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ISSN: 2278-1862

Journal of Applicable Chemistry

2019, 8 (1): 403-411

(International Peer Reviewed Journal)

Kinetics and Mechanism of Oxidation of Indigo Carmine with N-Bromosuccinimide-Effect of CTAB and SDS Micelles

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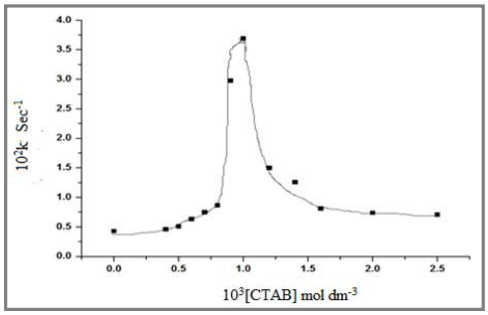
Accepted on 18th December, 2018

ABSTRACT

The kinetics and mechanism of oxidation of Indigo carmine is investigated in acetate buffer both in absence and presence of CTAB and SDS micelles. The kinetic runs are followed under pseudo first order conditions by maintaining [IC] < [NBS]. The reaction obeys first order kinetics with Indigo carmine, NBS and fractional order with [H^+] ion. The rate of the reaction is increased with increase in the [CTAB] and reached maximum. At [CTAB]> cmc the reaction rate is inhibited. The rate [surfactant]-profile shows maximum indicating a typical bimolecular reaction on micellar surface. There is no effect of varying [SDS] on reaction rate.

Graphical Abstract



Keywords: N-Bromosuccinimide (NBS), Indigo Carmine(IC), Sodium dodecyl sulphate (SDS), Cetyl Trimethyl ammonium bromide (CTAB).

INTRODUCTION

N-Bromosuccinimide (NBS) is a versatile analytical reagent in organic reactions. In most of the reactions, NBS acts as oxidizing as well as brominating agent [**1-6**]. It is used as one of analytical reagent because of its sensitivity, accuracy, simplicity and low cost. Surendra *et al*., reported the catalytic nature of NBS in organic transformations [**7**]. NBS has a significant role in the estimation of drugs by its oxidation [**8-15**]. In aqueous solutions the oxidizing properties of NBS were attributed by

the formation of hypobromous acid, an active species which is formed by hydrolysis. The formation of reactive species of NBS depends on the value of pH. The various reactive species of NBS in solutions [**16-18**] are

$$(CH_2CO)_2 N Br + H^+ \rightleftharpoons (CH_2CO)_2 N^+H Br$$

$$(CH_2CO)_2 N Br + H_2O \rightleftharpoons (CH_2CO)_2NH + HOBr$$

$$HOBr + OH^- \rightleftharpoons O Br^- + H_2O$$

$$(CH_2CO)_2 N Br + H^+ \rightleftharpoons (CH_2CO)_2 NH + Br^+$$

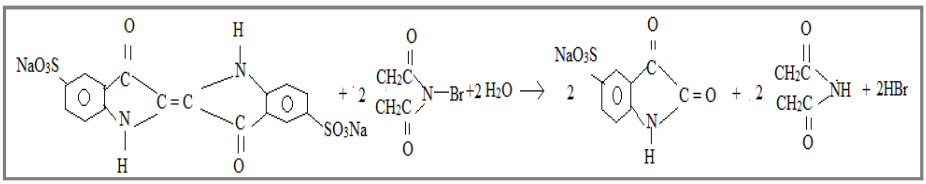
$$Br^+ + H_2O \rightleftharpoons (H_2OBr)^+$$

$$(CH_2CO)_2 N Br + OH^- \rightleftharpoons (CH_2CO)_2NH + Br^-$$

In acidic medium, the probable reactive species of NBS were NBS itself or Br^+ or protonated form of NBS i.e., $(CH_2CO)_2 N^+ HBr$. In basic medium, the reactive species of NBS will be NBS itself or HOBr or OBr^-. Oxidation of organic compounds with NBS in absence and presence of micelles were reported in the recent years [**19-35**]. From the literature survey, the authors have shown interest to investigate the kinetics and mechanism of oxidation of indigo carmine with NBS in presence and absence of cationic and anionic micelles.

