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A REVIEW OF SUBSTITUENT EFFECTS ON OXIDATION KINETICS: PHENYLIC ACID AND *m*-HYDROXY-NITROBENZENE

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ABSTRACT

This study explores the oxidation kinetics of phenylic acid and *meta*-hydroxy-nitrobenzene using pyrazinium chlorochromate (PzCC) in perchloric acid media. We examined how oxidant concentration, solvent composition, temperature, and additives like acrylonitrile and manganous sulfate influence the reaction. The reaction adhered to first-order kinetics concerning PzCC, showing slower rates at higher oxidant concentrations, suggesting possible inhibition. Increasing acetic acid concentration sped up the reaction due to its lower dielectric constant, while perchloric acid positively affected the rate, indicating its catalytic role. Sodium perchlorate and acrylonitrile had negligible impacts, with acrylonitrile not forming free radicals. Manganous sulfate inhibited the reaction. Higher temperatures accelerated the reaction by reducing activation energy and enhancing molecular collisions. The mechanism involves protonation of phenylic acid, electrophilic attack by molecular oxygen, and subsequent rearrangement, providing insights for optimizing oxidative processes.

Keywords: Phenylic Acid, Pyrazinium Chlorochromate, Solvent, Reaction Rate, Oxidation, Kinetics.

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INTRODUCTION

Phenylic acid, also known as phenyl alcohol, is a naturally occurring compound found in plants and foods. Although it does not pose significant environmental hazards in its pure form, its release into the environment can lead to water contamination.[1] Phenylic acid wastewaters, originating from various industries, are toxic and non-biodegradable. Phenylic acid, a byproduct in the oxidation of aromatic hydrocarbons, is a priority pollutant listed by the EPA.[2] It is used in industries including plastics, pharmaceuticals, petrochemicals, paints, refineries, pulp and paper, and petroleum.[3,4] Sources of such contamination include agricultural runoff, industrial discharges, and improper waste disposal. High concentrations of phenylic acid can disrupt aquatic ecosystems, impairing the health of aquatic life and potentially contaminating groundwater sources. Therefore, effective management of phenylic acid is crucial to mitigate its environmental impact. Several methods exist for removing phenylic acid, including extraction with organic solvents like ethyl acetate or diethyl ether, acid/base extraction to convert phenolic acids into phenolate salts, and adsorption using solid adsorbents such as activated carbon or resins.[5-7] Additionally, chelation methods and advanced oxidation processes can be employed.[8] Biological degradation processes involving microorganisms or enzymes also offer a means of breaking down phenylic acids.[9] Oxidation using several hexavalent chromium compounds, including benzimidazolium fluorochromate, morpholinium chlorochromate, isoquinolinium chlorochromate (IQC), morpholinium fluorochromate, quinaldinium chlorochromate (QnCC), tetraethylammonium chlorochromate, quinolinium fluorochromate, quinolinium bromochromate, imidazolium chlorochromate, tripropyl ammonium fluorochromate, benzyl trimethyl ammonium chlorochromate, and pyridinium dichromate, are used as multifunctional oxidizing agents under various conditions. These heterocyclic chromium compounds convert Cr (VI) to Cr (III) within their rings.[10-13] In this research paper the oxidant used is

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pyrazinium chlorochromate. The use of PzCC for the removal of phenylic acid offers several advantages. Its selectivity ensures that phenylic acid is efficiently oxidized into less problematic or more easily removable compounds. This is particularly important in scenarios where phenylic acid could interfere with subsequent reactions or affect the properties of the final product. Moreover, PzCC's compatibility with various reaction conditions and solvents allows it to be employed effectively in a wide range of synthetic and industrial processes without causing significant side reactions or adversely impacting other components. The combination of selectivity, efficiency, compatibility, and safety makes PzCC a valuable choice for the oxidation and removal of phenylic acid in various applications.

EXPERIMENTAL

Material and Methods

The chemicals used in this study were sourced from Merck, all of which were of analytical reagent (AR) grade with a purity of 100%. The oxidant, pyrazinium chlorochromate (PzCC), was synthesized following a standard procedure[13] to ensure consistency and accuracy in the experiments. The kinetics of the oxidation reaction were analyzed using a colorimeter, which provided precise measurements of absorbance over time, allowing for a detailed study of the reaction rates.

