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RESEARCH ARTICLE OPEN ACCESS

A COMPARATIVE ANALYSIS OF THE KINETIC OXIDATION OF MALTOSE BY N-BROMOSACCHARIN IN AQUEOUS ACETIC ACID WITH AND WITHOUT CTAB

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ABSTRACT

The kinetics of maltose oxidation by N-bromosaccharin (NBSA) was investigated in aqueous acetic acid medium under acidic conditions, with and without cetyltrimethylammonium bromide (CTAB), a cationic surfactant. Additionally, the Piszkiewicz model was utilized to elucidate the micellar effect. The reaction was studied at a constant ionic strength of 0.1 mol dm^{-3} (maintained using sodium perchlorate) and at a temperature range of 313.15-343.15 K. Under pseudo-first-order conditions ((maltose) ≫ (NBSA)), the reaction followed first-order kinetics with respect to (NBSA) and showed a fractional-order dependence on (maltose). Reactions were conducted in 10% v/v, 20% v/v and 30% v/v acetic acid-water mixtures, and the pH was maintained using perchloric acid. The rate increased with rising acetic acid content, indicating enhanced reactivity of protonated oxidant species. In the absence of CTAB, the oxidation proceeded at a moderate rate, while the addition of CTAB ($0–5 \times 10^{-3}$ mol dm^{-3}) led to a significant enhancement, confirming micellar catalysis. A break in the plot of rate constant versus (CTAB) corresponded to the critical micelle concentration (CMC), indicating the transition to micelle-assisted oxidation. Under various thermal conditions the Arrhenius activation energy along with various thermodynamic activation characteristics are evaluated. A mechanistic pathway is proposed involving the electrophilic attack of NBSA on maltose, facilitated by the microenvironment of the CTAB micelles. The study demonstrates that CTAB significantly enhances the oxidation rate by modifying the reaction pathway, which underscores the utility of surfactant systems in tuning the kinetics of carbohydrate oxidation.

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INTRODUCTION

Carbohydrate oxidation reactions play a pivotal role in various biochemicals and industrial processes, including energy metabolism, biosensor development, food chemistry, and pharmaceutical synthesis (1–3). Among disaccharides, maltose, composed of two glucose units linked by an α(1→4) glycosidic bond, serves as a model compound for exploring oxidative transformations due to its structural simplicity and biological relevance. Understanding the kinetics and mechanisms of maltose oxidation can provide insights into enzymatic mimetic and controlled oxidation strategies.

N-Bromosaccharin (NBSA) is a relatively less explored yet effective halogen-based oxidizing agent, structurally derived from saccharin. Compared to conventional oxidants such as N-bromosuccinimide (NBS) or potassium bromate, NBSA offers certain advantages, including better selectivity and stability under aqueous conditions. Its electrophilic bromine center facilitates redox reactions with a variety of organic substrates (4–6), but its use in sugar oxidation—particularly maltose—remains under-investigated.

In recent years, micellar catalysis has emerged as a powerful approach to modulate the rates and mechanisms of chemical reactions in aqueous media. Surfactants such as cetyltrimethylammonium bromide (CTAB), a cationic surfactant, can form micelles that alter the local polarity, substrate orientation, and reactant concentrations, often leading to significant changes in reaction kinetics (7–9). CTAB, in particular, can affect oxidation reactions by concentrating or repelling charged species within the micellar pseudophase, thereby either accelerating or retarding the reaction depending on the system (10). Despite the importance of micellar systems in kinetic studies, there is a notable gap in literature concerning the oxidation of maltose by N-bromosaccharin, both in micellar and non-micellar media. The kinetic oxidation of Maltose by a variety of oxidants has been studied extensively with Potassium Permanganate (11), vanadium pentoxide (12), Copper (II) Ion and Hexacyanoferrate (III) Ion (13), potassium iodate (14), $(H_2OBr)^+$(15), Pd(II) and Hg(II) (16), etc. and so many results were found. In this work, we report a comprehensive kinetic and mechanistic investigation of the oxidation of maltose by N-bromosaccharin in aqueous acidic medium, both in the absence and presence of CTAB. The reactions kinetics were monitored under varying conditions of temperature, oxidant concentration, substrate

concentration, acid strength, and surfactant concentration. The aim is to determine the rate law, evaluate activation parameters, and propose a plausible mechanism consistent with experimental observations. The results are discussed in light of established micellar reaction models and compared with analogous sugar oxidation systems.

