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Kinetics and Mechanism of Maltose Oxidation by N-Bromosaccharin with and without Non-Ionic Surfactant [Brij-35]

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Abstract:
The oxidation of maltose by N-bromosaccharin (NBSA) was examined in acidic aqueous medium at 313-343 K, both in the presence and absence of Brij-35 micelle. At initial concentration of NBSA, the reaction follows first-order kinetics, with or without non-ionic micelle. The reaction displays fractional-order dependence on maltose and slows with increasing $[H^+]$ concentration. The presence of Brij-35 significantly modifies the reaction rate, highlighting the role of micellar media. Kinetic studies suggest intermediate complex formation between the oxidant and Maltose. The reactions are acid-catalyzed, following both acid-dependent and independent pathways. Quantitative analysis was performed using the Menger–Portnoy and Piszkiewicz models, and parameters such as observed rate constants (k_{obs}), binding constants (K_M & K_S), and cooperativity index (n) were determined. Activation parameters were evaluated with and without Brij-35. Effects of medium polarity, addition of saccharin, and Hg (II) acetate were also examined, with micellar catalyzing the oxidation. A reaction mechanism consistent with the kinetics, stoichiometry, and product analysis is proposed.

Keywords: Micelle, Maltose, Oxidation, N-bromosaccharin, Brij-35, stoichiometry

Introduction:

The present work is undertaken to investigate the kinetics and mechanism of oxidation of maltose by *N*-bromosaccharin in aqueous acidic medium, both in the absence and presence of Brij-35 micelle. The effects of oxidant concentration, substrate concentration, acidity, temperature, and surfactant concentration have been systematically examined, and a plausible reaction mechanism has been proposed based on experimental findings.

Because of its essential significance in biological systems, analytical chemistry, and mechanistic organic chemistry, the oxidation of carbohydrates has been well studied. Reducing sugars are good model substrates for researching reaction kinetics and mechanisms because they easily undergo oxidation due to the presence of a free hemiacetal group. Such reactions provide important insights into the role of hydroxyl groups, aldehydic functions, and reaction intermediates in aqueous media, according to early and thorough research on carbohydrate oxidation [1, 2].

N-Halo compounds are widely recognized as mild, selective, and efficient oxidizing agents in kinetic studies. Among these, *N*-bromosaccharin (NBSA) has received considerable attention due to its stability, ease of handling, and predictable behavior in acidic solutions. Several studies have demonstrated that NBSA participates in oxidation reactions through the formation of reactive brominating species such as HOBr or Br^+, depending on the reaction conditions [3–5].

Oxidation reactions of sugars and related substrates by NBSA have been shown to follow well-defined kinetic patterns, making this oxidant particularly suitable for mechanistic investigations [6].

The speed and mechanism of chemical reactions are strongly influenced by the medium in which they occur. In this regard, micellar catalysis is an important area in solution chemistry. When surfactants are present above their critical micelle concentration (CMC), they form organized structures called micelles, which create a unique environment different from ordinary water. These micelles can change reaction rates by gathering reactants together, arranging them in a favorable way, and stabilizing transition states or reaction intermediates [7, 8].

Non-ionic surfactants such as Brij-35 are especially advantageous in kinetic studies because they minimize electrostatic interactions and emphasize hydrophobic and hydrogen-bonding effects. Several investigations have shown that micellar media can either accelerate or retard oxidation reactions depending on the nature of substrate–micelle interactions and the distribution of reactants between the aqueous and micellar pseudo phases [9–12]. However, detailed kinetic studies on carbohydrate oxidation by *N*-halo oxidants in the presence of non-ionic micelles remain relatively limited.

Maltose, a biologically important reducing disaccharide, has not been extensively studied in oxidation reactions involving *N*-bromosaccharin, particularly under micellar conditions. A comparative kinetic analysis in the absence and presence of Brij-35 micelle is therefore expected to provide valuable information regarding the reactive oxidizing species, the locus of reaction, and the role of organized media in modulating reaction pathways.

