The Inhibitive Action of Aniline on the Autoxidation of Sodium Sulfite in Acidic Medium

Abstract
The kinetics of atmospheric autoxidation of S(IV) by Ag(I) at pH 4.02-5.25 has been studied. The aqueous phase autoxidation of S(IV) is the major factor responsible for acidification of atmospheric aqueous system. The role of Aniline as an inhibitor of Ag(I) catalysed autoxidation of S(IV) in acidic medium has been identified, and based on the observed results following rate law given and a free radical mechanism has been proposed.

\[ \frac{d[S(IV)]}{dt} = \left( k_1 + k_2 [Ag(I)] \right) [S(IV)] + k_3 [Aniline] \]

Experiments were carried out at 30°C ≤ T ≤ 40°C, 4.02 ≤ pH ≤ 5.25, 1 × 10⁻⁴ mol/cm² ≤ [S(IV)] ≤ 1 × 10⁻³ mol/cm², 5 × 10⁻⁴ mol/cm² ≤ [Ag(I)] ≤ 2.5 × 10⁻³ mol/cm², 5 × 10⁻⁴ mol/cm² ≤ [Aniline] ≤ 8 × 10⁻³ mol/cm². Based on the experimental results, rate constants and orders of the reactions were determined. The reaction order in S(IV) were first order for both reactions in the presence and absence of Aniline. The effect of Ag(I) ion and Aniline concentrations as well as an initial pH of the solution on the S(IV) oxidation rate were discussed. It was found that the rate of the S(IV) oxidation depends on the initial pH of the solution but it is independent of the pH change during the reaction. Addition of Aniline leads to the introduction of an induction period and decrease in reaction rate, most likely due to SO₂⁻ radicals. The value of apparent energy and inhibition constant B were calculated in the presence of Aniline found as 26.43 kJ mol⁻¹ and 0.26x10⁻³ mol dm⁻³ respectively.

Keywords: Kinetics; Autoxidation; S(IV); Ag(I); Catalysis; Inhibition; Aniline; Oxidation; Concentrations

Introduction
Although the SO₂⁻ oxidation by O₂ has been the subject of many studies, the mechanism of the reaction is far from settled. Both non radical and radical mechanism have been proposed. The atmospheric oxidation of SO₂⁻ by O₃ in aqueous media has been the subject of numerous studies, and the subject matter of several reviews, monographs and papers, notably by Kuo et al. [1-11]. It is interesting to point out that reaction is strongly inhibited by alcohol, benzene, and other compounds. Pointing to the participation of radical intermediates [12-17]. Bigelow [18] was the first to observe the inhibiting effect of alcohols such as methanol, ethanol, propanol, butanol on the rate of the reaction between sodium sulfite and oxygen. The inhibiting effect of alcohols was investigated subsequently by Alvey et al. [19]. The other Organics studied are phenols [20] organic acids [21-23], benzene [24], toluene, naphthalene, paraffin oil [25], alpha-pinene, cis – verbenol [26], sobrerol [27], myrtenol [28]. The effect of aromatic amines i.e. aniline in atmospheric water on the transition metal-catalysed oxidation of S(IV) is not fully known yet and more work in this area is needed to understand these processes better. The purpose of the present study was to study the kinetics of the Ag(I) catalysed S(IV) oxidation and to determine the inhibiting effect of aniline on this process under different experimental conditions.

Experimental
The experimental procedure was exactly the same as described earlier [29]. All the chemicals used were AR grade and their solutions were prepared in doubly distilled water. The reaction were conducted in 0.15L Erlenmeyer flask, open to air and follow to passage of atmosphere oxygen. The flask was placed in a beaker which had an inlet at a lower part and an outlet at a outer part for circulating thermostatic water for maintaining the desired temperature 30±1°C. The reaction was initiated by adding the desired volume of Na₂SO₃ solution to the reaction mixture containing other additive such as buffer and catalyst. The reaction mixture was stirred continuously and magnetically at 1600±10 rpm to allow the passage of atmospheric oxygen and to save the reaction from becoming oxygen mass transfer controlled. The kinetics was studied in acetate buffered medium in which the pH remained fixed throughout the entire course of reaction. For this purpose 10cm⁻³ buffer made from sodium acetate (0.07mol L⁻¹) and acetic acid (0.03mol L⁻¹) for acidic medium were used (total volume 100cm³) for obtaining the desired pH. The kinetics were followed by withdrawing the aliquot samples periodically and titrating the unreacted S(IV) iodometrically. The reproducibility of replicate measurements was generally better than 10±1 %. All calculations were performed in MS Excel.
Product Analysis

The qualitative test shows sulphate to be only oxidation product. For quantitative analysis, the reaction mixture containing catalyst and S(IV) in appropriate buffered solutions were constantly stirred for a sufficiently long time so as to ensure complete oxidation of S(IV). When the reaction was complete then S(VI) estimated gravimetrically by precipitating sulphate ions as BaSO₄ using standard procedure.

