

The Inhibitive Action Of Isoamyl Alcohol On The Autoxidation Of Sodium Sulfite By Cu(II) In Alkaline Medium

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ABSTRACT:

The kinetics of the Isoamyl alcohol inhibited Cu(II) catalysed autoxidation of S(IV) in alkaline medium has been studied and based on the observed results following rate has been proposed. Rate constants and the order of reaction were calculated and the reaction was found to be pseudo- first order in all cases. The effect of pH and temperature are also discussed. The value of apparent activation (E_a) energy was calculated graphically by Arrhenius equation.

On the basis of inhibition parameters, the reaction follows free radical mechanism.

$$d[S(IV)]/dt = (k_1 + k_2[Cu(II)]) [S(IV)] / (1 + B [Isoamyl\ alcohol])$$

KEYWORDS: Kinetics; Autoxidation; Rate; Rate constant; SO₂; Cu(II); Catalysis; Inhibition ; Isoamyl alcohol

INTRODUCTION

The autoxidation of sodium sulfite in alkaline medium is significantly accelerated by transition metal ions like Cu(II), leading to rapid degradation of sulfite, which is widely used as a preservative and oxygen scavenger. Controlling this oxidation is crucial in industrial and environmental systems. Isoamyl alcohol, a branched-chain alcohol, has shown potential as an effective inhibitor by disrupting free radical chain mechanisms. The trace metal ions which are part of all atmospheric systems and are catalysing the oxidation of aqueous sulfur dioxide into acid sulphate. The reviews by Huie and Peterson¹, Hoffmann and Boyce², Hoffmann and Jacob³ deal with the oxidation of sulfur(IV) by transition metal ions and their role in catalysing the dioxygen-S(IV) systems. The metal oxides, which are released to the atmosphere as a result of combustion processes are integral part of suspended particulate matter. The catalytic role of several metal oxides such as CoO⁴; Co₂O₃⁵; Ni₂O₃⁶; CuO⁷; MnO₂⁸; and Cu₂O⁹; and MnO₂¹⁰ in acidic medium has been studied and given a two term rate law.

Biglow¹¹ (1898) was first to report that, alcohols slow down the reaction between sodium sulphite and oxygen. Aleya and Backstrom¹² studied the inhibiting effect of aliphatic alcohols such as ethanol, isopropanol, secondary butanol and benzyl alcohol on the oxidation of sodium sulphite in alkaline conditions. So far inhibiting effect of iso amyl alcohol on the metal oxide catalysed autoxidation of aqueous sulfur dioxide is not studied. So in view of the knowing the inhibiting effect of isoamyl alcohol on the Cu(II) catalysed autoxidation of sulfur dioxide in the alkaline medium the present study is under taken.

Experimental

The experimental procedure was exactly the methodology described earlier by Prasad et al¹³. All chemical substances utilized were of analytical grade, and their solutions were prepared using double-distilled water. The reactions were carried out in 0.15-liter Erlenmeyer flasks, exposed to ambient air to facilitate the exchange of atmospheric oxygen. Each flask was situated within a beaker with an inlet at the lower part and an outlet at the upper part to enable the circulation of thermostatic water, maintaining the desired temperature at 33±0.1°C. The reaction were started by adding the desired volume of standard Na₂SO₃ solution for the reaction combination containing different added substances like buffer and catalyst oxide. The reaction mixture was continuously and magnetically stirred at 1600 ± 100 rpm to permit the part of atmospheric oxygen and to save the reaction from becoming oxygen mass exchange controlled. The kinetic studies were conducted in a buffered medium, maintaining a constant pH throughout the entire reaction process. For this purpose, a 10 cm³ of buffer medium from Na₂HPO₄ (0.08 mol dm⁻³) and KH₂PO₄ (0.02 mol dm⁻³) was used to create a basic alkaline medium with a total volume of 100 cm³, thus achieving the desired pH. The kinetic were followed by withdrawing the aliquot examples periodically

and titrating the unreacted S(IV) iodometrically in slightly acidic medium as described earlier. The reproducibility of the replicate measurements was generally better than compared to $\pm 10\%$. All calculations were conducted using MS Excel¹⁴.

