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**Impact of Cationic surfactant on oxidation of d-Arabinose by
n- Bromophthalimide in the presence of Acidic medium:
A Kinetic Study**

Samta Deshpande^{1*}, Supriya Biswas² and Savita Nayak¹

1, Department of Applied Chemistry, Shri Shankaracharya Institute of Technology & Management
Junwani, Bhilai, (Chhattisgarh) - India

2, Department of Applied Chemistry, Shri Shankaracharya College of Engineering & Technology
Junwani, Bhilai, (Chhattisgarh) - India

Abstract

The impact of cationic surfactant (CPC) on the oxidation of D-Arabinose by N-bromophthalimide in the presence of acidic medium at 308 K has been studied. The order of reaction with respect to [D- Arabinose] has been found to be fractional. First and negative fractional order with respect to [NBP] and [H⁺] respectively. The presence of inorganic salts (KCl, KBr) exhibits a great hike in the reaction rate. Phthalimide has been studied as retarder. Mercuric acetate as scavenger in the temperature range of 298-318 K has been observed. The rate of reaction decreases with an increase in dielectric constant of the medium. The impact of cationic surfactant cetylpyridinium chloride (CPC) has been studied. CPC retards the rate of reaction. Thermodynamic and activation parameters have been calculated. A suitable mechanism in conformity with the kinetic data has been proposed.

Key-Words: N-bromophthalimide, Micellar catalysis, Oxidation, D- Arabinose

Introduction

Carbohydrates are the most important macronutrients as well as main source of energy. The interesting area is to study the chemical, biological and economic importance of carbohydrates with bio-chemical and physiochemical properties and reactivity's. Arabinoses are important for the understanding of carbohydrate metabolism because they undergo a wide variety of reactions and give rise to different products.¹⁻² One of the advantages of arabinose is that they can enter into the oxidation process much more quickly.

A lot of information is available on the oxidative degradation of organic substrate.³⁻⁸ kinetic studies of the oxidation of arabinose by NBP in the presence of micelle forming surfactants have been lacking in this paper, we report a study of the kinetics of the oxidation of D-arabinose by NBP in the absence and presence of cetylpyridinium chloride .

Number of reports on kinetic studies of oxidation of sugars by N halo compounds such as NBS, NBA, NCN,NBB, chloramine-T, Bromamine-T, CBT, NBSA,⁹⁻¹² as an oxidants have been reported. But N-bromophthalimide (NBP) is a potential oxidizing agent¹³ an eco friendly, cheap, non-hazardous and easily synthesized in chemical laboratories, available in the market. which has been extensively used in the estimation of organic substrates.¹⁴ Survey of literature revealed that Nbromophthalimide (NBP) is a source of positive halogen¹⁵⁻¹⁶, and this reagent has been selectively used as an oxidant for a variety of substrates in both acid and alkaline media. surfactants are amphiphilic and dissolve completely in water at very low concentrations, but above a certain level, the critical micelle concentration (CMC), the molecules form globular aggregates, called micelles. The presence of micelles can have marked effects on chemical reactions. Reaction rates can be either accelerated or decelerated, depending on the chemical system, the type and concentration of the surfactant, and other factors, such as pH, ionic strength, etc. Surfactants are widely used and found a large number of applications because of their remarkable ability to influence the property of surface and interfaces.¹⁷⁻²⁰

*** Corresponding Author**

E.mail: samta_chemistry@rediffmail.com

In the years that have passed, micellar solutions have proven to be an extremely versatile topic of research. The catalytic oxidation of sugars has been carried out using such oxidants as transition metal ions, inorganic acids, organometallic complexes, and enzymes.²¹⁻²⁵

The cationic surfactant, cetylpyridinium chloride (CPC) with strong bactericidal and resistance to fungi has a cetyl group attached to the nitrogen atom in the aromatic pyridyl group. It is used as an antibacterial agent in toothpaste, mouthwash and also used in hair conditioner.

The present work seeks to study the kinetics of catalytic oxidation of D-Arabinose by NBP acidic medium. The main aim of the present study is to: (a) elucidate a plausible mechanism, (b) identify the oxidation products, (c) deduce an appropriate rate law, (d) ascertain the reactive species of oxidant i.e. NBP, (e) calculate activation parameters, and (f) effect of CPC micelle on the rate of oxidation.