Relationship between PzCC Concentration and Reaction Rate

To explore the impact of varying concentrations of PzCC on the reaction rate, the oxidation process was carried out while keeping the concentrations of the acid and substrate constant. By altering only the concentration of PzCC, the experiment aimed to isolate its specific effects on the reaction kinetics. The results revealed that the reaction followed first-order kinetics concerning PzCC concentration, as demonstrated by the linear correlation observed in the plot of the concentration of the oxidant versus reaction rate as shown in the Fig.-1. As the concentration of PzCC increased, a steady decrease in the rate constant was observed. This counterintuitive finding suggests that higher concentrations of PzCC may introduce inhibitory effects or encounter other limiting factors that reduce the efficiency of the reaction. These inhibitory effects could be due to the formation of intermediate complexes or side reactions that become more prominent at higher oxidant concentrations. Additionally, it was noted that while PzCC served as a chromium-based oxidant, the actual concentration of Cr^{6+} ions present in the reaction medium was relatively low. This low presence of Cr^{6+} ions indicates that their direct involvement in the oxidation mechanism may be minimal. Instead, the primary role of PzCC likely involves the transfer of oxygen atoms, facilitating the oxidation of the substrate without extensive participation of Cr^{6+} ions.[14]

RESULTS AND DISCUSSION

Dissociation of PzCC

PzCC undergoes dissociation to release [Cr(VI)] and a pyrazinium cation [Pz]+.

$$PzCC \rightarrow [Cr\ (VI)] + [Pz]^{+}$$

Oxidation of Phenylic Acid

The phenylic acid reacts with the [Cr(VI)] ion and a proton (H^+) to form quinone, Cr(III), and water.

$$\text{Phenylic acid} + [Cr\ (VI)] + H^{+} \rightarrow \text{Quinone} + Cr\ (III) + H_2O$$

Potential Reaction with Cr(VI)

A secondary reaction occur where [Cr(VI)] interacts with a proton (H^+) to form a [Cr(VI)-H]+ complex. However, the low concentration of Cr^{6+} ions suggests that this step is not the primary pathway in the oxidation process.

$$[Cr\ (VI)] + H^{+} \rightarrow [Cr\ (VI)\text{-}H]^{+}$$

Substrate Influence on Reaction Kinetics

The concentration of the substrate, phenylic acid, was varied to assess its impact on the reaction rate. It was observed that the rate constant increased as the concentration of phenylic acid increased at a constant temperature. A linear relationship (Fig.-2) was found between the logarithm of the rate constant and the logarithm of the phenylic acid concentration, indicating that the reaction follows fractional-order kinetics

with respect to phenylic acid. The strong correlation, with a coefficient of (r = 0.997), underscores a significant relationship between the rate constant and phenylic acid concentration. These findings highlight the non-linear dependence of the reaction rate on substrate concentration, emphasizing the critical role that substrate concentration plays in influencing the reaction kinetics.