Product Analysis-

When the reaction was complete, the separation of Cu(II) was accomplished through filtration and the determination of Sulphate was estimated gravimetrically. This involved the precipitation of sulfate ions as BaSO₄, using standard procedure¹⁵

The product analysis showed the recovery of sulphate to be 98+2%, in all cases in agreement with eq. (1)



RESULTS

Preliminary Investigation—

The kinetics of both uncatalysed and Cu(II) Catalysed reaction were studied in alkaline medium in pH 7.30-9.40 and temperature 33°C. In both of the cases, the first order dependence of S(IV) was observed in the kinetics data treatment for the determination of first order rate constant k_1 was calculated from $\log [\text{S(IV)}]$ versus time, t . The plots were shown in fig 1 from the fig 1 it is observed that both the uncatalysed and Cu catalysed autoxidation of S (IV) reaction is inhibited by Isoamyl alcohol.

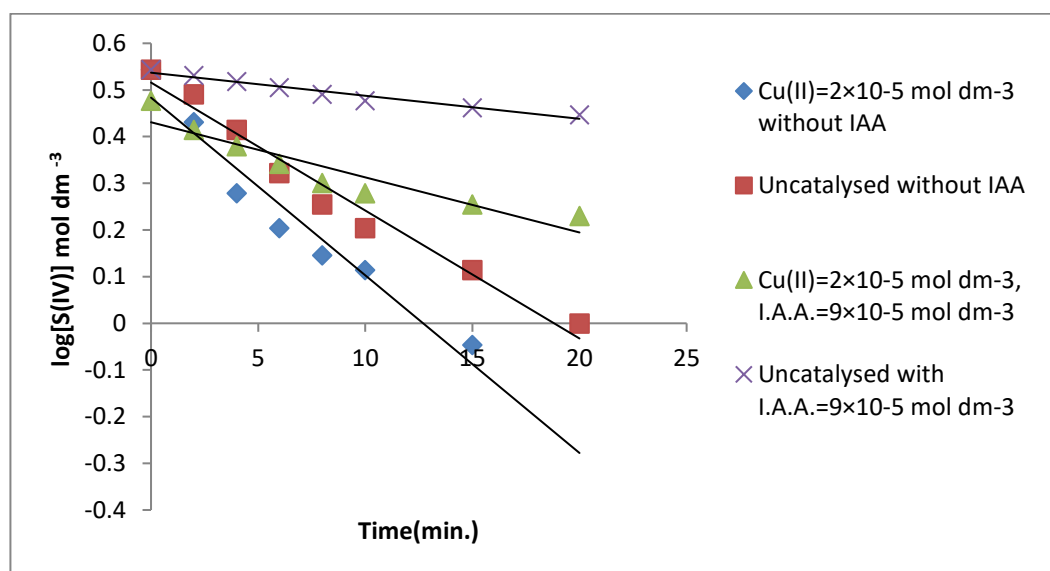


Fig.1 The disappearance of [S(IV)] with time in air saturated suspensions at [S(IV)] = $2 \times 10^{-3} \text{ mol dm}^{-3}$ at pH = 7.30, $t = 33^\circ\text{C}$

Uncatalysed Reaction

In this study, the reaction was examined without adding Cu(II). It is well known that the uncatalysed reaction is initiated by the presence of trace metal particle impurities in the reagent samples and in the distilled water utilized for the preparation of solutions.

Dependence of S (IV)

The dependence of the reaction rate on [S(IV)] was studied by varying sulphite in the range of $1 \times 10^{-3} \text{ mol dm}^{-3}$ to $9 \times 10^{-3} \text{ mol dm}^{-3}$ at pH = 7.30, $t = 33^\circ\text{C}$ in phosphate buffer medium. The kinetics was found to be first order in [S(IV)] as shown in Fig 1 and $\log [\text{S(IV)}]$ versus time plots were linear. The values of first order rate constant, k_1 are shown in table -1. The dependence of reaction rate on [S (IV)] follows the following rate law (2).