Experimental:

Maltose solution was prepared by dissolving in glacial acetic acid. The solution of Brij-35 (non-ionic) surfactant was prepared by dissolving its appropriate amount in distilled water. N-Bromosaccharin was prepared by the method suggested by Bacchawat and Mathur [13] and was standardized by iodometric method [14]

Reaction Kinetics:

Using Ostwald's isolation technique, the reaction was carried out at equilibrated temperature (±0.1 °C), and the residual NBSA was estimated iodometrically.

Stoichiometry and product analysis:

The stoichiometry of the reaction was determined by equilibrating a reaction mixture containing an excess of N-bromosaccharin (≈ten times) relative to maltose, while maintaining the concentrations of Brij-35 (when used) and acidic medium as in the kinetic experiments. Stoichiometric studies indicated that 1 mol of maltose reacts with 1 mol of NBSA showing 1:1 stoichiometric ratio both in the presence and absence of Brij-35 micelle. The stoichiometric equation can be represented as:

$C_{12}H_{22}O_{11}+2C_6H_4COSO_2NBr+H_2O \longrightarrow C_{12}H_{20}O_{12} + 2C_6H_4COSO_2NH + 2HBr$ …….. (1)

[Maltose] [NBSA] [Maltbionic Acid] [Saccharin]

Results and Discussion:

The oxidative kinetics of maltose by N-bromosaccharin was investigated in the presence and absence of Brij-35, keeping the concentrations of maltose and surfactant significantly higher than that of NBSA.

Dependency of Rate:

On substrate concentration

The first-order rate constant increases with increasing maltose concentration**. [Table 1]** At elevated maltose concentrations, the kinetic plot departs from linearity and approaches a limiting rate, beyond which additional substrate has little effect on the oxidation rate. The linear double-reciprocal plot of k_1 versus [maltose] with a positive y-intercept **(Figure 1)** provides kinetic evidence for the formation of an intermediate complex between the oxidant and substrate in a pre-equilibrium step.

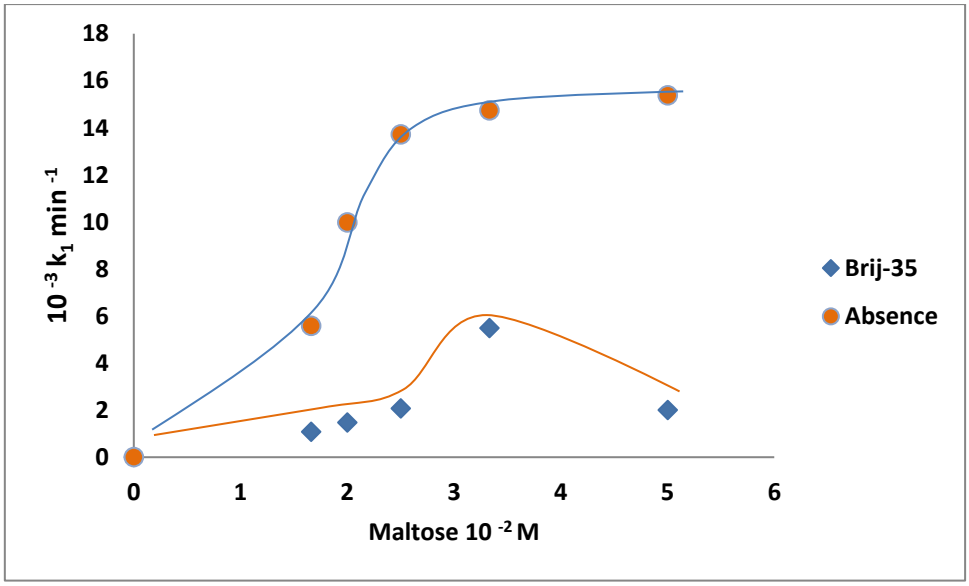


Figure: 1 Dependence of rate on Maltose shown in the plot of k versus [Maltose] at 60°C, where [NBSA] = 2.0 x 10^{-3}M; [$Hg(OAc)_2$]= 1.0 x 10^{-2}M; HOAc-H_2O = 20%v/v in the absence and presence of surfactant (Brij-35)

On Oxidant Concentration

The oxidation of maltose by N-bromosaccharin exhibits first-order dependence on oxidant concentration in both the presence and absence of Brij-35, as indicated by linear log (a–x) versus time plots. **(Figure 2)** However, an increase in initial N-bromosaccharin concentration results in a decrease in the pseudo-first-order rate constant, suggesting the involvement of a pre-equilibrium between N-bromosaccharin and water to form a less reactive species.

