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Mechanistic Investigation of Oxidative Decolorization of an Azo Dye Metanil Yellow by Chloramine - T in Hydrochloric Acid Medium: A Spectrophotometric Approach

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Abstract**: The kinetics of oxidation of Metanil Yellow (MY) by sodium-N-chloro-p-toluenesul fonamide or chloramine-T (CAT) was studied spectrophotometrically in HCl medium at 298K (λ_{max}= 384 nm). The reaction showed first order dependence of rate on both [CAT] and [Dye] and fractional order dependence of rate on [HCl]. Addition of halide ions and variation of ionic strength of the medium shown negligible effect on the rate of reaction. Addition of p-toluenesulfonamide, the reduction product shown negligible effect. Products of oxidation were isolated and identified as nitoso benzene and nitoso diphenyl amine. The impact of temperature on the rate was studied at different temperatures and activation parameters were evaluated. Rate law and plausible mechnism were deduced for the reaction studied.

Keywords: Mechanistic Investigation, Oxidation, Metanil Yellow, CAT, Hydrochloric acid.

I. INTRODUCTION

Metanil yellow (Also known as Acid yellow 36) is a mono azo dye which is highly water soluble in nature. It is widely used as a colouring agent in making of soap, shoe polish, wood stain, paper stain etc [1]. Metanil yellow is not a permitted colourant in food industry. Still it is used extensively as a colourant in sweet meat, ice creams, soft drinks and beverages. Metanil yellow is heavily used coating turmeric in countries like India due to its bright orange - yellow colour. Further, in making water - fast inks, this dye finds high suitability [2]Data on toxicity of Metanil yellow shown that, 13.6% of the orally induced dose of the dye is left out in the gastrointestinal track even after 96 hrs. This may cause decreased mucin secretion from the intestinal mucous cells [3]. Studies also revealed that oral feeding or intratesticular administering of Metanil yellow in animals produces testicular lesions which leads to the damage of somniferous tubules and results in the decreased rate of spermatogenesis [4]. Toxic methaemoglobinaemia and cyanosis [3] are caused by oral consumption of Mentanil yellow [5] in humans, while skin contact results into allergic dermatitis [6]. Metanil Yellow hastumour-producing effects. It can cause intestinal and enzymic disorders in human body [7]. Metanil yellow can also alter the expression of genes [7] but it is not mutagenic in nature. Therefore by keeping various hazardous effects of Metanil yellow on nature and on human health in particular, researchers proposed various methods for the removal of this dye from waste water[8-10]. Few photocatalytic degradation methods have been proposed for the treatment of this dye[11-12]. Photocatlytic membrane reactors[13] have been used to remove this dye from waste water. Pratibha and Meena [14] carried out the degradation of Metanil yellow with methylene blue immobilized resin dowex 11 photocatalyst. H D Revanasiddappa and Sajjad investigated the photocatalytic degradation of Metanil yellow using ZnO particles irradiated by UV light [15]. But these methods are bit costly and produce hazardous byproducts . The literature review reveals that there are no reports on the kinetic studies of declourization of Metanil yellow in aqueus medium by sodium-N-chloro-p-toluenesulphonamide (CAT).The chemistry of aromatic sulfonylhaloamines, generally known as N-haloamines, has created huge interest. This is due to thier ability to act as sources of both haloniumcations and N - anions which act as both bases and nucleophiles. These N - haloamines act as mild oxidants and they interact with a range of functional groups in aqueous, partially aqueous and non- aqueous media in the presence of an acid or alkali[16-21]. Sodium N-chloro-p-toluene sulfonamide, generally known as Chloramine - T (CAT), is a very prominent member of this group of N- haloamines. Chloramine - T is basically a source of positive halogen and thus it has been used as an oxidant for