MATERIALS AND METHODS

The analysis of the current work is done using analytical-grade chemicals and stock solutions of Maltose, cetyltrimethylammonium bromide (CTAB), Perchloric Acid, acetic Acid, Mercuric Acid, Sodium Perchlorate, Sodium Thiosulphate, Starch, Potassium Dichromate and Potassium iodide were prepared with doubly distilled deionised water.

Kinetic Measurements: The kinetics of the oxidation of maltose by N-bromosaccharin (NBSA) was studied under pseudo-first-order conditions by maintaining a large excess of maltose over NBSA. Kinetic runs were performed under identical conditions in the presence and absence of CTAB to ensure reliable comparison. The reaction was conducted in aqueous acidic medium at a constant ionic strength and at controlled temperatures (±0.1 K), typically in the range of 313.15-343.15 K.

Synthesis of NBSA: N-Bromosachharin (NBSA) was synthesized by the method suggested by Bacchawat and Mathur (17)

$$C_6H_4COSO_2NH \rightarrow C_6H_4COSO_2NBr + HBr \quad \ldots\ldots. (1)$$

Stoichiometry and Product Analysis: One mole of maltose is oxidized by one mole of NBSA, based on the quantitative measurement of the amount of unreacted NBSA after the reaction duration. This result provides strong confirmation that each molecule of maltose conducts a single-electron oxidation process with the aid of one equivalent of NBSA, confirming the 1:1 stoichiometric ratio for maltose given by the equation. We assume that one reducing end of maltose is oxidized, since each maltose molecule has only one available aldehyde group.

$$\underset{\text{(Maltose)}}{C_{12}H_{22}O_{11}} + NBSA \rightarrow \underset{\text{(maltobionic acid)}}{C_{12}H_{22}O_{12}} + saccharin + HBr \ldots.. (2)$$

RESULTS AND DISCUSSION

Under the conditions (Maltose) and (Hg(II)) >> (NBSA), the kinetics of Maltose oxidation by N-bromosaccharin in the presence and absence of CTAB were investigated.

Effect of NBSA Concentration on the Oxidation Rate: The reaction exhibits first-order kinetics with respect to the oxidant, as demonstrated by the linearity of the log (a–x) versus time plots in both the presence and absence of micelles as given in Table 1. Interestingly, on varying the initial concentration of N-bromosaccharin, the pseudo-first-order rate constant decreases with increasing oxidant concentration. This behavior indicates that NBSA likely participates in a pre-equilibrium with a nucleophilic species, possibly water, leading to the formation of a less reactive intermediate. Comparable trends have been observed in the oxidation of benzyl alcohol, α-hydroxy acids, benzhydrols, and primary aliphatic alcohols. (18-24)

Effect of Maltose Concentration on the Reaction Rate: The effect of maltose concentration on the oxidation kinetics was investigated in both the presence and absence of CTAB micelles. In both systems, the reaction exhibited first-order dependence on maltose. (Table 1) A plot of the observed pseudo-first-order rate constant (k) versus (maltose) produced a linear relationship (Figure 1), demonstrating that the reaction rate increases proportionally with the substrate concentration. The deviation from ideal second-order kinetics is evident from the non-constancy of the calculated second-order rate constants over the entire concentration range. At higher maltose concentrations, the kinetic plot deviates from linearity and gradually bends toward the x-axis, indicating a diminishing increase in reaction rate. Wherein the reaction approaches a limiting rate beyond which further increases in substrate concentration do not result in a significant enhancement of the oxidation rate.