Material and Methods

The surfactant N-cetylpyridinium chloride (CPC) (AR,SRL India), is used to study the effect of surfactant, N-bromophthalimide (NBP) as oxidant was used as obtained (Sigma-Aldrich, Germany, 99% pure). The melting point of the oxidant was found to be 481 K. Solution of NBP was prepared in 80% distilled acetic acid and stored in a dark colored flask to prevent photochemical deterioration.²⁶⁻²⁷ And standardized iodometrically against the standard solution of sodium thiosulphate (Qualigens). D-Arabinose (Loba Chem, Mumbai, India), potassium iodide (KI), potassium chloride (KCl), potassium bromide (KBr), phthalimide (NHP) (all S.d. fine), mercuric acetate (Hg(OAc)₂) (Loba Chem, Mumbai, India). Perchloric acid (S.d.fine) it maintains the hydrogen ion concentration constant. Distilled glacial acetic acid (s.d.fine) was used as solvent. Freshly prepared starch solution used as an indicator. Doubly distilled deionized water was used for preparation of solution. All the kinetic measurements were taken at constant temperature of 308K.

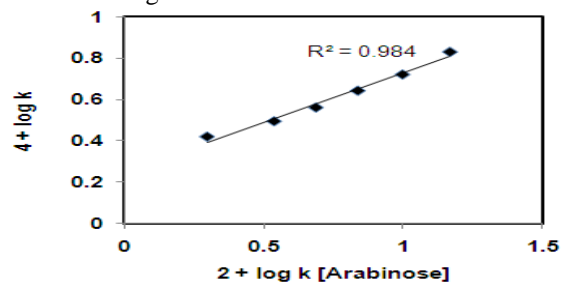
The reactions were carried out in glass-stopper Pyrex vessel whose outer surface was black coated to eliminate photochemical effects. To maintain the desired temperature the reaction mixture was kept in a thermostated water bath. D-Arabinose, CPC, Hg(OAc)₂, HClO₄, CH₃COOH, Hg(OAc)₂ and water were placed in a vessel and thermo-stated at 308 K for thermal equilibrium. A pre-equilibrated amount of the oxidant solution i.e. NBP which was also thermo stated at the same temperature was rapidly added to the reaction. The progress of the reaction was followed by

estimating the amount of unconsumed NBP iodometrically at regular time intervals using freshly prepared starch as indicator. The course of the reaction was studied for two half-lives.

Results and Discussion

Impact of varying - Reactant [D-Arabinose]

Varying concentration of Arabinose from 2.0×10^{-2} to $15 \times 10^{-2} \text{ mol l}^{-1}$ at 308 K. The rate of reaction increases from 2.23×10^{-4} to $6.81 \times 10^{-4} \text{ s}^{-1}$. The plot of $\log k$ versus $\log [\text{Arabinose}]$ has been found linear with a fractional slope (0.984), indicating fractional order with respect to [Arabinose]. The results are shown in Table 1 and Figure 1.



Impact of varying - Oxidant [NBP]

Varying concentration of NBP at 308 K, the k_{obs} values has been determined. The plots of $\log [\text{NBP}]$ versus time have been found to be straight lines, indicating that the order with respect to oxidant has one. The results are shown in Table 1.

Impact of varying - Acid [HClO₄]

Varying concentration of $[\text{H}^+]$ from $1.5 \times 10^{-4} \text{ mol .L}^{-1}$ to $4.0 \times 10^{-4} \text{ mol .L}^{-1}$. The rate of reaction decreases from 5.18×10^{-4} to $3.23 \times 10^{-4} \text{ s}^{-1}$. The plot of $\log k$ versus $\log [\text{H}^+]$ has been found straight line with a negative slope and less than unity indicating that order with respect to $[\text{H}^+]$ ion has negative fractional value. The results are shown in Table 1 and Figure 2.