Dependence of rate on the concentration of reactants:

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 Brij-35 [10^{-3} k_1 min^{-1}]
1.	2.0	2.0	-	1.0	5.11	1.61

2.	2.50	2.0	-	1.0	5.96	1.29
3.	3.30	2.0	-	1.0	7.89	1.08
4.	5.0	2.0	-	1.0	4.42	0.88
5.	10.0	2.0	-	1.0	3.73	0.57
6.	2.0	1.66	-	1.0	5.58	1.07
7.	2.0	2.0	-	1.0	9.97	1.47
8.	2.0	2.50	-	1.0	13.72	2.07
9.	2.0	3.30	-	1.0	14.74	5.48
10.	2.0	5.0	-	1.0	15.38	2.0
11.	2.0	2.0	0.0	1.0	9.88	6.47
12.	2.0	2.0	0.10	1.0	13.87	10.04
13.	2.0	2.0	0.20	1.0	17.49	14.21
14.	2.0	2.0	0.30	1.0	21.28	17.22
15.	2.0	2.0	0.40	1.0	27.33	24.87
16.	2.0	2.0	-	2.0	1.46	0.45
17.	2.0	2.0	-	2.50	2.89	0.66
18.	2.0	2.0	-	3.30	3.67	0.86
19.	2.0	2.0	-	5.0	4.76	9.74
20.	2.0	2.0	-	10.0	5.53	1.24

[HOAc-H_2O = 20% v/v]

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

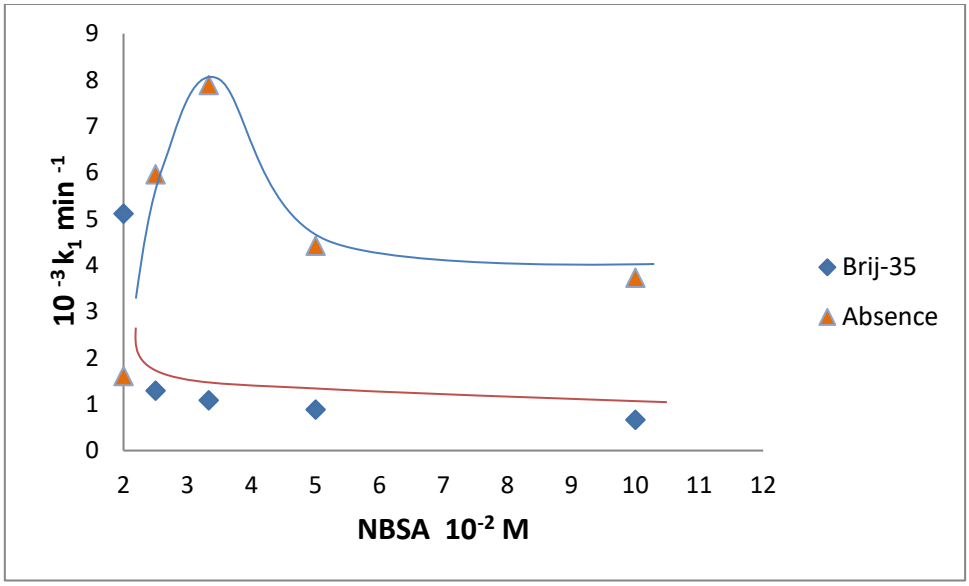


Figure: 2 plot of log [NBSA] at 60^oC, where [Maltose] = 2.0 x 10^{-3}M; [$Hg(OAc)_2$]= 1.0 x 10^{-2}M; HOAc-H_2O = 20%v/v in the absence and presence of surfactant (Brij-35)

On Brij-35 concentration

The rate increases with increasing Brij-35 concentration at lower levels and reaches a maximum near the cmc, followed by a slight decrease or leveling off at higher concentrations. This behavior reflects

micellar catalysis, arising from reactant incorporation into the micellar pseudophase and subsequent saturation effects. This indicates that the oxidation rate increases with rising surfactant concentration below the CMC and becomes nearly constant above the CMC at higher surfactant levels**. [Table 2]** The profile of the plot (Figure 1) indicates micellar catalysis and biphasic behavior, with the substrate Maltose distributed between the micellar and aqueous phases, allowing the reaction to occur in both environments.