$$-d[S(IV)]/dt = k_1[S(IV)] \quad (2)$$

Table 1-The values of k_1 for uncatalysed reaction at different $[S(IV)]$ at pH =7.30, $t=33^\circ\text{C}$ $\text{Na}_2\text{HPO}_4=8\times 10^{-2}\text{mol dm}^{-3}$,

$\text{KH}_2\text{PO}_4=2\times 10^{-2}\text{mol dm}^{-3}$

$[S(IV)] \text{ mol dm}^{-3}$	0.001	0.002	0.003	0.005	0.070	0.009
$(10^3) k_1 \text{ s}^{-1}$	1.04	1.37	1.50	1.65	2.01	2.30

Isoamyl alcohol Dependence

The dependence of Isoamyl alcohol (IAA) on the S(IV) autoxidation was studied by varying the [Isoamyl alcohol] from 1×10^{-6} mol dm^{-3} to 5×10^{-4} mol dm^{-3} in phosphate buffer medium at $[S(IV)]=3\times 10^{-3}$ mol dm^{-3} , pH =7.30, $t=33^\circ\text{C}$ and observed that the rate of the reaction was decreased by increasing Isoamyl alcohol. The results are given in Table 2. The nature of the $[S(IV)]$ dependence in presence of isoamyl alcohol did not change and remains first order. The first order rate constant k_{inh} , in the presence of ethyl alcohol was defined by rate law (3)

$$-d[S(IV)]/dt = k_{\text{inh}}[S(IV)] \quad (3)$$

The values of k_{inh} at different [Isoamyl alcohol] are given in table 2

Table 2 The values of k_{inh} at different [Isoamyl alcohol] at $[S(IV)]=3\times 10^{-3}$ mol dm^{-3} , pH = 7.30, $t = 33^\circ\text{C}$, $\text{Na}_2\text{HPO}_4=8\times 10^{-2}$ mol dm^{-3} , $\text{KH}_2\text{PO}_4=2\times 10^{-2}$ mol dm^{-3}

[Isoamyl Alcohol] mol dm^{-3}	$10^3 k_{\text{inh}} \text{ (S}^{-1}\text{)}$	$1/K_{\text{inh}} \text{ (S)}$
1×10^{-6}	0.576	1736
3×10^{-6}	0.523	1912
7×10^{-6}	0.493	2028
1×10^{-5}	0.468	2137
3×10^{-5}	0.394	2538
5×10^{-5}	0.382	2618
7×10^{-5}	0.320	3125
9×10^{-5}	0.286	3497
1×10^{-4}	0.262	3817
3×10^{-4}	0.212	4717
5×10^{-4}	0.178	5618

The values of first order rate constant k_{inh} in the presence of Isoamyl alcohol decreased with increasing [Isoamyl alcohol] is in agreement with the rate law.

$$k_{\text{inh}} = k_1/(1+B[\text{IAA}]) \quad (4)$$

Where B is inhibition parameter for rate inhibition by Isoamyl alcohol

The equation (4) on rearrangement becomes

$$1/k_{\text{inh}} = 1/k_1 + B[\text{IAA}]/k_1 \quad (5)$$

According to equation (5) the plot of $1/k_{\text{inh}}$ v/s [Isoamyl alcohol] was found to be linear with positive intercept. The values of intercept ($1/k_1$) and slope (B/k_1) were found to be 2.2×10^3 s and 7.4×10^6 mol $^{-1}$ dm^3 s at pH = 7.30, $t = 33^\circ\text{C}$. From these values, the value of slope/Intercept gives inhibition parameter B was found to be 3.28×10^3 mol $^{-1}$ dm^3 .

