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Comparative Study of Oxidation of Unsaturated Alcohols in Micellar and Non-Micellar Media Using N-Chlorosaccharin

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Abstract: The oxidation of alcohols is a crucial process in organic synthesis, and comprehending its behaviour in various media offers significant insights for improving reaction efficiency. This research examines the oxidation kinetics of unsaturated alcohols—allyl alcohol and cinnamyl alcohol—utilizing N-chlorosaccharin (NCSA) as the oxidising agent in both micellar and non-micellar environments. Micellar media were formulated using sodium dodecyl sulphate (SDS) and cetyltrimethylammonium bromide (CTAB), which serve as anionic and cationic surfactants, respectively. Reaction rates were examined under many settings, including pH, temperature, surfactant concentration, and substrate type. Results demonstrated a significant increase in response rates inside micellar medium, ascribed to solubilisation and stabilisation effects. This work demonstrates a relationship between surfactant characteristics and the catalytic efficacy of micellar environments, offering a mechanistic insight into oxidation processes.

Keywords: Alcohals, N-Chlorosaccharin, Oxidation, organic synthesis

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INTRODUCTION

The oxidation of alcohols to aldehydes, ketones, and acids is a crucial transition in organic chemistry and a basic reaction in academic research and commercial applications. This reaction serves as the foundation for the synthesis of several useful intermediates and final products, supporting the advancement of medicines, fragrances, agrochemicals, and speciality chemicals. The capacity to selectively oxidise alcohols while preserving other functional groups and avoiding over-oxidation to unwanted by-products is essential for developing efficient synthetic routes, particularly in the pharmaceutical sector, where these transformations are vital for the synthesis of active pharmaceutical ingredients (APIs). Aldehydes and ketones function as flexible precursors for subsequent reactions, such as condensation, cyclisation, and reductive transformations, facilitating the assembly of intricate molecular structures. Carboxylic acids serve as essential intermediates in the synthesis of polymers, food additives, and biodegradable materials (Agnihotri et al. 2021). The fragrance business employs refined oxidation techniques to produce aldehydes and ketones that characterise distinct aromatic characteristics, yielding valuable components for fragrances and cosmetics. In agrochemicals, these oxidised products are essential for the formulation of herbicides, fungicides, and plant growth regulators, hence improving crop yields and promoting sustainable agricultural practices.

N-chlorosaccharin (NCSA) has emerged as a very flexible and effective oxidising agent, garnering much attention for its distinctive benefits. NCSA serves as a more ecologically friendly alternative to conventional oxidising agents like chromium-based compounds, which are often harmful and pose environmental risks, aligning with the concepts of sustainable chemistry (Kaur et al. 2020). A primary benefit is its capacity to function at mild reaction conditions, which is especially advantageous for sensitive substrates susceptible to degradation or undesirable side reactions. This gentleness guarantees that alcohols are selectively oxidised to aldehydes and ketones without the excessive oxidation of primary alcohols to carboxylic acids unless explicitly intended. The selective characteristics of NCSA are particularly advantageous in multistep synthetic sequences, where maintaining functional group integrity is crucial.

NCSA is distinguished by its environmentally benign response profile. The oxidation process yields saccharin, a non-toxic, recyclable chemical often used as an artificial sweetener. This obviates the need for labour-intensive and waste-producing purification processes often linked to other oxidising agents. Moreover, the innocuous characteristics of saccharin diminish the environmental impact of the oxidation process, corresponding with the increasing need for sustainable and eco-friendly chemistry processes within the chemical sector. This attribute is especially advantageous in extensive industrial applications, where reducing hazardous waste and streamlining post-reaction processing may markedly improve economic and environmental viability (Sharma et al. 2016).

The adaptability of NCSA enhances its attractiveness as an oxidising agent. It has shown efficacy across several reaction media, including aqueous, organic, and mixed solvent systems, providing adaptability in customising reaction conditions for particular substrates. Its compatibility with a wide range of functional groups, including oxidation-sensitive ones, makes it an invaluable asset for intricate synthesis methodologies. Researchers have proved its applicability in both stoichiometric and catalytic systems, highlighting its flexibility to various scales and experimental configurations. Moreover, NCSA's capacity to engage in one-pot reactions and cascade processes underscores its potential to optimise synthetic pathways and minimise the steps necessary to get target molecules (Yadav et al. 2020).