On Perchloric acid concentration

In both micellar and non-micellar media, the first-order rate constant increases with increasing perchloric acid concentration, indicating acid catalysis. The fractional slope obtained from the log k_1 versus log [$HClO_4$] plot suggests partial acid dependence. **[Figure 3]** The linear k_1 versus [$HClO_4$] plot with a positive intercept indicates the operation of parallel acid-dependent and acid-independent reaction pathways.

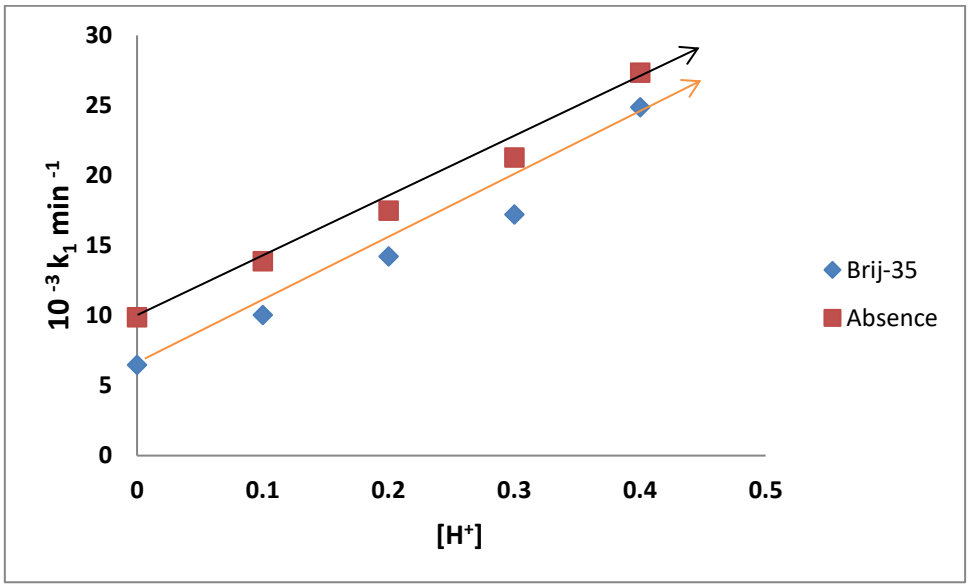


Figure: 3 plot of k versus [H^+] at 60°C, where [Maltose] = 2.0 x 10^{-3}M; [NBSA] = 2.0 x 10^{-3}M; ; [$Hg(OAc)_2$]= 1.0 x 10^{-2}M; HOAc-H_2O = 20%v/v in the absence and presence of surfactant (Brij-35)

On solvent composition

The effect of medium dielectric constant on the Maltose–NBSA reaction was examined using varying acetic acid–water mixtures at 60 °C. Increasing the acetic acid amount lowered the dielectric constant and enhanced the reaction rate, indicating that a lower dielectric medium favors the reaction. The linear relationship between log k_1 and the inverse dielectric constant suggests involvement of ion–dipole interactions or dipole–positive ion interaction in the rate-determining step. A similar behavior was reported by Banerji et al. [15] for the N-bromoacetamide oxidation of primary alcohols in an acetic acid medium. In CTAB-catalyzed oxidation, the decline in the rate constant at higher acetic acid concentrations (above 50%) suggests HOBr (dipolar species) as the more reactive oxidant.

On saccharin concentration

An increase in saccharin concentration leads to a decrease in reaction rate. The linear plot of $1/k_1$ versus [saccharin] indicates that saccharin participates in a pre-equilibrium step, thereby retarding the reaction. The pseudo–first-order rate constant decreases with increasing saccharin concentration in all media. The sigmoid nature of the plots indicates a linear decline in rate at low saccharin levels, followed by a much weaker decrease at higher concentrations, suggesting the attainment of a limiting inhibitory effect.