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variety of substrates in both acidic and alkaline medium. Usually, Chloramine -T undergoes two electron change in its reactions, the products obtained are P- toluene sulfonamide and sodium chloride[22]. This Chloramine -T is a by-product of saccharin manufacture. The N-Cl bond in CAT is strongly polar and thus it is considerably a strong electrophile. The redox potential of Chloramine - T is dependent on pH and it decreases with increase in pH of the medium. The property of the active oxidising species of Chlorami ne -T depends on the reaction conditions and also on the pH of the medium[23]. Along with the above convenient facts, Chloramine - T is easily available, non-toxic, water soluble or water tolerant and easy to handle. Though much of the information is available on mechanistic aspects of many of the reactions of Chloramine - T, literature survey indicates that, only few references are available on the kinetics and the mechanistic studies of oxidation of azo dyes [24-26].In this background, the current investigation was taken up with the purpose of studying decolourization of an azo dye Metanil yellow by using CAT as an oxidant.

II. EXPERIMENTAL

A. Materials and Methods

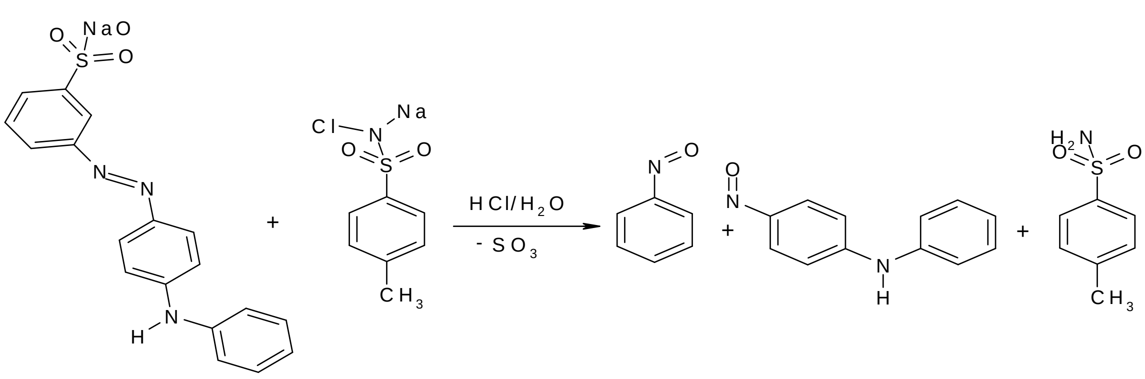
CAT (Merck) was purified by the method given by Morris et al [27]. The stock solution of CAT was freshly prepared in water, standardized iodometrically and stored in brown bottles to prevent photochemical degradation. MY (TCI chemicals) was used as received. Aqueous solution of MY was freshly prepared before the investigation process. All the remaining chemicals used were of analytical grade procured from sd fine chemicals. All the required solutions were prepared by using doubly distilled water.

B. Spectrophotometric Kinetic Procedure

Kinetic runs were carried out using a UV-visible spectrophotometer (Chemito UV - 2100 Spectrophotometer). In the present study, the temperature range was between 298K to 318K in which the kinetic experiments were carried out. With an accuracy of ±0.1K, a constant temperature was maintained using Raaga Ultra Cold chamber with digital temperature control. Kinetic runs of oxidation of MY dye by CAT in HCl medium was investigated under pseudo first-order conditions with excess of $[\text{oxidant}]_0$ over $[\text{MY}]_0$ at constant concentration of HCl at 298K. Reactions were carried out in glass stoppered Pyrex boiling tubes which are coated black on outer surface to rule out any photochemical effect. The required amounts of oxidant, dye, HCl solutions and water (to maintain the total volume constant for all the runs) were taken in separate tubes and thermostated at 298K for 30 minutes. Oxidation of the substrate (dye) was initiated by rapid pippeting out of requisite amount of CAT into the reaction mixture having substrate in the acidic medium. Immediately, 3ml of the reaction mixture was pipetted out into a cuvette placed in spectrophotometer. Absorbance or optical density was measured at 419 nm which is the λ_{max} of the dye MY. The kinetic run was followed upto two half lives. The absorbance readings at t = 0 (D_0) and t = t (D_t) were taken to obtain plots of log (D_0/ D_t) versus time. From the plots pseudo-first-order rate constants(k) were evaluated which were found to be reproducible within ±5%. On af$_x$ -100W calculator, regression analysis of the experimental data was carried out to evaluate the regression coefficient, r.