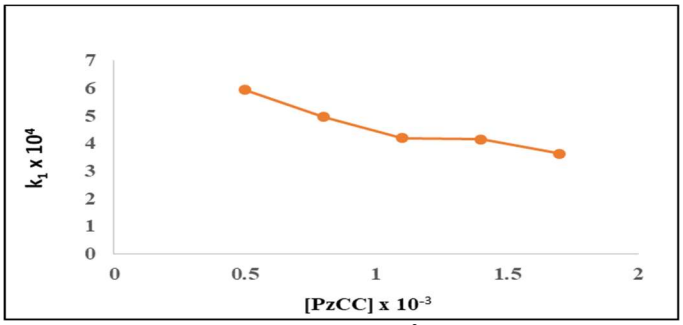


Fig.-1: Plot of [PzCC] 10^{-3} *versus* Rate

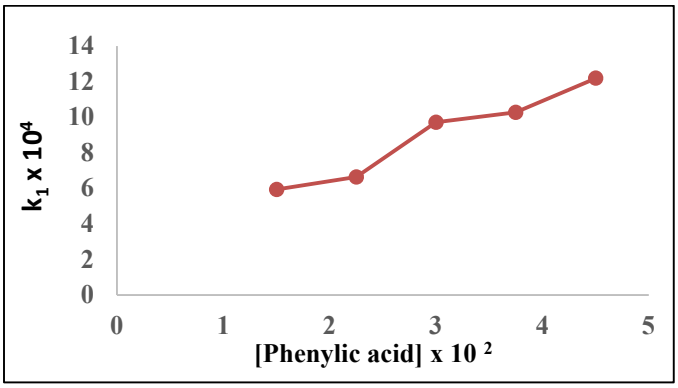


Fig.-2: Plot of [Phenylic acid] 10^{2} *versus* Rate

Quantifying the Effect of Varying Perchloric Acid Concentration on Reaction Kinetics

The concentration of perchloric acid, used as a catalyst in the oxidation reaction, was varied to explore its impact on the reaction rate. It was observed that the rate constant increased with the acid concentration, ranging from 1.08×10^{-1} to 2.3×10^{-1} mol dm^{-3}, and a plot of the logarithm of the rate constant against the logarithm of the acid concentration showed a linear relationship with a high correlation coefficient (r = 0.994). The observed fractional-order kinetics suggest that the reaction rate is dependent on the protonated form of the oxidant, highlighting the role of perchloric acid in enhancing catalytic efficiency. Higher acid concentrations likely stabilize reactive intermediates and facilitate ion pair formation, thereby lowering the activation energy and accelerating the reaction[14]. These findings emphasize the significant influence of perchloric acid concentration on reaction kinetics through various mechanistic pathways.

Oxidation of Phenylic Acid

$$\text{Phenylic acid} + \text{PzCC} + H^+ \rightarrow \text{Quinone} + \text{Pyrazinium} + \text{Chromate} + H_2O$$

Reaction with PzCC

$$\text{PzCC} \rightarrow \text{Pyrazinium} + \text{Chromate}$$

Potential Reaction with Chromate

$$\text{Chromate} + H^+ \rightarrow \text{Chromic Acid}$$

Assessing the Effect of Varying Solvent Composition on Product Selectivity

In the kinetic study, changes in acetic acid and water concentrations revealed a clear relationship between the rate constant and solvent properties. A plot of k_1 versus 1/D produced a linear graph with a positive slope (B = +11.34) and a high correlation coefficient (r = 0.998). This observation suggests that chromium (VI) species participate in the rate-determining step, with lower dielectric constants—resulting from

increased acetic acid concentrations—enhancing the reaction rate. The solvent composition significantly impacts product selectivity by modulating the stabilization of intermediates and transition states. Lower dielectric constants promote reactivity by favoring the formation of charged intermediates, particularly those involving *meta*-nitro phenol (*m*-hydroxy-nitrobenzene), which plays a key role in the reaction mechanism. *Meta* nitro phenol undergoes solvation and subsequent oxidation in the presence of acetic acid and the oxidant. The solvent composition influences the equilibrium between free and solvated forms of meta-nitro phenol, with acetic acid enhancing its solvation through hydrogen bonding. This stabilization facilitates the formation of charged transition states, driving the reaction forward and affecting product selectivity.

Dissociation of Acetic Acid

$$CH_3COOH \rightleftharpoons CH_3COO^- + H+$$

Solvation of *Meta* Nitro Phenol

$$\text{m-nitro phenol} + CH_3COOH \rightleftharpoons \text{m- nitro phenol-}CH_3COOH$$

Oxidation of Phenylic Acid

$$\text{m- nitro phenol} + \text{Oxidant} + H^+ \rightleftharpoons \text{Product(s)} + CH_3COOH$$

