



RESEARCH PAPER

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Kinetics of Succinic Acid Inhibited Uncatalyzed and CoO Catalyzed Atmospheric Autoxidation of S(IV) in Alkaline Medium

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Abstract

Air change is a major discuss of today's earth. The worry of weather changes along with the changeability of oil in the future. The burning of fossil power source, mostly coal is much accountable for global warming. The atmospheric response of sulfur dioxide is considerable acid rain predecessor and are accountable for acidification of various forms of climatic water. The purpose of present work was to study the influence of the succinic acid inhibited CoO catalyzed autoxidation of S(IV) in alkaline medium has been studied and on the basis of 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 by using Arrhenius equation. On the basis of calculated inhibition parameters, the reaction follows a free radical mechanism.

Keywords: Kinetics; Autoxidation; CoO; Catalysis; Inhibition; Succinic acid; Free radical mechanism

Introduction

The conversion of sulfur dioxide to acid sulphate is the major cause of climatic acid precipitation. It is usually agreed that the gas phase oxidation including photochemical oxidation by O_3 and H_2O_2 , which are produced in the climatic by photochemical reaction, and oxidation by O_2 in aqueous phase catalyzed by dissolved trace metal ions and by suspended particulate matter are major benefactor to acid precipitation (Calvert et al., 1986; Berreshim and Jaeshke, 1986). The oxidation of dissolved sulfur dioxide, henceforth known as sulfur (IV), proceeds via radical mechanism involving sulfoxyl radicals (Gupta et al., 1999; Sharma et al., 2017; Gupta et al., 2004). In acidic medium many metal oxides such as CoO (Mudgal et al., 2008); Ni_2O_3 (Sharma et al., 2018); CuO (Sharma et al., 2012); MnO_2 (Sharma et al., 2019) and Cu_2O have been reported. S(IV) autoxidation reactions are known to occur via both radical and non-radical mechanisms (Ghosh and Rajput, 2012). All type of VOCs, for example, aliphatic, aromatic, cyclic, polycyclic, heterocyclic, acyclic, saturated, unsaturated oxygenated, halogenated, alcohols, terpenes phenols, carbonyl compounds, carboxylic acid, esters, chloro-subordinates etc. are found noticeably in the surrounding, soil and water environments (Meena et al., 2017a; Meena et al., 2017b; Wilkosz and Mainka, 2008).

An interesting feature of numerous radical reactions are inhibited by ammonia, formic acid, benzamide, isopropyl alcohol, isoamyl alcohol, aniline, sodium benzoate (Sharma et al., 2015; Sharma et al., 2016; Sharma et al., 2017a; Sharma et al., 2017b). In the Indian subcontinent, the pH of the water reaches lies 6.5- 8.5. This requires the study of autoxidation of S(IV) in alkaline Medium (Prasad et al., 1991; Sharma et al., 2018). Autoxidation is catalyzed by CoO in the pH range 7.34- 9.40 and hence the effect of succinic acid on the reaction has been studied to know the nature of the mechanism.



Experimental

The experimental procedure was exactly as used earlier (Hussain et al., 2018a; Hussain et al., 2018b; Hussain et al., 2018c). 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. Erlenmeyer 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 $33 \pm 0.1^\circ\text{C}$. The reaction was started by adding the desired volume of standard Na_2SO_3 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 cm^3 of buffer medium from of Na_2HPO_4 (0.08 mol dm^{-3}) and KH_2PO_4 (0.02 mol dm^{-3}) was used to create a basic alkaline medium with a total volume of 100 cm^3 , 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 Excel (Hussain et al., 2018d).

Product analysis

The qualitative tests revealed sulfate to be the only oxidation product. For quantitative testing, the reaction mixture containing catalyst and S(IV) in appropriately buffered solutions was stirred continuously for a sufficiently long time to ensure complete. Oxidation of sulfur (IV). When the reaction was complete, the separation of CoO was filtered and the Sulphate was gravimetrically evaluated by precipitating the sulphate particles as BaSO_4 , using standard procedure (Begam et al., 2018).

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



Results

Preliminary investigation

The kinetics of both uncatalyzed and CoO catalyzed reactions were studied in alkaline medium at pH 7.34-9.40 and temperature 33°C . In both of the cases, the first order dependence of S(IV) was observed and the first order rate constant k_1 was calculated from $\log [\text{S(IV)}]$ versus time, t . The plots were shown in fig 1. it is observed that both the uncatalyzed and CoO catalyzed autoxidation of S (IV) reactions is inhibited by succinic acid.