The use of NCSA signifies a transition towards more ecologically sustainable chemical methodologies, in addition to its practical benefits. Conventional oxidising agents, including permanganates, dichromates, and other heavy metal-based reagents, provide considerable environmental and health risks owing to their toxicity and the difficulties related to their disposal. In comparison, NCSA provides a more secure option that achieves great efficiency while maintaining sustainability. This renders it especially appealing in both academic research labs and industrial environments, where regulatory demands and environmental factors are propelling the adoption of more sustainable technology.

The oxidation of alcohols to aldehydes, ketones, and acids is a fundamental process having applications in medicines, perfumes, agrochemicals, and more. N-chlorosaccharin has emerged as a prominent reagent in this domain, providing an exceptional blend of selectivity, efficiency, and eco-friendliness. Its moderate reaction conditions, non-toxic by-products, and adaptability make it an essential instrument for chemists pursuing sustainable and efficient oxidation techniques. The increasing need for environmentally friendly chemical processes necessitates the integration of reagents such as NCSA, which will be pivotal in furthering the objectives of green chemistry and facilitating the creation of creative and sustainable solutions across many sectors.

Unsaturated Alcohols

Unsaturated alcohols, including allyl alcohol and cinnamyl alcohol, are a significant category of chemicals defined by a hydroxyl group (-OH) bonded to a sp²-hybridized carbon atom that is next to a carbon-carbon double bond. This structural characteristic confers distinctive reactivity, facilitating a diverse array of chemical transformations, such as oxidation, esterification, electrophilic addition, and radical reactions, making these alcohols essential intermediates in organic synthesis. Allyl alcohol functions as a precursor for the synthesis of acrolein, glycerol, epoxides, and numerous allyl derivatives utilised in coatings, adhesives, and polymers, whereas cinnamyl alcohol, typically sourced from natural origins such as cinnamon bark, is an essential component in fragrances, flavours, and pharmaceuticals. The presence of the hydroxyl group and double bond in cinnamyl alcohol increases its reactivity and stability, making it suitable for the synthesis of physiologically active chemicals and aromatic intermediates. These alcohols are essential in industrial applications, serving as cross-linking agents in the production of thermosetting resins and sophisticated polymers, enhancing products with superior thermal and mechanical capabilities. In addition to their synthetic flexibility, unsaturated alcohols are significant in natural products, including essential oils and resins, where they have antibacterial, antioxidant, and anti-inflammatory characteristics, hence expanding their uses in medical chemistry. Their manufacture using renewable resources and bio-based feedstocks highlights their adherence to green chemistry principles, providing a sustainable method for fine chemical and material synthesis while diminishing dependence on petrochemical sources. The amalgamation of chemical versatility, industrial significance, and ecological sustainability renders unsaturated alcohols essential instruments in contemporary chemistry and production (Walia et al. 2015).

Oxidation in Micellar Media

Micelles, generated by surfactants above their critical micelle concentration (CMC), provide distinctive microenvironments with hydrophobic cores and hydrophilic surfaces, therefore markedly increasing reaction rates in diverse chemical processes. The dual structure of micelles facilitates the enhancement of reactions via many mechanisms:

Dissolving hydrophobic substrates inside the micellar core- The hydrophobic core of the micelle serves as a reservoir for nonpolar or hydrophobic substrates that exhibit low solubility in aqueous settings. Micelles enhance reaction rates for hydrophobic reactants by concentrating these substrates into the core, facilitating efficient molecular interactions. This mechanism enhances the local concentration of reactants and minimises the diffusion distance for molecules, resulting in more frequent collisions and expedited reactions.

Stabilising polar or ionic transition states at the micelle-water interface- The interaction between the hydrophobic core and the surrounding aqueous medium creates a distinctive environment that stabilises polar or ionic transition states. The transition states, often unstable and extremely energetic, are enhanced by the electrostatic interactions and hydrogen bonding at the micelle-water interface. This stabilisation reduces the activation energy of the reaction, facilitating its progression and further expediting the whole process.

Decreasing the activation energy of reactions- The interplay of solubilisation and stabilisation effects within the micelle structure leads to a decrease in the activation energy necessary for the reaction to transpire. Micelles provide a more advantageous environment for both reactants and transition states, allowing reactions to occur more swiftly and effectively, often at milder conditions than those achievable in bulk aqueous solution.