MATERIALS AND METHODS

All the chemicals used are AR Grade type. The solutions are prepared in triple distilled water rendered free from ionic salts. A stock solution of Indigo carmine, NBS (E.Merck India Pvt Ltd, Mumbai) of 1.0 x 10^{-3} mol dm^{-3}, strength are prepared and standardized by the method available in the literature[**36, 37**]. SDS (AG, Fluka) is prepared as per the literature and its cmc is determined. CTAB (Sigma Aldrich) was directly used without purification and a stock solution of 1x10^{-1} $moldm^{-3}$ is prepared. The solutions are standardized as per the procedure available in the literature.[**38, 39**].The stoichiometry of the reaction was determined by mole ratio method[40] and it is observed that 1 mole of Indigo carmine reacts with 2 moles of NBS.



The authors identified one of the product as Isatin-5-monosulphonic acid by the formation of blue color precipitate when magnesium nitrate is added to alkaline solution of the product containing 4-Nito phenyl hydrazine and succinimide as reduction product of NBS. After the decolorization of blue color of Indigo Carmine, the resultant solution is distilled with Zn dust so that pyrrole in gas phase is obtained which when allowed to come in contact with p-dimethylaminobenzaldehyde gave blue-violet color as prescribed by Feigl [**41**].

Kinetic Procedure: The reaction is followed spectrophotometrically at known intervals of time using systronics 106 visible spectrophotometer. The kinetics of the reaction is followed at 570 nm in presence of CTAB micelles and 610 nm in SDS micelles till the completion of 90% of the reaction. The kinetic runs were carried out under pseudo first order conditions [NBS] >> [IC].The pseudo first order rate constants were determined from the slopes of the straight lines plots drawn between log(absorbance) versus time. The kinetic runs were carried out in duplicate and rate constants found to agree within ± 5%.

RESULTS AND DISCUSSION

The kinetic results: The linear plots of log (absorbance) and time show that the order with respect to Indigo carmine is one both in absence and presence of CTAB micelles under the condition [NBS] ›› [I.C] (Table 1).

Table 1. Effect of Indigo carmine concentration on rate in absence and presence of CTAB micelle

In aqueous media		In CTAB	
10^5 [IC] moldm^{-3}	10^4 k_1 Sec^{-1}	10^5 [IC] moldm^{-3}	10^4 k_1 sec^{-1}
0.7	6.56	1.654	2.86
1.0	6.49	2.481	2.63
1.5	6.47	3.308	2.41
1.8	6.06	4.135	2.52

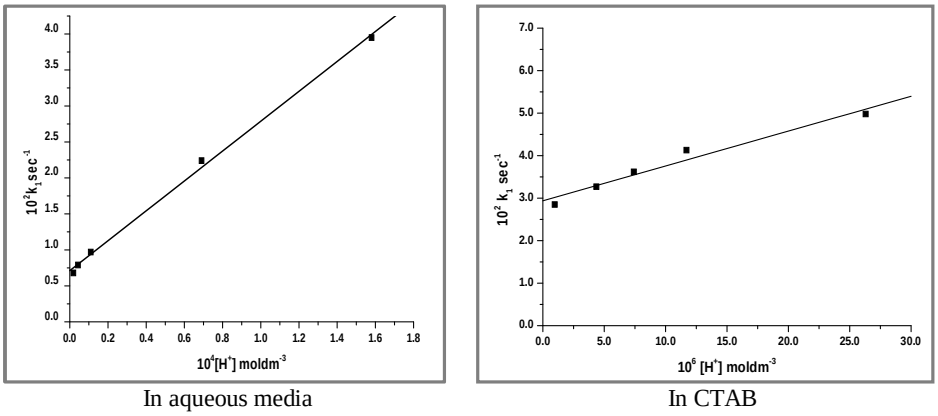
In aqueous media: [NBS] = $1.0x10^{-4}$ mol dm^{-3}, pH = 4.58, Temperature = 30.0°C ± 0.1°C,
In CTAB: [NBS] = $1.34x10^{-4}$ mol dm^{-3}, pH = 5.13, [CTAB] = $1.0 x10^{-3}$ mol dm^{-3}

The reaction exhibits H^+ ion independent and H^+ ion dependent paths in absence and presence of CTAB micelles which is evidenced by the linear plots of k_1 and [NBS] with positive intercept under pseudo first order conditions (Figures 1, Table 2). From this observation it can be explained that protonated and unprotonated form of NBS will be involved in the rate determining stages of the reaction.