C. Reaction Stoichiometry

Different ratios of CAT to MY dye in the presence of 2×10^{-2} mol dm^{-3} HCl were equilibrated at 298K for 48 hours. The unreacted CAT in the reaction mixture was found out by iodometric titration. This analysis showed that one mole of MY dye consumed one mole of the oxidant CAT at 298K. The observed reaction stoichiometry is represented in the below scheme.



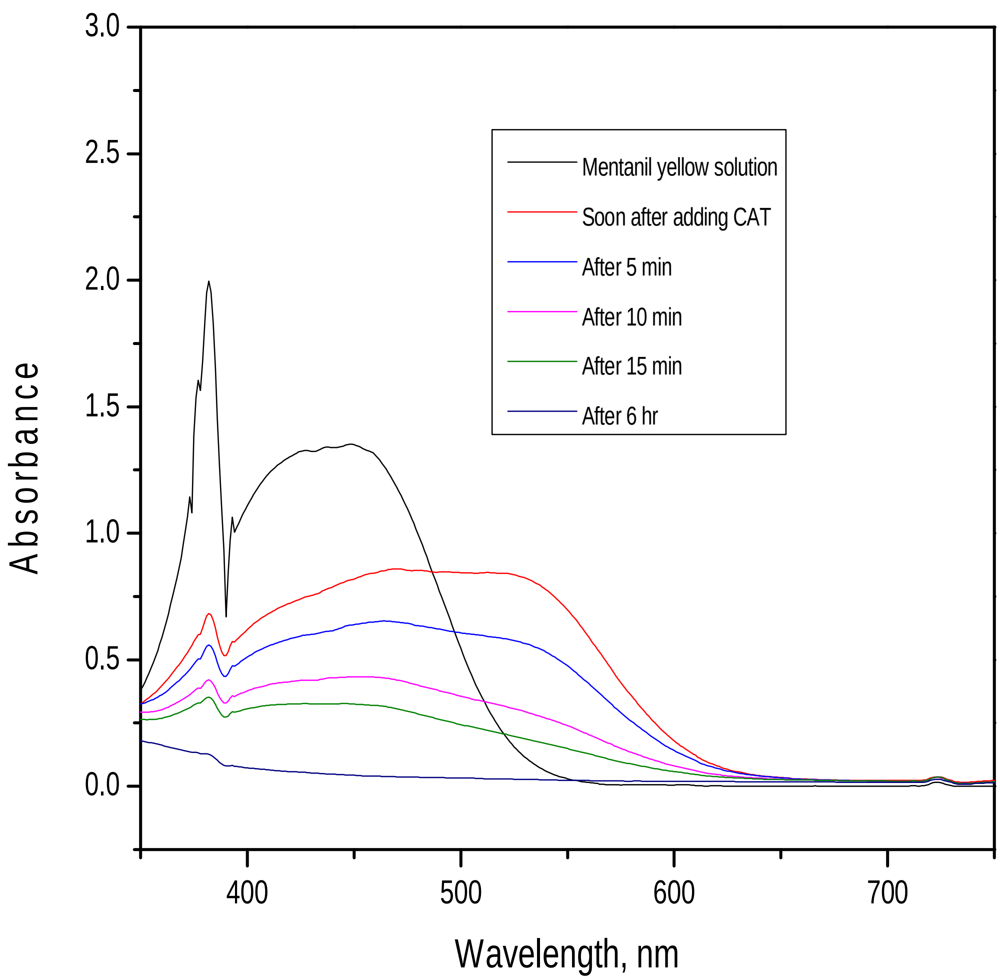
Scheme 1 : Stoichiometric Oxidation of MY Dye by Chloramine-T