Potential Reaction with Oxidant

$$\text{Oxidant} + H^+ \rightleftharpoons \text{Oxidant-}H^+$$

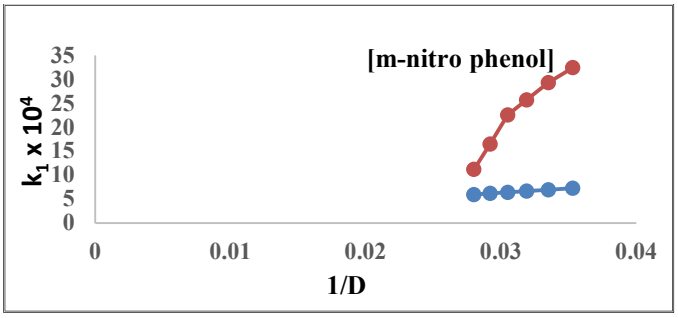


Fig.-3: Correlating The Dielectric Constant with the Reaction Rate Constant

Elucidating the Influence of Acrylonitrile Concentration on the Reaction Mechanism

In the reaction mixture of phenylic acid, acetic acid, and pyrazinium chlorochromate, adding acrylonitrile showed no precipitate after a day. This absence of precipitate indicates that acrylonitrile did not generate free radicals, suggesting that the reaction mechanism did not involve significant free radical intermediates. A similar effect was observed in 3-hydroxy-nitrobenzene.

Influence of Salt Effect

The investigation into the concentration-dependent effects of sodium perchlorate ($NaClO_4$) and manganous sulfate ($MnSO_4$) on reaction kinetics revealed distinct influences on the rate constant. While varying $NaClO_4$ concentrations had no effect, indicating that the ionic species Na^+ and ClO_4^- do not significantly alter the rate-determining step, increasing $MnSO_4$ concentrations led to a progressive decrease in the rate constant. This suggests that Mn^{2+} ions likely participate in electron transfer processes, potentially inhibiting the reaction through competitive interactions, inactive complex formation, side reactions, or altered pathways. These results highlight the contrasting roles of $NaClO_4$ and $MnSO_4$ in the reaction mechanism, with Mn^{2+} exerting a more direct influence on the reaction's efficiency and kinetics.

Oxidation of Phenylic Acid

$$\text{Phenylic acid} + \text{Oxidant} + H^+ \rightarrow \text{Quinone} + Na^+ + ClO_4^- + H_2O$$

Reaction with $NaClO_4$

$$NaClO_4 \rightarrow Na^+ + ClO_4^-$$

Potential Reaction with ClO_4^-

$$ClO_4^- + H^+ \rightarrow HClO_4$$

Reaction with Manganous Sulfate

$$MnSO_4 \rightarrow Mn^{2+} + SO_4^{2-}$$

Potential Electron Transfer

$$Mn^{2+} + 2e^- \rightarrow Mn$$

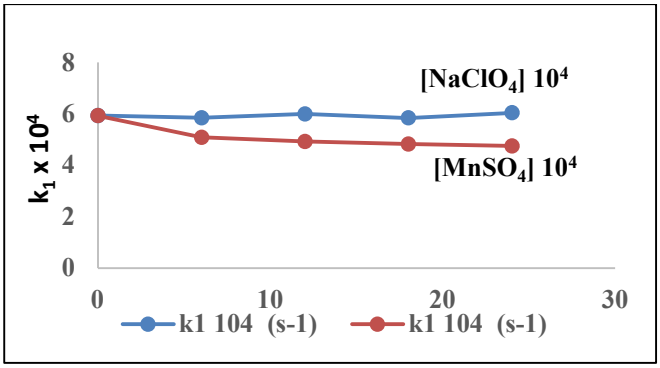


Fig.-4: Effect of Ionic Strength and the Reaction Rate

Thermodynamic and Kinetic Effects of Temperature Variation

The effect of varying the temperature on the oxidation reaction of phenylic acid and *m*-hydroxy-nitrobenzene was investigated at different temperatures (298K, 303K, 313K, and 323K) while keeping other parameters constant. The rate constant values were determined, and it was observed that the rate constant increased with an increase in temperature. To further analyze the relationship between the rate constant and temperature, Eyring's least square method was used to assess the activation parameters and thermodynamic parameters of the reaction. By plotting the logarithm of the rate constant divided by temperature (log k_2/T) against the reciprocal of temperature (1/T), a linear plot was obtained.[15] The linear relationship observed in the plot confirms the validity of the Arrhenius equation and indicates the temperature dependence of the reaction rate. The positive slope in the plot suggests an increase in the rate constant as temperature rises, which is consistent with the general trend observed for many chemical reactions. The activation parameters, such as activation energy (E_a), pre-exponential factor (A), and frequency factor, can be calculated using the slope and intercept of the linear plot. These parameters provide insights into the energy barrier and the efficiency of the reaction at different temperatures. The thermodynamic parameters, such as enthalpy (ΔH), entropy (ΔS), and Gibbs free energy (ΔG), can also be assessed from the temperature dependence of the rate constant. These parameters provide information about the overall energy changes and spontaneity of the reaction.[16,17]

