

## Synthesis and catalytic activity of Cu (II) complex with 4-phenyl 2, 6-di (thiazol-2-yl) pyridine in the oxidation of para-aminophenol

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### ABSTRACT

In the present work the synthesis of 4-phenyl-2, 6-di (thiazol-2-yl) pyridine copper chloride (abbreviated as Cu-DTPy) complex using copper (II) chloride and 2, 6-di (thiazol-2-yl) pyridine has been carried out. The complex has been specified by UV-Vis, FT-IR, CHNS and ESI-MS methods of analysis. The synthesized copper complex has been used as catalyst in the oxidation of *p*-aminophenol using hexacyanoferrate (III) ions as an oxidant in aqueous alkaline medium by kinetic-spectrophotometric method. This experimental work was carried out at optimum pH 11.0 and temperature 25°C. The variation of effect of concentration of *p*-aminophenol (abbreviated as PAP), hexacyanoferrate (III) ions, and Cu-DTPy catalyst on reaction rate has been studied. Thermodynamic parameters such as free energy of activation, activation energy, enthalpy and frequency factor have been evaluated with the change in rate of the reaction at four disparate temperatures i.e. 25-40°C. The oxidation products have been identified by FT-IR and ESI-MS spectroscopic analytical techniques. The major product of this reaction is NN'-bis-(*p*-hydroxyphenyl)-2-hydroxy-5-amino-1,4-benzoquinone di-imine. Thus, this method is highly efficient and environmentally safe to remove PAP from polluted water resources.

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## 1. Introduction

During the past few decades, transition metals<sup>1</sup>, and transition metal complexes<sup>2</sup> mediated catalytic oxidation of organic substrates with different oxidants have fascinated much interest of researchers<sup>3</sup>. In chemical industries, there are many applications of these oxidative transformation reactions such as removal of antibiotics from water, eradication of heavy metals and industrial waste water treatment etc. Now a days, several metal complexes are used as highly efficient catalyst as 2-hydroxy-1-naphthaldehyde Schiff base derived nickel(II) complex and metal complex of Ru<sup>3+</sup>, Pd<sup>2+</sup> and Ni<sup>2+</sup> with 2-hydroxy-1-naphthaldehyde(H<sub>2</sub>L)<sup>4</sup>.

Copper (II) complexes based terpyridine and their derivatives are special to chemists for investigation, owing to their impending applications<sup>5</sup>. The drainage of contaminated industrial water causes water pollution<sup>6</sup>. A well-known hazardous pollutant in the environment is *p*-aminophenol PAP<sup>7</sup>. It is a raw material in many industrial products<sup>8</sup> such as petroleum, rubber, dye, medicine and photographic etc<sup>9</sup>. At large scale, in the formation of many commercial permanent hair dyes *p*-aminophenol is used as a precursor<sup>10</sup>. Paracetamol tablet is an anti-pyretic drug, after expiry date degrades into PAP<sup>11</sup>. Skin, eye and respiratory system irritation<sup>12</sup>, bronchial asthma, dermatitis, headaches and severe allergies are caused by the exposure of *p*-aminophenol<sup>13</sup>. For both animal and environment, *p*-aminophenol is a major environmental remediation issue due to its toxicity<sup>14</sup>.

Many traditional methods such as electro-chemical oxidation, adsorption, ozonation, coagulation, ion-exchange and flocculation etc are used for the eradication of organic pollutants, but high cost, sludge formation, time consuming and operationally tuff are some obstacles in these methods<sup>15</sup>. In this present study the work is carried out on remediation of PAP by catalytic oxidative degradation method using hexacyanoferrate (III) ions as oxidant in aqueous alkaline medium. Cu-DTPy complex is used as a catalyst which is synthesized by reported method.

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## 2. Results and discussion

### 2.1. Synthesis of Ligand (4-phenyl-2, 6-di (thiazol-2-yl) pyridine)

“2-Acetylthiazole (0.128 gram, 1.0 mmol) was added into a solution of benzaldehyde (0.053 gram, 0.5 mmol) in ethyl alcohol (10 ml). KOH pellets (0.077 gram, 1.0 mmol) and  $\text{NH}_3$  (aqu., 0.149 ml, 25%, 2.0 mmol) were added to the mixture of 2-acetylthiazol and benzaldehyde<sup>16</sup>”. After stirring at room temperature (28°C) for 6 hours, a solid (silver-white) was collected by filtration process and washed with ethyl alcohol. The yield was about 60%. The ligand was used for the synthesis of copper complex without further purification. Synthesized ligand was characterized by CHNS determinator, FTIR and ESI-MS method of analysis. Analytically calculated ratio of CHNS in ligand for molecular formula  $\text{C}_{17}\text{H}_{11}\text{N}_3\text{S}_2$  is (%) C, 63.53; H, 3.45; N, 13.07; which are similar to observed values (%)C, 63.748; H, 3.444; N, 13.052. ESI-MS ( $\text{CH}_3\text{OH}$ )  $m/z$ : 322 $[\text{M}+\text{H}]^+$ . FTIR: (3414), (2923), (2855), (1744), (1581), (1369) and (1207) also confirms the synthesis of ligands.