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

\[ S(IV) + 0.5 O_2 \rightarrow S(VI) \]  (1)

Results

Preliminary investigation

The kinetics of both uncatalysed and Ag(I) catalysed and aniline inhibited reaction were studied in acidic medium in pH 4.95 and temperature 30°C. In both the cases the first order dependence of S(IV) was observed in the kinetics data treatment for the determination of first order rate constant \( k \). The plots were shown in Figure 1. It was observed from Figure 1 that both the uncatalysed and Ag(I) catalysed autoxidation of S(IV) reaction are inhibited by aniline.

Uncatalysed reaction

Uncatalysed reaction was studied in the absence of Ag(I) and all the solutions were prepared in doubly distilled water.

Dependence of S(IV)

The detail dependence of the reaction rate on [S(IV)] was studied by varying it in the range 1x10⁻³mol dm⁻³ to 4x10⁻³mol dm⁻³ at pH = 4.95, t = 30°C in acetate buffered medium. The kinetics was found to be first order in [S(IV)] and values of \( k_{1} \) was calculated from log [S(IV)] v/s time plots which was linear. The values of first order rate constant \( k_{1} \), are given in Table 1. The dependence of the reaction rate on [S(IV)] follows the rate law (2).

\[ -d[S(IV)]/dt = k_{1} [S(IV)] \]  (2)

Table 1: The values of \( k_{1} \) for uncatalysed reaction at different [S(IV)], pH = 4.95, t = 30°C, CH₃COONa = 7x10⁻²mol L⁻¹, CH₃COOH = 3x10⁻²mol L⁻¹.

| [S(IV)] mol dm⁻³ | \( k_{1} \) s⁻¹ |
|-----------------|----------------|
| 0.001           | 1.04           |
| 0.002           | 1.06           |
| 0.003           | 1.09           |
| 0.004           | 1.09           |

[Aniline] dependence

The major aim of this study was to examine the effect of aniline on the autoxidation of S(IV) in acetate buffer medium and varying the [aniline] from 5x10⁻⁷mol dm⁻³ to 8x10⁻³mol dm⁻³, we observed the rate of the reaction decreased by increasing [aniline]. The results are given in Table 2. However the nature of the [S(IV)] dependence in presence of aniline did not change and remains first order. The first order rate constant \( k_{inh} \) in the presence of aniline was defined by rate law (3).

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

Table 2: The values of \( k_{inh} \) at different [Aniline], pH = 4.95, t = 30°C, CH₃COONa = 7x10⁻²mol L⁻¹, CH₃COOH = 3x10⁻²mol L⁻¹.

| [Aniline] mol dm⁻³ | \( 10^3 k_{inh} \) s⁻¹ | \( 1/k_{inh} \) s⁻¹ |
|-------------------|------------------------|---------------------|
| 5.0x10⁻⁷          | 0.94                   | 1064                |
| 5.0x10⁻³           | 0.827                  | 1209                |
| 8.0x10⁻⁷           | 0.707                  | 1414                |
| 8.0x10⁻⁵           | 0.513                  | 1949                |
| 1.0x10⁻⁴           | 0.418                  | 2392                |
| 1.0x10⁻³           | 0.36                   | 2778                |
| 5.0x10⁻⁷           | 0.284                  | 3521                |
| 8.0x10⁻⁵           | 0.189                  | 5291                |
| 3.0x10⁻³           | 0.138                  | 7246                |
| 5.0x10⁻⁷           | 0.075                  | 13333               |
| 8.0x10⁻³           | 0.037                  | 27027               |

The values of \( k_{inh} \) in the presence of aniline decreased with increasing [Aniline] are given in Table 2 which are in agreement with the rate law (4).

\[ k_{inh} = k_{1} / \left(1 + B [aniline]\right) \]  (4)
Where $B$ is inhibition parameter for rate inhibition by aniline. The equation (4) on rearrangement becomes

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

In accordance with the equation (5) the plot of $1 / k_{inh} \text{ v/s } [\text{aniline}]$ was found to be linear with non-zero intercept. The values of intercept $(1 / k_1)$ and slope $(B / k_1)$ were found to be $1.53 \times 10^3$ and $2.86 \times 10^6 \text{ mol dm}^{-3} \text{ s}$ at $pH = 4.95, t = 30 \degree C$ from these values the value of inhibition parameter $B$ was found to be $1.86 \times 10^3 \text{ mol dm}^{-3}$.

**Ag(I) catalysed reaction**

At first the kinetics of Ag(I) Catalysed reaction in the absence of inhibitor was studied.