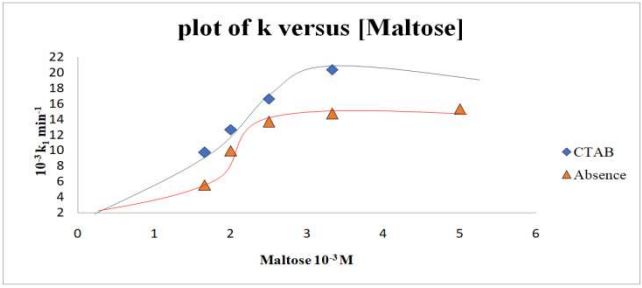

Figure 1. Dependence of rate on Maltose shown in the plot of k versus (Maltose) at 60°C, where (NBSA) = 2.0 x 10^{-3}M; ($Hg(OAc)_2$)= 1.0 x 10^{-2}M; HOAc-H_2O = 20%v/v in the absence and presence of surfactant (CTAB)

Dependence of rate on the concentration of reactants:

(HOAc-H_2O = 20% v/v)

Influence of CTAB Concentration on the Oxidation Kinetics: The effect of CTAB concentration on the rate of oxidation of maltose by NBSA was examined by varying the surfactant concentration while keeping other reaction parameters constant.

Table 1. Dependence of rate constants on (NBSA), (Maltose), ClO_4^-, and ($Hg(OAc)_2$) for the oxidation of Maltose by NBSA at 60^0C

S. No.	(NBSA) 10^{-3} M	(Maltose) 10^{-2} M	($HClO_4$) 10^2 M	$Hg(OAc)_2$ 10^{-2} M	Without Micelles (10^{-3} k_1 min^{-1})	With CTAB (10^{-3} k_1 min^{-1})
1.	2.0	2.0	-	1.0	1.61	12.68
2.	2.50	2.0	-	1.0	5.96	11.17
3.	3.30	2.0	-	1.0	7.89	10.11
4.	5.0	2.0	-	1.0	4.42	5.92
5.	10.0	2.0	-	1.0	3.73	3.19
6.	2.0	1.66	-	1.0	5.58	9.76
7.	2.0	2.0	-	1.0	9.97	12.69
8.	2.0	2.50	-	1.0	13.72	16.67
9.	2.0	3.30	-	1.0	14.74	20.43
10.	2.0	5.0	-	1.0	15.38	22.53
11.	2.0	2.0	0.0	1.0	9.88	17.55
12.	2.0	2.0	0.10	1.0	13.87	21.56
13.	2.0	2.0	0.20	1.0	17.49	24.59
14.	2.0	2.0	0.30	1.0	21.28	29.26
15.	2.0	2.0	0.40	1.0	27.33	32.03
16.	2.0	2.0	-	2.0	1.46	3.31
17.	2.0	2.0	-	2.50	2.89	5.38
18.	2.0	2.0	-	3.30	3.67	7.87
19.	2.0	2.0	-	5.0	4.76	9.74
20.	2.0	2.0	-	10.0	5.53	12.68

Cationic surfactant (CTAB) was employed for the oxidation of Maltose. The reaction rate increased with surfactant concentration at low levels and then remained nearly constant beyond the CMC. (Figure 2) The CMC values and corresponding profiles are presented in Table 2.

Effect of Perchloric Acid Concentration on the Oxidation Rate: The oxidation rate of maltose increases with perchloric acid concentration, confirming the acid-catalyzed nature of the reaction. Under micellar conditions, the reaction remains first-order with respect to NBSA. The linear plot of *k* versus ($HClO_4$) with a positive y-intercept indicates that protonation facilitates the reaction, while the non-zero intercept reveals a minor acid-independent pathway. (Figure 3).