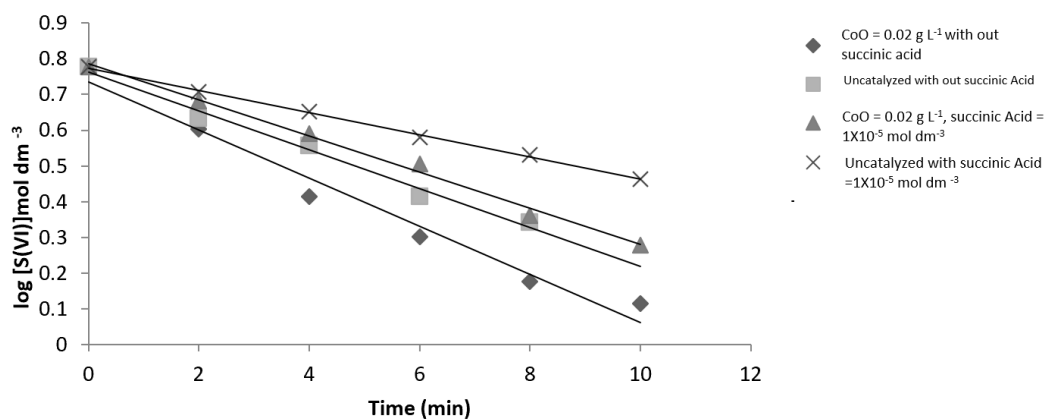


Fig. 1 The disappearance of $[\text{S(IV)}]$ with time in air saturated suspensions at $[\text{S(IV)}] = 3 \times 10^{-3}\text{ mol dm}^{-3}$ at pH = 7.34 and temp = 33°C , (×) Uncatalyzed with Succinic Acid = $1 \times 10^{-5}\text{ mol dm}^{-3}$, (Δ) CoO = 0.020 g L^{-1} Succinic Acid = $1 \times 10^{-5}\text{ mol dm}^{-3}$, (■) Uncatalyzed Without Succinic Acid, (◇) CoO = 0.020 g L^{-1} , without succinic acid.

Uncatalyzed reaction

The reaction was examined without adding CoO. As it is well known that the uncatalyzed 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 solution.

Dependence of S (IV)

The dependence of the reaction rate on [S(IV)] was studied by varying sulphate in the range of 1×10^{-3} mol dm⁻³ to 5×10^{-3} mol dm⁻³ at pH = 7.34, t = 33°C in phosphate buffer medium. The kinetic was found to be first order in [S(IV)] as shown in Fig. 1 and log [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 uncatalyzed reaction at different [S(IV)] at pH = 7.34 and t = 33°C.

[S(IV)] mol dm ⁻³	(10 ³) k_1 s ⁻¹
0.001	1.38
0.002	1.75
0.003	2.09
0.004	2.37
0.005	2.60

[Succinic acid] dependence

Aim of the study was to examine the effect of succinic acid inhibitors on the autoxidation of S(IV) in alkaline medium so for this purpose succinic acid was varied the from 8×10^{-6} mol dm⁻³ to 1×10^{-4} mol dm⁻³ and observed the rate of the reaction decreased by increasing succinic acid. The results are given in table 2. The nature of the [S(IV)] dependence in presence of succinic acid did not change and remains first order. The first order rate constant k_{inh} , in the presence of succinic acid was defined by rate law (3).

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

The values of k_{inh} at different [succinic acid] are given in table 2.

Table 2. The values of k_{inh} at different [succinic acid] at pH = 7.34 and t = 33°C.