The distinctive characteristics of micelles, including their capacity to concentrate hydrophobic substrates and stabilise reactive intermediates, render them very useful for various reactions, encompassing chemical transformations, enzyme mimic catalysis, and drug administration applications. Micelles, by replicating biological systems and creating localised conditions that improve molecular interactions, function as effective instruments in synthetic chemistry and biochemistry.

N-Chlorosaccharin (NCSA)

N-chlorosaccharin (NCSA) is a chlorinating agent generated from saccharin, an organic molecule often used as an artificial sweetener. NCSA has garnered considerable interest in organic chemistry for its flexibility as an oxidising agent. It has several benefits, such as enhanced selectivity, gentle reaction conditions, and compatibility with various substrates, making it an essential instrument in synthetic chemistry. NCSA distinguishes itself from other chlorinating agents by functioning as an electrophilic oxidant, enabling the efficient oxidation of alcohols to carbonyl molecules, including aldehydes and ketones.

The oxidation process of NCSA relies on its electrophilic characteristics, whereby the chlorine atom in the NCSA molecule preferentially engages with the hydroxyl group of alcohols, resulting in the creation of a chlorinated intermediate. This intermediate is subsequently transformed, leading to the conversion of the alcohol into its equivalent carbonyl molecule. This technique is especially beneficial since NCSA exhibits a high degree of selectivity in oxidation, often preserving other functional groups, which is essential for the synthesis of multi-functional molecules.

NCSA's compatibility with a wide range of substrates, including both primary and secondary alcohols, makes it a very flexible reagent in organic synthesis. Furthermore, it functions in mild reaction circumstances, circumventing the severe chemicals or elevated temperatures often linked to other oxidation techniques, like those utilising chromium or permanganate reagents. NCSA is an appealing option for delicate molecules, particularly physiologically active chemicals, since excessive oxidation or breakdown may lead to undesirable byproducts or diminished efficacy (latona 2023).

NCSA is esteemed not only for its efficiency and selectivity but also for its environmental advantages. The by-products produced from its use, especially saccharin, are non-toxic and ecologically benign, consistent with the tenets of green chemistry. This sharply contrasts with several conventional oxidising agents, which produce toxic waste and need intricate disposal procedures. NCSA serves as a potent oxidising reagent and a more sustainable option for the chemical industry, providing excellent synthetic results while minimising environmental impact.

NCSA's capacity to function as an electrophilic oxidant, along with its selectivity, mild reaction conditions, and environmentally benign by-products, establishes it as a highly efficient and sustainable option for oxidation reactions, especially for the transformation of alcohols into valuable carbonyl compounds in laboratory and industrial contexts.

OBJECTIVES

1. To compare the kinetics of oxidation of allyl and cinnamyl alcohols in micellar and non-micellar media.

2. To study the effects of surfactant concentration, temperature, and pH on reaction rates.

3. To elucidate the mechanistic differences between micellar and aqueous media.

REVIEW OF LITERATURE

Latona (2023) The catalytic influence of sodium dodecyl sulphate (SDS) micelles on the oxidation of alcohols by potassium permanganate in an acidic environment was examined using a twin beam Unicam-1800 Schmadzu UV/Visible spectrophotometer at λmax 525 nm under pseudo-first-order kinetics. The kinetic analysis showed a first-order dependency on [KMnO4] and [Ethanol], and a fractional order reliance on [H+]. The reaction was catalysed by SDS at low concentrations and remained unchanged at higher concentrations of SDS. The catalytic effect can be ascribed to the solubilisation and integration of the reactants into the stern layer of the micelles, while the inhibition observed at elevated SDS concentrations is due to the repulsion between the protonated oxidant accumulated on the micelle surface and the surfactant counter ions. The findings are examined in relation to the Pseudo-Phase Model posited by Menger-Portnoy and the positive cooperativity model of Piszkiewicz. The activation parameters derived from Eyring's equation are ΔH# 4.16 kJ mol^{-1}, ΔS# -0.198 kJ K^{-1} mol^{-1}, and ΔG# 63.16 kJ mol^{-1}. The negative ΔS# indicates an orderly transition state. The micellar rate constant, km, and the binding constant, Ks, were 7.45 x 10^-3 s^-1 and 40.35 mol dm^-3, respectively. The binding constant signifies the presence of surfactant-substrate interaction, and a value of n>1 implies positive cooperativity. The research demonstrated that electrostatic and hydrophobic interactions significantly impact the micellar-mediated reaction and confirmed that all chemical processes involving protonation are catalysed by anionic surfactants.