Effect of Solvent Polarity on the Reaction Kinetics: The effect of solvent polarity was examined using acetic acid–water mixtures of varying composition. The pseudo-first-order rate constant decreased with increasing acetic acid content, keeping all other reaction conditions unchanged. A linear plot of log k_1 versus the inverse of the dielectric constant reveals that the reaction rate increases progressively with an increase in the proportion of acetic acid in the reaction medium. This enhancement in rate correlates with a concomitant decrease in the dielectric constant of the medium, indicating that a less polar environment is more conducive to the advancement of the reaction. Moreover, the plot of the reaction rate against the dielectric constant of the medium initially shows a linear relationship followed by a curved deviation towards the x-axis.

Table 2. CMC values of the surfactants Used in Maltose Oxidation by N-Bromosaccharin, measured conductometric method

Determination of Critical Micelle Concentration		
S.no.	**Concentration 10^5 M**	**CTAB 10^3 k_1 min^{-1}**
1.	0.00	0.00
2.	0.30	4.80
3.	0.75	5.14
4.	0.99	6.32
5.	1.50	8.50
6.	1.88	4.80
7.	3.00	5.57
8.	5.00	5.70

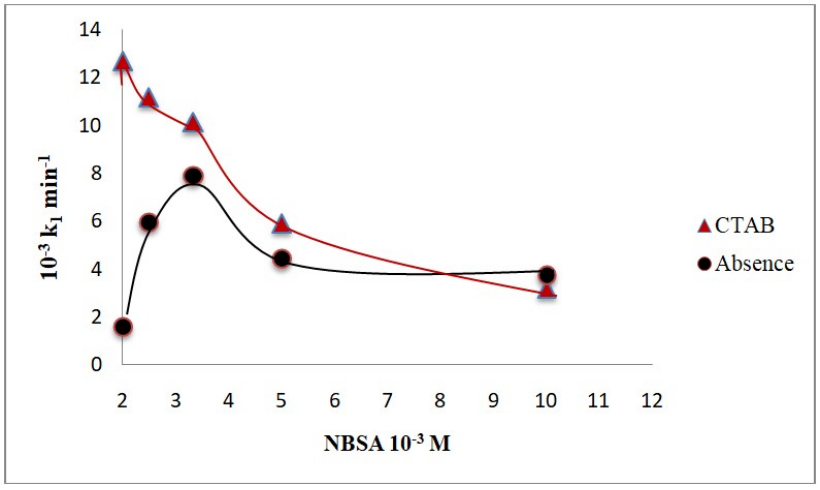

Figure 2. plot of log (NBSA) at 60°C, where (Maltose) = 2.0 x 10^{-3}M; ($Hg(OAc)_2$)= 1.0 x 10^{-2}M; HOAc-H_2O = 20%v/v in the absence and presence of surfactant (CTAB)

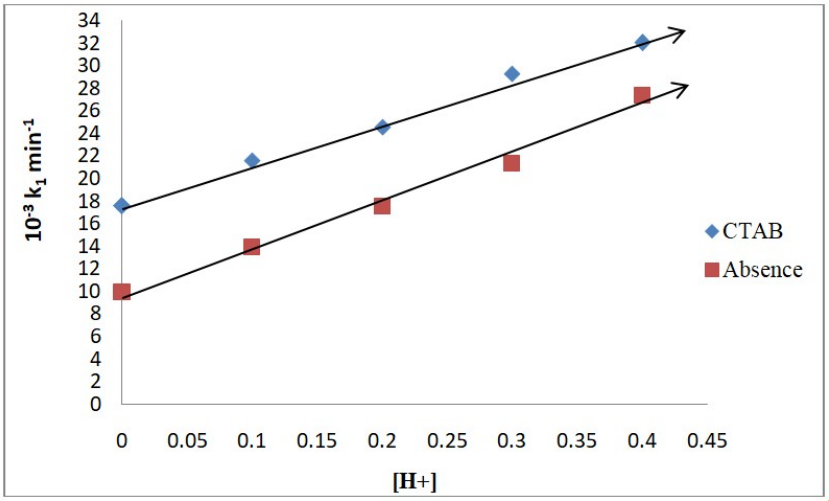

Figure 3. plot of k versus (H^+) at 60°C, where (Maltose) = 2.0 x 10^{-3}M; (NBSA) = 2.0 x 10^{-3}M; ; ($Hg(OAc)_2$)= 1.0 x 10^{-2}M; HOAc-H_2O = 20%v/v in the absence and presence of surfactant (CTAB)