[Succinic acid] mol dm ⁻³	10 ³ k_{inh} , s ⁻¹	1/ k_{inh} , s
8.0×10^{-6}	1.22	819
1.0×10^{-5}	1.19	840
3.0×10^{-5}	1.13	884
6.0×10^{-5}	0.98	1020
8.0×10^{-5}	0.94	1063
1.0×10^{-4}	0.93	1075

The value of first order rate constant k_{inh} in the presence of succinic acid decreased with increasing [succinic acid] in agreement with the rate law

$$k_{inh} = k_1/(1+B[S.A.]) \quad (4)$$

Where B is inhibition parameter for rate inhibition by succinic acid

The equation (4) on rearrangement becomes

$$1/k_{inh} = 1/k_1 + B[S.A.]/k_1 \quad (5)$$

The equation (5) the plot of $1/k_{inh}$ v/s [succinic acid] was found to be linear with positive intercept fig 2. The values of intercept ($1/k_1$) and slope (B/k_1) were found to be 8.07×10^2 s and 2.9×10^6 mol⁻¹ dm³s at pH = 7.34 and t = 33°C From these values the value of inhibition parameter B was found to be 3.6×10^3 mol⁻¹ dm³

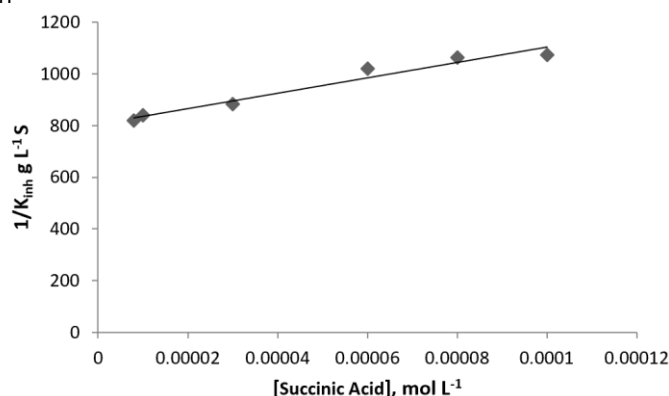


Fig. 2 Effect of succinic acid at [S(IV)] = 3×10^{-3} mol dm⁻³, pH = 7.34 and temp = 33°C in alkaline buffered medium.

CoO catalyzed reaction

The kinetics of CoO catalyzed autoxidation of S(IV) was studied in alkaline medium in the absence of inhibitor succinic acid

[S(IV)] Variation

The dependence of S(IV) on reaction rate was studied by varying [S(IV)] from $1 \times 10^{-3} \text{ mol dm}^{-3}$ to $5 \times 10^{-3} \text{ mol dm}^{-3}$ at two different but fixed [CoO] of 0.01 and 0.02 g dm^{-3} at pH = 7.34 and $t = 33^\circ$. The kinetics was found to be first order in [S(IV)].

CoO variations

The effect of CoO on the reaction rate was studied by varying CoO from 0.01 to 0.05 gm dm^{-3} at $S(IV) = 3 \times 10^{-3} \text{ mol dm}^{-3}$, pH=7.34, $t = 33^\circ\text{C}$ in alkaline buffer medium. The values of first order rate constant k_{cat} given in Table-3 were plotted against [CoO] and are shown in fig 3. The nature of dependence of k_{cat} on CoO was indicated as two term rate law (6)

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

$$\text{Or } k_{\text{cat}} = k_1 + k[\text{CoO}] \quad (7)$$

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 $4.6 \times 10^2 \text{ s}$ and $1.7 \times 10^3 \text{ mol}^{-1} \text{ dm}^3 \text{ s}$ at pH = 7.34, $t = 33^\circ\text{C}$, in alkaline buffered medium.

Table 3. The value of k_{cat} at different [CoO] at pH = 7.34 and Temp. = 33°C , $\text{Na}_2\text{HPO}_4 = 8 \times 10^{-2} \text{ mol L}^{-1}$, $\text{KH}_2\text{PO}_4 = 2 \times 10^{-2} \text{ mol L}^{-1}$

CoO (g dm^{-3})	$10^3 k_{\text{cat}} \text{ s}^{-1}$
0.01	2.20
0.02	2.31
0.03	2.45
0.04	2.54
0.05	2.60

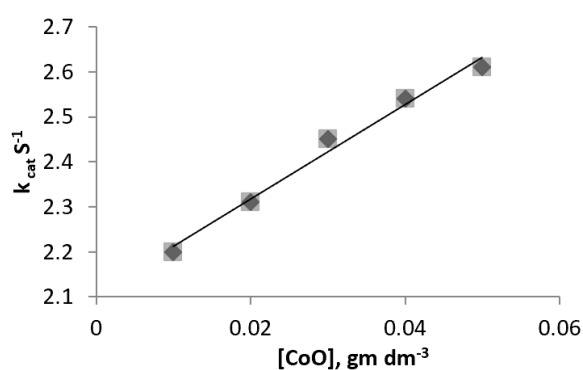


Fig. 3 The dependence of catalyst concentration at $[S(IV)] = 3 \times 10^{-3} \text{ mol dm}^{-3}$, pH = 7.34, $t = 33^\circ\text{C}$ in alkaline buffered medium.