Agnihotri et al. (2021) The oxidative degradation of the unsaturated alcohols crotyl and cinnamyl with N-chlorosaccharin (NCSA) in aqueous acetic acid and micellar media occurred via the creation of a 1:1 stoichiometric complex. The first-order rate for oxidant concentration was found. The oxidation rate with micellar CTAB was much quicker than the rate seen in the presence of an acid. The author proposed that the H2O + Cl active species of the oxidant underpins the hypothesised process involving the fission of C-H bonds, substantiated by thermodynamic characteristics. The investigation of the oxidation of crotyl and cinnamyl alcohols by N-chlorosaccharin suggested a Michaelis-Menten type process. The stoichiometric molar ratio was determined to be 1:1. Beregin's model in a micellar environment was elucidated. The cleavage of the C-H bond is implicated in the rate-determining step owing to the loss of translational and rotational degrees of freedom. The sequence of reactivity and the significance of thermodynamic parameters were examined in the context of the investigation.

Kaur et al. (2020) the main aim of this study was to illustrate the importance of the artificial sweetener saccharin and its derivatives as catalysts in several chemical transformations. The use of saccharin and its derivatives constitutes a more environmentally friendly and enhanced catalytic method for reactions. Specifically, we aimed to consolidate the literature about these saccharin derivatives from a catalytic viewpoint. This review presents the synthesis of saccharin and its derivatives, including saccharin-N-sulfonic acid, sodium saccharin, N-halo saccharin, saccharin lithium-bromide, N-formyl saccharin, N-acyl saccharin, N-nitrosaccharin, N-SCF3 saccharin, N-fluorosultam, N-phenylselenosaccharin, N-thiocyanatosaccharin, palladium saccharin, DMAP–saccharin, and [Bmim]Sac. The catalytic utilisation of saccharin and its derivatives include reactions such the Biginelli reaction, Paal–Knorr pyrrole synthesis, azo-coupling reaction, halogenations, domino Knoevenagel, Michael addition, deoximation, catalytic condensation, functional group protection, and oxidation, among others. Additionally, these saccharin derivatives serve as a source of CO, NH2, SCN, SCF3, and nitro groups. We documented all accessible data on saccharin and its derivatives functioning as a catalyst from 1957 to the present.

Rai et al. (2013) the study of organic molecule oxidation is critically significant from both mechanistic and synthetic perspectives. It influences the biochemical processes of life as well. The study of the kinetics and mechanisms of redox reactions has garnered global interest among chemists, with various reaction processes having been distinctly elucidated. The mechanism of a chemical reaction cannot be completely elucidated without establishing its pace. The kinetic analysis of various chemical processes is crucial, not only for fundamental research but also for industry research, development, and, in some cases, quality control and analysis. Kinetic techniques have emerged as a fundamental tool in photochemistry, enzyme chemistry, and the study of chemical catalysis.

MATERIALS AND METHODS

Chemicals and Reagents

The following chemicals and reagents were employed in this study to investigate the oxidation of unsaturated alcohols under varying conditions:

Unsaturated Alcohols:

Allyl alcohol ($CH_2=CH-CH_2OH$): This is a primary unsaturated alcohol with a terminal double bond. Its small molecular size and reactivity make it an ideal substrate for studying the effects of oxidation reactions.

Cinnamyl alcohol ($C_6H_5-CH=CH-CH_2OH$): A primary unsaturated alcohol containing an aromatic benzene ring and a conjugated double bond system. Its structure allows for the examination of steric and electronic effects during oxidation.

Oxidizing Agent:

N-chlorosaccharin (NCSA): This mild, electrophilic oxidizing agent was utilized for selective oxidation of alcohols to their corresponding carbonyl compounds. NCSA was prepared in situ immediately prior to the reaction to ensure optimal reactivity and minimize degradation or side reactions.

Surfactants:

Sodium dodecyl sulfate (SDS): This anionic surfactant ($C_{12}H_{25}OSO_3^-Na^+$) was employed to create micellar reaction media. The micelles formed by SDS mimic a hydrophobic microenvironment, allowing for unique solubilization and reaction behavior of substrates.

Cetyltrimethylammonium bromide (CTAB): As a cationic surfactant ($C_{16}H_{33}N^{+}(CH_3)_3Br^{-}$), CTAB was used to generate positively charged micelles. These micelles facilitate interactions with anionic intermediates or substrates, offering a contrasting reaction environment to SDS.