Table 2. Effect of pH on rate of reaction in absence and presence of CTAB micelles

In aqueous media		In CTAB	
pH	10^2 $k_1$sec^{-1}	pH	10^2 $k_1$sec^{-1}
3.8	3.95	4.58	4.86
4.16	2.24	4.93	4.13
4.93	0.97	5.13	3.62
5.36	0.79	5.36	3.27
5.71	0.68	6.04	2.78

In aqueous media: [IC] = $4.0x10^{-5}$ mol dm^{-3}, [NBS] = $1.0x10^{-4}$ mol dm^{-3}
In CTAB: [IC]=$4.0x10^{-5}$ mol dm^{-3}, [NBS] = $1.0x10^{-4}$ mol dm^{-3}, [CTAB] = $9.0x10^{-4}$ mol dm^{-3}



In aqueous media In CTAB

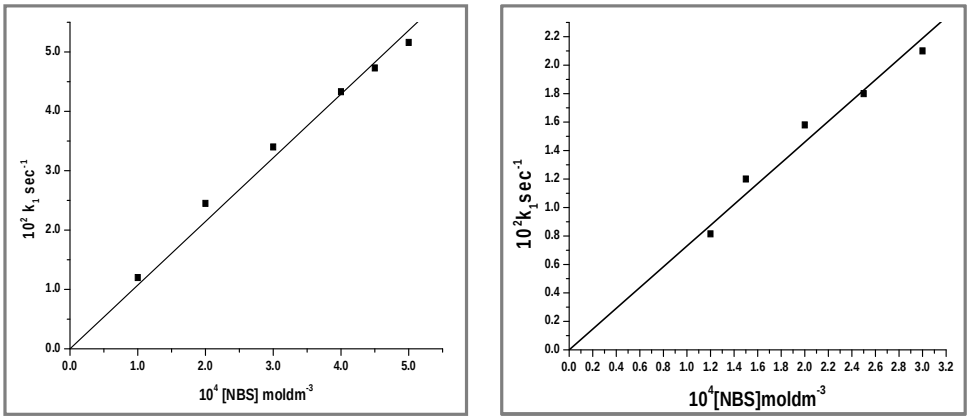
Figure 1. Effect of varying pH on reaction rate in absence and in CTAB micelle

The reaction shows first order kinetics with NBS both in absence and presence of CTAB micelles. Plots drawn between k_1 versus [NBS] are linear passing through origin (Figure 2) (Table 3).

Table 3. Effect of [NBS] on rate of reaction in absence and presence of CTAB micelle

In aqueous media		In CTAB	
10^4 [NBS] moldm^{-3}	$10^2 k_1$ sec^{-1}	10^4 [NBS] moldm^{-3}	$10^2 k_1$ sec^{-1}
1.0	1.20	1.2	0.81
2.0	2.45	1.5	1.20
3.0	3.40	2.0	1.58
4.0	4.33	2.5	1.80
4.5	4.73	3.0	2.10
5.0	5.16	-	-

In aqueous media:[IC] =1.0x10^{-5} mol dm^{-3}, pH =4.58
In CTAB: [IC] = 4.0x10^{-5} mol dm^{-3}, pH = 4.58, [CTAB] = 1.0x10^{-4} mol dm^{-3}



In aqueous media In CTAB

Figure 2. Effect of varying [NBS] on reaction rate in absence and in presence of CTAB micelles.