### 2.2 Synthesis of Cu-DTPy complex

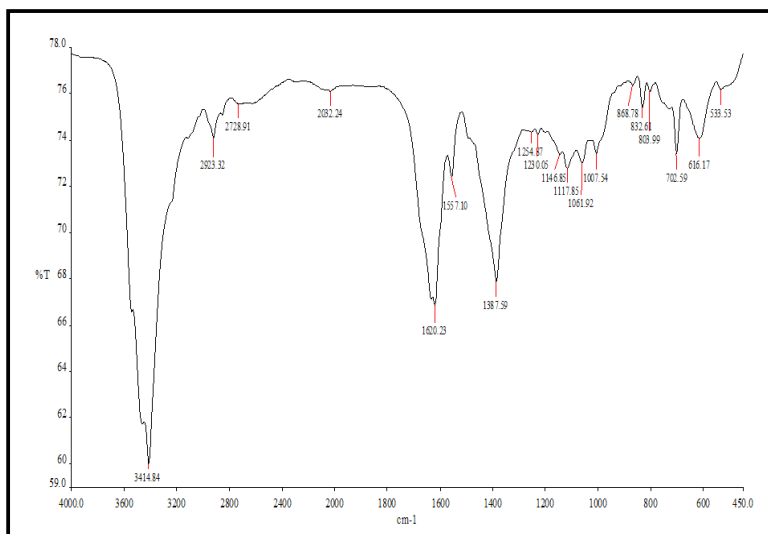
The solution of methanol (10 ml) and  $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$  (0.017 gram, 0.1 mmol) was added drop by drop into a hot methanolic solution of ligand 4-phenyl-DTPy (0.0322 gram, 0.1 mmol). After stirring the solution for 4-5 hours at 50°C, the solution turned clear green. The resulting solution was filtered while it was hot, and then filtrate was left unperturbed in air to allow slow evaporation. Dark-green crystals were obtained after one week. The yield was approx. 80%. Again the 4-phenyl-DTPy complex was characterized by CHNS determinator, FTIR and ES-MS method of analysis. Analytically calculated ratio of CHNS for molecular formula  $\text{C}_{17}\text{H}_{11}\text{N}_3\text{S}_2\text{Cl}_2\text{Cu}$  is (%) C, 44.79; H, 2.43; N, 9.22; S, 14.07, found (%)C, 44.10; H, 2.31; N, 9.24; and S, 14.0 respectively. ESI-MS ( $\text{CH}_3\text{OH}$ )  $m/z$ : 421  $[\text{M}-\text{Cl}]^+$ , FTIR: (3322), (2944), (2832), (1660), (1450) and (1020) also confirm the synthesis of Cu-DTPy complex.

### 2.3. FT-IR

FT-IR techniques help to verify the existence of molecules and functional groups in the ligand and complex.

#### FT-IR of Ligand

The IR spectra of ligands had peaks in the region 3414.84  $\text{cm}^{-1}$ , 2032.24  $\text{cm}^{-1}$ , 1620.23  $\text{cm}^{-1}$ , 1061.92  $\text{cm}^{-1}$ , 702.59  $\text{cm}^{-1}$ . The peak lies 3414.84  $\text{cm}^{-1}$  indicating the region of secondary amine. 2032.24  $\text{cm}^{-1}$  peak showing bond (N=C=S), and 1620.23  $\text{cm}^{-1}$  peak in region (-CN) group. Peak at the 1061.92  $\text{cm}^{-1}$  falls in (-C=S) group and peak at the 702.59  $\text{cm}^{-1}$  is showing meta substituted aromatic compounds.

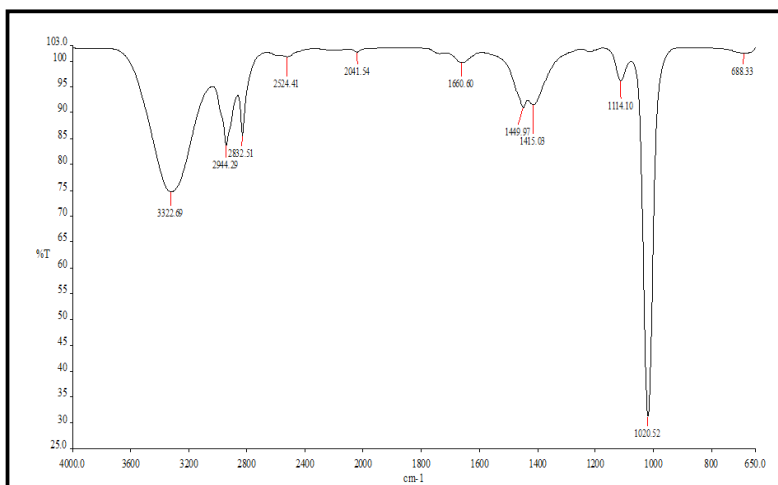


**Fig. 1.** FT-IR Analysis of Ligand

#### FT-IR of Cu-Complex

The peaks present in the ligand are also shown in complex with some shifting such as peak at 3414.84  $\text{cm}^{-1}$  shifts to 3322.69  $\text{cm}^{-1}$ , peak of (N=C=S) group shifted from 2032.24 to 2041.54 and 1620.23  $\text{cm}^{-1}$  for (-CN) group in ligand is now at 1660.60

