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Synthesis, Spectroscopic Investigation and Catalytic Behaviour of Histidine Substituted Nickel(II) Phthalocyanine

K.R. VENUGOPALA REDDY[1,*], T.M. SHARANAKUMAR[1,2], MOUNESH[1], N.Y. PRAVEENKUMAR[1], SURESH[2] and N.H.M NANDINIBABY[3]

[1]Department of Studies and Research in Chemistry Vijayanagara Sri Krishnadevaraya University, Bellary-583105, India
[2]Department of Chemistry, Ballari Institute of Technology and Management, Jnana Gangotri Campus, Bellary-583104, India
[3]Department of Physics, Ballari Institute of Technology and Management, Jnana Gangotri Campus, Bellary-583104, India

*Corresponding author: E-mail: venurashmi30@gmail.com

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In this work, the oxidation behaviour of phenol and chlorophenol contaminants present in the water catalyzed by histidine substituted nickel(II) phthalocyanine (NiPc) is reported. This work comprises synthesis, characterization and study the catalytic behaviour of histidine substituted NiPc on the oxidation of phenol and their substrates like 2- and 4-chlorophenols with 4-aminoantipyrine utilizing molecular oxygen. The products from the oxidation can be analyzed by UV-Vis spectroscopy and the results confirmed the formation of superoxide anion radical were crucial for the formation of product and a feasible mechanism including a successive transfer of single electron from phenolic compounds to O_2 through the axis of histidine Ni(II)Pc was reported.

Keywords: Histidine, Nickel(II) phthalocyanine, 4-Aminoantipyrine, Phenol, Chlorophenol, 2,4-dichlorophenol, Catalytic oxidation.

INTRODUCTION

Chlorophenols, which are employed as intermediates in the synthesis of pesticides, herbicides, fungicides, paper industries and dyes are the one of the most abundant environmental contaminants [1-5]. Contamination with numerous phenol substrates is observed in drinking water and industrial wastewaters, and toxic compounds are found in the atmosphere [6,7]. Presently, several methods are used for the estimation and analysis of phenol pollutants in soil or water. An estimation method involves the oxidation of phenol substrate with 4-aminoantipyrine (4-AAP) to form a pink antipyrilquinoneimine dye. This catalytic oxidation reaction (COR) can be catalyzed through many catalysts [8-12]. The reactive ability and nature of catalysts directly influences its sensitivity and accuracy. With 4-aminoantipyrine, phenol substrates undergo oxidation through molecular oxygen [13]. Molecular oxygen is readily available, eco-friendly, presents outstanding oxidative behaviour and has led to green chemistry development [14]. 4-Aminoantipyrine is a metabolite of antipyrine, which serves as anti-inflammatory and antipyretic agents [15]. 4-Aminoantipyrine causes cations and anions to become detectable in the chemosensing method [16-18]. The products of 4-aminoantipyrine can be highly effectively used to estimate phenols, phenol substrates, glucose contents in phenols, uric acid and peroxidase in hydrogen peroxide [19].

The complexes of metal phthalocyanine (MPc) exhibit low environmental hazards, show an excellent catalytic activity, and present critical applications dye industries. MPcs have stable higher oxidation states and electro-active central metal ions; thus, they are important in chlorophenol COR. MPcs assist in numerous oxidation reactions, such as nitrile and nitrate oxidation [20], L-cysteine oxidation [21], molecular O_2 reduction [22], olefin oxygenation [23] and water electrolysis [24]. Few researchers [25,26] have reported catalyst applications to acquire a substitute for activators and found the means to conduct reactions at low temperatures with less reaction time.

In this study, the HisNiPc complex was synthesized through a reaction between histidine, tetracarboxylic acid and Ni(II) phthalocyanine (NiTcPc). The FTIR, UV-visible, elemental analysis results supported HisNiPc formation. The thermal stability of the complex was studied using thermogravimetric estimation and its molecular morphology was investigated through PXRD. The HisNiPc was used for the first time in the catalytic oxidation reaction (COR) of phenol contaminant substrates and phenol in water. Phenolic compounds, including phenol, *p*-chlorophenol (*p*-CP), *o*-chlorophenol (*o*-CP) and 2,4-dichlorophenol (DCP), readily undergo oxidation by using dissolved oxygen in the presence of prepared catalyst, HisNiPc. Phenol substrates vigorously react with 4-aminoantipyrine to produce a pink dye. The catalytic oxidation reaction (COR) and their products were investigated through UV-visible spectroscopy. The results confirmed the chromogenic recognition of phenolic pollutants.

EXPERIMENTAL

All chemical compounds, solvents and reagents were of high grade and used without purification (Sigma-Aldrich, India). The reaction was performed under the nitrogen atmosphere. Some chemicals were prepared in oxygen-free distilled water. Nickel(II) phthalocyanine (NiTcPc) was synthesized by known procedure [27,28]. The FT-IR spectra have been recorded with a Perkin-Elmer 1600 FT-IR spectrophotometer using KBr pellets. Electronic spectra were recorded on a Perkin-Elmer Lambda 25 spectrophotometer in DMF. The XRD spectra was measured by Bruker D8 diffractometer Cu*K*α radiation source. Thermal stability of the synthesized HisNiPc was analyzed by thermogravimetric analysis method by way of STA-6000 system in the temperature range of 0 to 1000 ºC with the scan rate of 20 ºC min^{-1} under blow rate of 20 mL/min oxygen.