This non-linearity indicates the involvement of dipolar species and positively charged ionic intermediates in the reaction mechanism. This behaviour suggests that the rate-determining step is strongly influenced by changes in solvent polarity, which controls the stabilization of charged intermediates or transition states.

Influence of Mercuric Acetate: Mercuric acetate was used as a bromide ion scavenger to prevent the in-situ formation of molecular bromine, which could otherwise initiate a competitive oxidation pathway. By efficiently removing Br^-, $Hg(OAc)_2$ prevents this side reaction and ensures that oxidation proceeds solely via N-bromosaccharin (NBSA). The pseudo-first-order rate constant increases with increasing $Hg(OAc)_2$ concentration, indicating that preventing Br^- formation enhances the main reaction pathway. At higher $Hg(OAc)_2$ concentrations, the rate constant reaches a limiting value,(Table 1) exhibiting saturation behavior. Therefore, plots of k versus ($Hg(OAc)_2$) are linear at low concentrations and curve towards the x-axis. (Figure 4) At higher concentrations, suggesting that the scavenging effect becomes negligible after all available Br^- has been removed.

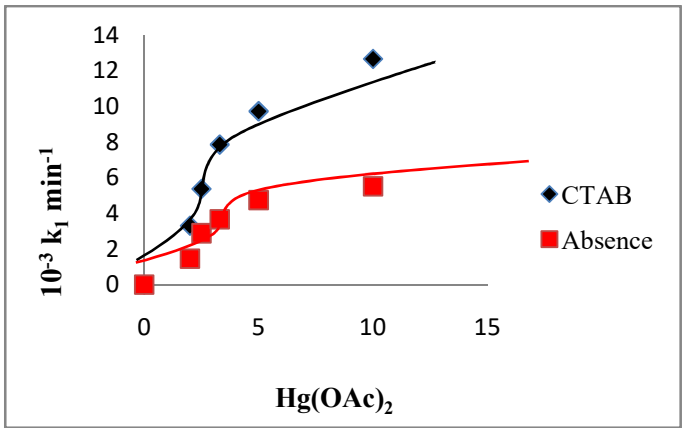

Figure 4.plot of k versus $Hg(OAc)_2$ at 60°C, where (Maltose) = 2.0 x 10^{-3}M; (NBSA) = 2.0 x 10^{-3}M; HOAc-H_2O = 20%v/v in the absence and presence of surfactant (CTAB)

Influence of Saccharin concentration on the Reaction Rate: The effect of saccharin concentration on the reaction rate was systematically investigated. The effect of saccharin on the reaction rate shows a dependence on concentration. At low saccharin concentrations, the rate constant decreases almost linearly with increasing (saccharin), indicating a direct inhibitory effect, possibly due to interaction with a reactive intermediate. At higher concentrations, the rate decrease becomes subtle, and the rate constant reaches a height, suggesting saturation-like behaviour in which additional saccharin has little further effect on the reaction kinetics. This trend suggests that saccharin participates in a pre-equilibrium step that becomes effectively saturated at higher concentrations.

