**A Kinetic and thermodynamic study of oxidation of selective alcohols using Chromium-based reagents and silver nanoparticle catalyst: A spectrophotometric approach**

**1. abstract**

Oxidation is a fundamental industrial process due to its ability to produce valuable compounds, particularly the transformation of alcohols into carbonyl derivatives. While extensive research has explored this conversion, limited attention has been given to the kinetic and thermodynamic aspects. This study investigates the oxidation kinetics of selected aliphatic and cyclic alcohols using PCC and PDC in an acidic medium. These oxidants are stable, easily accessible, and efficiently convert a variety of alcohols into corresponding carbonyl compounds. The reactions were found to follow first-order kinetics with respect to oxidant concentration, and the reaction progress was monitored spectrophotometrically by tracking the decrease in absorbance at 354 nm for PCC and 470 nm for PDC. The influence of temperature was analyzed using the Arrhenius equation, while the impact of ionic strength followed the Bronsted-Bjerrum equation. An increase in alcohol concentration led to a higher oxidation rate. Additionally, this work highlights the potential of silver nanoparticles derived from plant sources as effective catalysts for alcohol oxidation. Silver nanocatalysts are highly effective in enhancing the kinetics of alcohol oxidation due to their large surface area, which provides abundant active sites for catalytic interactions. Their excellent electron transfer capability facilitates rapid redox reactions, promoting efficient conversion of alcohols to aldehydes or ketones.

**Keywords:** Chemical kinetics, oxidation of alcohols, Pyridinium chlorochromate, Pyridinium dichromate, Spectrophotometer

**2. INTRODUCTION**

The oxidation of alcohols holds significant industrial importance, as it leads to the production of valuable compounds. In India, the thriving indigenous perfumery sector enjoys a global market presence, highlighting the pivotal role of the study of perfumes, fragrances, and cosmetic formulations. While organic oxidants have traditionally dominated alcohol oxidation reactions, the utilization of inorganic oxidants remains relatively scarce [1, 2]. This paper presents Cr(VI) as an oxidant for the oxidation of various perfumery alcohols, offering promising avenues for enhancing the efficiency and sustainability of these processes within the industry.

In this study, Pyridinium chlorochromate (PCC) and Pyridinium dichromate (PDC) were used as oxidants for converting alcohols into carbonyl compounds. It is well-established that the oxidation mechanism is influenced by the specific counter-ion linked to the chromium anion. This study explores the kinetic and mechanistic of oxidation by complexed Cr(VI) species, as dichromate-based systems have already been reported [3, 4]. Herein, a detailed analysis of the kinetics and mechanisms governing the oxidation of both aliphatic and cyclic alcohols using these reagents under acidic conditions is presented [5-10]. The main aims of the present investigation are to determine kinetic parameters and to evaluate the rate equations, study the correlation analysis of effect of temperature on reactivity, study effect of ionic strength, study effect of silver nanoparticle [11] and providing a suitable mechanism for the oxidation process [12-14]. There are various reviews, focusing on the mechanisms, catalyst design, applications, and future directions for selective oxidation of alcohols using various catalytic strategies [15-21]. Another purpose of this study is to highlight the potential of plant-derived silver nanoparticles as highly efficient catalysts for alcohol oxidation, suggesting the development of more effective and sustainable industrial oxidation methods. Table-1 and Table-2 includes the information of selected alcohols and oxidizing agents for the study.

Table 1-General information about used alcohols

Name of alcohol

Molecular formula

Molar mass (g/mol)

Structural formula

n

-

butanol

C

4

H

10

O

74.12

2

-

chloroethanol

C

2

H

5

OCl

80.52

Cyclohexanol

C

6

H

12

O

100.61

Cinnamyl alcohol

C

9

H

10

O

134.17

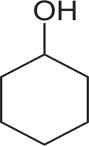
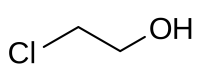


Table 2- General information about Oxidizing agents

|  |  |  |  |
| --- | --- | --- | --- |
| Name | Molecular formula | Molar mass(g/mol) | Structural formula |
| Pyridinium  chlorochromate  Pyridinium dichromate | C5H5NHClCrO3  C10H12N2Cr2O7 | 215.55  376.24 |  |

**3. material and methods**

**3.1 Kinetic measurements**

All chemicals used were of analytical reagent (AR) grade. The oxidant solution in the acid-water medium adheres to Beer-Lambert's law, meaning the absorbance correlates linearly with the oxidant concentration [6]. To investigate the reaction under first-order conditions, an excess of substrate was maintained over the oxidant [4]. The reaction mixture was prepared by combining the appropriate amounts of substrate, H2SO4, and water, then allowed to equilibrate in a thermostatic bath for a specific time. Upon addition of the oxidant solution, the reaction commenced [4]. The absorbance of the reaction mixture was monitored spectrophotometrically at 354 nm for PCC and 470 nm for PDC using a spectrophotometer.