Synthesis of histidine substituted Ni(II)phthalocyanine (HisNiPc) complex: A mixture of histidine (0.05 mmol), tetracarboxylic-Ni(II)-phthalocyanine (NiTcPc) (0.003 M), K_2CO_3 (0.06 mM), DCC (10 mg) and DMF 30 mL in 250 mL and kept for stirring under nitrogen environment for 30 h at 28 ºC (**Scheme-I**). The green colour product was filtered, washed with warm water then rinsed with ethanol followed by distilled water. Finally, the HisNiPc precipitate was dried at 40 ºC for 1 h Yield: 70%. Anal. calcd. (found) % for $C_{60}H_{46}N_{20}O_{12}Ni$ (*m.w.*: 1295.82): C, 55.527 (55.501); H, 3.572 (3.551); N, 21.584 (21.576); O, 14.793 (14.773), Ni, 4.522 (4.506).

RESULTS AND DISCUSSION

FT-IR studies: The key IR bands of HisNiPc complex was observed at 1652-1621, 1560-1480, 1440-1410, 1325-1315, 1290-1245, 1231-1210, 1140-1120, 1110-1050, 935-930, 830-820, 780-740 and 710-670 cm^{-1}. While for NiTcPc complex, the key IR bands were observed at 3400-3200 (amide peak), 3000-2800 (C=N), 1652-1621 (C=C), 1560-1480 (-C=C-N=), 1325, 1315, 1299, 1245, 1230, 1210, 1137 (C-O), 1120, 1050, 930, 820, 740, 710 cm^{-1} (Fig. 1).

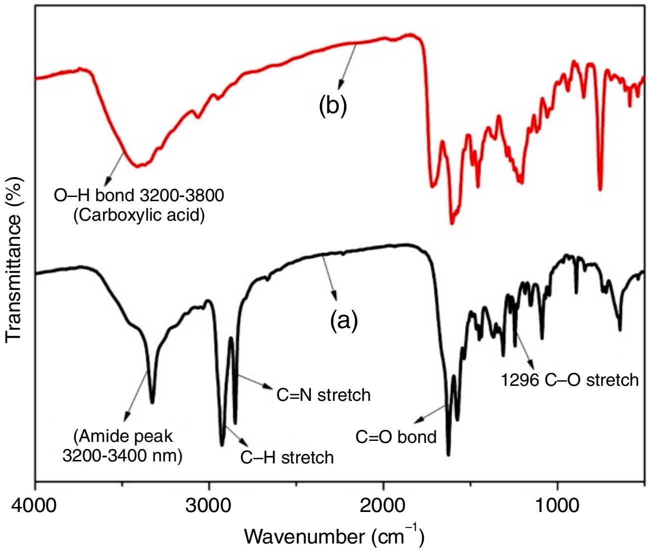
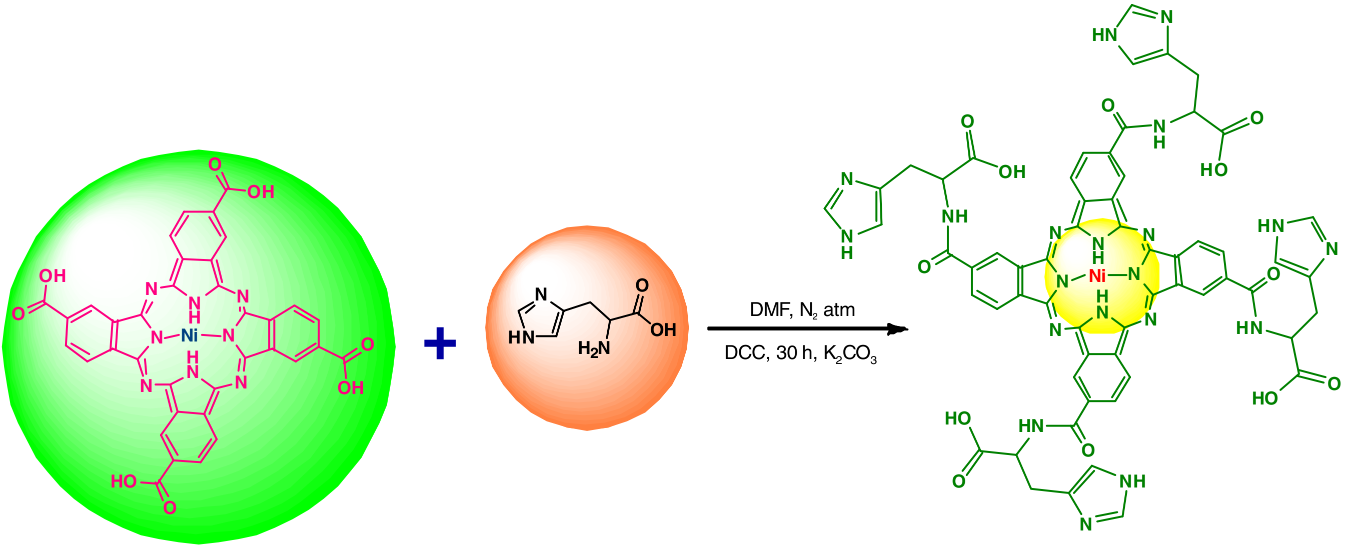