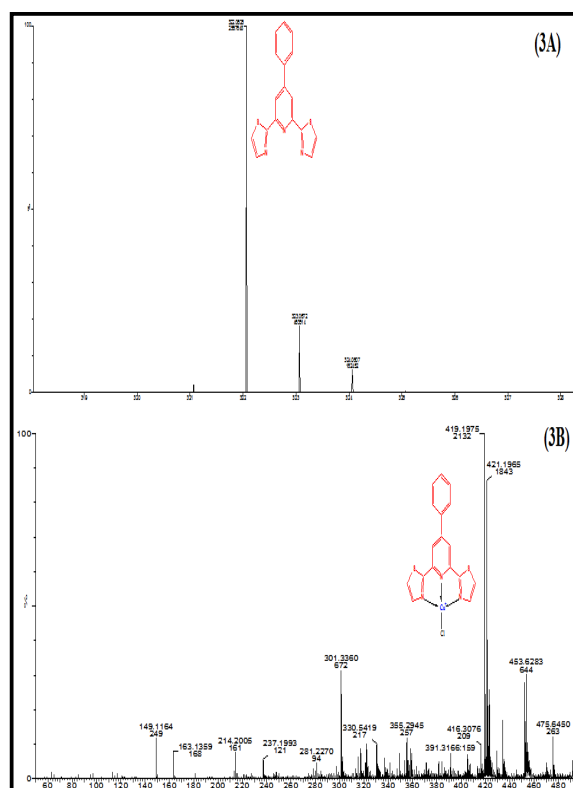
$\text{cm}^{-1}$ . A sharp peak develops in complex at  $1020.52 \text{ cm}^{-1}$  for ( $-\text{C}=\text{S}$ ) while in ligand it is at  $1061.92 \text{ cm}^{-1}$  (a small peak). Peak showing the aromatic meta substitution in the complex also shifted from  $702.59 \text{ cm}^{-1}$  to  $688.33 \text{ cm}^{-1}$ .



**Fig. 2.** FT-IR Analysis of Cu-complex

#### 2.4. ESI-Mass spectroscopy of Ligand and Cu-complex

ESI-Mass spectroscopy (ESI-MS) is a technique which investigates the compound by its  $m/z$  values. In mass spectra of ligand and complex **Fig. 3A** shows at the  $322.06 \text{ m/z}$  value showing the formation of ligand DTPy. **Fig. 3B** peak at  $421.19$  shows DTPy- $\text{CuCl}_2$  complex.



**Fig. 3.** Mass spectra of Ligand (3A) and complex (3B)

Based on the above results following structural formula has been proposed for DTPy- $\text{CuCl}_2$  complex.

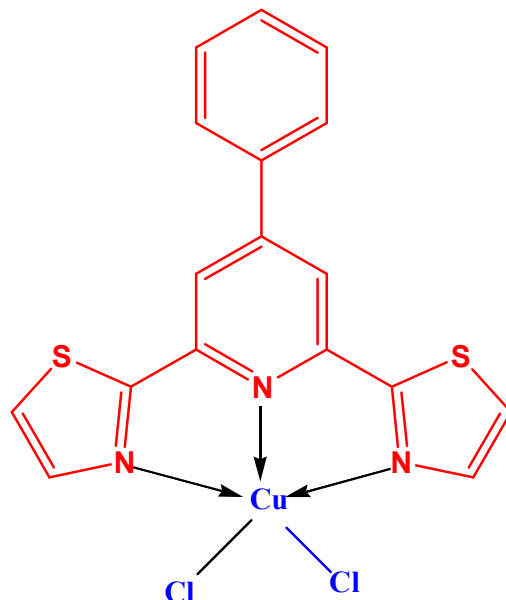


Fig. 4. Proposed structure of 4-phenyl-2, 6-di (thiazol-2-yl) pyridine copper chloride

### 3. Catalytic activity of Cu-DTPy in the oxidation of p-aminophenol by hexacyanoferrate (iii) ion

The catalytic activity of DTPy-CuCl<sub>2</sub> complex in the oxidation of PAP by alkaline HCF (III) was done at optimum pH 11 and fixed temperature 25±0.1°C in a thermostatic water bath with an electronically controlled magnetic stirrer. The study was made using the kinetic-spectrophotometric method at  $\lambda$  max 415 nm. For all the experiments 100 ml iodometric flasks were used. Buffer solution of KH<sub>2</sub>PO<sub>4</sub> and NaOH was used to adjust to the pH at desirable value by pH meter. The reactions were initiated by adding the required amount of HCF (III) ions, p-aminophenol and catalyst were varied 10 times from its initial value. A double beam spectrophotometer was used for monitoring the progress of oxidation of p-aminophenol at various time intervals.