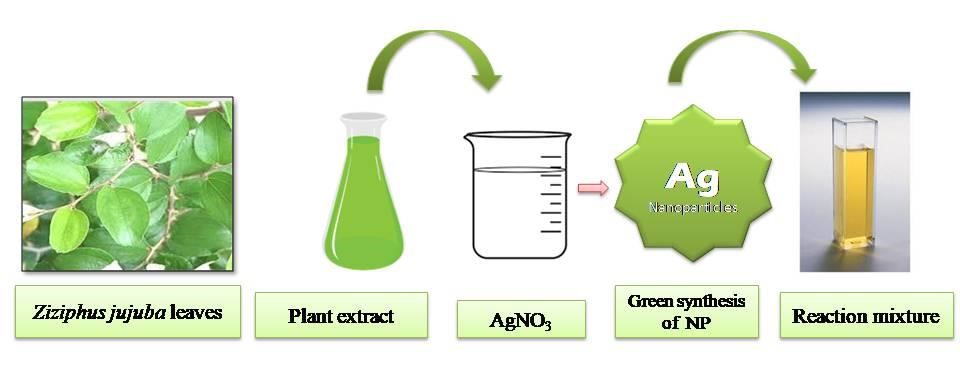
**3.2 Product Analysis**

A large volume containing an excess of alcohol relative to the oxidant was allowed to react with 2, 4-dinitrophenylhydrazine [14]. The formation of a yellow-orange precipitate ensued, which was then filtered, washed, dried, and weighed. The confirmation of the carbonyl compound (whether aldehyde or ketone) was performed by determining its melting point.

**3.3 Green synthesis of Silver nanoparticles**

The green synthesis of nanoparticles is an eco-friendly and sustainable alternative to conventional chemical methods, as it eliminates the use of harmful reagents and reduces environmental impact [21-23]. The silver nanoparticles were extracted from leaves of *Ziziphus jujuba* plant [21] and used in the reaction which resulted into increased in rate of reaction for alcohols (fig.1)

Fig.1. Synthesis of AgNP



To prepare AgNPs from *Ziziphus jujuba* leaves, leaves were collected locally in Mumbai, India,and carefully washed. They were dried in a hot air oven at a temperature of 65-70°C, finely crushed and 5 grams of the resultingmaterial was weighed and brought to boiled with distilled water. The solution was filtered and 2 ml of thisleaf extract was added to 100 ml of0.001M AgNO3 solution with continuous stirring on amagnetic plate. After 15-20 minutes, the formation of brown-colored AgNPs was observed. The UV spectrum showed the presence of Ag NP.

Characterization of the synthesized AgNPs was performed using UV-Vis spectra. The UV-Vis absorption graph shows a peak around 450-480 nm. This peak is characteristic of silver nanoparticles (AgNPs), indicating the presence of AgNPs synthesized using *Ziziphus jujuba* leaves (fig.2)