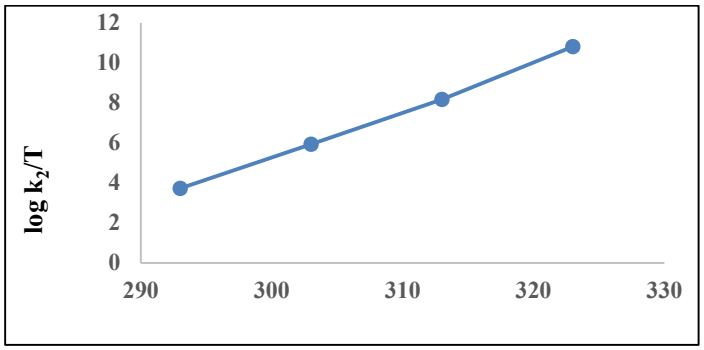


Fig.-5: Plot of log k_2/T *versus* 1/T

Increase in Rate of *m*-Hydroxy-Nitrobenzene Substrate and its Influence

The increased oxidation rate of substituted phenylic acids, such as *meta*-hydroxy-nitrobenzene, compared to phenylic acid can be attributed to several key factors related to the electronic effects of the substituents and their influence on the reaction mechanism. Firstly, electron-withdrawing groups, like the nitro group in

meta-hydroxy-nitrobenzene, decrease the electron density around the hydroxyl group of the aromatic ring through inductive effects. This reduction in electron density enhances the electrophilicity of the hydroxyl group, making it more reactive towards oxidation by the oxidant, such as pyrazinium chlorochromate (PzCC). Secondly, in acidic conditions, the protonation of the hydroxyl group increases its electrophilicity further. For substituted phenylic acids with electron-withdrawing groups, this protonation becomes more favorable, facilitating a faster reaction with the oxidant. Thirdly, the resonance effects of the substituents can stabilize the transition state or intermediate species in the reaction, leading to a lower activation energy and thus a higher reaction rate. Lastly, the solvent composition also plays a crucial role. In a solvent mixture of acetic acid and water, the lower dielectric constant of acetic acid enhances charge transfer and solvation of reactants, which can accentuate the impact of substituents on the oxidation rate. The combination of these factors—enhanced electrophilicity, favorable protonation, resonance stabilization, and solvent effects—contributes to the faster oxidation rate observed for substituted phenylic acids compared to phenylic acid.

Table 1: Rate values of Phenylic Acid and *m*-hydroxy-nitrobenzene with Varying Temperature and [S]

Phenylic acids	Temperature *K*	[S] $10^2 mol\ dm^{-3}$	$k_1\ 10^4 s^{-1}$	Order with respect to [S]
Phenylic acid (-H) ¤	303	1.50	5.93	0.551
	303	2.25	6.63	
	303	3.00	9.7	
	303	3.75	10.27	
	303	4.50	12.19	
Phenylic acid (-H)❁	293	1.50	3.73	-
	303	1.50	5.93	
	313	1.50	8.17	
	323	1.50	10.81	
m-NO_2¤	303	1.50	13.65	0.380
	303	2.25	18.94	
	303	3.00	24.41	
	303	3.75	27.57	
	303	4.50	29.23	
m-NO_2❁	293	1.50	19.39	-
	303	1.50	23.81	
	313	1.50	26.84	
	323	1.50	31.95	