#### 3.1. Effect of pH

The variation of pH is shown in Fig. 5. It was studied in the range of pH 8.0 to 12.0 for both the catalysed and uncatalysed reaction. Results show that the optimum pH for the oxidation reaction of PAP by alkaline HCF(III) is 11 either catalysed or uncatalysed reaction.

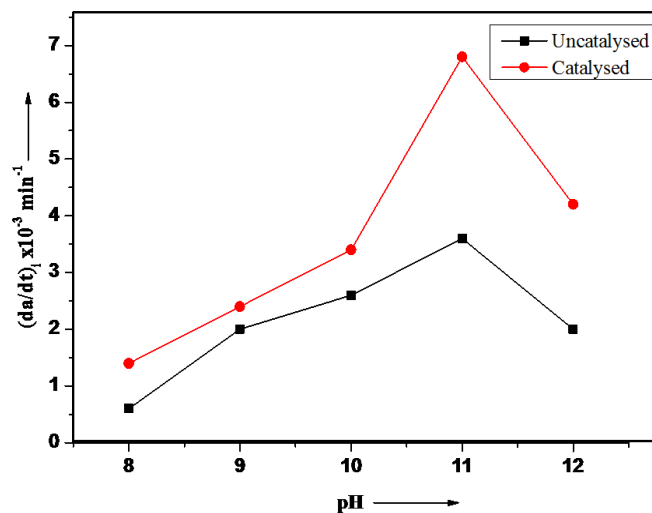


Fig. 5. Effect of pH on the oxidation of p-aminophenol. Temp. 25°C,  $\lambda$  max 415 nm, [HCF(III)]  $3 \times 10^{-4}$  mol/dm<sup>3</sup>, [PAP]  $3 \times 10^{-5}$  mol/dm<sup>3</sup>

### 3.2. Effect of concentration of HCF (III) ion

To analyze the effect of oxidant [HCF (III)] ions on the oxidation of p-aminophenol, the pH, substrate concentration [p-aminophenol], and temperature were kept stable and the concentration of HCF (III) ions was varied from  $1 \times 10^{-4}$  to  $9 \times 10^{-4}$  mol/dm<sup>3</sup>. The outcomes given in Fig. 6 represented that rate of reaction of substrate enhances linearly for both uncatalyzed and catalyzed reaction on enhancing the concentration of oxidant.

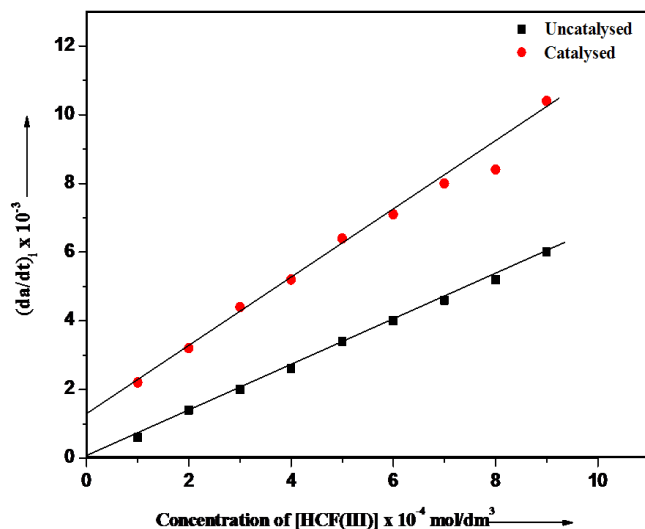


Fig. 6. Effect of [HCF (III)] on the rate of oxidation of p- aminophenol. Temp. 25°C,  $\lambda$  max 415 nm, pH 11, [PAP]  $3 \times 10^{-5}$  mol/dm<sup>3</sup>

### 3.3. Effect of concentration of para-aminophenol

The effect of variation of p-aminophenol concentration on rate of its oxidation by HCF (III), as shown in Fig. 7, was studied from  $1 \times 10^{-5}$  to  $9 \times 10^{-5}$  mol/dm<sup>3</sup>. The other parameters were kept constant throughout the study. Results demonstrate that increase in the concentrations of p-aminophenol increases the rate of reactions continuously. A straight-line plot is obtained in both uncatalyzed and catalyzed oxidation reaction showing first order kinetics with respect to substrate concentration both in catalysed and uncatalysed reaction.