0

0.5

1

1.5

2

2.5

3

3.5

4

4.5

200

300

400

500

600

700

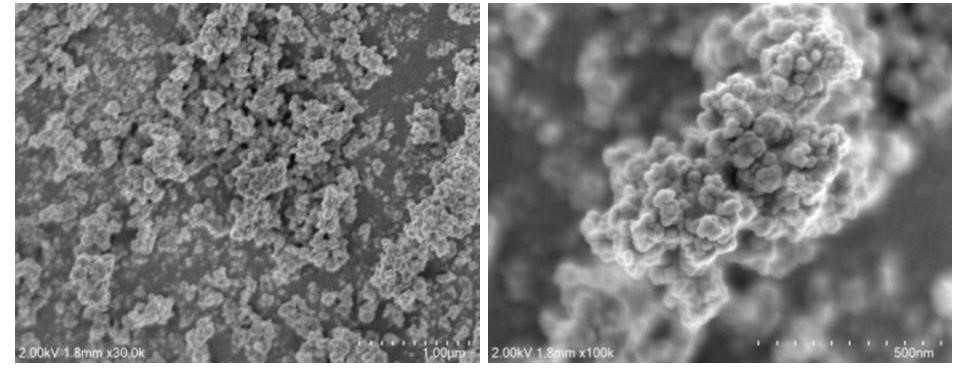
800

**Abs.**

**Wavelength (nm)**

Fig.2 UV graph of AgNP Using Ziziphus jujuba leaves

Fig.3.(a). shows a densely packed aggregation of silver nanoparticles. The particles appear to have a rough, clustered surface, indicative of the typical morphology of AgNPs synthesized through biological methods. The high surface area is beneficial for catalytic applications due to more active sites being available for reactions. The higher magnification reveals more details of the surface structure, showing individual nanoparticles and their interconnections (fig.3b).



(

a

)

(

b

)

Fig 3.(a),(b): SEM images of AgNP prepared from Ziziphus jujuba leaves

These characteristics suggest that the AgNPs will have a high surface area and numerous active sites, making them effective catalysts in chemical reactions, such as the oxidation of alcohols.

The spectrum indicates that plant-derived organic compounds, such as polyphenols, proteins, or other metabolites, might have capped the silver nanoparticles, facilitating their stabilization (fig. 4). The peak at 537 cm⁻¹ can be related to metal-oxygen bonding, suggesting interactions between the silver nanoparticles and plant-based molecules or a direct interaction with oxygen from the stabilizing compounds in the plant extract. The involvement of hydroxyl, carbonyl, and aromatic functional groups is common in green synthesis methods involving plant extracts.

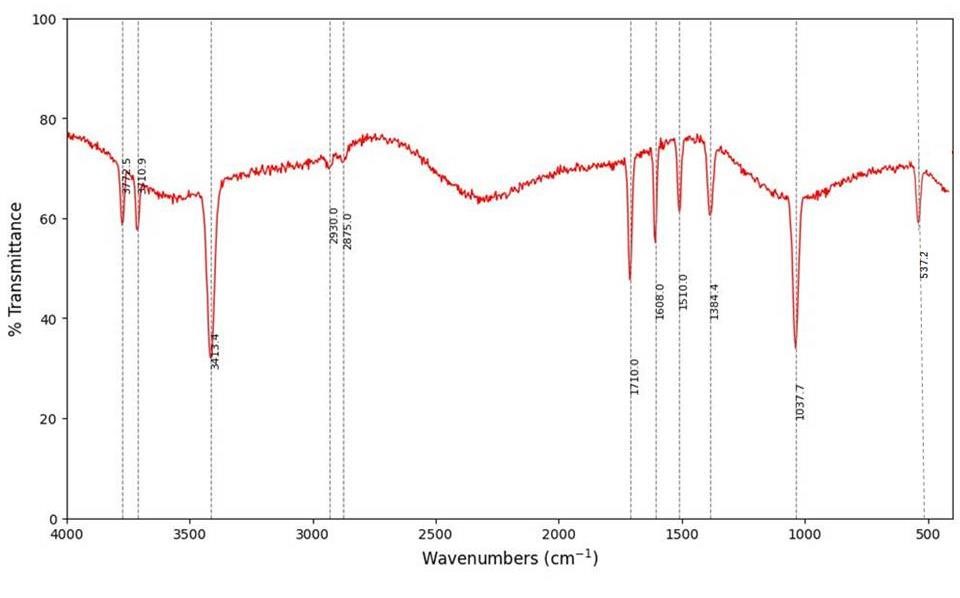


Fig.4 FTIR Spectra of AgNP derived from Ziziphus jujuba leaves

**4. RESULTS AND DISCUSSION**

**4.1 Using oxidizing agents**

The reactions exhibit first-order kinetics with respect to PCC, indicating a linear relationship between the logarithm of absorbance and time. Table 3 given below shows the increasing rate constant with increased alcohol concentration. Duplicate runs demonstrated the reproducibility of the rate constants.