On [Hg(OAc)$_2$] concentration

Mercuric acetate was used to scavenge Br^-, preventing Br_2 formation and side oxidation, ensuring N-bromosaccharin (NBSA) drives the reaction. The pseudo–first-order rate constant increases with [Hg(Oac)$_2$], indicating enhanced primary pathway, but stabilizes at higher concentrations, reflecting saturation. The k versus [Hg(Oac)$_2$] plot is initially linear but curves at higher levels, showing the limited effect once Br^- is fully scavenged. **[Figure 4]**

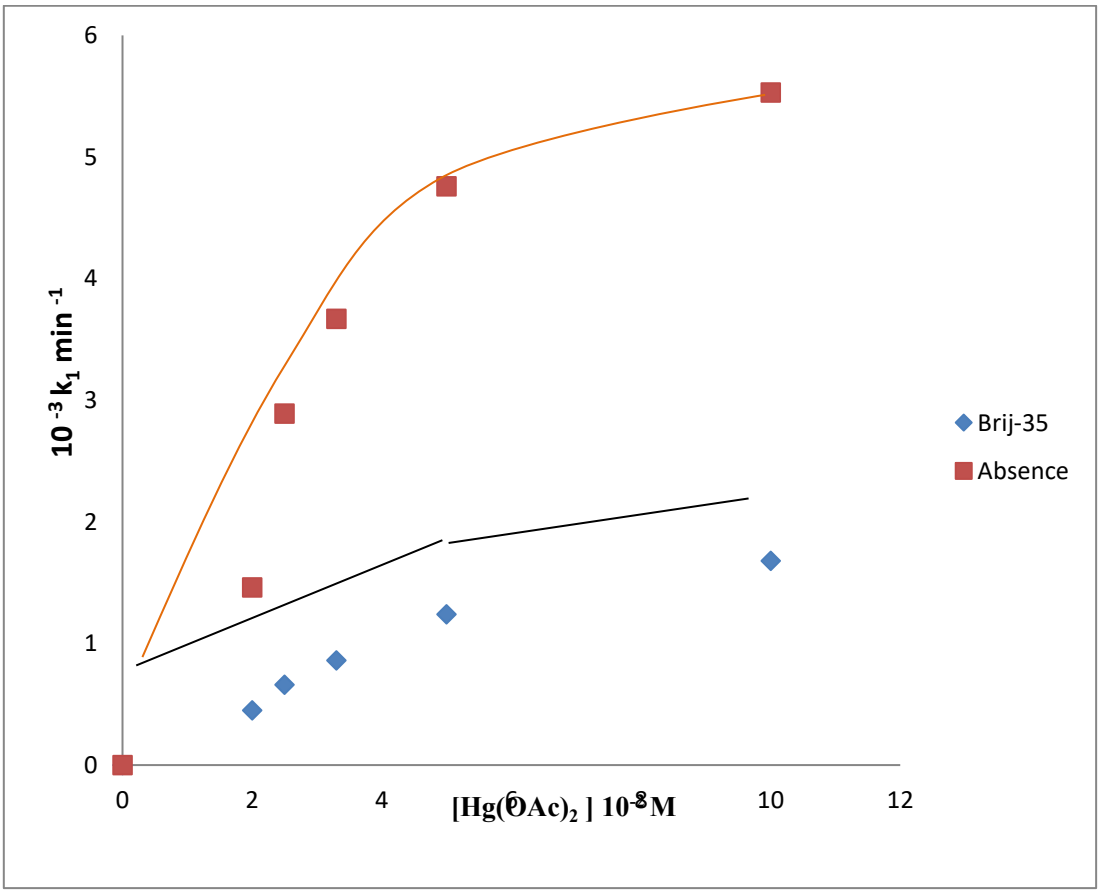
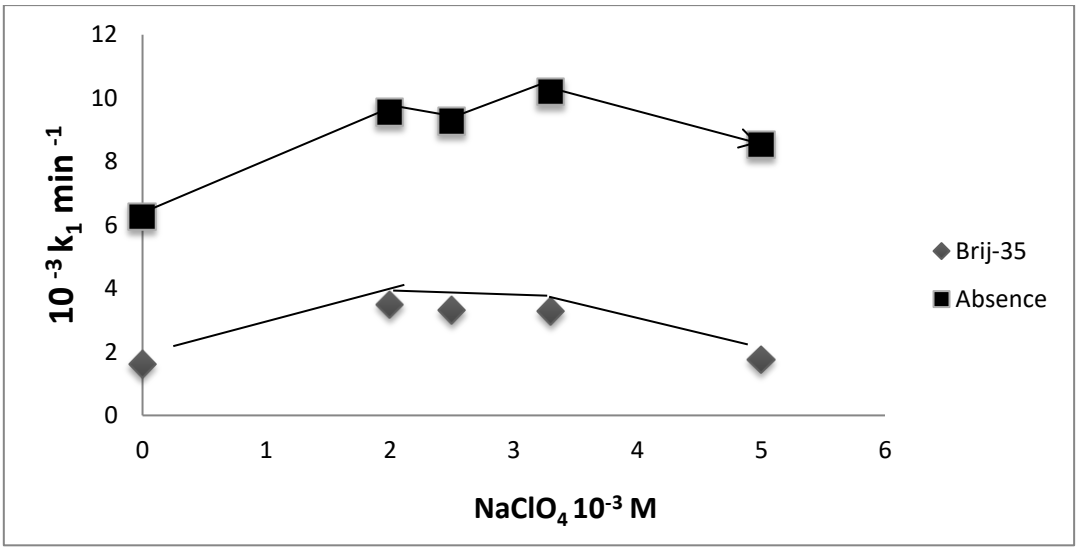