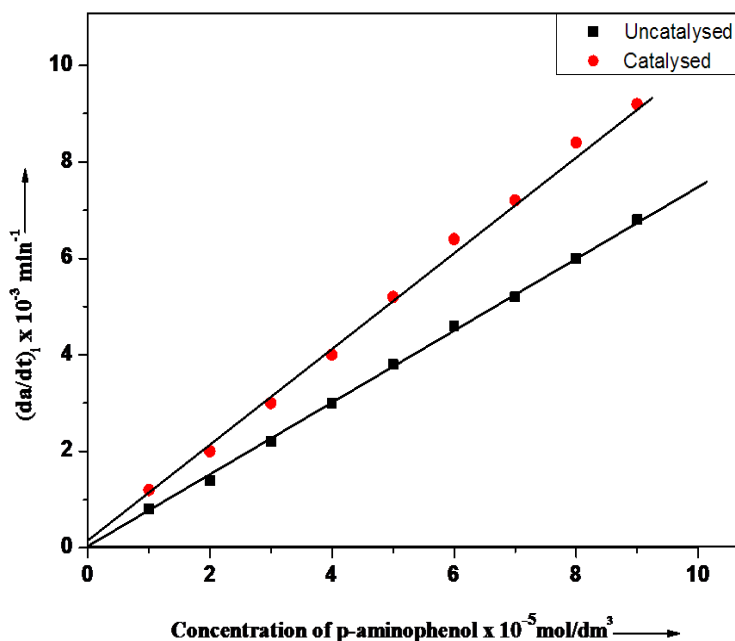
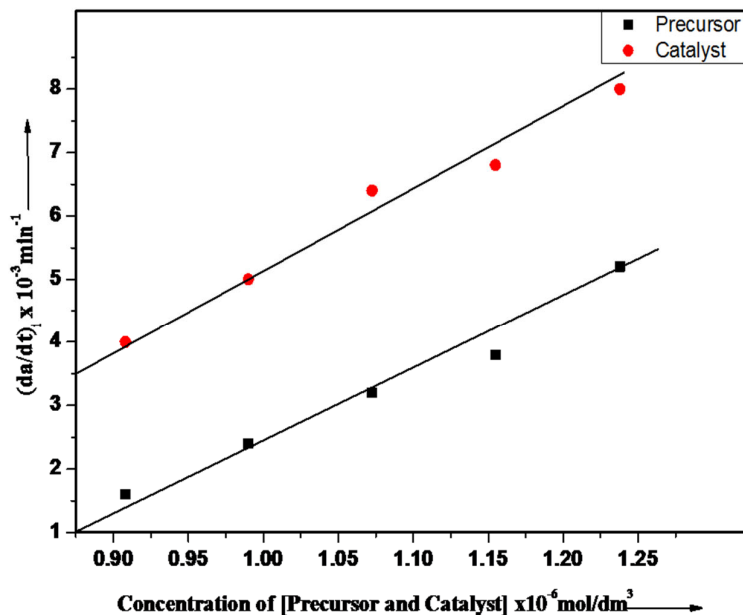


Fig. 7. Effect of concentration of p-aminophenol on its rate of oxidation. Temp. 25°C,  $\lambda$  max 415 nm, pH 11, [HCF(III)]  $3 \times 10^{-4}$  mol/dm<sup>3</sup>

### 3.4. Effect of concentration of catalyst

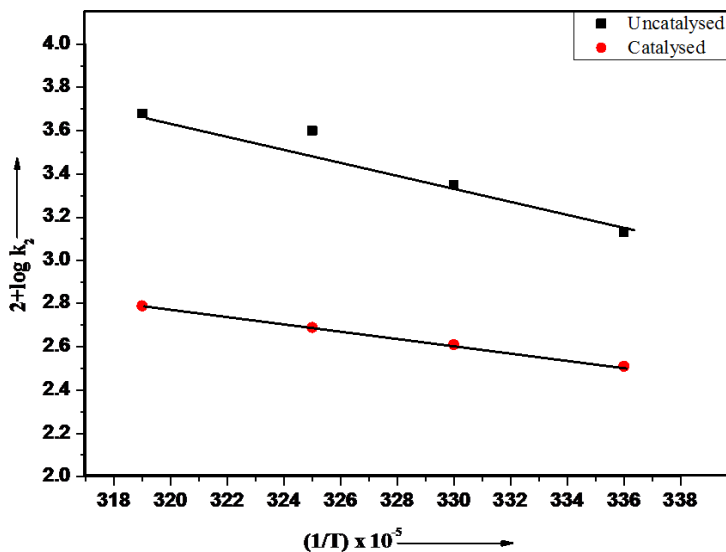
The variation of concentration of catalyst and precursor from  $0.908 \times 10^{-6}$  to  $1.1238 \times 10^{-6}$  mol/dm<sup>3</sup> are shown in **Fig. 8**. The rate of reaction increases linearly to indicate first order of reaction.



**Fig. 8.** Effect of concentration of Catalyst and Precursor on the rate of oxidation. Temp. 25°C,  $\lambda$  max 415 nm, pH 11, [HCF(III)]  $3 \times 10^{-4}$  mol/dm<sup>3</sup>, [PAP]  $3 \times 10^{-5}$  mol/dm<sup>3</sup>

### 3.5. Effect of temperature

To analyze the variation of temperature as shown in **Fig. 9**, reaction has been studied at four different temperatures i.e. 25-40°C. The Arrhenius plot between reaction rate and  $1/T$  is used for calculating activation energy ( $E_a$ ), entropy ( $\Delta S^\ddagger$ ), frequency factor ( $A$ ), free energy of activation ( $\Delta F^\ddagger$ ) and enthalpy ( $\Delta H^\ddagger$ ).