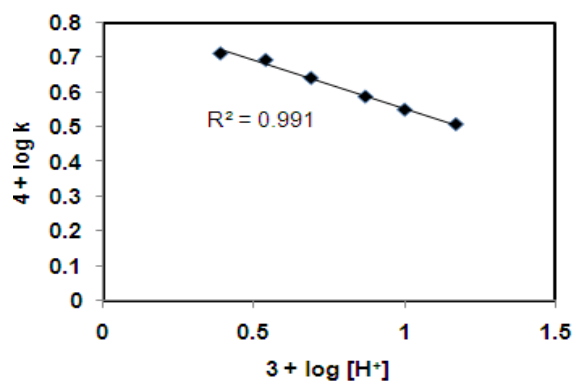


Table 1: Impact of [D-Arabinose], [NBP], [H⁺], [Hg⁺⁺], [CPC] and acetic acid % on the rate of oxidation reactions

10 ² [D-Ara] (mol l ⁻¹)	10 ⁴ [NBP] (mol l ⁻¹)	10 ³ [H ⁺] (mol l ⁻¹)	10 ⁴ [Hg ⁺⁺] (mol l ⁻¹)	[CH ₃ COOH] %	10 ³ [CPC] (mol l ⁻¹)	10 ⁴ k _{obs} s ⁻¹
2.0	1.0	2.0	2.0	50	5.0	2.63
3.5	1.0	2.0	2.0	50	5.0	3.13
5.0	1.0	2.0	2.0	50	5.0	3.65
7.0	1.0	2.0	2.0	50	5.0	4.41
10.0	1.0	2.0	2.0	50	5.0	5.29
15.0	1.0	2.0	2.0	50	5.0	6.81
5.0	1.0	2.0	2.0	50	5.0	4.41
5.0	2.0	2.0	2.0	50	5.0	4.40
5.0	3.0	2.0	2.0	50	5.	4.40
5.0	4.0	2.0	2.0	50	5.0	4.40
5.0	1.0	1.5	2.0	50	5.0	5.18
5.0	1.0	2.0	2.0	50	5.0	4.96
5.0	1.0	2.5	2.0	50	5.0	4.4
5.0	1.0	3.0	2.0	50	5.0	3.88
5.0	1.0	3.5	2.0	50	5.0	3.56
5.0	1.0	4.0	2.0	50	5.0	3.23
5.0	1.0	2.0	2.0	50	5.0	4.2
5.0	1.0	2.0	4.0	50	5.0	4.44
5.0	1.0	2.0	6.0	50	5.0	4.44
5.0	1.0	2.0	8.0	50	5.0	4.42
5.0	1.0	2.0	2.0	30	5.0	6.11
5.0	1.0	2.0	2.0	40	5.0	5.44
5.0	1.0	2.0	2.0	50	5.0	4.44
5.0	1.0	2.0	2.0	60	5.0	3.78
5.0	1.0	2.0	2.0	70	5.0	2.77
5.0	1.0	2.0	2.0	50	2.0	6.93
5.0	1.0	2.0	2.0	50	3.0	6.22
5.0	1.0	2.0	2.0	50	4.0	5.21
5.0	1.0	2.0	2.0	50	5.0	4.44
5.0	1.0	2.0	2.0	50	6.0	3.67
5.0	1.0	2.0	2.0	50	7.0	3.22
5.0	1.0	2.0	2.0	50	8.0	2.55

Temp=308K

Impact of varying - Acetic acid [CH₃COOH]

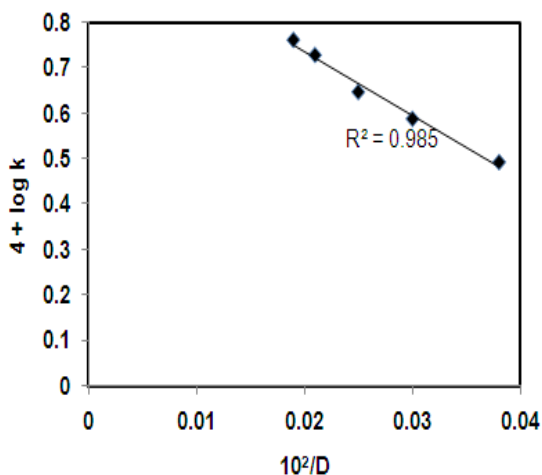
Varying concentration of acetic acid percentage from 30 to 70%. The rate of the reaction decreases from 6.11x10⁻⁴ to 2.77x10⁻⁴ s⁻¹. The rate of oxidation has been found to depend on the dielectric constant (polarity) of the medium. To study the impact of dielectric constant of the medium on the rate of reaction, the micellar catalyzed oxidation of Arabinose by NBP was studied in various compositions of acetic

acid. The data clearly reveal that the rate decreases with an increase in the percentage of acetic acid.