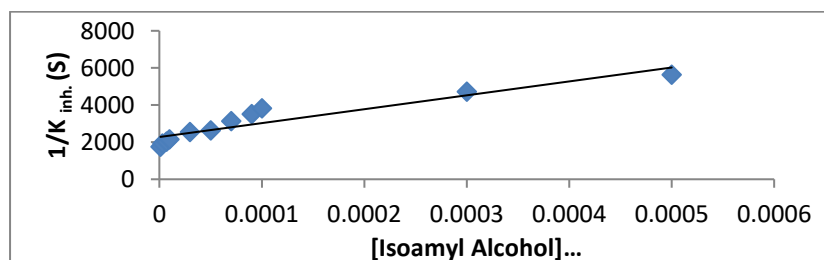


Fig.2-Effect of Isoamyl alcohol at $[S(IV)]=3\times 10^{-3}$ mol dm^{-3} pH=7.30 and temp= 33°C in Phosphate buffered medium.

Cu(II) Catalysed Reaction

The kinetics of Cu(II) Catalysed autoxidation of S(IV) was studied in alkaline medium in the absence of inhibitor Isoamyl alcohol.

[S(IV)] Variation

The effect of S(IV) on reaction rate was investigated by increasing [S(IV)] from $1 \times 10^{-3} \text{ mol dm}^{-3}$ to $9 \times 10^{-3} \text{ mol dm}^{-3}$ at various but fixed Cu(II) concentrations of 2×10^{-5} , 4×10^{-5} , and $6 \times 10^{-5} \text{ mol dm}^{-3}$ at pH = 7.30, $t = 33^\circ\text{C}$. The kinetics was found to be first order in S(IV) v/s time are linear, as illustrated in Fig. 1.

Cu(II) variations

The effect of Cu(II) on the reaction rate was studied by varying Cu(II) from 1×10^{-5} to $1 \times 10^{-4} \text{ mol dm}^{-3}$ at S(IV) = $2 \times 10^{-3} \text{ mol dm}^{-3}$, pH = 7.30, $t = 33^\circ\text{C}$ in alkaline buffer medium. The values of first order rate constant k_{cat} for S(IV) oxidation was determined are shown in fig 3. The nature of dependence of k_{cat} on Cu(II) was indicated as two term rate law (6)

$$-d[S(IV)]/dt = k_{\text{cat}}[S(IV)] = (k_1 + k_2[\text{Cu(II)}])[S(IV)] \quad (6)$$

$$\text{Or } k_{\text{cat}} = k_1 + k_2[\text{Cu(II)}] \quad (7)$$

From the plot in fig.3 the values of intercept is equal to k_1 and slope is equal to k_2 were found to be $7.09 \times 10^2 \text{ s}$ and $4.39 \times 10^6 \text{ mol dm}^{-3} \text{ s}$ at pH = 7.30, $t = 33^\circ\text{C}$, in phosphate buffered medium.

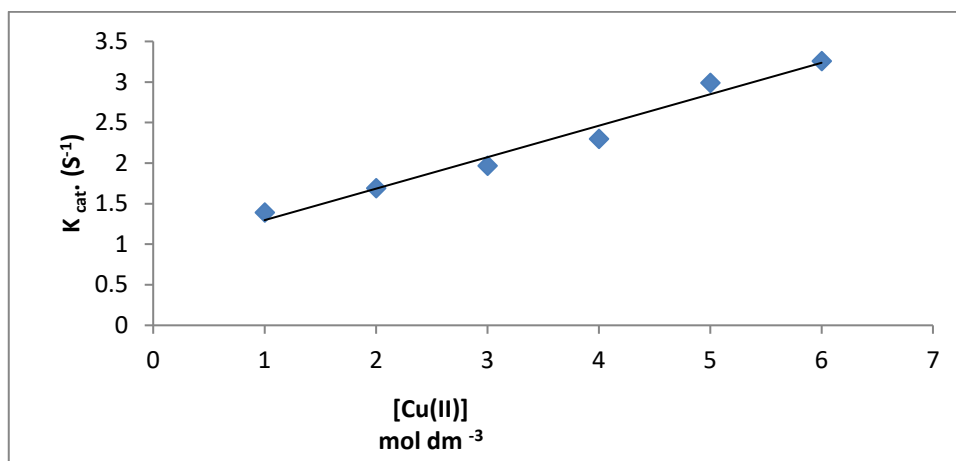


Fig.3 The dependence of Cu(II) concentration at [S(IV)] = $2 \times 10^{-3} \text{ mol dm}^{-3}$, pH = 7.30, $t = 33^\circ\text{C}$, in Phosphate buffered medium.