Variation of pH

pH variation within the range 7.34 to 9.40 at fixed different concentrations of S(IV), CoO, and Succinic acid, temperatures. The rate constant show that the rate was not affected by varying pH. The results are show in table 4.

Table 4. Effect of pH at $[S(IV)] = 3 \times 10^{-3} \text{ mol dm}^{-3}$ at $\text{CoO} = 0.02 \text{ gm dm}^{-3}$, $[\text{Succinic acid}] = 6 \times 10^{-4} \text{ mol dm}^{-3}$

pH	7.34	8.55	8.96	9.40
$10^3 k_{S,A} \text{ s}^{-1}$	1.94	1.80	1.95	2.00

Rate law in the presence of succinic acid

A detailed study of dependence of rate on S(IV), [CoO] and pH with in the presence of Succinic acid revealed that the kinetics remain first order both in S(IV) and [CoO] and independence of pH Table-5 obeys the following rate law.

$$-d[S(IV)]/dt = (k_1 + k_2[CoO] [S(IV)] / 1 + B (S.A.)) \quad (8)$$

$$\text{Where } k_{inh} = (k_1 + k_2[CoO] / 1 + B (S.A.)) = k_{cat} / 1 + B (S.A.) \quad (9)$$

$$1/k_{inh} = 1 + B (S.A.) / k_{cat} \quad (10)$$

$$1/k_{inh} = 1/k_{cat} + B (S.A.) / k_{cat} \quad (11)$$

Table 5. The values of k_{inh} at different [succinic acid] at $[S(IV)] = 3 \times 10^{-3} \text{ mol dm}^{-3}$, $CoO = 0.02 \text{ gm dm}^{-3}$ at $pH = 7.34$ and $t = 33^\circ C$

[Succinic acid] mol dm ⁻³	$10^3 k_{s,A} s^{-1}$	$1/k_{s,A} s$
8.0×10^{-6}	2.13	469
1.0×10^{-5}	1.94	515
3.0×10^{-5}	1.82	549
6.0×10^{-4}	1.77	564
8.0×10^{-5}	1.67	598
1.0×10^{-4}	1.56	641

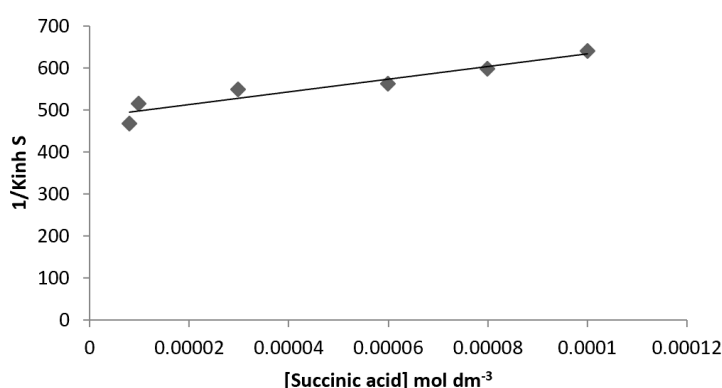


Fig. 4 Effect of succinic acid at $[S(IV)] = 3 \times 10^{-3} \text{ mol dm}^{-3}$, $[CoO] = 0.02 \text{ gm dm}^{-3}$, $pH = 7.34$ and $temp = 33^\circ C$ in alkaline buffered medium.

By plotting a graph between $1/k_{inh}$ versus [succinic acid] 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 $4.8 \times 10^2 \text{ s}$ and $1.5 \times 10^6 \text{ mol}^{-1} \text{ dm}^3 \text{ s}$ respectively. From these values the value of inhibition parameter B can be determined, inhibition parameter $B = \text{slope}/\text{Intercept}$ that is $B = 3.1 \times 10^3 \text{ mol}^{-1} \text{ dm}^3$.