¤ – Effect of varying the [S] ❁ - Effect of varying the temperature

Varying Temperature

As the temperature of the reaction medium for the oxidation of *m*-hydroxy-nitrobenzene increases from 293K to 323K, the rate of the reaction also increases. This phenomenon can be attributed to two primary factors: activation energy and collision frequency. Firstly, the increase in temperature affects the activation energy of the reaction. According to the Arrhenius equation, the rate constant of a reaction is exponentially dependent on the activation energy. As the temperature rises, the value of 1/T decreases, leading to a larger exponential term in the equation. This implies that more reactant molecules possess sufficient energy to surpass the activation energy barrier, allowing them to participate in the reaction. Consequently, the reaction rate increases as more reactants can effectively undergo the necessary chemical transformations. Secondly, higher temperatures result in an increase in the kinetic energy of the molecules in the reaction medium. This elevated kinetic energy leads to more frequent and energetic collisions between reactant molecules. With a greater collision frequency and energy, the chances of successful molecular rearrangements and the formation of products are enhanced. Therefore, the overall reaction rate is accelerated.

The difference in products observed when phenylic acid and *meta*-hydroxy-nitrobenzene are oxidized with pyrazinium chlorochromate (PzCC) arises from the positions of their substituents on the aromatic ring and their influence on the oxidation reaction. Phenylic acid, which has a hydroxyl group at the para position

relative to itself, upon oxidation with PzCC, is converted into 1,4-benzoquinone. This product is formed because the hydroxyl group is oxidized to a carbonyl group, resulting in a fully conjugated system with two adjacent carbonyl groups. In contrast, *meta*-hydroxy-nitrobenzene has the hydroxyl and nitro groups positioned in a *meta* arrangement relative to each other. When oxidized, the hydroxyl group in *meta*-hydroxy-nitrobenzene is converted to a carbonyl group, but the nitro group remains at the *meta* position relative to the newly formed carbonyl groups (Scheme-1). This leads to the formation of 3-nitroquinone, where the *meta* position of the nitro group disrupts the conjugation seen in quinones derived from phenylic acid. Thus, the oxidation product differs because the *meta*-hydroxy-nitrobenzene structure affects the conjugation and symmetry of the resulting quinone system, resulting in 3-nitroquinone rather than the symmetric quinone observed with phenylic acid.

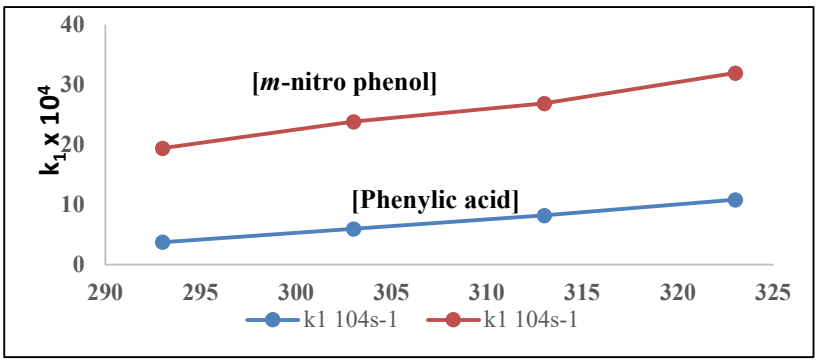
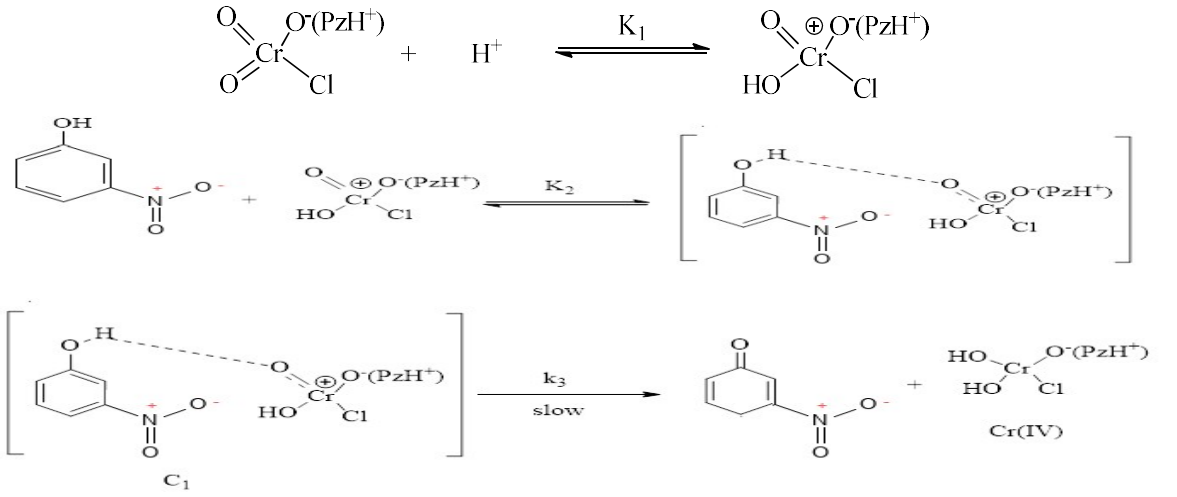