D. Product Analysis

The reaction mixture of dye, oxidant and HCl in the stoichiometric ratio was taken and allowed react for 48 hours at 298K. After the reaction was over, the products were neutralized with NaOH and extracted with ether. The completion of the reaction was monitored by thin layer chromatography, which revealed the formation of oxidation products. The products were identified as benzene and biphenyl amine. These two products were confirmed by LC-MS analysis. It was also noticed that, there was no reaction between benzene and biphenyl amine with CAT under the present experimental conditions. The reduction product of CAT, p-toluenesulfonamide (PTS) was detected by paper chromatography confirmed by LC-MS analysis.

III.RESULTS

A UV-visible spectra of the reaction mixture containing known concentarations of dye, oxidant and HCl were taken at different time intervals. The oxidative decolourization of the dye is clearly shown in the spectra given in figure 1. It is apparent from the spectra that 75% decolourization of the dye is completed in 45 minutes and 100% decolourization of the dye is observed in 24 hours.



A. Effect of Varying Concentration of MY Dye on the Reaction Rate

The oxidation of MY by CAT (hereafter abridged as oxidant) was kinetically studied at different concentrations of reactants (MY) under pseudo first-order conditions of $[oxidant]_o >> [MY]_o$, in HCl medium at 298K. Under the conditions of $[oxidant]_o >> [MY]_o$, at constant $[CAT]_0$, HCl and temperature, plots of log (Absorbance) versus time were linear ($r > 0.9965$) indicating first-order dependence of rate on $[MY]_o$. The values of pseudo first-order rate constants ($k^l s^{-1}$) are recorded in Table I which shows they are independent of $[MY]_o$, confirming the first-order dependence on $[MY]_o$.

B. Effect of Varying Concentration of Oxidant on the Reaction Rate.

It was observed that, the rate of the reaction increased with increase in [oxidant]. A plot of log k^1 versus log[oxidant] gave a straight line ($r > 0.9986$) with a slope of unity indicating that the reaction is of first-order with respect to [oxidant]. In addition to this, a plot of k^1 versus log[oxidant] was linear passing through the origin ($r > 0.9817$) confirming the first-order dependence of rate on [oxidant].

C. Effect of Varying HCl Concentration on the Reaction Rate.

It was observed that rate increased with increase in [HCl] as shown in Table 1. A plot of log k^1 versus log [HCl] was linear ($r > 0.9976$) with a slope less than unity clearly shows a fractional-order dependence on [HCl].

Table I. Effect of Varying Concentrations of Oxidant, Substrate and Medium on the Reaction Rate at 298 K.

$10^4\times[\text{oxidant}]_o$ mol dm^{-3}	$10^5\times[\text{MY}]_o$ mol dm^{-3}	$10^2\times[\text{HCl}]$ mol dm^{-3}	$10^5k^1(s^{-1})$
2.0	2.0	2.0	8.67
3.0	2.0	2.0	12.97
4.0	2.0	2.0	20.53
6.0	2.0	2.0	26.6
8.0	2.0	2.0	37.8
4.0	2.0	1.0	14.81
4.0	2.0	2.0	20.53
4.0	2.0	3.0	30.6
4.0	2.0	4.0	41.1
4.0	2.0	5.0	50.5
4.0	1.0	2.0	19.78
4.0	2.0	2.0	20.53
4.0	3.0	2.0	20.98
4.0	4.0	2.0	21.2
4.0	5.0	2.0	20.14

D. Effect of Varying Halide ion Concentration and Ionic Strength on the Reaction Rate

Addition of Cl^- and Br^- ions (from1.0 x 10^{-3} to 8.0 x 10^{-3}mol dm^{-3}) in the form of their sodium salt solutions to the reaction mixture was done. Increase in concentration of halide ions did not show any considerable effect on the reaction rate ruling out their involvement in the rate-determining step.