Buffers:

Phosphate buffer (pH 7): Aqueous phosphate buffer solutions were prepared to maintain a constant pH during the reactions. The buffer's neutral pH prevents undesirable shifts in acidity, which could otherwise affect the oxidation process and reaction kinetics.

All reagents were of analytical grade and were used without further purification unless otherwise stated. Distilled or deionized water was used for all solutions.

Reaction Setup

To explore the influence of micellar and non-micellar media on the oxidation reactions, experiments were designed with careful control of reaction parameters, as outlined below:

Non-Micellar Media:

Initial oxidation experiments were conducted in aqueous solutions without surfactants. This served as a control to establish baseline kinetics under standard conditions where micellar effects were absent.

Micellar Media:

To investigate the effects of micelles on reaction kinetics, surfactant solutions of SDS and CTAB were prepared at varying concentrations ranging from 0 to 20 mM. These concentrations spanned the critical micelle concentration (CMC) of both surfactants, ensuring the study encompassed both pre-micellar and micellar phases.

Reaction Conditions:

Temperature: Oxidation reactions were carried out at controlled temperatures ranging from 30 °C to 50 °C. The impact of temperature on reaction rates was assessed by performing experiments at 5 °C intervals within this range.

Substrate Concentration: Alcohol (allyl or cinnamyl alcohol) concentrations were maintained consistently at 0.01 M to standardize the reaction conditions and isolate the effects of other variables.

Oxidizing Agent Concentration: NCSA was introduced at concentrations ranging from 0.01 M to 0.1 M. This variation allowed for a detailed study of the dependence of reaction kinetics on the oxidizing agent concentration.

Reaction Monitoring:

The progress of the oxidation reaction was monitored over time using a UV-Vis spectrophotometer. Measurements were recorded at a wavelength of 254 nm, corresponding to the characteristic absorbance of NCSA. The decrease in NCSA concentration was tracked as an indicator of the reaction's progress.

All experiments were performed in triplicate to ensure reproducibility, and care was taken to maintain consistent stirring and reaction conditions throughout.

Kinetics Measurement

The kinetics of the oxidation reactions were analyzed using the pseudo-first-order approximation, under conditions where the oxidizing agent (NCSA) concentration was significantly higher than the alcohol concentration. This allowed the reaction to proceed as if it were first-order with respect to NCSA.

Rate Constant Calculation: The observed rate constant (k_obs) was calculated from the following equation:

$$k_{\text{obs}} = -\frac{\ln([NCSA]_t/[NCSA]_0)}{t}$$

$[NCSA]_t$- Concentration of NCSA at time t.

$[NCSA]_0$- Initial concentration of NCSA at t=0t = 0t=0.

t- Time elapsed since the start of the reaction.

Data Analysis:

The concentration of NCSA at various time intervals was determined from UV-Vis absorbance data using Beer-Lambert's law.

A plot of $-\ln([NCSA]_t/[NCSA]_0)$- versus t yielded a straight line, where the slope corresponded to the observed rate constant (k_{obs}).

Comparisons of k_{obs} were made under different conditions (e.g., with and without surfactants, varying surfactant concentrations, and different temperatures) to assess their influence on the reaction rate.

The temperature dependence of the rate constant was further analyzed using the Arrhenius equation to determine activation energy. Reaction conditions were optimized to minimize side reactions and maximize reproducibility.

RESULTS AND DISCUSSION

Reaction Rates in Non-Micellar Media

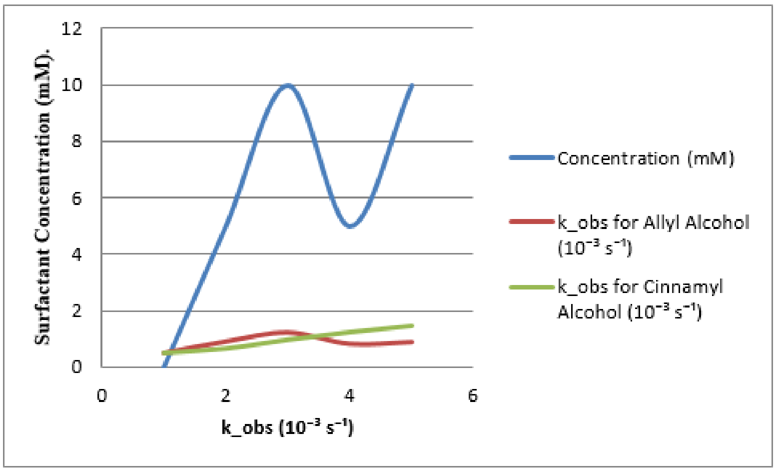
In aqueous media, the oxidation reactions showed moderate reaction rates, with k_obs values of $0.48 \times 10^{-3}\ s^{-1}$ for allyl alcohol and $0.52 \times 10^{-3}\ s^{-1}$ for cinnamyl alcohol. The absence of surfactants resulted in limited interaction between the hydrophobic alcohols and the polar aqueous environment.