The effect of dielectric constant of the medium on the rate constant of a reaction between two ions has been described by the well-known equation given.²⁸

$$\log k_{\text{obs}} = \log k'_0 - \frac{Z_A Z_B e^2 N}{2.303 (4\pi\epsilon_0) d_{AB} RT} \times \frac{1}{D}$$

where k_{obs} is the rate constant in a medium of infinite dielectric constant, Z_A and Z_B are the charges of reacting ions, d_{AB} refers to the size of activated complex and T is absolute temperature and D is the dielectric constant of the medium. A plot has been made between $4 + \log k$ and $1/D$, which gave a straight line having a slope equal to $\{-Z_A Z_B e^2 N\} / 2.303(4\pi\epsilon_0) d_{AB} RT$. The results are shown in Table 1 and Figure 3.



Impact of varying - Mercuric acetate [Hg(OAc)₂]

Varying concentration of [Hg(OAc)₂] from 2.0×10^{-4} to 8.0×10^{-4} mol·L⁻¹ has been found to be an insignificant effect on the rate of oxidation. An insignificant effect of mercuric acetate

Shows the possibility of its involvement either as a catalyst, co-catalyst or as an oxidant because it does not help the reaction proceed without NBP.²⁹ Thus in view of such kinetic observations, Hg(OAc)₂ acts only as a scavenger for any Br⁻ formed and inhibits the formation of free bromine. According to Bailar (1956), mercuric acetate reacts with Br⁻ and exists as HgBr₂⁴⁻ or unionized HgBr₂.³⁰⁻³¹ Therefore; all the experiments have been carried out in presence of Hg(OAc)₂. The results are shown in Table 1.

Impact of varying - Initially added product [NHP]

The impact of added phthalimide has been studied, which has been found to decrease the rate of oxidation reaction. Thus, the retardation of reaction rate on the addition of phthalimide suggests a pre equilibrium step involving a process in which phthalimide is one of the products.



If this equilibrium is involved in the oxidation process, the rate should be an inverse function of phthalimide

concentration because additions of phthalimide increase the aggregation number, due to that these reduce the catalytic efficiency of surfactants and decrease the number of micelles.

Free radical – Test

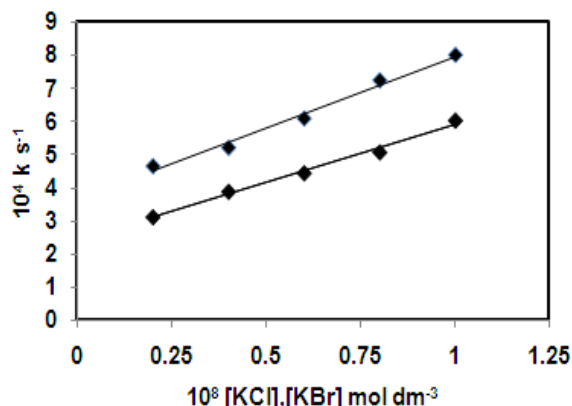
To test the presence of free radicals in the reaction, a known amount of acrylonitrile has been added in a reaction mixture. The reaction neither induces polymerization nor retards the reaction rate which may be attributed to the inertness shown by free radicals.

Impact of varying – Salts

Varying concentration of [Br⁻] and [Cl⁻]. The rate of reaction increases. The salt impact on micellar catalyzed reactions has been rationalized by assuming a competition between the reactant and the counter ion for a “binding site” on or in the micelle. The results are shown in Table 3 and Figure 4.