Fig. 1. FT-IR spectra (cm^{-1}) of (a) HisNiPc complex (b) NiTcPc complex

UV-Vis studies: The UV-Vis spectra of HisTcPc and NiTcPc in DMSO (Fig. 2), which is the characteristic absorptions range between 600-700 nm in the Q band region [29,30]. The Q band observed was ascribed to the π-π* transition from the HOMO to the LUMO of phthalocyanine (Pc) ring. The other bands (B) in the UV region at 290-390 nm were observed due to the transitions from the deeper π levels to the LUMO. The HisNiPc complex obtained in this study did not give



Scheme-I: The synthetic route for the preparation of HisNiP complex

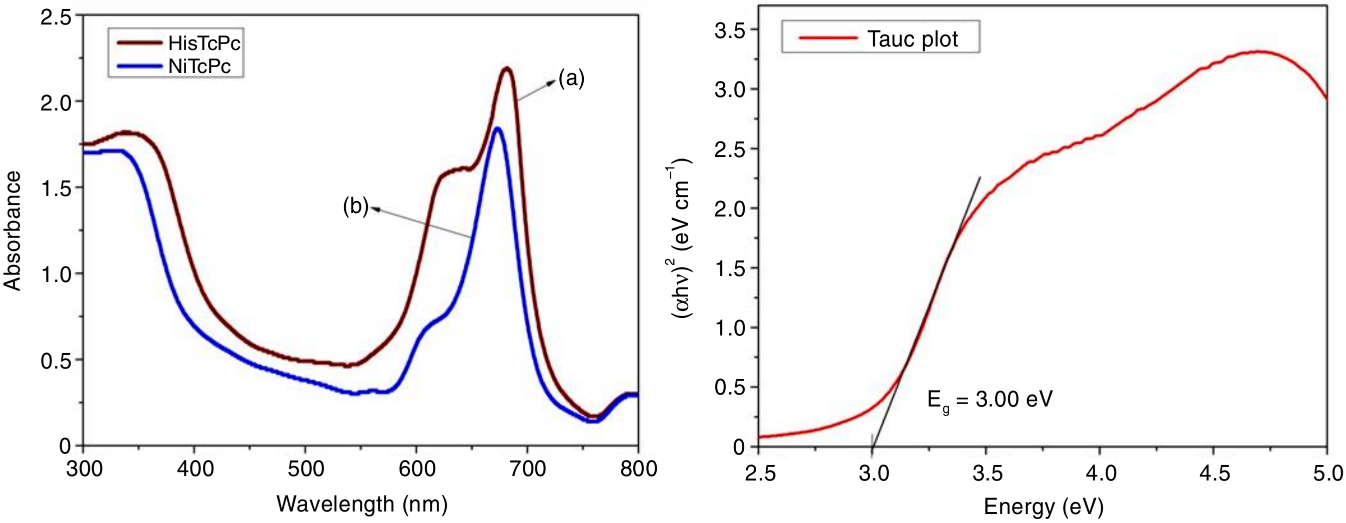


Fig. 2. UV-vis spectra of (a) HisNiPc complex (b) NiTcPc complex (S2) Tauc Plot of the HisNiPc complex

shoulder spectrum in DMSO while NiTcPc gave shoulder at 608 nm. The electronic spectrum showed monomeric behaviour evidenced by a single Q band, typical for metal bonded phthalocyanine complexes.The optical band gap was determined from the Tauc plot using eqn. 1:

$$\alpha h\nu = \alpha_0(h\nu - E_g)^n \qquad (1)$$

Mass studies: The theoretical observation of mass spectra of HisNiPc complex was 1297.82 and the experimental mass of the HisNiPc exhibits molecular ion peak at 1295.82 (Fig. 3), which is in the agreement with the the proposed structure of the compound.

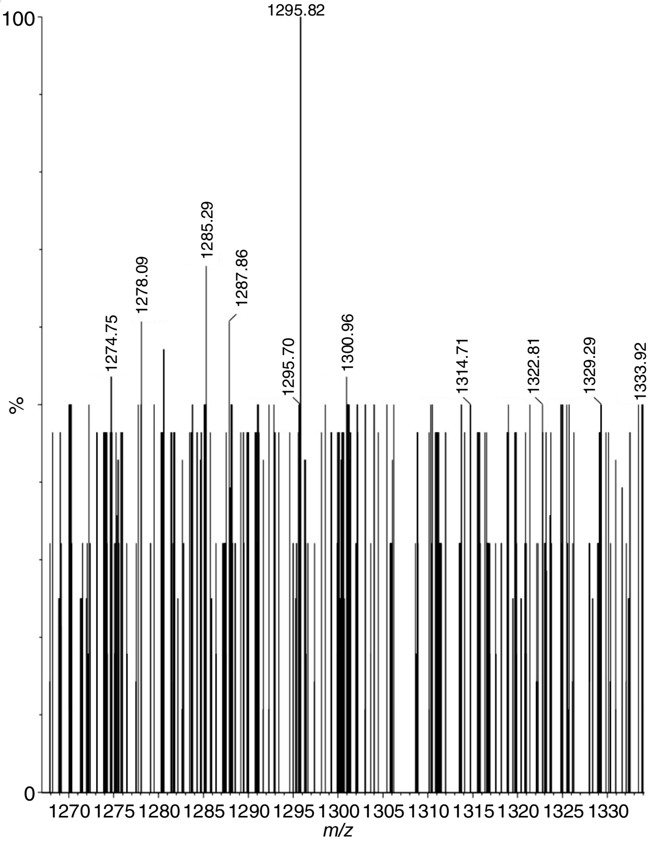


Fig. 3. Mass spectrum of HisNiPc complex

Thermal studies: Fig. 4 showed the thermal stability of HisNiPc complex. In the first step, the evaporation of water took place at 235.75 ºC; in the second step, HisNiPc undergoes the degradation and decomposition of the substituent with 29.6% of weight loss occured at 286.66 ºC and in the third step, the phthalocyanine ring decomposed with 7.45% of weight loss, finally, metal was converted into metal oxide 20.23% of weight loss at 580.60 ºC [31,32]. The experimental results showed that the HisNiPc complex exhibited good thermal stability at 235.75 ºC. It indicates that the melting point and stability of the HisNiPc complex was high ≥ 580.60 ºC. Therefore, the HisNiPc complex was suitable for an electrochemical and chemical reaction.