Variation of pH

The pH range of 7.30 to 9.40 is used for pH fluctuation, with fixed S(IV), Cu(II), and Isoamyl alcohol, temperatures. It was discovered that the rate remained constant when the pH was changed. The impact of [buffer] was investigated by altering the concentrations of KH_2PO_4 and Na_2HPO_4 . pH stayed constant as long as the ratio $[\text{Na}_2\text{HPO}_4]/[\text{KH}_2\text{PO}_4]$ stayed constant. The findings showed that the buffer concentration had no effect on the reaction rate. The results are shown in Table 3.

Table-3 Effect of pH at [S(IV)] = $3 \times 10^{-3} \text{ mol dm}^{-3}$, [Cu(II)] = $2 \times 10^{-5} \text{ mol dm}^{-3}$, [Isoamyl alcohol] = $9 \times 10^{-5} \text{ mol dm}^{-3}$, $t = 33^\circ\text{C}$, $\text{Na}_2\text{HPO}_4 = 8 \times 10^{-2} \text{ mol dm}^{-3}$, $\text{KH}_2\text{PO}_4 = 2 \times 10^{-2} \text{ mol dm}^{-3}$

pH	7.30	7.90	8.50	8.90	9.20	9.40
$10^3 K (S^{-1})$	1.16	1.18	1.23	1.26	1.20	1.30

Rate law in the Presence of Isoamyl alcohol

A detailed study of dependence of rate on S(IV), Cu(II) and pH with in the presence of isoamyl alcohol revealed that the kinetics remain first order both in S(IV) and Cu(II) and independence of pH obeys the following rate law.

$$-d[S(IV)]/dt = (k_1 + k_2[\text{Cu(II)}])[S(IV)]/(1 + B[\text{IAA}]) \quad (8)$$

$$\text{Where } k_{\text{inh}} = (k_1 + k_2[\text{Cu(II)}]) / (1 + B[\text{IAA}]) = k_{\text{cat}} / (1 + B[\text{IAA}]) \quad (9)$$

$$1/k_{inh} = (1+B [IAA]) / k_{cat} \quad (10)$$

$$1/k_{inh} = 1/k_{cat} + B [IAA] / k_{cat} \quad (11)$$

Table 4 The value of K_{inh} at different [Isoamyl alcohol] , $S(IV)=3 \times 10^{-3} \text{ mol dm}^{-3}$, $Cu(II) = 2 \times 10^{-5} \text{ mol dm}^{-3}$, $pH=7.30$,

$t=33^\circ\text{C}$, $Na_2HPO_4=8 \times 10^{-2} \text{ mol dm}^{-3}$, $KH_2PO_4=2 \times 10^{-2} \text{ mol dm}^{-3}$

[Isoamyl Alcohol] mol dm^{-3}	$10^3 K_{inh} \cdot (S^{-1})$	$1/ K_{inh} \cdot (S)$
1×10^{-6}	2.11	474
3×10^{-6}	1.92	521
7×10^{-6}	1.65	606
1×10^{-5}	1.42	704
3×10^{-5}	1.38	725
5×10^{-5}	1.36	735
7×10^{-5}	1.28	781
9×10^{-5}	1.16	862
1×10^{-4}	0.99	1010
3×10^{-4}	0.857	1167
5×10^{-4}	0.644	1553