Effect of temperature

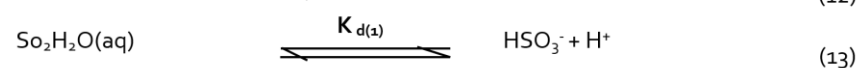
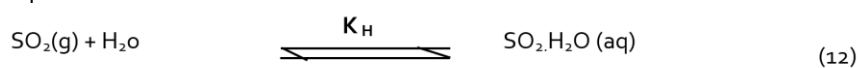
The values of k_{obs} were determined at different temperatures in the range of $33^\circ C$ to $48^\circ C$. The results are given in Table 6. By plotting a graph between $\log k$ v/s $1/T$ yield us an apparent empirical energy of activation $45.26 \text{ kJ mol}^{-1}$ in the presence of succinic acid.

Table 6. Effect of temperature at $[S(IV)] = 3 \times 10^{-3} \text{ mol dm}^{-3}$ at $CoO = 0.02 \text{ g dm}^{-3}$ [succinic acid] = $6 \times 10^{-4} \text{ mol dm}^{-3}$ $pH = 7.34$ $Na_2HPO_4 = 8 \times 10^{-2} \text{ mol L}^{-1}$ $KH_2PO_4 = 2 \times 10^{-2} \text{ mol L}^{-1}$.

t °C	33°C	38 °C	43 °C	48°C
$10^3 k_{s,A} s^{-1}$	1.94	2.72	3.56	4.39

Discussion

In aqueous solution SO_2 is present in four forms $SO_2 \cdot H_2O$, HSO_3^- , SO_3^{2-} , $S_2O_3^{2-}$ governed by the following equations



K_H is Henry's constant and K_1 , K_2 are acid dissociation constants. K_3 is the consistent arrangement for $S_2O_3^{2-}$ at $25^\circ C$ the qualities are $K_H = 1.23 \text{ mol}^{-1} \text{ atm}^{-1}$, $K_1 = 1.4 \times 10^{-2}$, $K_2 = 6.24 \times 10^{-8}$, also $K_3 = 7.6$

$\times 10^{-2}$. In this test concentrated in the pH range (7.34 - 9.40), S(IV) would generally be present as SO_3^{2-} . So, in the present study we consider only SO_3^{2-} species to be reactive. A previous study found that oxalate has a strong inhibiting effect on autoxidation of S(IV) in the presence of Fe (III) or (II). Another study also reported that mono-carboxylic acids repress the oxidation, out of which formic acid shows strong inhibiting effect. The probable reason for the inhibition is the interaction between sulfate radicals and carboxylic acid. Organic compound that doesn't form solid complexes have also been show an inhibiting effect on Fe (III) catalyzed oxidation of S(IV) at high pH. It was also found that acetate and formate ions show substantial inhibiting effect at $\text{pH} \geq 5$ but negligible inhibition at $\text{pH} \geq 3$.

In a several transition metal oxide catalyzed heterogeneous aqueous phase autoxidation reactions of S (IV) the development of surficial complexes by adsorption of S(IV) and O_2 on the molecule surface and oxidation of sulfur S(IV) occur through the mediation of different oxidation states has been proposed. in the heterogeneous liquid phase reaction of MnO_2 and S(IV), that the sulfite particle makes bond through oxygen molecule and MnO_2 . In the current investigation, the formation of surficial complex by adsorption of O_2 and SO_3^{2-} on the molecule surface of CoO through the fast steps.

In alkaline medium, the rate of the reaction catalyzed by CoO is exceptionally slowed by the addition of succinic acid, such as that of ethanol, reported by Gupta et al. (2012).



The inhibition is caused by inhibitors such as ethanol and benzene as reported by Gupta et al. (2004) lie the range of 10^3 - 10^5 . In the current study, the inhibition parameter is found to be $1.0 \times 10^3 \text{ mol dm}^{-3}$, which lies in a similar range. This firmly clears the operation of radical mechanism is the most study for the CoO catalyst reaction in the presence of succinic acid. Based on the observed results, including the inhibition by succinic acids, the following radical mechanism is proposed which similar to that proposed by Gupta et al in the ethanol proposed inhibition of the CoO catalyst reaction (Sharma et al., 2019; Sharma et al., 2019; Sharma et al., 2017a; Sharma et al., 2017b; Bhargava et al., 1993).