Table 3: Rate constant of oxidation of alcohol using PCC and PDC reagent

|  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **[alcohol] mol dm-3** | **[oxidant] mol dm-3** |  |  |  | **k x 103 s-1** | |  |  |  |
|  |  | **PCC** |  |  |  | **PDC** |  |
| **n-butanol** | **2-chloro ethanol** | **cyclohexanol** | **Cinnamyl alcohol** | **n-butanol** | **2-chloro ethanol** | **cyclohexanol** | **Cinnamyl alcohol** |
| **SET I** |  |  |  |  |  |  |  |  |  |
| 1.00 | 2.50 | 5.56 | 8.50 | 1.61 | 0.31 | 2.90 | 4.91 | 0.75 | 0.52 |
| 1.00 | 5.00 | 4.57 | 7.01 | 0.69 | 0.28 | 2.89 | 4.62 | 0.69 | 0.50 |
| 1.00 | 10.0 | 4.32 | 6.89 | 0.46 | 0.19 | 1.83 | 2.30 | 0.64 | 0.50 |
| 1.00 | 15.0 | 3.11 | 6.32 | 0.46 | 0.11 | 0.90 | 0.46 | 0.51 | 0.16 |
| 1.00 | 20.0 | 3.06 | 5.41 | 0.23 | 0.02 | 0.21 | 0.20 | 0.46 | 0.09 |
| 1.00 | 25.0 | 3.04 | 4.22 | 0.10 | 0.02 | 0.13 | 0.16 | 0.38 | 0.06 |
| **SET II** |  |  |  |  |  |  |  |  |  |
| 0.25 | 5.00 | 4.01 | 5.29 | 1.09 | 0.04 | 0.23 | 2.76 | 0.27 | 0.06 |
| 0.50 | 5.00 | 4.38 | 6.63 | 1.19 | 0.23 | 0.46 | 3.22 | 0.41 | 0.15 |
| 0.63 | 5.00 | 4.46 | 7.91 | 1.28 | 0.29 | 0.69 | 3.45 | 0.53 | 0.20 |
| 0.75 | 5.00 | 5.57 | 8.52 | 1.62 | 0.38 | 0.92 | 3.68 | 0.60 | 0.26 |
| 0.88 | 5.00 | 5.82 | 9.60 | 1.76 | 0.41 | 1.38 | 4.37 | 0.68 | 0.28 |
| 1.00 | 5.00 | 6.17 | 9.82 | 1.98 | 0.52 | 1.94 | 5.12 | 1.01 | 0.33 |

The oxidation rate intensified with higher concentrations of alcohols. Comparing the rate constant data for aliphatic and cyclic alcohols revealed that 2-chloroethanol exhibits a higher reaction rate than n-butanol, whereas Cinnamyl alcohol oxidizes slower than Cyclohexanol for both the oxidants (Fig.5 and Fig.6). The faster oxidation of alcohols can be attributed to several factors like length of the Carbon chain, electrophilicity of atoms, steric hinderance, etc [12]. The activation energy of such alcohols is likely to be lower than that of other alcohols, enabling a faster reaction rate [14].

0

2

4

6

8

10

0.2

0.4

0.6

0.8

1

1.2

**Rate constant (k x 10**

**3**

**s**

**-**

**1**

**)**

**[**

**Alcohol] mol dm**

**-**

**3**

2-

chloroethanol

n-butanol

cyclohexanol

cinnamyl alcohol

Fig. 5 Rate constant data for oxidation of alcohols using PCC Oxidizing agent

0

1

2

3

4

5

6

0.2

0.4

0.6

0.8

1

1.2

**Rate constant (k x 10**

**3**

**s**

**-**

**1**

**)**

**[**

**Alcohol] mol dm**

**-**

**3**

2-

chloroethanol

n-butanol

cyclohexanol

cinnamyl alcohol

Fig. 6. Rate constant data for oxidation of alcohols using PDC Oxidizing agent

**4.2 Effect of ionic strength**

The use of Na2SO4 was aimed to examine the impact of altering ionic strength within the range of µ = 5 to 25 x 10-2 mol dm-3 at 313K on the oxidation rate [11]. The resulting graphs (Fig. 7 and Fig. 8), depicting log k against √µ, displayed linear trends running parallel to the √µ axis. These outcomes imply that fluctuations in ionic strength do not influence the oxidation rate. The Bronsted- Bjerrum equation was employed to ascertain the impact of ionic strength (µ) on the oxidation rate.

log k = logk0 +1.02 ZA ZB√µ

Furthermore, this assertion finds reinforcement in the underlying reaction mechanism.