Figure: 4 plot of k versus Hg(OAc)$_2$ at 60°C, where [Maltose] = 2.0 x 10^{-3}M; [NBSA] = 2.0 x 10^{-3}M; HOAc-H$_2$O = 20%v/v in the absence and presence of surfactant (Brij-35)

On added salt

The reaction rate increases with increasing sodium perchlorate concentration, indicating a positive ion–dipole type interaction. Mercuric acetate effectively scavenges bromide ions under the experimental conditions.



Plot of k versus [salt] at 60°C, where [Galactose]=2.0 x 10^{-2}M; [NBSA]= 2.0 x 10^{-3}M; surfactant=1.0 x 10^{-2}M; [$Hg(OAc)_2$]= 1.0 x 10^{-2}M; HOAc-H_2O = 20%v/v in the absence and presence of surfactants [Brij-35]

On activation parameters and temperature

The reaction was studied at different temperatures range between 40–70 °C, and the evaluated activation parameters support the proposed reaction mechanism. More negative activation entropy (ΔS) values in the presence of surfactants indicate a highly ordered transition state, suggesting that the reactants undergo significant structural organization, likely stabilized by the micellar environment. A comparatively larger negative ΔS is observed with the non-ionic surfactant, indicating the formation of a more ordered activated complex. **[Table 3]** The relatively high positive ΔH and ΔG values suggest that the transition state is highly solvated.

Reactive species and micellar reaction pathway

NBSA hydrolyzes to HOBr, which is protonated under acidic conditions to form the active electrophilic species. Both oxidant and maltose partition between aqueous and Brij-35 micellar phases. Oxidation occurs mainly at the micellar interface, where local concentration of reactants enhances the rate, while a parallel aqueous pathway contributes minimally. At higher surfactant concentrations, redistribution of reactants among excess micelles leads to saturation of the rate.

Generation of active oxidant:

NBSA + H_2O ⇌ HOBr + Saccharin

HOBr + H^+ ⇌ $[Br–OH_2]^+$

Phase distribution equilibria:

$[Br–OH_2]^+$_(aq) ⇌ $[Br–OH_2]^+$_(micellar)

Maltose_(aq) ⇌ Maltose_(micellar)

Rate-controlling step (micellar domain):

$[Br–OH_2]^+$_(micellar) + Maltose_(micellar) → Maltbionic Acid + Br^-

Competing aqueous pathway:

$[Br–OH_2]^+$_ (aq) + Maltose_ (aq) → products

Bromide removal:

$Br^- + Hg(OAc)_2 \rightarrow HgBr_2$

Mechanistic rationale

The overall kinetics represents the combined contributions of reactions occurring in the aqueous phase and within the Brij-35 micellar pseudophase. The micellar medium modulates the rate by selectively concentrating the substrate and the electrophilic oxidant at the interface, while excess surfactant leads to dilution of reactants and diminished catalytic efficiency.