XRD studies: The powder XRD of HisNiPc complex was obtained by a Cu*K*α radiation source (λ = 1.540 Å) [31,33-36]. The diffraction pattern of HisNiPc complex shows the broad peaks with different diffused intensity, the intensity of peak increased by histidine group (Fig. 5). The less intensity and short peak observed at 2θ values of 100º, 150º, 160º, 170º, 190º, 200º, 300º and 450º. A highly long and sharp peak was observed at 2θ values of 50º, 60º, 120º, 130º and 180º indicates that HisNiPc was amorphous. The XRD analysis also shows the good microstrain and dislocation density of the HisNiPc complex.

Catalytic oxidation: The catalytic oxidation reactions (CORs) of substituted phenols with 4-aminoantipyrine (4-AAP) using HisNiPc as catalyst were carried out (**Schemes II and III**). In a 100 mL beaker, 30 mL demineralized water, 6 mL of 4-AAP (1.5×10^{-3} mol/L) and 6 mL of substituted phenol were thoroughly mixed followed by the addition of 0.01 mM of HisNiPc. The reaction mixture was ultrasonicated for 5 min with continous stirring at 30 ºC. The product was filtered and analyzed with a UV-visible spectrophotometer. The product was then washed with deionized water until pink colour disappeared and kept for drying in 30 ºC in an oven. The dry HisNiPc powder was washed finally with hot water and dried at 110 ºC.

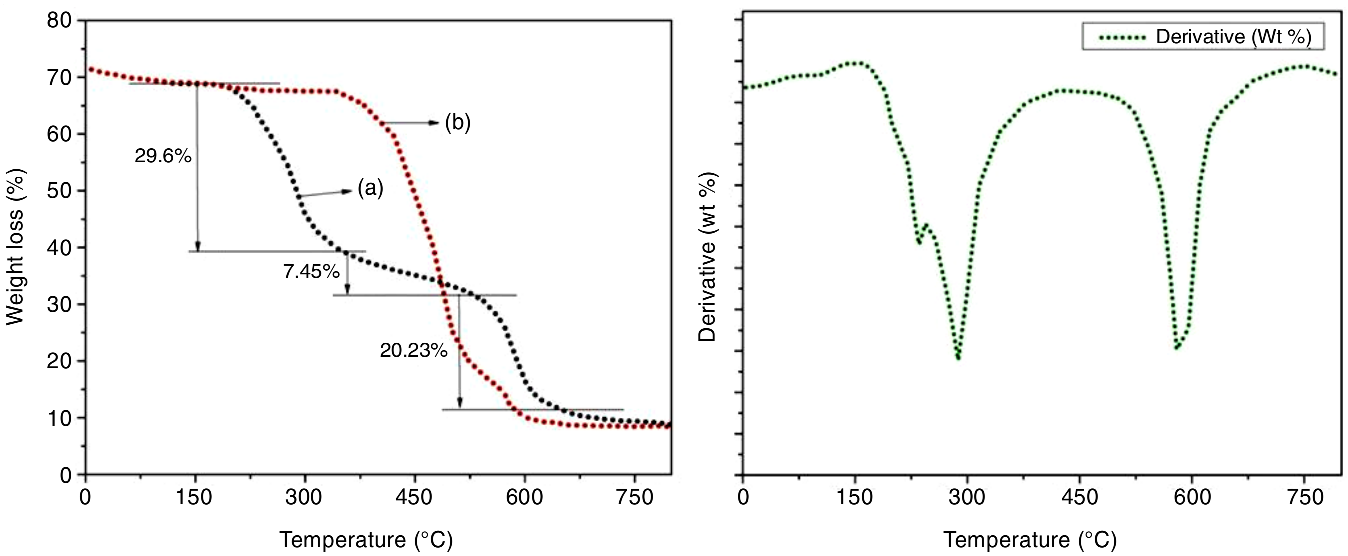


Fig. 4. Thermo gravimetric analysis of (a) HisNiPc complex (b) NiTcPc complex derivative (wt.%) of HisNiPc complex