By assuming long chain hypothesis and steady state approximation $d[\text{SO}_3^{\cdot-}]/dt$, $d[\text{SO}_4^{\cdot-}]/dt$ and $d[\text{SO}_5^{\cdot-}]/dt$ to zero it can be shown that the rate of initiation is equal to the rate of termination. (Eq. 26)

$$k_1[\text{CoO}(\text{SO}_3^{2-})(\text{O}_2)] = \{k_7[\text{X}] + k_8[\text{Succinic acid}]\}[\text{SO}_4^{\cdot-}] \quad (26)$$

Since the reaction is completely stopped in the presence of succinic acid at $1 \times 10^{-3} \text{ mol dm}^{-3}$, So the steps (18) and (22) appear to be unimportant. The contribution of propagation reaction (Eq. 21)

been significant in the CoO catalyzed .Reaction where the autoxidation reaction should have occurred even in the presence of high succinic acid concentration i.e. the reaction is completed seized in the presence of high concentration of succinic acid .This drove us to the rate of reaction given by equation- (27) .

$$-d[S(IV)]/dt = R_{cat} = k_6[SO_4^-][SO_3^{2-}] \quad (27)$$

By substituting the value of (SO_4^-) we get

$$R_{cat} = \frac{k_6 k_1 [CoO \cdot SO_3^{2-} \cdot O_2][SO_3^{2-}]}{k_7[x] + k_8 [S.A.]} \quad (28)$$

From equilibrium Eq. 16 and Eq. 17

$$R_{cat} = \frac{k_6 K_1 K_2 [CoO][S(IV)][O_2]}{\{1+k_1[S(IV)]k_7[x] + k_8 [S.A.]\}} \quad (29)$$

At fixed O_2 , replacing $k_6 K_1 K_2 [O_2]$ by k^1 we get

$$R_{cat} = \frac{k^1 [CoO][S(IV)]}{\{1+k_1[S(IV)]k_7[x] + k_8 [S.A.]\}} \quad (30)$$

Since we observe a first order in $[S(IV)]$, The value of $k_1[S(IV)] \ll 1$ so the above rate law can be reduce to

$$R_{cat} = \frac{k^1 [CoO][S(IV)]}{k_7[x] + k_8 [S.A.]} \quad (31)$$

Gupta et al. (2012) and Sharma et al. (2017) proposed a similar mechanism for the CoO catalyzed autoxidation of sulfur dioxide inhibited by ethanol, which leads to the same rate law. By comparing the derived rate law with the experimental rate law, we observe the similarity in these two. The calculated value of inhibition parameter B is $3.1 \times 10^3 \text{ mol}^{-1} \text{ dm}^3$. Which is in the range of 10^3 to 10^5 . So, on the basis of calculated value of B, we concluded that succinic acid act as a free radical scavenger in the CoO catalyzed autoxidation of aqueous sulfur dioxide in alkaline medium and a free radical mechanism can work in this system.

Conclusion

The following culmination are deduced from the results of succinic acid react as an inhibitor in CoO catalyzed autoxidation of sulfur dioxide in alkaline medium has been get going. Succinic acid are good to inhibit the oxidation of sulfur dioxide, so rain water acidity can be managed. They enlarge the life span of sulfur dioxide so vegetation, civil buildings, monuments, fisher of water bodies and life are not affected by rain water. The ecological effects of acid rain are most understandably seen in aquatic environments, such as streams, marshes and lakes where it can be dangerous to fish and other wildlife. So, the inhibiting effect of succinic acid can be used to reduce rain water acidity. The result is functional for modeling rain water acidity and therefore a considerable use meteorology and climatic chemistry. The value of Inhibition constant in uncatalyzed and CoO catalyzed autoxidation of $S(IV)$ in the present study are $3.6 \times 10^3 \text{ mol}^{-1} \text{ dm}^3$ and $3.1 \times 10^3 \text{ mol}^{-1} \text{ dm}^3$ respectively which one in the range of 10^3 - 10^5 which shows that free radical mechanism is operative in this reaction.

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Author Contributions

AK, HS, AKG and DSNP conceived the concept, wrote and approved the manuscript.

Acknowledgements

Not applicable.

Funding

Not applicable.

Availability of data and materials

Not applicable.

Competing interest

The authors declare no competing interests.

Ethics approval

Not applicable.



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Citation: Kumar A, Sharma H, Gupta AK and Prasad DSN (2024) Kinetics of Succinic Acid Inhibited Uncatalyzed and CoO Catalyzed Atmospheric Autoxidation of S(IV) in Alkaline Medium. *Environmental Science Archives* 3(2): 149-157.