Ionic strength of the medium was varied from 0.1 to 0.4 moldm^{-3} with $NaClO_4$ solution keeping other experimental conditions constant. It was observed that variation of ionic strength had negligible effect on the reaction rate. This confirms the involvement of non-ionic species in the rate-determining step of the reaction. Hence, no efforts were put to maintain ionic strength of the medium stagnant during the kinetic runs.

E. Effect of Varying PTS Concentration on the Reaction Rate

Addition of reduction product PTS to the reaction mixture (1.0×10^{-4} to 6.0×10^{-4}) did not show any significant effect on the reaction rate as shown in table II. It indicates that, PTS is not involved in the rate-determining step of the reaction.

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Table II. Effect of varying concentration of methanol on the reaction rate at 298K.

$10^4 \times$ [PTS] mol dm^{-3}	10^5 k^1 (s^{-1})	Methanol (%)	Dielectric constant (D)	10^5 k^1 (s^{-1})
1.0	20.68	0	76.73	20.53
2.0	19.2	10	72.37	20.68
3.0	19.7	20	67.48	20.84
4.0	19.2	30	62.71	22.72
5.0	20.1	40	58.06	22.45

[CAT] = 4×10^{-4}mol dm^{-3}, [MY] = 2×10^{-5}mol dm^{-3} and [HCl] = 2×10^{-2}mol dm^{-3}

F. Effect of Varying Dielectric Constant of the Medium on the Reaction Rate

The dielectric constant (D) of the medium was varied by adding MeOH (0-40 % v/v) to the reaction mixture by keeping other experimental conditions constant at 298K. But the reaction rate was not altered significantly as shown in the Table 2. The dielectric constant values of MeOH-H_2O mixtures were taken from the literature[28].

G. Effect of Varying Temperature on the Reaction Rate

The reaction rates were studied at various temperatures (305K to 318K) by keeping other parameters constant. From the Arrhenius plots of log k^1 versus 1/T (r> 0.9988) shown in figure 2, the values of composite activation parameters (Ea, ΔH≠, ΔS≠, ΔG≠ and log A) were computed for the oxidation of MY by CAT. Corresponding data are summarized in Table III

Table III. Effect of Temperature on the Reaction Rate.

Temperature (K)	10^4 k^1(s^{-1})	Activation parameters	
305	3.16	Ea (kJ mol^{-1})	54.859
308	3.89	Log A	44.28
311	4.82	#ΔH (kJ mol^{-1})	52.267
314	6.03	#ΔS (J $K^{-1}mol^{-1}$)	-140.75
318	7.56	#ΔG (kJ mol^{-1})	96.066

[CAT] = 4×10^{-4}mol dm^{-3}, [MY] = 2×10^{-5}mol dm^{-3} and [HCl] = 2×10^{-2}mol dm^{-3}