Table 4- Effect of ionic strength using Na2SO4

|  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| µ×102 moldm-3 |  | **PCC** | |  |  | **PDC** | |  |
| **n-butanol** | **2-chloro ethanol** | **cyclohexanol** | **Cinnamyl alcohol** | **n-butanol** | **2-chloro ethanol** | **cyclohexanol** | **Cinnamyl alcohol** |
| 05 | 1.88 | 1.69 | 0.86 | 1.59 | 5.04 | 1.39 | 2.64 | 1.87 |
| 10 | 1.88 | 1.65 | 0.92 | 1.59 | 4.20 | 1.00 | 2.94 | 1.93 |
| 15 | 1.88 | 1.65 | 0.92 | 1.49 | 5.03 | 1.20 | 3.45 | 1.88 |
| 20 | 1.68 | 1.71 | 0.92 | 1.49 | 4.21 | 1.46 | 3.29 | 1.64 |
| 25 | 1.84 | 1.65 | 1.84 | 1.49 | 4.22 | 1.37 | 3.59 | 1.69 |

0.6

1

1.4

1.8

2.2

5

15

25

35

**logk**

**√µ**

2-

chloroethanol

n-butanol

cyclohexanol

cinnamyl alcohol

Fig.7. Effect of ionic strength on the oxidation rates of alcohols by PCC in 0.2M H2SO4 [alc.]= 0.2 mol dm-3, [PCC] = 2.5 x 10-2 mol dm-3, Temperature = 313K

1

1.5

2

2.5

3

3.5

4

4.5

5

10

15

20

25

30

**logk**

**√µ**

n-butanol

2-

chloroethanol

cyclohexanol

cinnamyl alcohol

Fig.8. Effect of ionic strength on the oxidation rates of alcohols by PDC in 0.2M H2SO4.[alc]=0.2 mol dm-3, [PDC] = 2.5 x 10-2 mol dm-3, Temperature = 313K

**4.2 Effect of temperature**

The oxidation process was investigated within the temperature range of 303-318K tabulated in Table 5 and Table 6, and an assessment of the thermodynamic activation parameters was conducted. Across all the alcohols examined, the rate constants (k) exhibited an upward trend with increasing temperature, demonstrating an inverse relationship with the energy of activation (fig. 9 and fig.10).

Table 5- Effect of temperature of PCC and PDC (303K to 318K)

|  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Temperature (K) |  |  |  | **k x 102 s-1** | |  |  |  |
|  |  | **PCC** |  |  |  | **PDC** |  |
| **n-butanol** | **2-chloro ethanol** | **cyclohexanol** | **Cinnamyl alcohol** | **n-butanol** | **2-chloro ethanol** | **cyclohexanol** | **Cinnamyl alcohol** |
| 303 | 2.101 | 4.021 | 0.819 | 0.026 | 0.928 | 1.810 | 0.296 | 0.0890 |
| 308 | 2.138 | 4.376 | 0.904 | 0.032 | 1.382 | 2.336 | 0.345 | 0.0921 |
| 313 | 2.142 | 4.603 | 0.972 | 0.038 | 1.614 | 3.614 | 0.368 | 0.0964 |
| 318 | 2.163 | 4.835 | 1.030 | 0.041 | 1.846 | 3.895 | 0.391 | 0.0982 |

Table 6- Temperature parameters of PCC and PDC

|  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **Parameters** |  |  | **PCC** | |  | | **PDC** |  |
| **n-butanol** | **2-chloro ethanol** | **cyclohexanol** | **Cinnamyl alcohol** | **n-butanol** | **2-chloro ethanol** | **cyclohexanol** | **Cinnamyl alcohol** |
| **∆E**  (kJmol-1) | 21.1 | 9.65 | 62.88 | 71.76 | 23.25 | 16.96 | 34.19 | 53.56 |
| **∆G**  (kJmol-1) | -80.03 | -93.96 | -88.19 | -83.18 | -86.07 | -90.12 | -83.31 | -86.79 |
| **∆H**  (kJmol-1) | 18.58 | 7.13 | 60.36 | 69.24 | 20.73 | 14.44 | 31.67 | 51.04 |
| **∆S**  (kJmol-1 K-1) | -0.202 | -0.286 | -0.091 | -0.046 | -0.215 | -0.249 | -0.170 | -0.118 |