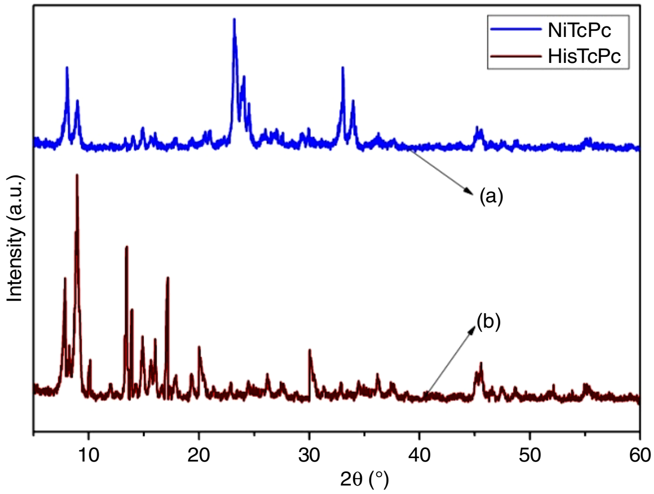
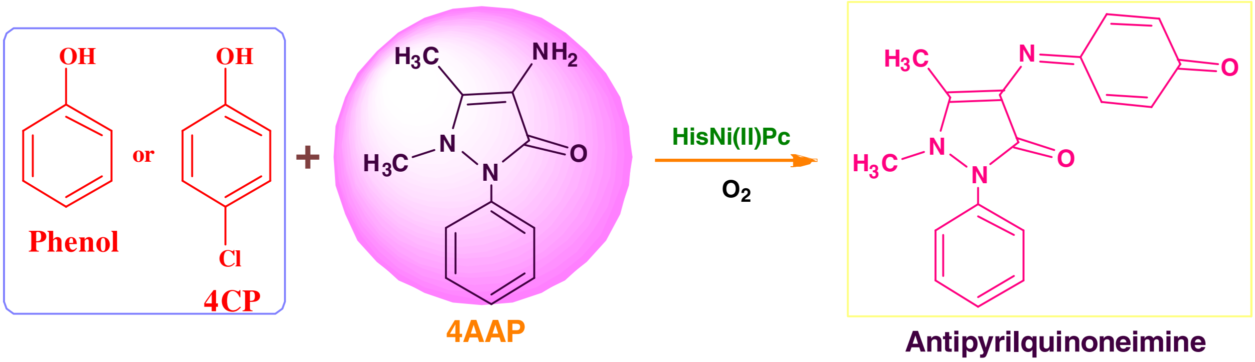


Fig. 5. XRD spectra of (a) NiTcPc (b) HisNiPc

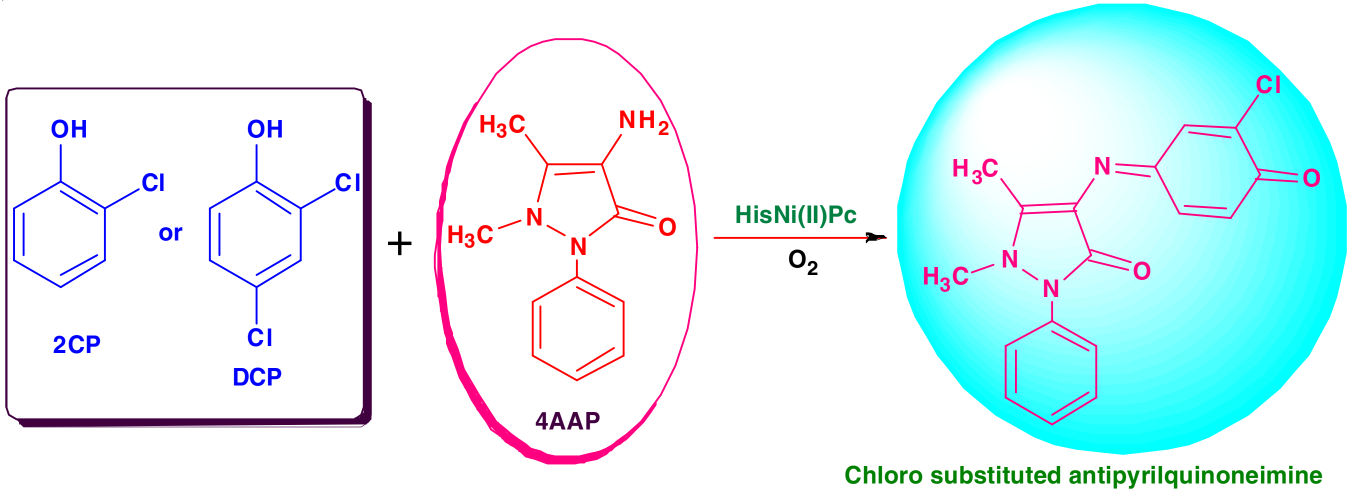
Catalytic-oxidation of phenols and chloro phenols by UV-visible method: The UV-Vis chromogenic absorption spectra of *o*-CP, DCP, *p*-CP and phenol with 4-aminoantipyrine in the presence of HisNiPc as catalyst at regular intervals is shown in Fig. 6. The characteristics peaks corresponds to the formation of dye at 520 nm indicates that COR of phenols converted into chloro substituted anti-pyrilquinoneimine dye [37]. The intensity of dye can be observed more in the range of 500-530 nm or observed at low intense in the range of 210-350 nm within 120 min. After 120 min, no further change was observed in the absorption, thus considered as the endpoint of a reaction. The absorption intensity was observed at 520 nm, the catalytic reactivity trends for various phenol compounds were in the order of *o*-CP > DCP > phenol > *p*-CP.

Catalytic oxidation of phenol substrates by varying pH: The COR of phenols with 4-aminoantipyrine (4-AAP) was carried out with different pH conditions [38]. The pK_a value for the deprotonation of *o*-CP and DCP, the hydroxyl species is 8.49 and 7.86, respectively; therefore, phosphate buffer solution (PBS, pH 9 and 10) and acetic acid buffer solution (pH 4 and 5) as well as demineralized water was utilized to study the catalytic reaction by varying with pH.