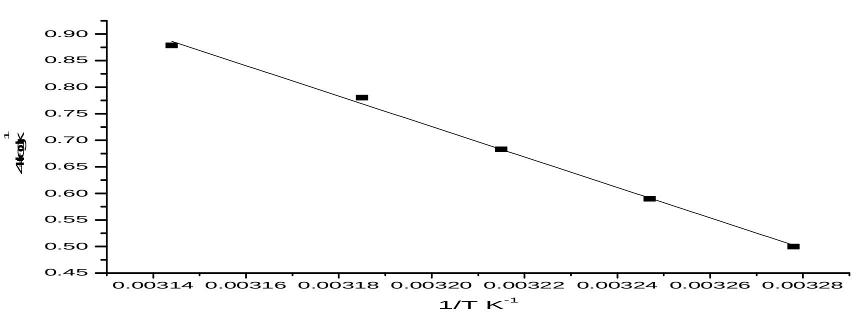

Figure 2: A plot of log k^1 vs 1/T

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IV. DISCUSSION

A. Reactive Species of Chloramine-T

Chloramint-T acts as an oxidising agent in both acidic and alkaline media[22]. It generally undergoes two elctron change forming the reduction product para-toluenesulfonamide ($TsNH_2$) and sodium chloride[23]. The potential of the CAT-PTS redox couple is dependent on pH of the medium. It is 1.138 V, 1.778 V, 0.614 V and 0.5 V at pH 0.65, pH7.0, pH9.7 and pH 12 respectively[27]. Chloramine-T acts as a strong electrolye and produces different reactive species in the solution based on pH of the medium[27]. Based on pH of the medium, Chloramine-T furnishes variety of reactive species as shown in the reaction scheme 2.
(Ts - $CH_3C_6H_5SO_2$, it can also be represented as Ar SO_2)

$$TsNClNa \rightleftharpoons TsNCl^- + Na^+$$

$$TsNCl^- + H^+ \rightleftharpoons TsNHCl$$

$$2TsNHCl \rightleftharpoons TsNH_2 + TsNCl_2$$

$$TsNHCl + H_2O \rightleftharpoons TsNH_2 + HOCl$$

$$TsNCl_2 + H_2O \rightleftharpoons TsNHCl + HOCl$$

$$HOCl \rightleftharpoons H^+ + OCl^-$$

$$HOCl + H^+ \rightleftharpoons H_2OCl^+$$

$$TsNHCl + H^+ \rightleftharpoons TsNH_2Cl^+$$

Scheme 2: Reactive Species of Chloramine-T.

The species responsible for oxidizing nature of chloramine-T is dependent on pH of the medium. In acidic solutions, the species responsible for oxidation of substrate are the conjugate free acid (TsNHCl), dichloramine-T ($TsNCl_2$), hypochlorous acid (HOCl) and possibly H_2OCl^+. Further, formation of the species like $TsNH_2Cl^+$ has been reported.

If $TsNCl_2$and HOCl were to act as reactive species, then the rate would be showing second-order dependence on $[oxidant]_0$ and first-order retardation by the addition of [$TsNH_2$]. But, the experimental evidences are contrary to these expectations.

In the present investigation, the fractional order in [HCl] suggests that, the protonation of TsNHCl results in the formation of $TsNH_2Cl^+$ which is most likely active oxidizing species in the oxidation mechanism of the present reaction.

The first-order dependence of rate on $[oxidant]_0$and a fractional-order dependence of rate on $[HCl]_0$ clearly indicates that $TsNH_2Cl^+$ is the most probable reactive species.

$$TsNHCl + H^+ \overset{K_1}{\rightleftharpoons} TsNH_2Cl^+ \qquad (1)\ \text{fast}$$

$$TsNH_2Cl^+ + Dye \xrightarrow{k_2} X + [TsNH_2] \qquad (2)\ \text{slow and rate limiting step}$$

$$X + H_2O \xrightarrow{k_3} \text{Products} \qquad (3)\ \text{fast}$$

Scheme 3: A general Reaction Scheme for the Oxidation of MY dye by Chloramine-T in HCl Medium.

In the first step, conjugate free acisTsNHCl(one of the species of the Chloramine-T in the given medium) picks up a proton to give cation as shown in equation (1). The resulting cation attacks the sustrate (MY) forming an intermediate complex (X) and para-toluenesulfonamide as shown in equation (2). This is the slow and rate limiting step. In the last step, the complex (X) undergoes hydrolysis forming the oxidized products.