Table 3: The impact of varying concentration of inorganic salts on the rate of reaction at 308k

10 ⁸ mol.L ⁻¹	[KCl] k.10 s	[KBr] k.10 s
0.2	4.66	3.11
0.4	5.21	3.88
0.6	6.08	4.44
0.8	7.22	5.07
1.0	7.98	6.03



Impact of varying - Temperature and activation parameters

The impact of temperature on rate constant has been studied in the temperature range of 298-318 K. From Arrhenius plots, the value of activation energy (E_a) has been calculated and the values of entropy of activation (ΔS) and free energy of activation (ΔG) has computed from Eyring equation. The values of negative ΔS and positive (ΔH) suggest the formation of more ordered activated complexes and transition state is highly solvated. The value of energy of activation shows that

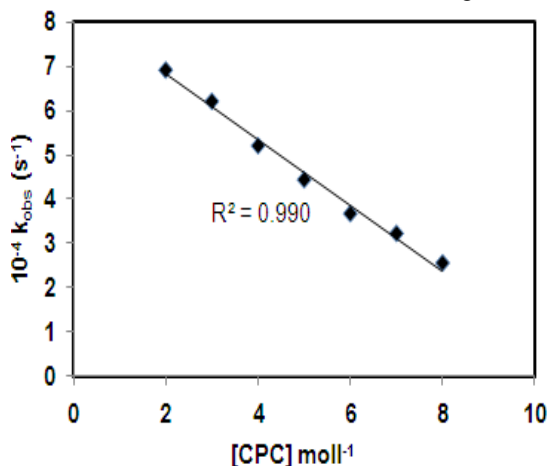
the reaction is slow and enthalpy is controlled. The results are shown in Table 2.

Table 2: Activation parameters for CPC catalyzed reactions of oxidation of D-Arabinose by NBP

Parameters	Without CPC k.10 ⁴ s ⁻¹	With CPC k.10 ⁴ s ⁻¹
298K	4.65	3.23
303K	5.34	4.01
308K	6.11	4.44
313K	6.98	5.21
318K	7.56	6.43
ΔEa (kJ.mol ⁻¹)	22.88 KJ/mol	22.52 KJ/mol
ΔH(kJ.mol ⁻¹)	20.32 KJ/mol	19.96 J/mol
ΔS(Jk.mol ⁻¹)	-56.41 Jk ⁻¹ mol ⁻¹	-57.35 Jk ⁻¹ mol ⁻¹
ΔG(kJ.mol ⁻¹)	37.69 KJ/mol	37.35 KJ/mol
log P _Z	0.671	0.467

Effect of variation of surfactant [CPC]

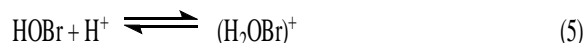
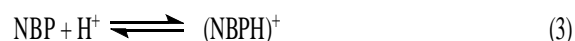
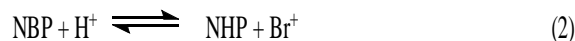
Varying concentration of CPC, a cationic surfactant has been found to retard the rate of reaction. The plot of k_{obs} versus [CPC] indicates that the rate decreases in a continuous fashion and it tends to level off at a higher concentration of CPC. Bunton and Cerichelli noted a similar observation in the oxidation of ferrocene by Fe (III) salts in the presence of cationic cetyl trimethylammonium bromide (CTAB).³² Similar micellar effects have been noted in the oxidation of ethanol and D-sorbitol by pentavalent vanadium ion.³³ The results are shown in Table 1 and Figure 5.



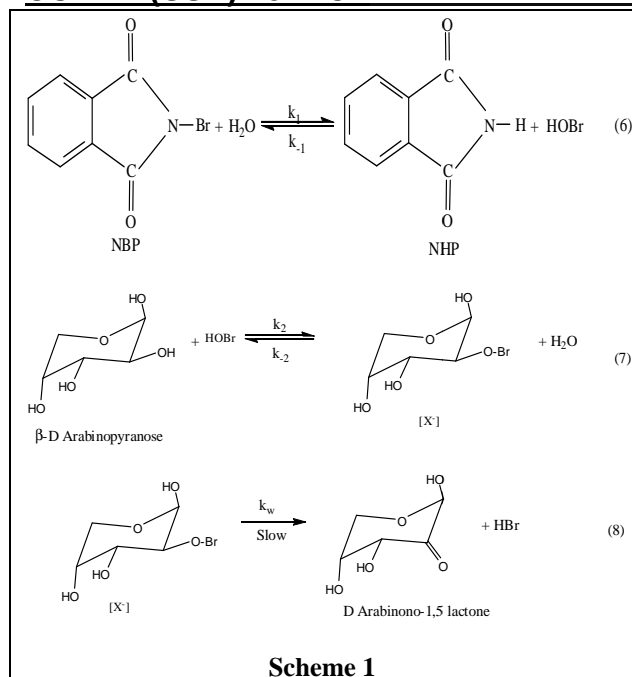
Reaction Mechanism and rate law

On the basis of the above findings the following mechanism may be proposed in order to explain the Kinetics of the oxidation of D-Arabinose. It has been already reported that the oxidation by NBP is good because of large polarity of NBP. NBP, like other