-4

-3.2

-2.4

-1.6

-0.8

0

0.00314

0.00319

0.00324

0.00329

0.00334

**log k**

**1**

**/T**

n-butanol

2-

chloroethanol

cinnamyl alcohol

cyclohexanol

Fig. 9 Temperature graph of PCC

-3.2

-2.4

-1.6

0.00314

0.00319

0.00324

0.00329

0.00334

**log k**

**1**

**/T**

2-

chloroethanol

cyclohexanol

cinnamyl alcohol

n-butanol

Fig. 10. Temperature graph of PDC

**4.3 Effect of silver nanoparticle**

The rate of the reaction increased when nano catalyst was used (Table 7). From Fig.11 and Fig. 12, it is observed that the alcohols containing smaller carbon chains have higher rates than the ones which have more carbon atoms.

Table 7: Rate constant data for the alcohol oxidation by PCC and PDC in 0.2 M H2SO4 and AgNP from *Ziziphus* leaves at temperature 303K

|  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| **[alcohol] mol dm-3** | **[oxidant] mol dm-3** |  |  | | **k x 103 s-1** | |  |  |  |
|  | **PCC** | |  |  |  | **PDC** |  |
| **n-butanol** | **2-chloro ethanol** | **cyclohexanol** | **Cinnamyl alcohol** | **n-butanol** | **2-chloro ethanol** | **cyclohexanol** | **Cinnamyl alcohol** |
| **SET I** |  |  |  |  |  |  |  |  |  |
| 0.25 | 5.0 | 0.06 | 5.29 | 1.61 | 0.04 | 0.87 | 2.76 | 0.70 | 0.06 |
| 0.50 | 5.0 | 1.38 | 6.63 | 1.84 | 0.23 | 2.02 | 3.22 | 0.96 | 0.15 |
| 0.63 | 5.0 | 1.84 | 7.91 | 2.07 | 0.29 | 2.31 | 3.45 | 1.12 | 0.20 |
| 0.75 | 5.0 | 2.07 | 8.52 | 2.53 | 0.38 | 2.44 | 3.68 | 2.32 | 0.26 |
| 0.88 | 5.0 | 2.33 | 9.60 | 2.79 | 0.41 | 2.50 | 4.37 | 2.94 | 0.28 |
| 1.00 | 5.0 | 4.66 | 9.82 | 3.31 | 0.52 | 0.87 | 5.12 | 3.80 | 0.33 |

0

2

4

6

8

10

12

0.2

0.4

0.6

0.8

1

1.2

**Rate constant (k x 10**

**3**

**s**

**-**

**1**

**)**

**[**

**Alcohol] mol dm**

**-**

**3**

chloroethanol

2-

n-butanol

cyclohexanol

cinnamyl alcohol

Fig. 11 Rate constant data for alcohol oxidation using PCC and AgNP from Ziziphus leaves