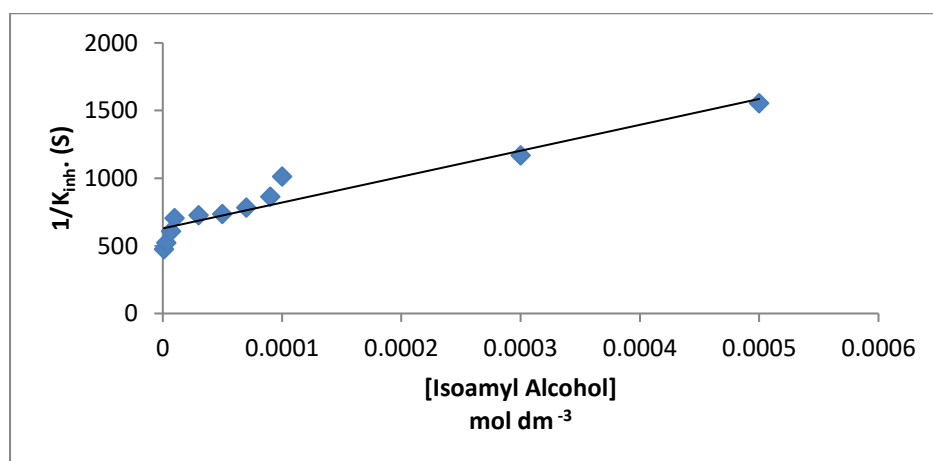


Fig.4-Effect of [Isoamyl alcohol] at $S(IV)=3 \times 10^{-3} \text{ mol dm}^{-3}$ at $t=33^\circ\text{C}$, $Cu(II) = 2 \times 10^{-5} \text{ mol dm}^{-3}$ and $pH=7.30$ in phosphate buffered medium. By plotting a graph between $1/k_{inh}$ versus [Isoamyl alcohol] gives a straight line with positive intercept fig.4. The values of intercept = $1/k_{cat}$ and slope= B/k_{cat} from the graph these values are found to be $6.2 \times 10^2 \text{ s}$ and $1.9 \times 10^6 \text{ mol}^{-1} \text{ dm}^3 \text{ s}$ separately. From these values the value of inhibition parameter B can be determined. Inhibition parameter $B = \text{slope}/\text{Intercept}$ that is $B = 3.04 \times 10^3 \text{ mol}^{-1} \text{ dm}^3$.

Effect of temperature-

The values of k_{obs} were determined at different temperatures in the range of 33°C to 43°C . The results are given in Table 5. By plotting a graph between $\log k$ v/s $1/T$ yield us an apparent empirical energy of activation $47.76 \text{ kJ mol}^{-1}$ in the presence of isoamyl alcohol.

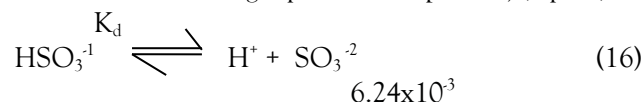
Table 5 The Effect of temp. on K_{obs} at $S(IV)=3 \times 10^{-3} \text{ mol dm}^{-3}$, $Cu(II) = 2 \times 10^{-5} \text{ mol dm}^{-3}$, [Isoamyl alcohol] $= 9 \times 10^{-5}$

mol dm^{-3} , $Na_2HPO_4=8 \times 10^{-2} \text{ mol dm}^{-3}$, $KH_2PO_4=2 \times 10^{-2} \text{ mol dm}^{-3}$

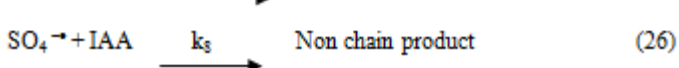
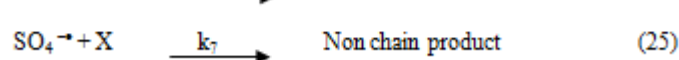
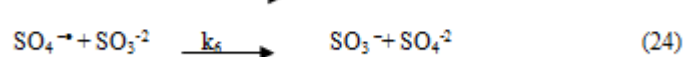
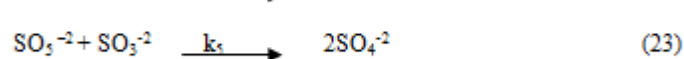
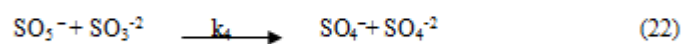
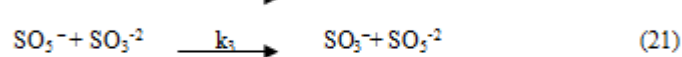
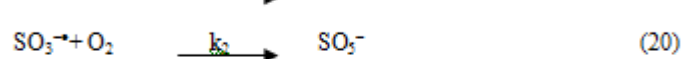
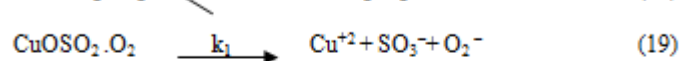
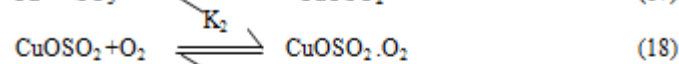
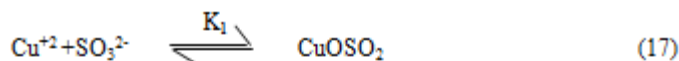
Temp.	33°C	38°C	43°C
$10^3 K_{obs} (S^{-1})$	1.16	1.54	2.19