From the rate limiting step of scheme 2, if $[CAT]_t$ is the total effective concentration of CAT, then

$$[CAT]_t = [TsNHCl] + [TsNH_2^+Cl] \quad (4)$$

From equation (1),

$$[TsNHCl] = \frac{[TsNH_2^+Cl]}{K_1[H^+]} \quad (5)$$

Substituting equation (5) in (4), and solving for $[TsNH_2^+Cl]$

$$[TsNH_2^+Cl] = \frac{K_1[H^+][CAT]_t}{1+K_1[H^+]} \quad (6)$$

Also from equation (2),

$$Rate = k_2[Dye][TsNH_2^+Cl] \quad (7)$$

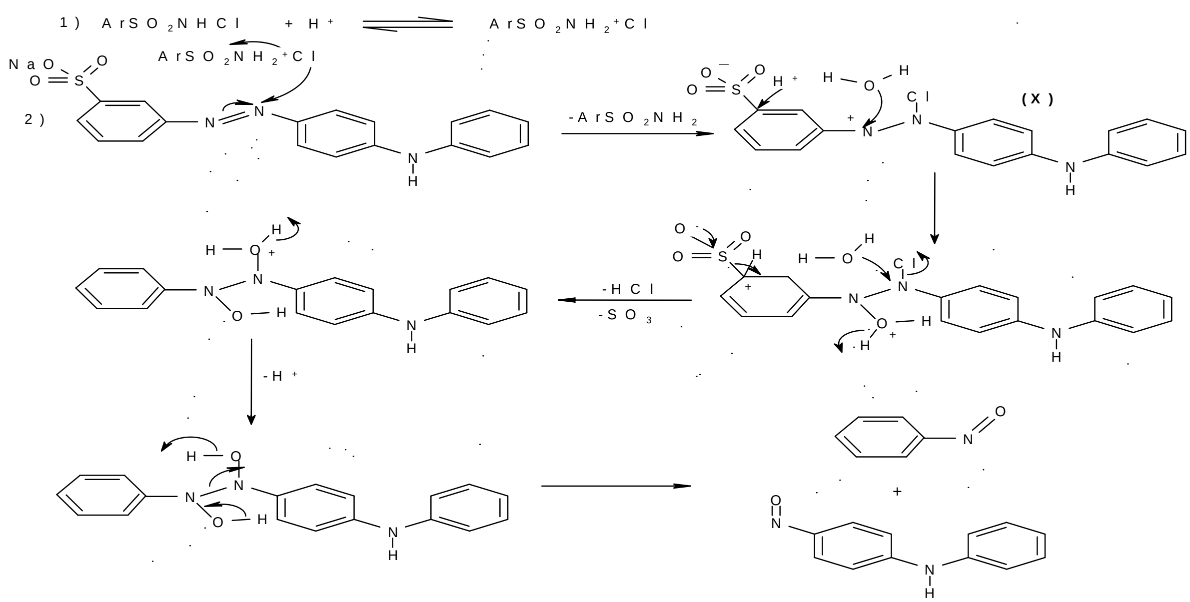
Finally Substituting equation (6) in (7),

$$[Rate] = \frac{K_1k_2[H^+][CAT]_t[Dye]}{1+K_1[H^+]} \quad (8)$$

The derived rate law fits well to the observed kinetic data which reveals a first order dependence of rate on [Dye] and [CAT] and fractional order dependence of rate on $[H^+]$.

B. Reaction Mechanism

Most probable mechanism for the oxidation of MY dye by CAT in the HCl medium is given in the scheme 4. The reactive species of CAT, $TsNH_2Cl^+$ attacks the dye MY forming the intermediate X. It is followed by desulfonation and hydrolysis forming the products nitrosobenzene and nitosobiphenylamine.



Scheme 4 : Plausible mechanism for the oxidation of MY dye by CAT in HCl medium

V. CONCLUSION

Chloramine- T has been proven effective in the oxidation of Metanil yellow dye in the acid medium. Kinetic studies revealed a pseudo first-order dependence of rate on oxidant, dye and fractional-order dependence on HCl. Kinetic rate law in agreement with experimental data is deduced and a plausible reaction mechanism is put forward. Out of these observations, CAT can be used effective oxidant in decolorizing the MY dye. Thus, decolourization thereby removal of the hazardous dye Metanil yellow by using CAT from industrial effluents is advantageous and hence can be used to remove effluent toxicity present in the form of MY dye.