**Fig. 9.** Effect of Temperature on the oxidation of p-Aminophenol. pH 11,  $\lambda$  max 415 nm, [HCF(III)]  $3 \times 10^{-4}$  mol/dm<sup>3</sup>, [PAP]  $3 \times 10^{-5}$  mol/dm<sup>3</sup>.

### 3.5.1 Calculation of thermodynamic parameters

Thermodynamic parameters including activation energy ( $E_a$ ), enthalpy of activation ( $\Delta H^\ddagger$ ), pre-exponential factor ( $A$ ), energy of formation ( $\Delta F^\ddagger$ ) and entropy of activation ( $\Delta S^\ddagger$ ) for the oxidation of p-aminophenol by HCF(III) ions in presence of catalyst have been evaluated using Arrhenius equation and plot (Fig. 9). The data reveals low energy of activation for catalyzed reaction as compared to uncatalyzed reaction. It reveals good catalytic activity of copper complexes of 4-phenyl 2, 6-di (thiazol-2-yl) pyridine. The reaction was characterized by a more negative value of entropy ( $S^\ddagger$ ) of activation that shows the formation of polar species during the reaction. Positive value of enthalpy of activation ( $\Delta H^\ddagger$ ) shows reaction is endothermic because the enthalpy of the product is greater than the enthalpy of the reactant. The Arrhenius equation is mentioned as follows:

$$K = A \cdot e^{E_a/RT} \quad (1)$$

where;  $k$  is the rate constant,  $T$  is the absolute temperature,  $A$  is Arrhenius factor,  $E_a$  is activation energy,  $R$  is universal gas constant.

**Table 1.** Calculation of thermodynamic parameters

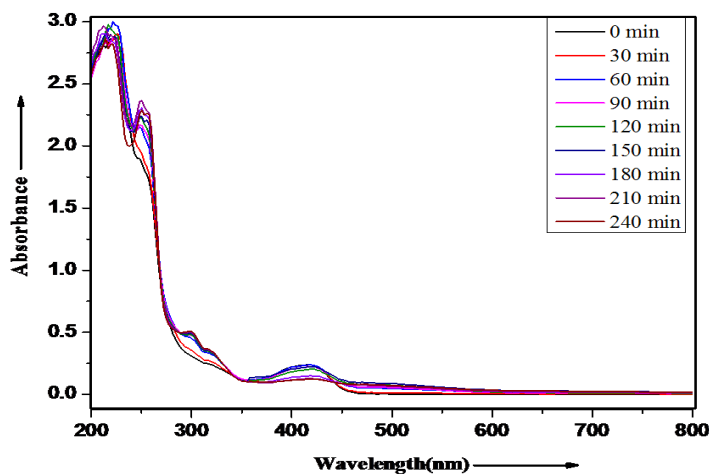
S. No	Thermodynamic Parameter	Uncatalyzed Values	Catalyzed Values
1	$E_a$ (kcal/mol)	14.6	7.32
2	$A$ (l/mol sec)	$2.77 \times 10^{11}$	$7.67 \times 10^5$
3	$\Delta S^\ddagger$ (e.u.)	-10.1	-34.4
4	$\Delta H^\ddagger$ (kcal/mol)	14.0	6.72
5	$\Delta F^\ddagger$ (kcal/mol)	17.1	17.2

## 4. Separation and identification of product

After completion of reaction i.e. 24 hours the reaction mixture was extracted using ethyl acetate and the resulting organic layer of resultant was separated and concentrated. Chromatographic separation was carried out based on the literature<sup>17</sup> with mobile phase of methanol, ethyl acetate and chloroform under composition ratio 2:2:6 (v/v/v) respectively<sup>13</sup>. Then the product was analyzed by ESI-MS, UV-Vis, and FTIR methods of analysis.

### 4.1. UV-Vis spectroscopy of product

The monitoring of reaction progress was done by the double-beam UV-Vis spectrophotometer. Fig. 10 shows that with the passage of time  $\lambda_{max}$  of the reaction mixture has been shifted from 415nm to 305nm showing the formation of some new product. The peak present at 415 nm completely disappears within about 4 hours.