0

1

2

3

4

5

6

7

0.2

0.4

0.6

0.8

1

1.2

**Rate constant (k x 10**

**3**

**s**

**-**

**1**

**)**

**[**

**Alcohol] mol dm**

**-**

**3**

2-

chloroethanol

n-butanol

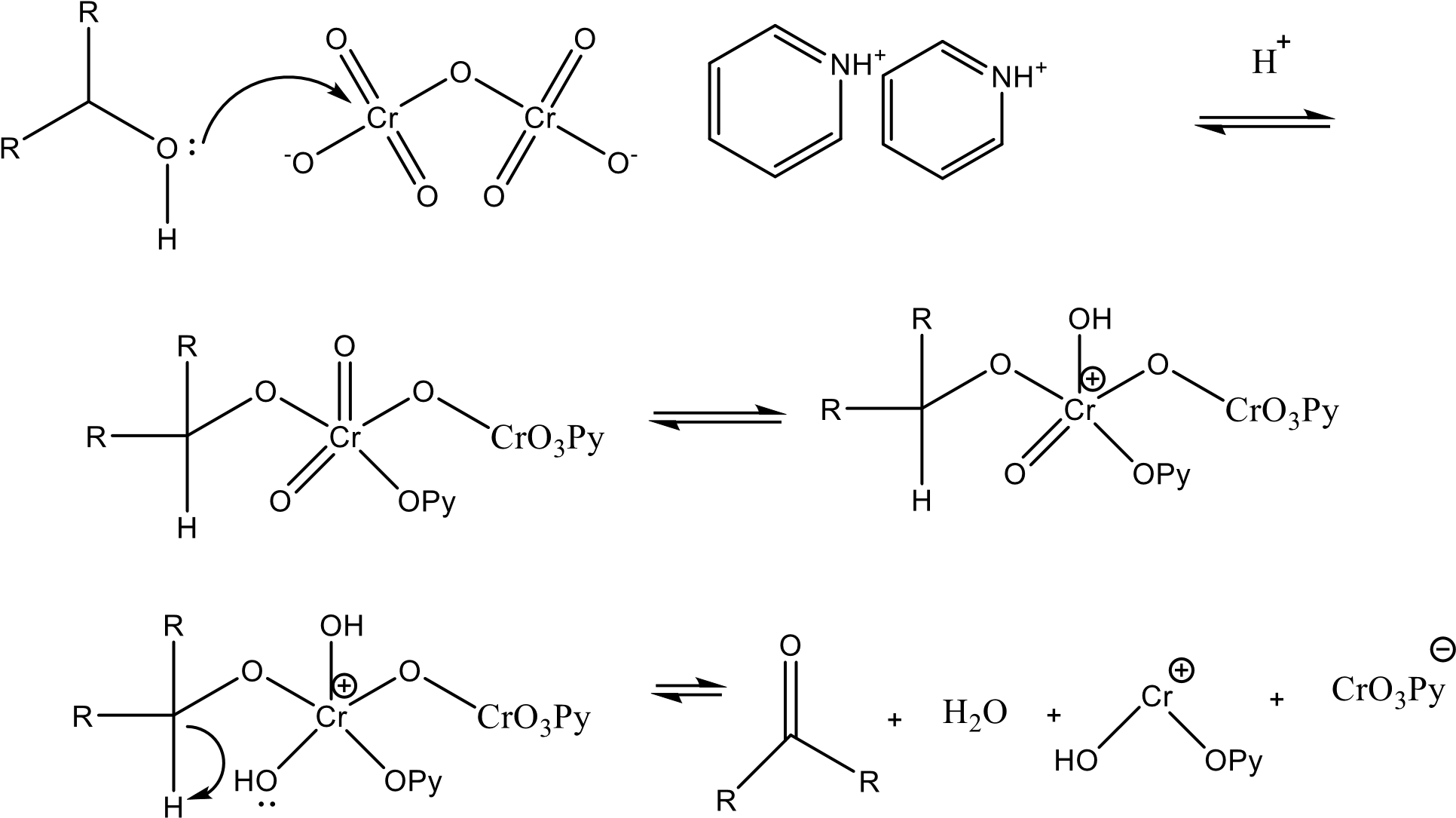
cyclohexanol

cinnamyl alcohol

Fig. 12 Rate constant data for alcohol oxidation using PDC and AgNPs from Ziziphus leaves

The concluded reaction mechanism is given below:

**PDC Mechanism**



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# **5. Conclusion**

Though PCC and PDC themselves are sufficient for the oxidation, the presence of silver can potentially improve the efficiency of oxidation of alcohols. Silver facilitates electron transfer reaction as it activates oxidant species. It stabilizes intermediates by interacting with them on their surface.

Considering the aliphatic alcohols, 2-chloroethanol oxidizes faster than n-butanol due to the electron withdrawing effect of the chlorine atom, which makes the hydroxyl group more susceptible to oxidation. This effect increases the reactivity of 2-chloroethanol towards oxidizing agents, facilitating a quicker oxidation process compared to n-butanol. While in the case of cyclic alcohols, the steric hindrance around the hydroxyl group in cinnamyl alcohol due to the aromatic ring and double bond could slow down its oxidation compared to the less hindered cyclohexanol.

**6. ACKNOWLEDGMENTS**

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**7. COMPETING INTERESTS**

Authors have declared that no competing interests exist.

**8. AUTHORS CONTRIBUTIONS**

All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication.

9. authors contribution

‘Ms. Anjali Tiwari’ designed the study, performed the statistical analysis, wrote the protocol, and wrote the first draft of the manuscript. ‘Dr. D.V. Prabhu’ and ‘Prof. H. A. Parbat’ managed the analyses of the study. All authors read and approved the final manuscript.”

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