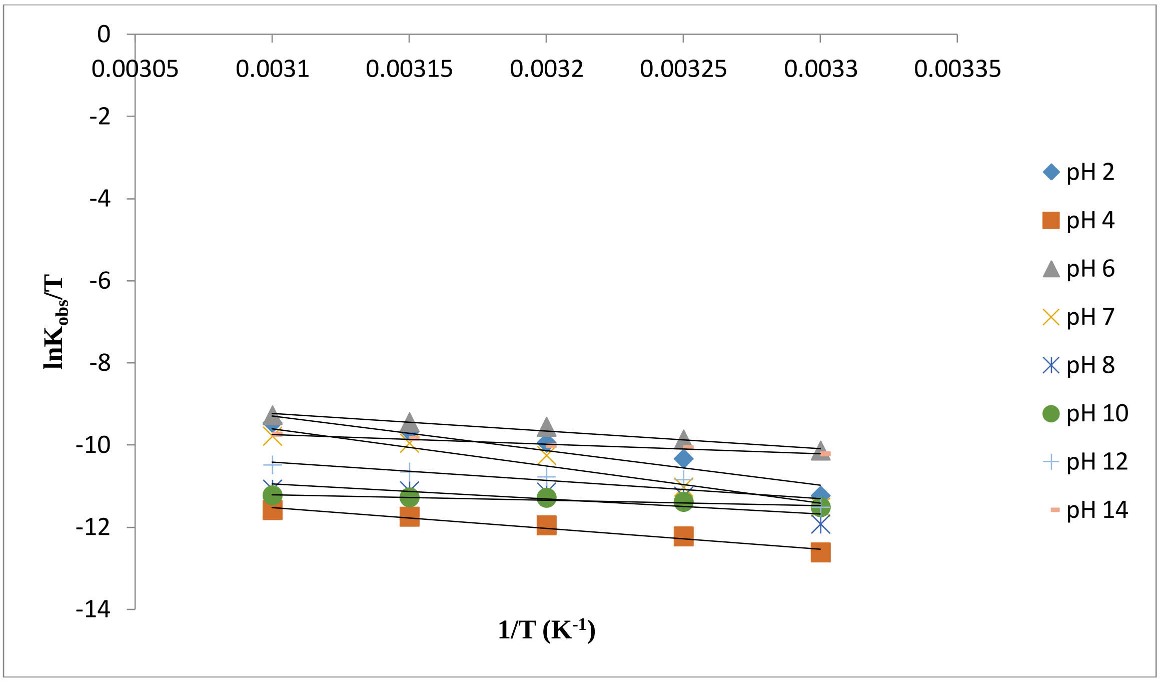

Fig. 10. A plot of ln(k_{obs}/T) vs 1/T for the reduction of Mn(VII) by $Na_2S_2O_5$

IV. Discussion

Kinetic of Mn(VII) Reduction by $Na_2S_2O_5$ at Different Temperature and pH

The results of the observed rate constant of Mn(VII) reduction by $Na_2S_2O_5$ (table 1) was found to be large at an alkaline pH (10) and increases as temperature increases. This shows that the reduction of Mn(VII) was faster at a higher pH (alkaline) and increases as temperature increases. The fast reduction at an alkaline pH can be attributed to the fact that, the concentration of protons (H^+) was lower than hydroxide ions (OH^-) which was not able to deplete electrons transfer thereby causing reduction at a higher rate (Batie and Kamin, 1981). Increased temperature increases the rate of interaction between reacting species; this is why the k_{obs} increased when the temperature was increased (Li *et al*., 2013). In the study, the smallest value of activation energy was obtained at an alkaline pH while the largest value was obtained at an acidic pH which demonstrates that, the rate of reduction of Mn(VII) by $Na_2S_2O_5$ was fast in basic medium and slow in acidic medium (Li *et al.*, 2013). Therefore, basic environment is beneficial to the reduction of Mn(VII) by $Na_2S_2O_5$ while acidic environment is not beneficial.

Thermodynamics of Mn(VII) Reduction by $Na_2S_2O_5$ at Different Temperature and pH

Thermodynamics of Mn(VII) reduction by $Na_2S_2O_5$ at different temperature and pH result are presented in table 2. The values of results showed that $Na_2S_2O_5$ activation enthalpy and entropy vary at a given temperature and pH. In this study, the largest value of activation enthalpy was obtained at a pH of 10. The negative values of activation enthalpy (ΔH^o) of the reduction indicate a reaction in which heat is evolved (Iorungwa *et al*., 2019). And this was found to vary with pH, with alkaline environment (pH of 10) been more exothermic than acidic and neutral environment but remained constant with change in temperature and the positive values of activation entropy (ΔS^o) and its slight variation with pH and temperature suggests that the reduction is driven by solution dynamics and the reduction at pH of 10 been more driven by solution dynamics.

Reaction Stoichiometry

While it is known that the reduction of Mn(VII) results in the production of Mn(II) species (Iorungwa *et al.*, 2019), the reaction with $Na_2S_2O_5$ generates a theoretical stoichiometric equation:

$4KMnO_4 + 5Na_2S_2O_5 + H_2SO_4 \rightarrow 4MnSO_4 + 5Na_2SO_4 + 2K_2SO_4 + H_2O$ ---------- (3)

Table 4 shows the amount (mg/L) of Mn(VII) consumed when the concentration of $Na_2S_2O_5$ was varied. Here, it was observed that the 4 mg/L of $Na_2S_2O_5$ consumed exactly 2.644mg/L of Mn(VII) giving a stoichiometric mass balance Mn(VII)/ $Na_2S_2O_5$ ratio of 0.66:1. The ratio was maintained when the concentration of $Na_2S_2O_5$ was varied from 4 to 12 mg/L and the result obtained from the experiment were in agreement with the theoretical ratio obtained elsewhere (Iorungwa *et al*., 2014).

V. Conclusion

In conclusion, this study has demonstrated that $Na_2S_2O_5$ reduced Mn(VII) in aqueous phase, the reduction rate of Mn(VII) by $Na_2S_2O_5$ was exothermic and entropic in a alkaline environment suggesting that the reduction was beneficial in an alkaline environment and driven by solution dynamics. The stoichiometry suggests that the Mn(VII):$Na_2S_2O_5$ ratio of 0.66:1, was found to be in agreement with the theoretically obtained result for both acidic and alkaline regimes. The activation energy obtained was positive indicating that the product molecules were bonded by strong bonds. The findings from the research show that $Na_2S_2O_5$ could be used for the reductive detoxification of Mn(VII) in polluted areas.

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