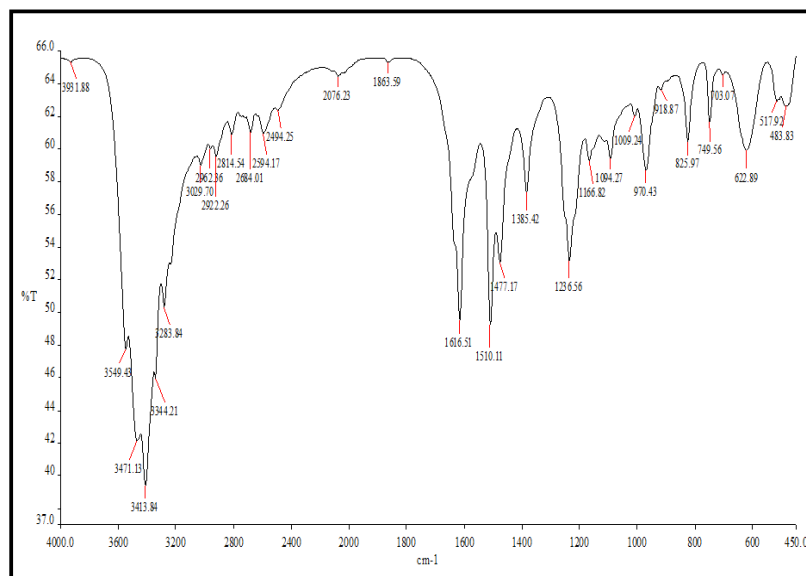


**Fig. 10.** UV-Vis spectra of PAP oxidation reaction

### 4.2. FT-IR of Product

The peaks present at different regions explain the existence of functional groups in FT-IR spectra of products. The peak region are  $3549.43 \text{ cm}^{-1}$ ,  $3413.84 \text{ cm}^{-1}$ ,  $1616.51 \text{ cm}^{-1}$ ,  $1510.11 \text{ cm}^{-1}$ ,  $1385.42 \text{ cm}^{-1}$ ,  $1236.56 \text{ cm}^{-1}$ ,  $1166.82 \text{ cm}^{-1}$ ,  $825.97 \text{ cm}^{-1}$ ,  $622.89 \text{ cm}^{-1}$ . The peak at  $3549.43 \text{ cm}^{-1}$  is showing internally bonded and a broad peak at  $3413.84 \text{ cm}^{-1}$  (-OH) group stretching. Peak at  $1616.51 \text{ cm}^{-1}$  it falls in the region of secondary amine. Aromatic ring stretching shows at  $1510.11 \text{ cm}^{-1}$ .

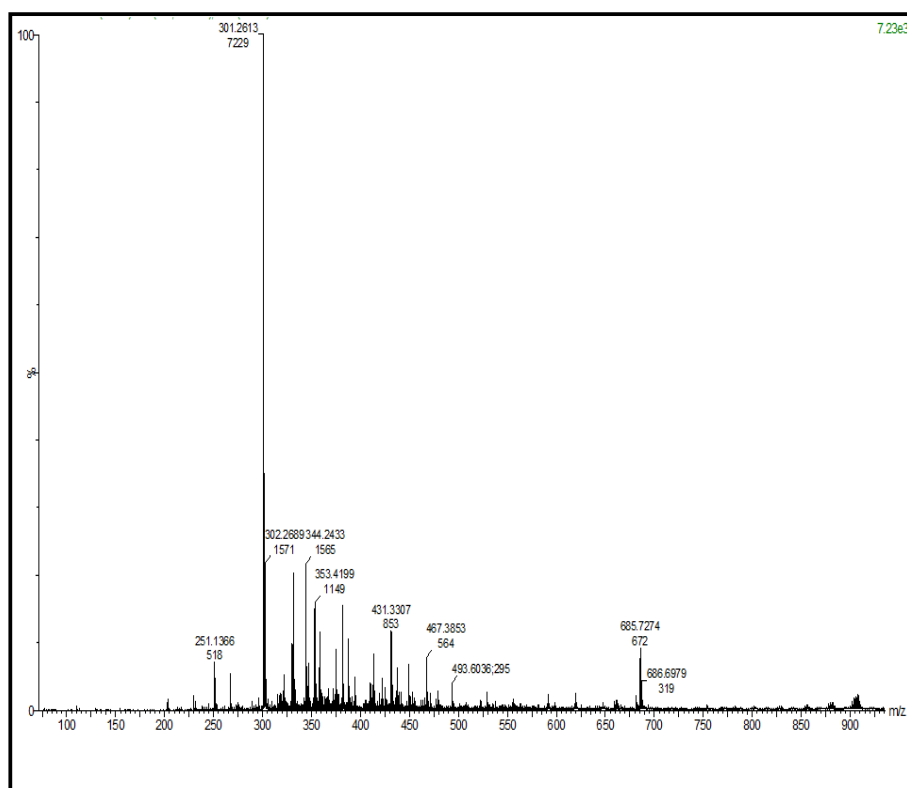
1236.56  $\text{cm}^{-1}$  peak is indicated to the aromatic ether group. Peak at 1385.42  $\text{cm}^{-1}$  and 1166.82  $\text{cm}^{-1}$  is for stretching of tertiary amine. A peak present at the 825.97  $\text{cm}^{-1}$  is falls in the region of para substituted aromatic group. The bending of the (-OH) group is represented by the peak at 622.89<sup>18</sup>  $\text{cm}^{-1}$ .



**Fig. 11.** FT-IR Analysis of Product

#### 4.3. ESI-Mass spectroscopy of product

The mass spectra of products given below in **Fig. 12** show only one major peak corresponding to  $m/z$  value of 301, which may be attributed to the NN'-bis-(p-hydroxyphenyl)-2-hydroxy-5-amino-1,4-benzoquinone di-imine which is an oxidized form of trimer of PAP with the loss of one water molecule<sup>19</sup>.