Effect of Neutral Salt on Reaction Rate: The influence of neutral salt on the oxidation rate was studied by varying the concentration of sodium perchlorate ($NaClO_4$). In the presence of micelles, the pseudo-first-order rate constant remains essentially unchanged with increasing sodium perchlorate concentration, indicating that the oxidation rate of maltose is largely unaffected by the salt under micellar conditions**.**

Temperature Dependence of the Oxidation Rate: The reaction was studied at different temperatures (40^0 -70^0 C), and the activation parameters were assessed. (Table 3) Evaluation of the temperature dependence and thermodynamic parameters offers clear insights into the structural and energetic characteristics of the transition state. The larger negative values of the activation entropy (ΔS) observed in the presence of surfactants suggest the formation of a more ordered and structured activated complex during the transition state. Additionally, the relatively high positive values of the activation enthalpy (ΔH) and Gibbs free energy (ΔG) indicate that the transition state is energetically less favorable and requires a substantial input of energy to form. These positive ΔH and ΔG values also reflect extensive solvation of the transition state, consistent with the role of the micellar medium in stabilizing polar or charged transition structures through solvation and micro environmental effects. Collectively, these observations highlight the influence of both temperature and micellar environments on the energetics and structural organization of the transition state.

Table 3. Thermodynamic parameters and Fighting Parameters

Activation Parameters			
S. No.	Parameters	CTAB	Absence
1.	E_a (KJ mol^{-1})	66.76	45.52
2.	A (sec^{-1})	2.93×10^9	8.96×10^7
3.	Δ H (KJ mol^{-1})	63.99	42.75
4.	Δ G (KJ mol^{-1})	104.9	105.03
5.	Δ S (Jk mol^{-1})	-122.8	-186.7
6.	K_M	28.70×10^{-3}	5.42×10^{-3}
7.	K_S	42.27×10^{-3}	19.28×10^{-3}

Reactive species of NBSA

In aqueous medium, NBSA undergoes hydrolysis as presented below:

$$NBSA + H_2O \rightleftharpoons \text{sachharin} + HOBr \quad \text{..... (3)}$$

Reactive species of NBSA in aqueous medium are as follows:

$$NBSA + H_2O \rightleftharpoons \text{sachharin} + HOBr \quad \text{..... (4)}$$

In the presence of mineral acid, the above species get protonated yielding

$$HOBr + H^+ \rightleftharpoons (H_2OBr)^+ \quad \text{..... (5)}$$

$$NBSA + H^+ \rightleftharpoons (NBSAH)^+ \quad \text{..... (6)}$$

$$(NBSAH)^+ + H_2O \rightleftharpoons (H_2OBr)^+ + \text{saccharin} \quad \text{..... (7)}$$

$$NBSA + H^+ \rightleftharpoons \text{sacharrin} + Br^+ \quad \text{..... (8)}$$

The linear relationship observed in the plot of the inverse of k_1 against (saccharin) indicates that HOBr is the active oxidizing species. The fact that the reactions are acid-catalyzed implies the participation of protonated intermediates, such as $H_2O{\cdot}Br^+$ or Br^+. To produce the protonated form ($NBSAH^+$), the hydrolyzed species may go through further protonation in an acidic environment. However, it appears that neither NBSA nor $NBSAH^+$ function as the direct oxidizing agents because of the observed slowdown in the oxidation rate following the addition of free saccharin. This result suggests that there is a pre-equilibrium hydrolysis step that occurs before the actual oxidation event, and that the rate law is insufficient to explain saccharin's inhibitory action. Kinetic properties suggest the active oxidant is electrophilic brominating species like Br^+ or H_2O^+Br, resulting in a rate law that describes observed behavior along with various instances.

Mechanism in the absence of micelles

$$NBSA + H_2O \leftrightharpoons^{K_1} HOBr + \text{saccharin} \quad \text{..... (9)}$$

$$HOBr + H^{\oplus} \rightleftharpoons H_2O^{\oplus}Br \quad \text{..... (10)}$$

$$HOBr + \text{Carb.} \rightleftharpoons C_1 + H_2O \quad \text{..... (11)}$$

$$H_2O^{\oplus}Br + \text{Carb.} \rightleftharpoons C_2{}^{\oplus} + H_2O \quad \text{..... (12)}$$

$$C_1 + H_2O \xrightarrow{K_5} \text{Lactone} + H_3O^{\oplus} + Br^{\ominus} \quad \text{..... (13)}$$

$$C_2{}^{\oplus} + H_2O \xrightarrow{K_6} \text{Lactone} + H_3O^{\oplus} + HBr \quad \text{..... (14)}$$

$$\text{Lactone} + H_2 \xrightarrow{Fast} \text{Maltobionic acid} \quad \text{..... (15)}$$