Fig. 7 shows the dye formation process in *o*-CP, DCP, *p*-CP and phenol system at various pH. In *o*-CP system (Fig. 7a), the catalytic reaction was over after 2 h at pH-7.0 and 2.5 h at pH 4 and 5, while the reaction was continued after 4 h at pH 9 and 10. From these results, the COR of *o*-CP in demineralized water, more dye formed at pH 7 within 120 min. The formation of dye at pH 4 and 5 occured after 150 min, and at pH 9 and 10 occured at ≥ 240 min. The reactions of DCP at studied pH (Fig. 7b) was also similar to *o*-CP.



Scheme-II: Phenol and *p*-CP oxidized with 4-AAP in the existence of dioxygen and HisNiPc catalyst



Scheme-III: Catalytic oxidation of *o*-CP and DCP with 4-aminoantipyrine in the existence of dioxygen and HisNiPc catalyst

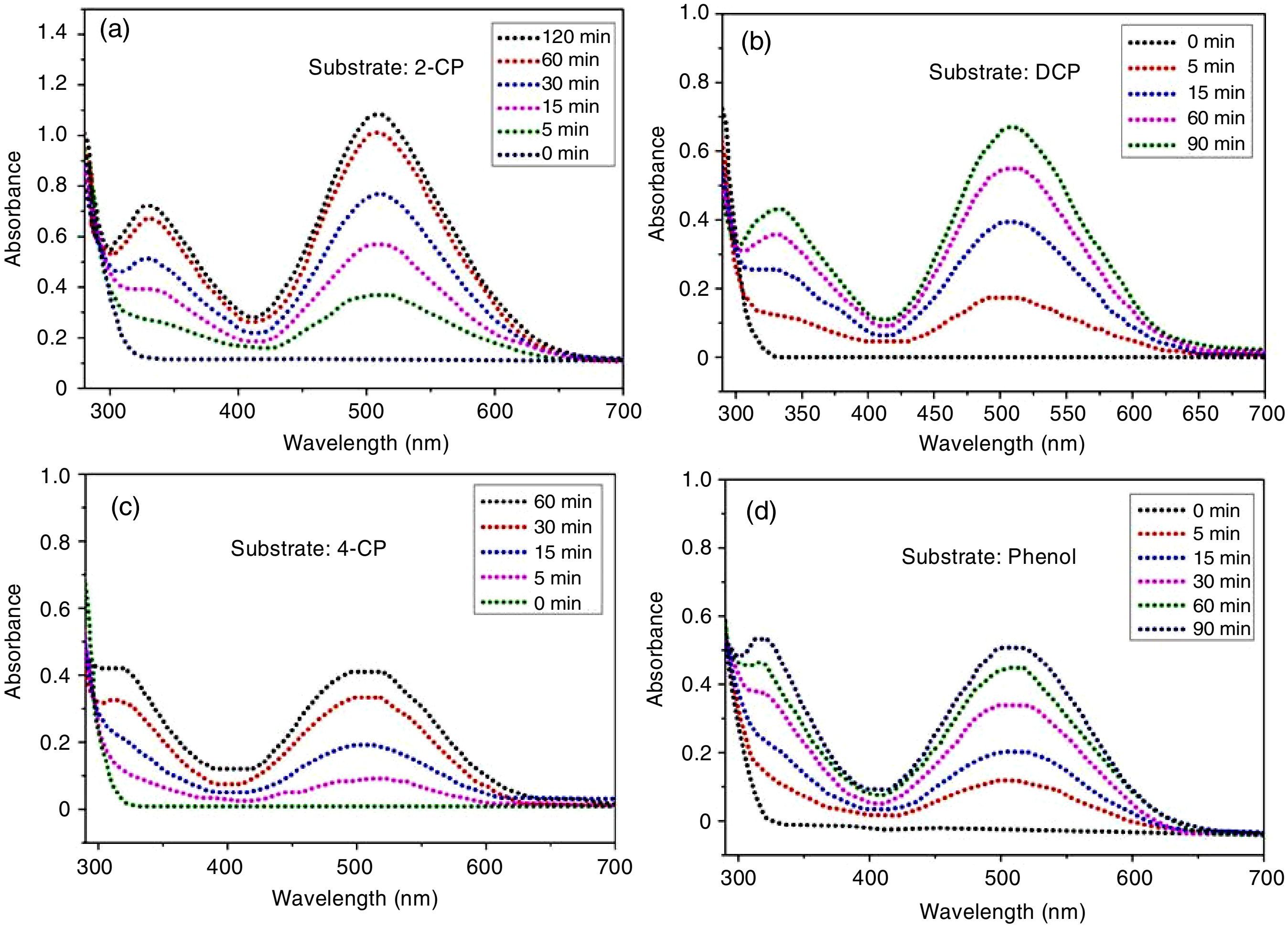


Fig. 6. Catalytic oxidation of phenol substrate with 4-aminoantipyrine in the existence of O_2 and HisNi(II)Pc catalyst (a) *o*-CP (b) DCP (c) *p*-CP and (d) phenol

In *p*-CP (Fig.7c), the reaction was over after 2.5 h at pH 7.0 and 3.0 h at pH 4 and 5, while the reaction proceeded after 5 h at pH 9 and 10. From these results, the COR of *p*-CP in demineralized water, maximum dye yield at pH 7.0 was occured at ≥ 150 min. The formation of dye at pH 4 and 5 occured after 180 min and at pH 9 and 10, the dye formed after 300 min. The reaction of phenol of studied pH was similar to *p*-CP but small variation was observed at pH 9 and 10, as shown in Fig. 7d.