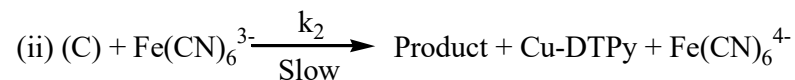
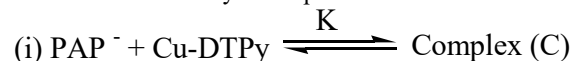


**Fig. 12.** Mass spectra of Product



#### 4.4. Proposed Mechanism for oxidation of PAP

The kinetic study and reported work<sup>20</sup> show that the reaction proceeds through complex formation as follows,



In the above mechanism, it is assumed that in aqueous basic medium p-aminophenol exists as anion (PAP<sup>-</sup>), which slowly forms a loosely bonded complex (C) with Cu-DTPy catalyst. This complex (C) slowly reacts with HCF (III) ions converting into a product along with Cu-DTPy and Fe(CN)<sub>6</sub><sup>4-</sup>.

On the basis kinetic study and above mechanism, following rate law is derived as follows,

$$r = k_2 K [\text{PAP}] [\text{Cu-DTPy}] [\text{Fe(CN)}_6^{3-}]^3$$

where K = equilibrium constant

k<sub>2</sub> = Rate constant

## 5. Conclusion

We have concluded that the synthesis of the copper complex of 4-phenyl 2, 6-di (thiazol-2-yl) pyridine was monitored spectrophotometrically. The results presented in **Fig. 1**, **Fig. 2** and **Fig. 3** show that FTIR and Mass spectra are in favour of synthesis. The catalytic oxidative kinetics of p-aminophenol follows a first order kinetic model with respect to p-aminophenol, catalyst and HCF (III) ions at constant temperature 25°C with optimum pH 11. In a strong alkaline medium, p-aminophenol is oxidized into its trimer and tetramer. In **Fig. 12** the mass spectra we get 301 which represents the oxidized form of trimer (with loss of water molecule) p-aminophenol. Basically we know that the trimers and tetramers cannot be produced directly without its monoamine (m/z 108) and diphenylamine derivative (dimer, m/z 217.2). So we conclude that both these were formed in the reaction mixture and consumed to form trimers and tetramers. The tetramers are precipitated out and filtered from the reaction mixture<sup>19</sup>.

The major product formed by oxidation of PAP is NN'-bis-(p-hydroxyphenyl)-2-hydroxy-5-amino-1, 4-benzoquinone di-imine an oxidized form of its trimer with loss of water molecule. Thus, this method is efficient, easier in handling, and environmentally friendly to removal of p-aminophenol from water bodies. Based on kinetics and the product analysis of the reaction a possible pathway has been proposed. Thus, the results reported here are of significance in understanding the oxidation of p-aminophenol.

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## 6. Experimental

### 6.1. Method

The synthesis of catalyst 4-phenyl-2, 6-di (thiazol-2-yl) pyridine (abbreviated 4-phenyl-DTPy) ligand has been carried out by Kröhnke pyridine synthesis (Ring forming reaction) method. Now the ligand is reacted with CuCl<sub>2</sub>.2H<sub>2</sub>O to form Copper-4-phenyl-DTPy complex by chelation reaction. The synthesized ligand and Cu-DTPy complex was characterized by CHNS, ESI-MS, UV-Vis and FT-IR methods of analysis. The synthesized Cu-DTPy complex has been used as catalyst in the oxidation of p-aminophenol using hexacyanoferrate (III) ions in aqueous alkaline medium by kinetic spectrophotometric method of analysis. The effect of different parameters on the change in reaction rate such as concentration of [HCF (III)], [p-aminophenol] and pH of solution have been studied at pH 11.0 and temperature 25°C. The impact of temperature was examined at pH 11 within the range 298-313K and the corresponding enthalpy (ΔH<sup>#</sup>), activation energy (E<sub>a</sub>), entropy (ΔS<sup>#</sup>), frequency factor (A) and free energy of activation (ΔF<sup>#</sup>) have been calculated. The FTIR and ESI-Mass spectroscopy techniques are used to confirm the oxidation of para-aminophenol.

### 6.2. Instruments and Reagents

2-acetylthiazol was purchased from Sigma-Aldrich. p-aminophenol was purchased from CDH (Central Drug House (P) Ltd, New Delhi, India) and used as received. The chemicals and reagents used during this work were analytical grade. Systronics μ-pH System 361 model digital pH meter was used to measure the pH of reaction mixture. Systronics Double-beam spectrophotometer-2203 was used to note λ max of reaction in the range of 200-800. FT-Infra Red spectrophotometer

(model no. RZX Perkin Elmer) instrument was used to record the FT-IR spectra of the ligand, complex and oxidized product of p-aminophenol in the range of 4000-450 cm<sup>-1</sup> using the KBr cell and nujol mull method technique. The mass spectra were measured by LC-MS spectrophotometer (Q-ToF Micro Waters model) equipped with electrospray ionization (ESI). All the experiments were done by de-ionized water. Freshly prepared solutions were used in all experimental work.

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