$NBSA + H_2O \rightleftharpoons HOBr + Sac$ (K_1, pre-equilibrium) ……. (2)

$HOBr + H^+ \rightleftharpoons H_2OBr^+$ (K_2, protonation equilibrium) …….. (3)

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

$HOBr + H+ \rightleftharpoons (H2OBr)^+$ ….. (4)

$NBSA + H+ \rightleftharpoons (NBSAH)^+$ ….. (5)

$(NBSAH)^+ + H2O \rightleftharpoons (H_2OBr)^+ +$ saccharin ….. (6)

$NBSA + H+ \rightleftharpoons$ sacharrin + Br+….. (7)

Acidic conditions facilitate the protonation of the hydrolyzed NBSA, resulting in the formation of $NBSAH^+$. However, the observed decrease in the oxidation rate upon the addition of free saccharin indicates that neither NBSA nor $NBSAH^+$ serves as the direct oxidizing agent. This observation supports the presence of a pre-equilibrium hydrolysis step that precedes the actual oxidation reaction. Moreover, the standard rate law is insufficient to account for the inhibitory effect of saccharin, highlighting the involvement of additional reactive species.

The kinetic evidence indicates that the actual oxidizing agent is an electrophilic bromine species, such as Br^+ or the hydrated bromonium ion (H_2OBr^+), generated during the reaction and mediating the oxidation. The involvement of these electrophilic intermediates accounts for the observed reaction kinetics, including the inhibitory effect of saccharin providing a coherent mechanistic explanation.

Mechanism in the absence of micelles

$NBSA + H_2O \overset{K_1}{\leftrightharpoons} HOBr +$saccharin …….. (8)

$HOBr + H^{\oplus} \rightleftharpoons H_2O^{\oplus}Br$ ….. (9)

$HOBr + Carb. \rightleftharpoons C_1 + H_2O$ ….. (10)

$H_2O^{\oplus}Br + Carb. \rightleftharpoons C_2{}^{\oplus} + H_2O$ ….. (11)

$C_1 + H_2O \overset{K_5}{\rightarrow} Lactone + H_3O^{\oplus} + Br^{\ominus}$ ….. (12)

$C_2{}^{\oplus} + H_2O \overset{K_6}{\rightarrow} Lactone + H_3O^{\oplus} + HBr$ ….. (13)

$Lactone + H_2 \overset{Fast}{\longrightarrow} Maltobionic\ acid$ ….. (14)

Under the experimental conditions used, HOBr is identified as the primary reactive species of NBSA, with the oxidation proceeding in a 1:1 stoichiometry—one mole of Maltose reacts with one mole of NBSA, as shown in the scheme below:

$$k_{obs} = \frac{k_1 k_3 k_5 [Maltose]\,[H_2O]}{(k_{-3}+k_5)k_{-1}[S]+k_3 k_5 [Maltose]} \text{ …. (15)}$$

The kinetic expression in Equation (15) agrees with the experimental data, supporting the proposed mechanism. Equation (16) describes the consumption of [NBSA], accounting for HOBr formation and its subsequent reaction with the substrate.

Rate = k_{obs} $[NBSA]_T$ ….. (16)

Oxidation of Maltose in presence of non-ionic micelle (Brij-35):

The oxidation of Maltose was performed using non-ionic (Brij-35) surfactant. The results indicate that the reaction rate increases with surfactant concentration below the CMC, but becomes nearly constant above it. The CMC profiles are presented in Figure 1 and summarized in Table 2. Micellar systems can either accelerate or inhibit chemical reactions.