DISCUSSION-

In the experimental range of pH = 7.30-9.40 the following equilibrium operates, (eq. 16):



In the pH range 7.30 to 9.40 SO_3^{-2} present predominantly. The rate of reaction indicates that it is independent of pH. It is reported that radical mechanism operates in those reactions in which the inhibition parameters lies $10^3 - 10^4$ as reported by gupta et al¹⁶. In the present study, The value of inhibition parameter for uncatalysed and Cu (II) catalysed autoxidation of S (IV) by isoamyl alcohol is found to be $3.28 \times 10^3 \text{ mol}^{-1} \text{ dm}^3$ & $3.04 \times 10^3 \text{ mol}^{-1} \text{ dm}^3$ respectively. This strongly supports the radical mechanism in the present case too based on the observed results¹⁷⁻²⁴, (eqs. 17-26):



In the mechanism, no role is assigned to $\text{O}_2^{-\cdot}$, which is also known to react with sulfur (IV) slowly²⁵. It may disproportionate to form H_2O_2 and O_2 or may be scavenged by impurities. By assuming long chain hypothesis and steady state approximation $d[\text{SO}_3]/dt$, $d[\text{SO}_4]/dt$, $d[\text{SO}_5]/dt$, to zero. It can be shown that the rate of initiation is equal to rate of termination (eq. 27):

$$k_1[\text{Cu(II)}](\text{SO}_3^{-2})(\text{O}_2) = \{k_7[\text{X}] + k_8[\text{IAA}]\} [\text{SO}_4^{-1}] \dots\dots\dots (27)$$

Since the reaction is completely stopped in the presence of $[\text{IAA}] = 1 \times 10^{-4} \text{ mol dm}^{-3}$, so the steps (20) and (23) appear to be unimportant. From the above mechanism the following rate law can be obtained (eq. 28):

$$R_{\text{cat}} = k_1 [\text{Cu(II)}] [\text{S(IV)}] / \{k_7[\text{X}] + k_8[\text{IAA}]\} \dots\dots\dots (28)$$

By comparing the derived rate of law with the experimental rate of law we observed the similarity in these two. The calculated value of inhibition constant B is $3.28 \times 10^3 \text{ mol}^{-1} \text{ dm}^3$ which is in the range of $10^3 - 10^4$ and also coincide with earlier reported value of B of Cu(II) catalyzed autoxidation of S(IV) by isoamyl alcohol is $3.04 \times 10^3 \text{ mol}^{-1} \text{ dm}^3$ so on the basis of the calculated value of B we concluded that isoamyl alcohol acts as a free radical scavenger in Cu (II) catalyzed autoxidation of aqueous SO_2 in alkaline medium and a free radical mechanism can work in this system²⁶.

CONCLUSION

The conclusions are deduced from the results of the Isoamyl alcohol inhibited Cu (II) catalysed autoxidation of S(IV) was that inhibit the oxidation with the fast influence. The value of Inhibition factor of both uncatalysed and Cu(II) catalysed autoxidation of S(IV) in the present study are in the range of $10^3 - 10^4$ which shows that free radical mechanism is operative.

FUTURE SCOPE

The results are useful for modeling rain water acidity and therefore a great use of meteorology and impressive chemistry. This study is important in understanding the mechanism of the atmospheric oxidation of S(IV) by O₂.

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