Catalytic oxidation of phenol substrates by the catalytic quantity: The catalytic role is important in the formation of dye yield. The HisNiPc catalytic dosage of 10 mg, 15 mg, 20

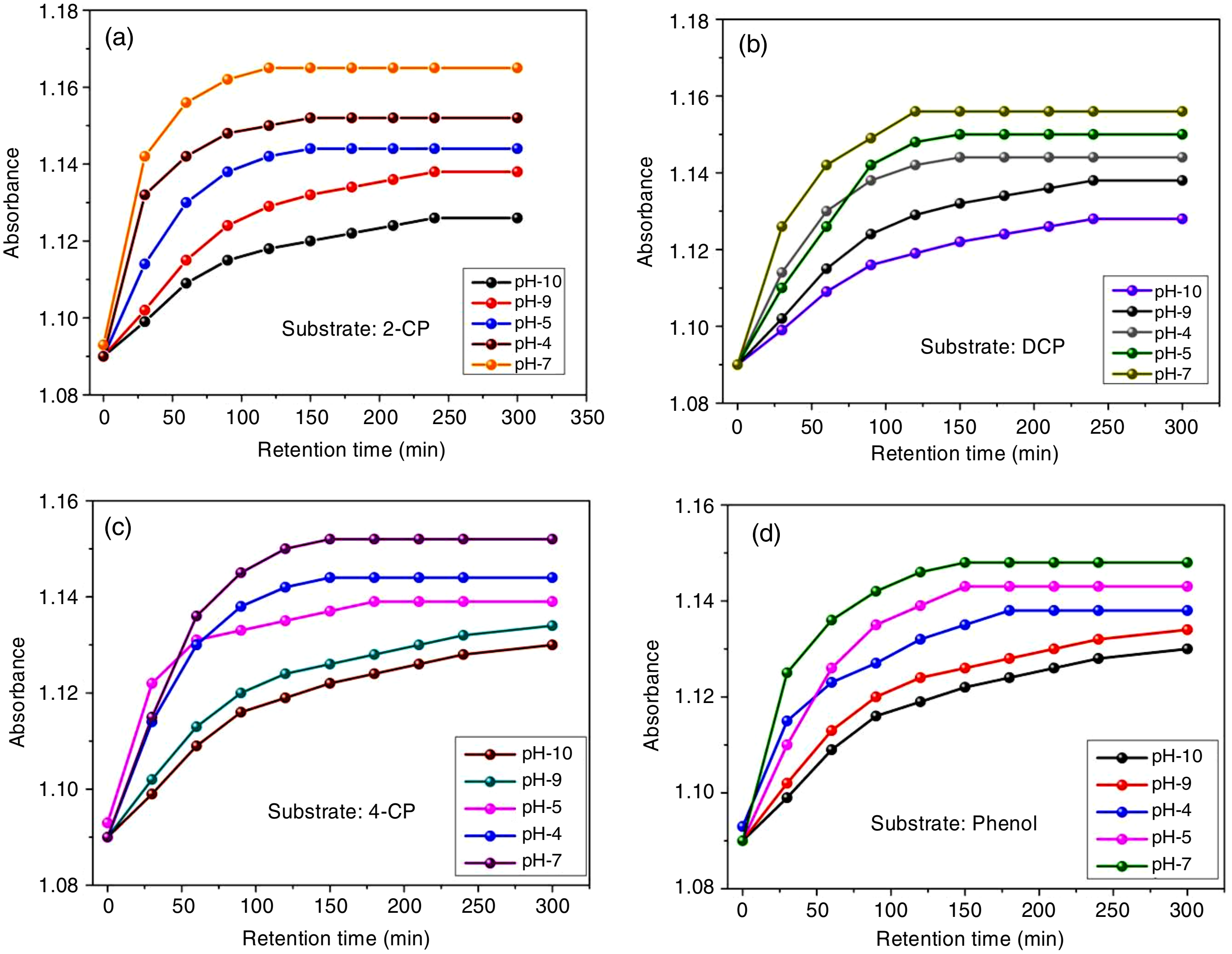


Fig. 7. Catalytic-oxidation of phenol substrate by UV-vis absorption method by variation of pH of (a) *o*-CP (b) DCP (c) *p*-CP and (d) phenol

mg, 25 mg and 30 mg was used for the catalytic oxidation of *o*-CP, DCP, *p*-CP and phenol. The maximum absorption was monitored within 300 min as shown in Fig. 8. The *o*-CP, DCP, *p*-CP and phenol results showed that 20 mg of HisNiPc catalyst contributed to the maximum formation of dye, since at this amount, the activation energy reduces more and the reactants easy to convert into the product. However, excess of catalyst 30 mg did not contributed the good catalytic efficiency because of the dye deposited on the catalytic site. Thus, 20 mg HisNiPc catalyst was optimized for the formation of dye in *o*-CP, DCP, *p*-CP and phenol after 180 min.