From the results obtained, the authors proposed the following scheme for the oxidation of Indigo carmine with NBS in absence of micelles.

$$NBS + H^+ \underset{k_1}{\overset{K}{\rightleftharpoons}} NBSH^+ \quad (1)$$

$$NBS + IC \xrightarrow{k_2} Products \quad (2)$$

$$NBSH^+ + IC \xrightarrow{} Products \quad (3)$$

Equations 2 and 3 are the rate determining steps, hence

$$Rate = k_1[NBS][IC] + k_2[NBSH^+][IC] \quad (4)$$

$$K = \frac{[NBSH^+]}{[NBS]\,[H^+]} \quad (5)$$

$$[NBSH^+] = K\,[NBS]\,[H^+] \quad (6)$$

$$Rate = k_1\,[NBS]\,[IC] + k_2K\,[NBS]\,[H^+]\,[IC] \quad (7)$$

$$Rate = [NBS]\,[IC]\,\{k_1 + k_2K\,[H^+]\} \quad (8)$$

Effect of Micelles: The reaction kinetics has been investigated in presence of cetyltrimethyl ammonium bromide (CTAB) and sodium dodecyl sulphate (SDS) at different concentrations of these

surfactants to understand the nature and kinetic features of micellar effects. Under the experimental conditions employed, NBS does not oxidise either of these surfactants with a detectable rate. The reaction obeys first order kinetics with IC, NBS and fractional order kinetics with H^+ ion in CTAB micelles.

SDS Effect: SDS has been found to have no effect on the rate of the reaction as shown by the results presented in table 4.The authors found that the SDS micelles do not have any appreciable interaction with either reactant from the spectrophotometric investigation.

Table 4. Effect of [SDS] on reaction rate

10^3 [SDS] moldm^{-3}	$10^2 k_1$ sec^{-1}
0.0	1.21
1.0	1.22
2.0	1.25
3.0	1.27
4.0	1.27

[IC] = 1.0x10^{-5} moldm^{-3}, [NBS] = 1.0x10^{-4} mol dm^{-3}, pH = 4.58

CTAB Effect: The reaction is appreciably accelerated by CTAB (Table 5, Fig. 3). The plot of k versus C (C = [CTAB] – cmc) exhibiting a maximum typical of bimolecular micellar – catalyzed reactions. The kinetic data can be treated by using Berezin approach [**42-47**]. According to Berezin the increase in concentration of micelles has two opposing effects.

1. The increase in concentration of surfactant increases concentration of micelles leading to greater binding of reactants in micelles and increase in rate. This is predominant at lower surfactant concentration.

2. The increase in the micellar concentration i.e., increase in micellar volume dilute the reactants and produce decrease in rate and this effect is predominant at higher values of C and both these effects together are responsible for the existence of maximum in the rate [Surfactant]-profile. The latter effect predominates at higher surfactant concentration.

Table 5. Effect of varying [CTAB] on rate of reaction

10^3 [CTAB] moldm^{-3}	10^2 k sec^{-1}
0.0	0.43
0.4	0.46
0.5	0.51
0.6	0.63
0.7	0.75
0.8	0.87
0.9	2.98
1.0	3.69
1.2	1.50
1.4	1.26
1.6	0.81
2.0	0.74
2.5	0.71
3.0	0.79

[IC] = 4.0x10^{-5} moldm^{-3}, [NBS] = 1.0x10^{-4} mol dm^{-3}, pH = 5.13

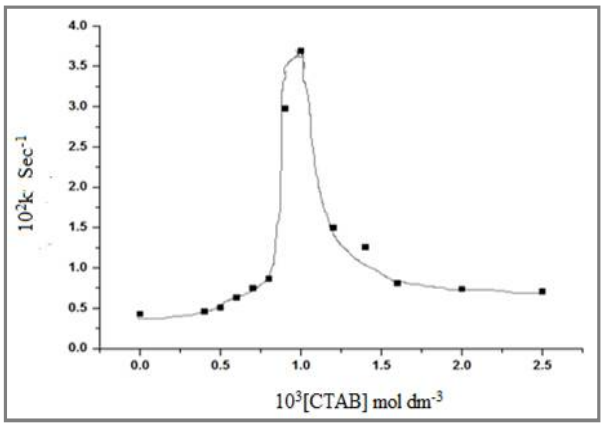


Figure 3. Effect of Varying [CTAB] on reaction rate
(Plot of 10^2k versus 10^3[CTAB]).