**[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 $10 \times 10^{-3} \text{ mol dm}^{-3}$ at two different but fixed $Ag(I)$ of $5 \times 10^{-6} \text{ mol dm}^{-3}$and $1 \times 10^{-5} \text{ mol dm}^{-3}$ at $pH = 4.95, t = 30 \degree C$. The kinetics was found to be first order in $[S(IV)]$ v/s time were linear as shown in Figure 1.

**Ag(I) variations**

The dependence of Ag(I) on the reaction rate was studied by varying Ag (I) from $5 \times 10^{-6} \text{ mol dm}^{-3}$ to $2.5 \times 10^{-5} \text{ mol dm}^{-3}$ at $S(IV) = 2 \times 10^{-3} \text{ mol dm}^{-3}$ pH= 4.95, $t = 30 \degree C$ in acetate buffer medium. The values of First order rate constant $k_{cat}$ for S(IV) oxidation was determine are shown in Figure 2. The nature of dependence of $k_{cat}$ on Ag (I) was indicated as two term rate law (6).

$$-\frac{d [S(IV)]}{dt} = k_{cat} [S(IV)] = (k_1 + k_2 [Ag(I)]) [S(IV)] \quad (6)$$

Or $$k_{cat} = k_1 + k_2 [Ag(I)] \quad (7)$$

From the plot in Figure 2 the values of intercept is equal to $k_1$ and slope is equal to $k_2$ were found to be $0.72 \times 10^1 \text{ s}$ and $8.6 \times 10^{-3} \text{ mol dm}^{-3} \text{ s}$ respectively at $pH = 4.95, t = 30 \degree C$ in acetate buffered medium.

**Variation of pH**

Variation of pH was carried out from 4.02 - 5.25 at different $[S(IV)], [Ag(I)], [Aniline]$ and temperatures. The rate decreases slightly by varying pH is inverse $H^+$ ion dependence was observed. From the plot of log $k_1$ v/s log $(H^+)$ the order with respect to $H^+$ is 0.16 which is a fractional order and can be neglected as shown in Figure 3 (Table 3 & 4).

**Table 3:** Rate of Ag (I) catalysed autoxidation in the presence of Aniline.

| [Aniline], mol dm$^{-3}$ | Ag (I) =5x10$^{-6}$ mol dm$^{-3}$ | Ag (I) =1x10$^{-5}$mol dm$^{-3}$ | Ag (I) =1.5 x 10$^{-5}$ |
|--------------------------|----------------------------------|---------------------------------|-------------------------|
| pH = 4.02                |                                  |                                 |                         |
| 5.0 x 10$^{-4}$          | 0.470 x 10$^{-3}$                | 0.564 x 10$^{-3}$               | 0.717 x 10$^{-3}$      |
| 8.0 x 10$^{-4}$          | 0.499 x 10$^{-3}$                | 0.590 x 10$^{-3}$               | 0.788 x 10$^{-3}$      |
| 3.0 x 10$^{-3}$          | 0.471 x 10$^{-3}$                | 0.577 x 10$^{-3}$               | 0.942 x 10$^{-3}$      |
| pH = 4.50                |                                  |                                 |                         |
| 5.0 x 10$^{-4}$          | 0.499 x 10$^{-3}$                | 0.590 x 10$^{-3}$               | 0.788 x 10$^{-3}$      |
| 8.0 x 10$^{-4}$          | 0.471 x 10$^{-3}$                | 0.577 x 10$^{-3}$               | 0.942 x 10$^{-3}$      |
| 3.0 x 10$^{-3}$          | 0.402 x 10$^{-3}$                | 0.516 x 10$^{-3}$               | 0.942 x 10$^{-3}$      |
| pH = 5.25                |                                  |                                 |                         |
| 5.0 x 10$^{-4}$          | 0.586 x 10$^{-3}$                | 0.718 x 10$^{-3}$               | 0.942 x 10$^{-3}$      |
| 8.0 x 10$^{-4}$          | 0.577 x 10$^{-3}$                | 0.718 x 10$^{-3}$               | 0.942 x 10$^{-3}$      |
| 3.0 x 10$^{-3}$          | 0.516 x 10$^{-3}$                | 0.718 x 10$^{-3}$               | 0.942 x 10$^{-3}$      |
Table 4: Ratio of rates for Ag (I) catalysed oxidation in the absence and in the presence of Aniline.

| [Aniline] 5.0 x 10^{-4} mol dm^{-3} | Ag (I) = 5 x 10^{-6} mol dm^{-3} | Ag (I) = 1 x 10^{-5} mol dm^{-3} | Ag (I) = 1.5 x 10^{-5} mol dm^{-3} |
|-----------------------------------|-----------------------------------|-----------------------------------|-----------------------------------|
| pH = 4.50                         | 3.02                              | 3.82                              | 3.54                              |

Figure 3: Effect of pH at [S(IV)] = 2 x 10^{-3} mol dm^{-3}, Ag (I) = 5 x 10^{-6} mol dm^{-3}, [Aniline] = 5.0 x 10^{-4} mol dm^{-3}, pH = 4.95, t = 30°C in acetate buffered medium.