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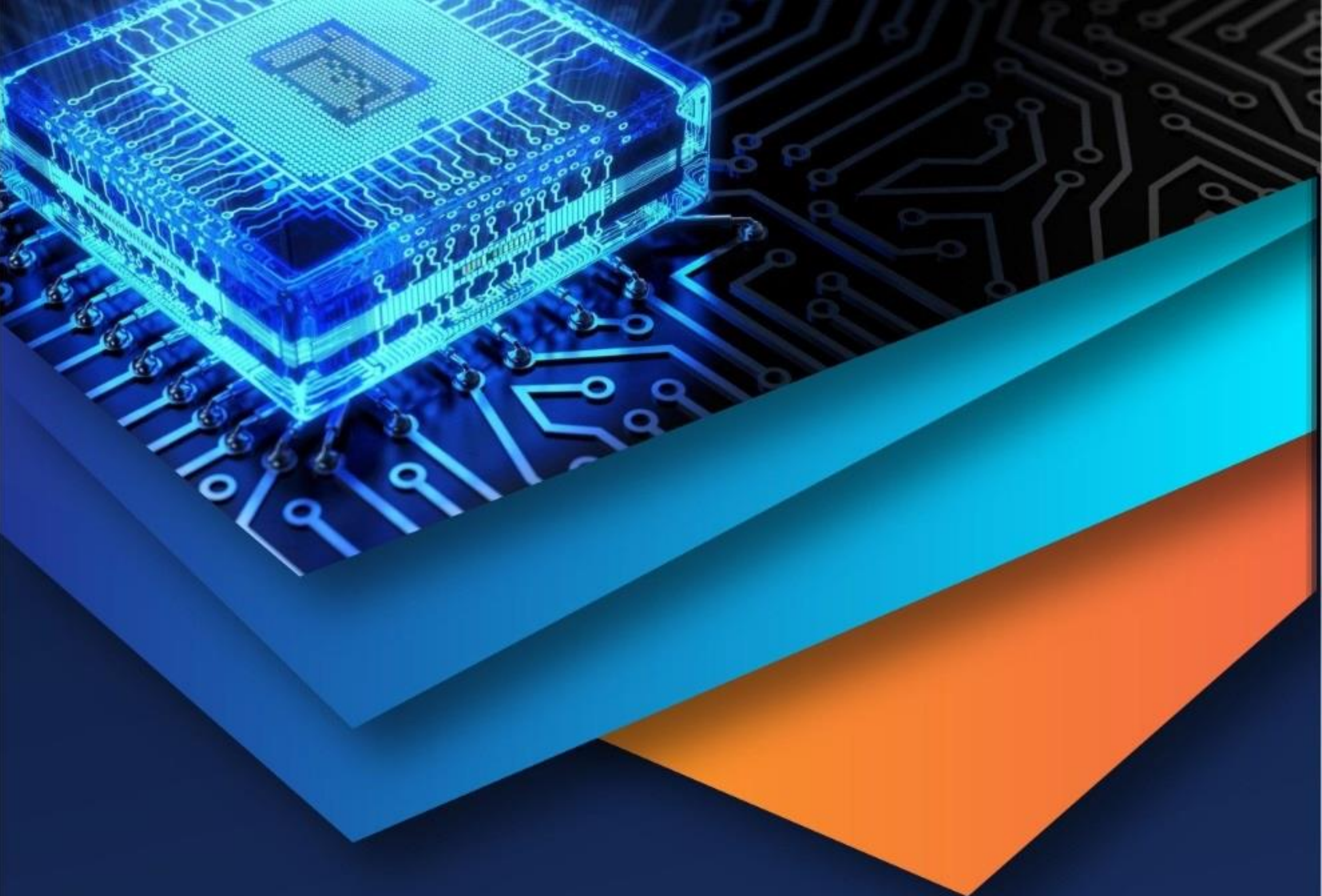

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