The berezin equation can be written as

$$k = \frac{k_w + \bar{k}_m K_A K_B C}{(1 + K_A C)(1+K_B C)} \quad (9)$$

K_A, K_B are the binding constants of NBS and IC respectively, C = [CTAB]-cmc. Since the binding constant of Indigo Carmine is high, k_w can be assumed to be very small in comparison with $\bar{k}_m K_A K_B C$ and $(1+ K_B C) = K_B C$ and hence

$$k = \frac{\bar{k}_m K_A}{1 + K_A C} \quad (10)$$

$$\frac{1}{k} = \frac{1}{\bar{k}_m K_A} + \frac{K_A C}{\bar{k}_m K_A} \quad (11)$$

$$\frac{1}{k} = \frac{1}{\bar{k}_m K_A} + \frac{C}{\bar{k}_m} \quad (12)$$

Plot of 1/kversus C (Fig. 4) must be linear with a positive intercept ($1/\bar{k}_m K_A$) and slope ($1/\bar{k}_m$) and Slope/intercept = K_A (the binding constant of NBS with CTAB).From the observed values of slope and intercept, K_A and $\bar{k}_m$ is found to be 935.0±46.7 dm^{3}mol^{1-} and 1.73x10^{-5} Sec^{-1}. This has been obtained from the experimental data showing the assumptions made by the author are reasonable
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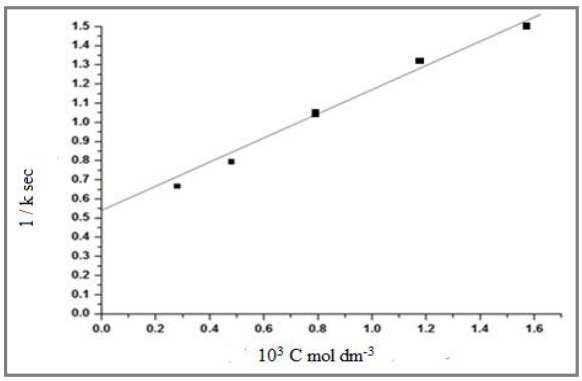

Figure 4. Plot between 1/k sec versus 10^3 C (CTAB Effect).

APPLICATION

The effluent water from textile industries contains unused dyes which cause harm to aquatic life when dispersed in various water bodies. This effluent water may sometimes contaminate the drinking waters causing carcinogenic diseases. To reduce the effects, the textile industries were following certain oxidation methods where the dyes are converted to non-toxic substances. The present work is a methodology where oxidation of dyes is done by selective oxidant and the rate of oxidation is increased by using micelles as catalysts whose concentration is maintained in the multiples of 10^{-3} $moldm^{-3}$. As the concentration of the catalyst employed in the present work is low, it can be followed comfortably for oxidation of dyes.

CONCLUSION

Oxidation of Indigo carmine with N-Bromosuccinimide is done in presence of acetate buffer in absence and in presence of CTAB micelles. The reaction is following first order kinetics with respect to [Indigo carmine], fractional order with respect to [H^+] and first order with respect to [NBS] in absence and in presence of CTAB micelles. The rate of the reaction is enhanced with increase in the [CTAB] reached maximum and at [CTAB] ›cmc, the rate of the reaction is inhibited. The rate [surfactant] profile is showing a maximum which is indicating a typical bimolecular reaction on micellar surface. Berezin kinetic model is followed and the binding constant of NBS with CTAB is determined and is found to be 935.0 ± 46.7 dm^3 mol^{-1}. The rate of reaction is unaffected with change in the [SDS].

ACKNOWLEDGEMENTS

We acknowledge University grants commission, SERO for sanctioning a Minor research project to one of the author Kalyana Chakravarthy for carrying this research work.

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