**[Aniline] dependence**

To know the effect of aniline on Ag(I) catalysed autoxidation of S(IV) aniline variation was carried out from 5 x 10^{-7} mol dm^{-3} to 3 x 10^{-5} mol dm^{-3} at two different Ag (I) that is 5 x 10^{-6} mol dm^{-3} to 1 x 10^{-5} mol dm^{-3} but fixed S(IV) = 2 x 10^{-3} mol dm^{-3}, pH = 4.95, t = 30°C. The results indicated that by increasing aniline the rate becomes decelerates (Figure 4 & 5).

\[
-d \left[ S(IV) \right] / dt = (k_1 + k_2 \left[ Ag(I) \right] \left[ S(IV) \right]) / (1 + B \left[ Aniline \right])
\]  

Where

\[
k_{inh} = (k_1 + k_2 \left[ Ag(I) \right]) / (1 + B \left[ Aniline \right]) = k_{cat} / (1 + B \left[ Aniline \right])
\]

\[1 / k_{inh} = 1 + B \left[ Aniline \right] / k_{cat}\]  

\[1 / k_{inh} = 1 / k_{cat} + B \left[ Aniline \right] / k_{cat}\]

**Effect of temperature**

The values of \(k_{obs}\) were determined at three different temperatures in the range of 30°C to 40°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 26.43 KJ mol^{-1}.

**Table 5: Effect of temperature k_{obs} saturated suspensions at [S(IV)] = 2 x 10^{-3} mol dm^{-3}, Ag (I) = 5 x 10^{-6} mol dm^{-3}, [Aniline] = 5.0 x 10^{-4} mol dm^{-3}, pH = 4.95.**

| t°C | 10^4 k_{obs} S^{-1} |
|-----|---------------------|
| 30  | 0.686               |
| 35  | 0.781               |
| 40  | 0.915               |

**Discussion**

In aqueous solutions SO_3 is present in four forms SO_2H_2O, HSO_3^{-}, SO_3^{-2}, S_2O_3^{-2} in the experimental range of pH the following equilibrium operates

\[ HSO_3^{-} + H^+ ⇌ SO_3^{-2} \]  

The equilibrium constant is 5.07 x 10^{-6}. In the experimental range of pH both species HSO_3^{-}, SO_3^{-2} are present but former one present predominantly. During the course of reaction fraction order obtain is 0.18 indicates that it is almost independent of pH which is co-relate with the work of Irena [30]. Prasad et al. [31] studied the inhibiting effect of formic acid [31], isopropyl alcohol [32], isooamyl alcohol [33] in the presence of Ag(I) catalysed autoxidation of S(IV) and reported the they all are influence the S(IV) oxidation in atmosphere with moderate rate. Gupta et al. [34] studied the role of some organic organics on the oxidation of dissolved sulfur dioxide by oxygen in rain water medium and found it follows radical mechanism so free radical scavengers such
as VOCs decelerate the S(IV) oxidation and control the rain water acidity [35]. Bigelow et al. [18] studied the effect of alcohols on the reactions between sodium sulphite and O₂, and found that the alcohols inhibited the reaction rate. Alyea et al. [19] studied the inhibiting effect of aromatic alcohols on sodium sulphite in alkaline medium. Gupta et al. [34] studied the inhibition of aniline on the oxidation of S(IV) by oxygen, and found that the oxidation of S(IV) is complete in the presence of higher concentration of aniline. The rate of uncatalyzed and Ag(I) catalyzed reaction is decelerated by the addition of aniline in the present study. Gupta et al. [37] reported that radical mechanism can operate in this system.

By assuming long chain hypothesis and steady state approximation d[S(IV)]/dt, d[S(IV)]/dt, d[S(IV)]/dt, to zero. It can be shown that rate of initialisation is equal to rate of termination (eq. 29).

$$R_{cat} = k_1 \left[ Ag(I) \right] \left[ SO_3^{-2} \right] \left( O_2 \right) / \left[ k_5 \left[ X \right] + k_8 \left[ Aniline \right] \right] \left[ SO_4^{-1} \right]$$ (30)
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Citation: Sharma AK, Parashar P, Prasad DSN, Sharma R (2017) The Inhibitive Action of Aniline on the Autoxidation of Sodium Sulfite in Acidic Medium. J Anal Pharm Res 4(1): 00091. DOI: 10.15406/japlr.2017.04.00091

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