Determination of Critical Micelle Concentration		
S.no.	**Concentration 10^5 M**	**Brij-35 10^3 k_1 min^{-1}**
1.	0.00	0.00
2.	0.30	3.20
3.	0.75	2.76
4.	0.99	2.43
5.	1.50	1.40
6.	1.88	2.69
7.	3.00	3.54
8.	5.00	3.98

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

These results align with Berezin's model [16], which suggests that when concentrations exceed the CMC, the system separates into two phases: an aqueous phase and a micellar pseudophase. The observations are also consistent with the Menger–Portnoy model [17] and the Piszkiewicz model [18], both of which account for the catalytic effects and kinetic characteristics of micellar systems.

To study the effect of surfactants on the Maltose–NBSA system, varying concentrations of Brij-35 were added. The plot of k_1 versus [surfactant] shows a peak, indicating micellar catalysis and a biphasic reaction, with oxidation occurring in both aqueous and micellar phases. The kinetics are interpreted using the Piszkiewicz model, where the linear plot of $\log[(k\psi - kW)/(kM - k\psi)]$ versus $\log[D]$ yields the cooperativity index "n." The oxidation rate follows the order Micellar > non micellar, consistent with the thermodynamic parameters listed in Table 3.

Activation Parameters			
S. No.	**Parameters**	**Brij-35**	**Absence**
1.	**E_a (KJ mol^{-1})**	36.41	45.52
2.	**A (sec^{-1})**	4.41×10^5	8.96×10^7
3.	**Δ H (KJ mol^{-1})**	33.64	42.75
4.	**Δ G (KJ mol^{-1})**	106.29	105.03
5.	**Δ S (Jk mol^{-1})**	-217.9	-186.7

6.	**K_M**	5.42×10^{-3}	-
7.	**K_S**	19.28×10^{-3}	-
8.	**n**	2.82	-
9.	**Log $[D]_{50}$**	1.702	-
10.	**10^3 K_D**	1.00	-

Table: 3 Thermodynamic parameters and Fighting Parameters

This can be attributed to the preferential interaction of the cationic oxidant species H_2O^+Br with the non-ionic Brij-35 micelle, which facilitates its attack on the Substrate more effectively than without micelle. The nearly constant ΔG values for both the Maltose–NBSA–surfactant and Maltose–NBSA systems indicate a common reaction mechanism. Addition of non-ionic micelle reduces the activation entropy and enthalpy, reflecting greater stabilization of the transition state, with Brij-35 providing stronger stabilization than absence of it.

Oxidation of Maltose in absence of non-ionic micelle (Brij-35):

The oxidation of maltose under non-micellar conditions was studied to establish the baseline kinetics in the absence of surfactant-mediated effects. The reaction proceeds through the formation of an intermediate complex between the oxidant and maltose, as evidenced by the dependence of the rate on substrate concentration. Oxidation in such an environment clearly depends on the acid concentration, dielectric constant, and additives such as saccharin or Hg(OAc)4. At lower maltose concentrations, the reaction rate increases linearly, whereas at higher concentrations the rate gradually approaches a limiting value, indicating saturation behavior. The reaction is influenced by acid catalysis, following both acid-dependent and acid-independent pathways, and the kinetic data suggest that electrophilic bromine species act as the primary oxidant in the process.

Conclusion:

The kinetic and mechanistic study of maltose oxidation by *N*-bromosaccharin (NBSA) indicates the formation of an intermediate complex between the substrate and oxidant, with electrophilic bromine species as the active oxidants. Kinetic results identify HOBr and H_2O^+Br as the reactive species. The reaction rate is lower under non-micellar conditions due to insufficient microenvironmental polarity tuning and electrostatic stabilization of charged intermediates. The system also exhibits substrate-dependent saturation behavior and proceeds via both acid-dependent and acid-independent pathways. The presence of the non-ionic surfactant Brij-35 enhances the reaction rate at concentrations below the CMC, indicating micellar catalysis and a biphasic reaction in which the substrate partitions between aqueous and micellar phases. Thermodynamic parameters suggest that the transition state is more stabilized in the micellar medium, highlighting the role of surfactants in facilitating structural organization and lowering the activation barrier. Overall, Brij-35 significantly influences the reaction kinetics and provides mechanistic insight into the micelle-assisted oxidation process.

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