similar *N*-halo imides, may exist in various forms in acidic medium, i.e., free NBP, protonated NBP, Br⁺, HOBr, (H₂OBr)⁺. The monosaccharides are considered as a polyol and the reactivities of -OH groups can be influenced by the presence of the carbonyl group. Aldohexoses exist mainly as pyranoid and furanoid forms, the former being more stable. The pyranoid form mainly exists in a chair conformation. On the contrary, various species of NBP in aqueous media are given in reactions (2-5).



It has observed that addition of phthalimide in the reaction mixture decreases the rate of oxidation in acidic media. This suggests that the pre equilibrium step involves a process in which phthalimide is one of the products. When NBP or (NBPH)⁺ is assumed as the reactive species, the derived rate laws fail to explain the negative effect of phthalimide. Hence neither of these species can be considered as the reactive species. When (H₂OBr)⁺ is taken as the reactive species, the rate law obtained shows first order kinetics with respect to hydrogen ion concentrations, contrary to our observed negative fractional order with respect to HClO₄. Although equilibrium (4) fully explains the negative effect of phthalimide, but the contrary findings of HClO₄ dependence rules out the possibility of cationic bromine (Br⁺) as reactive species. Thus, the only choice left is HOBr, which, when considered as the reactive species of NBP, leads to a rate law capable of explaining all the kinetic findings and other impacts in the light of kinetic observations, HOBr can safely assumed to be the main reactive species of NBP for the present reaction. On the basis of above experimental findings Scheme -1 can be proposed for effect of cationic surfactant on the oxidation of D-Arabinose by *N*- bromophthalimide in the presence of acidic medium.



On the basis of scheme 1, it is evident that equation (8) is the rate determining step. Let [X] be the intermediate formed out of the equation (7) which acts a reactant in equation (8) of scheme 1.

Then rate shall be:

$$\text{rate} = k [X] \quad (9)$$

$$K_1 = \frac{[NHP][HOBr]}{[NBP]} \quad (10)$$

$$K_2 = \frac{[X][H^+]}{[Arabinose][HOBr]} \quad (11)$$

$$\text{rate} = \frac{kK_1K_2[Arabinose][NBP]}{[H^+][NHP]} \quad (12)$$

$$\text{rate} = \frac{kK_1K_2[Arabinose][NBP]_T}{[H^+][NHP] + K_1[H^+] + K_1K_2[Arabinose]} \quad (13)$$

The above rate law is in good agreement with our experimental findings i.e. addition of $[H^+]$ decreases the rate, addition of $[NHP]$ decreases the rate and increasing $[D\text{-Arabinose}]$ increases the rate. This confirms the validity of the proposed mechanism.

Conclusion

During the course of this work following conclusions behind Impact of Cationic surfactant on Oxidation of D-Arabinose by N- Bromophthalimide in the presence of Acidic Medium is as follows - The reactive species of oxidation process by NBP is HOBr not NBP itself. Oxidation products have been identified and activation parameters are evaluated for the catalyzed and uncatalyzed reaction. A plausible mechanism and a related rate law have been given. CPC has been found to retard the rate of reaction. In this paper the entropy of activation for the catalyzed and uncatalyzed reaction was found as $-57.35 \text{ Jk}^{-1}\text{mol}^{-1}$ and $-56.41 \text{ Jk}^{-1}\text{mol}^{-1}$ respectively. The observed negative entropy of activation clearly supports the interaction between the species leading to the formation of an activated complex. In conclusion, it can be said that cationic micelle of CPC is an efficient catalyst for the oxidation of the D-Arabinose by NBP in acidic medium.

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