Fig.- 6: Temperature Effect and the Reaction Rate

Mechanism for the Oxidation of 3-nitro Phenol Involves the Following Steps



Scheme-1: Mechanism of Oxidation of 3-nitrophenol by Pyrazinium Chlorochromate

Reaction Rate [14]

$$\text{Rate} = \frac{-d[PzCC]}{dt} = k_3[\text{complex}]$$

$$= \frac{k_3 K_2 [S][C_1]}{1 + K_2[S]}$$

$$= \frac{k_3 K_2 K_1 [S][PzCC][H^+]}{(1 + K_2[S])(1 + K_1[H^+])}$$

$$= \frac{k_3 K_2 K_1 [S][PzCC][H^+]}{1 + K_2[S] + K_1[H^+]}$$

Product Characterization and Verification

These reactions may lead to the formation of quinones. The overall rate of the reaction may be influenced by factors such as the concentration of the oxidizing agent, phenylic acid, and perchloric acid, as well as the nature of the solvent. In the oxidation of phenylic acid and *meta* with pyrazinium chlorochromate (PzCC), 1,4-benzoquinone and 3-nitroquinone are formed, respectively, and their characterization can be achieved using UV-Vis and IR spectroscopy. 1,4-Benzoquinone, derived from phenylic acid, is identified by its UV-Vis absorbance around 250 nm, which corresponds to the π-π* transitions in the quinone structure[14]. IR spectroscopy confirms its presence with strong peaks around 1650-1700 cm^{-1} for carbonyl stretching. On the other hand, 3-nitroquinone, the product of *meta*-hydroxy-nitrobenzene oxidation, also shows absorbance near 250 nm, though with shifts due to the nitro group. Its IR spectrum reveals similar carbonyl stretching peaks but also includes additional bands around 1500-1600 cm^{-1} (asymmetric NO_2 stretching) and 1300-1400 cm^{-1} (symmetric NO_2 stretching), indicating the presence of the nitro group. 1,4-Benzoquinone, derived from phenylic acid, is characterized by a melting point around 114°C, whereas 3-nitroquinone, from *meta*-hydroxy-nitrobenzene, has a slightly lower melting point of about 112°C due to the influence of the nitro group on its crystal structure. These techniques together confirm the distinct structures and substituent effects of the two quinones.

CONCLUSION

The oxidation of phenylic acid, *meta* hydroxy-nitrobenzene and reaction conditions was explored in this discussion. The choice of oxidants such as pyrazinium chlorochromate (PZCC) offers advantages in terms of selectivity, efficiency, and compatibility. The reaction rates were found to be influenced by factors such as solvent composition, temperature, and catalyst concentration. Varying the concentration of manganous sulfate showed a gradual decrease in the rate constant, indicating the involvement of Mn^{2+} ions in electron transfer. The activation parameters and thermodynamic parameters were assessed using Eyring's least square method, providing insights into the energy barrier and spontaneity of the reaction. The addition of acrylonitrile did not induce free radical formation, as confirmed by the absence of precipitate. Varying the concentration of sodium perchlorate did not significantly affect the rate constant, suggesting that the rate-determining step involves ionic species.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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