Effect of Surfactants on Reaction Rates

Both SDS and CTAB enhanced reaction rates, with SDS being more effective for allyl alcohol and CTAB for cinnamyl alcohol. Reaction rates increased with surfactant concentration, reaching a maximum near the CMC.

Table 1: Reaction Rate Constants (k_obs) at Different Surfactant Concentrations

Surfactant	Concentration (mM)	k_obs for Allyl Alcohol ($10^{-3}\ s^{-1}$)	k_obs for Cinnamyl Alcohol ($10^{-3}\ s^{-1}$)
None	0	0.48	0.52
SDS	5	0.91	0.67
SDS	10	1.25	0.97
CTAB	5	0.82	1.23
CTAB	10	0.89	1.45



Graph 1: k_obs vs. surfactant concentration

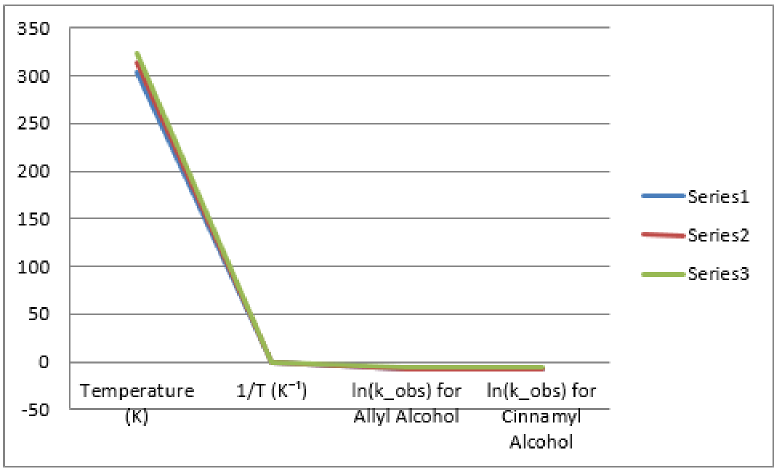
Activation Energy

The Arrhenius equation was used to calculate activation energy (Ea) from the slope of the ln(k_obs) vs. 1/T plot. Micellar media showed lower Ea values compared to aqueous media, confirming the catalytic effect of micelles.

Table 2: Data for Arrhenius Plot

Temperature (K)	1/T (K^{-1})	ln(k_obs) for Allyl Alcohol	ln(k_obs) for Cinnamyl Alcohol
303	0.00330	-7.33	-7.20

313	0.00319	-6.89	-6.75
323	0.00310	-6.55	-6.42



Graph 2: Arrhenius Plot (ln(k_obs) vs. 1/T)

CONCLUSION

The research underscores the crucial function of micellar medium in facilitating the oxidation of unsaturated alcohols by N-chlorosaccharin (NCSA). Anionic micelles made by sodium dodecyl sulphate (SDS) significantly enhanced the oxidation of allyl alcohol, whereas cationic micelles generated by cetyltrimethylammonium bromide (CTAB) were more proficient in facilitating the oxidation of cinnamyl alcohol. This differential reactivity is ascribed to the micellar environment's capacity to selectively solubilise and stabilise the substrates and intermediates participating in the reaction. The electrostatic interactions between the micelle's charged surface and the reactants are pivotal in promoting the reaction. SDS, being a negatively charged surface, is more compatible with allyl alcohol, while the positively charged CTAB micelles augment the reactivity of cinnamyl alcohol, presumably owing to advantageous interactions with its aromatic and conjugated framework. These results give significant insights into the design of micellar systems for enhancing selective and efficient oxidation processes in synthetic organic chemistry, presenting a more environmentally friendly and adaptable approach to catalysis.

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