The overall reaction is:

$$R{-}CHO + C_6H_4COSO_2NBr \rightarrow R{-}COOH + C_6H_4COSO_2NH + HBr + H2O \quad \ldots.. (16)$$

Under the specified experimental conditions, HOBr appears to be the most reactive form of NBSA, with the reaction proceeding such that one mole of maltose is oxidized by one mole of NBSA, as illustrated in the scheme below.

$$k_{obs} = \frac{k_1 k_3 k_5 [\text{Maltose}]\,(H_2O)}{(k_{-3}+k_5)k_{-1}[S]+k_3 k_5 [\text{Maltose}]} \quad \ldots. (17)$$

The kinetic expression presented in Equation (17) is consistent with the experimental data, thereby supporting the proposed reaction mechanism. Moreover, the rate of (NBSA) depletion, corresponding to the consumption of the oxidizing agent, is described by Equation (18), which accounts for both the formation of HOBr via the established pre-equilibrium and its subsequent reaction with the substrate.

$$\text{rate} = k_{obs}[NBSA]_T \quad \ldots.. (18)$$

Oxidation of Maltose under Cationic Micellar condition: Micellar systems can significantly influence reaction rates by modifying the local environment of reactants. The effect of the cationic surfactant CTAB on the oxidation of maltose by NBSA was studied by varying its concentration. The plot of k1k_1k1 versus (CTAB) exhibits a bell-shaped curve with a maximum, indicating that the reaction is micellar-catalyzed and proceeds via a biphasic mechanism involving both aqueous and micellar phases. At low CTAB concentrations, micelles enhance the local concentration of reactants, increasing the reaction rate, whereas higher concentrations lead to micellar saturation, which reduces the rate. The kinetic behavior is well-described by the Piszkiewicz model, which considers the binding of substrate molecules at the micellar interface, highlighting the catalytic role of CTAB in facilitating the oxidation of maltose.

Oxidation of Maltose under non-micellar condition: Under non-micellar condition, the kinetic oxidation of maltose by N-bromosaccharin (NBSA) occurs in a homogeneous aqueous phase. Under these conditions, there is no organized microenvironment to alter the orientation, local concentration, or reactivity of the maltose molecules. Both the substrate and the oxidant are uniformly distributed throughout the solution. Consequently, the reaction rate is primarily determined by the intrinsic molecular characteristics of maltose and NBSA, as well as the bulk solution properties. Oxidation in such an environment clearly depends on the acid concentration, dielectric constant, and additives such as saccharin or $Hg(OAc)_2$. This demonstrates that solution parameters significantly influence the reaction even in homogeneous media. Due to the uniform reaction environment, the system exhibits simple and linear kinetic behavior. Unlike in micellar systems, no rate maxima or biphasic kinetic patterns are observed.

CONCLUSION

The effect of the cationic surfactant CTAB (Cetyle Trimethylammonium bromide) on the oxidation of maltose by N-bromosaccharin (NBSA) was investigated by varying its concentration CTAB micelles enhance the local concentration and orientation of reactants at the micellar interface, resulting in significant rate acceleration. Experimental data shows that, the presence of CTAB significantly accelerates maltose oxidation compared to the surfactant-free system, demonstrating the catalytic effect of the micellar medium.

The oxidations are prompted by acid and proceeded in both acid-dependent and acid-independent ways, revealing that HOBr and H2O+Br are active oxidant species. Additionally, kinetic data supports the production of inter-mediary complex between the substrate species and the active oxidant. The kinetic behavior is described by the Piszkiewicz model, which accounts for substrate binding at the micellar interface.

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