Catalytic oxidation of phenol substrates by varying temperature: The COR of phenol substrates was performed at various temperatures *viz*. 5, 25, 40 and 60 ºC, the maximum absorption was monitored within 300 min. The results revealed that at 40 ºC, the reaction rate was faster due to COR of phenols related to the fast concussion of the radical species, therefore more dye formation takes place as shown in Fig. 9. At 60 ºC, less dye formed due to the small effect of rate of reaction on COR and it involves the pseudo-first-order reaction [37] and according to the Arrhenius theory, the rate of side reactions depends upon the yield of the dye. At 15 ºC and 25 ºC, the rate of reaction was slow thus less yield formed at low temperature. The above data indicates that the temperature was also affected by the COR of phenols and chloro phenols reactions.

Catalytic oxidation under aerobic and anaerobic conditions: The catalytic oxidation reactions of *o*-CP, DCP, *p*-CP and phenol were carried out in presence of air and no other oxidant was used. The reaction was done in a neutral atmosphere by the continuous supply of N_2 gas in the reaction mixture. Under anaerobic conditions (Fig. 10a), the rate of dye formation was more forbidden compared to typical catalysis (Fig. 10d), which indicates that oxygen is essential for the oxidation of phenols. Usually, the electrons gained by oxygen molecule to become active oxygen radical ($O_2^{\bullet-}$ and OH^-) and thus facilitating the oxidation reaction (**Scheme-IV**) [39]. The $O_2^{\bullet-}$ is also an important for the chromogenic reaction and an $O_2^{\bullet-}$ mediator in the oxidation of phenol mechanism of HisNiPc catalysis was confirmed. Further, the typical catalysis could also proceed efficiently in absence of light (Fig. 10b), which gives the evidence that in the absence of sunlight and with the presence of catalyst *o*-CP, DCP, *p*-CP and phenol, the catalytic oxidation process proceeds. The overall reaction mechanism proceeds in three steps [40] (**Scheme-V**) *viz*. step-I: formation

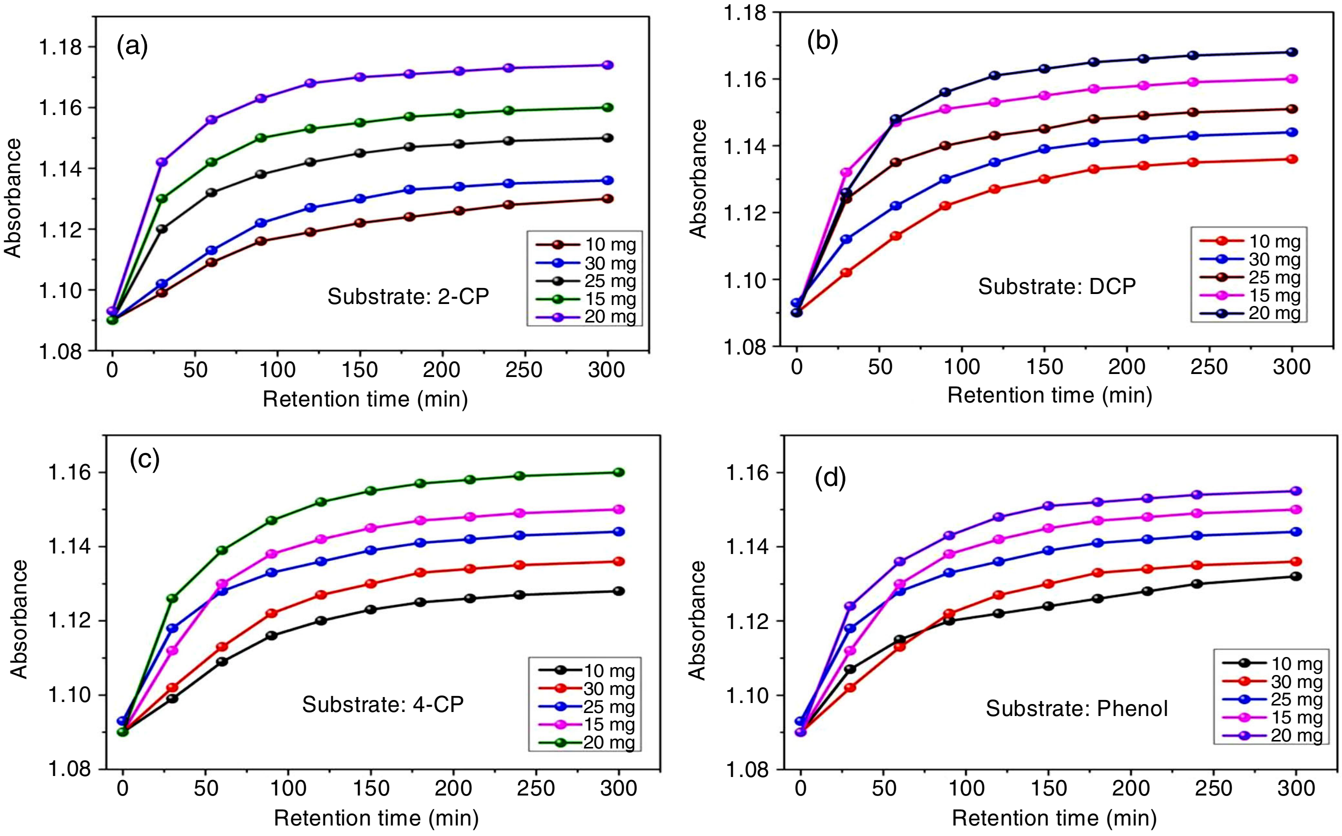


Fig. 8. Catalytic-oxidation of phenol substrate by UV-vis absorption method by variation of catalytic quantities of (a) *o*-CP (b) DCP (c) *p*-CP and (d) phenol

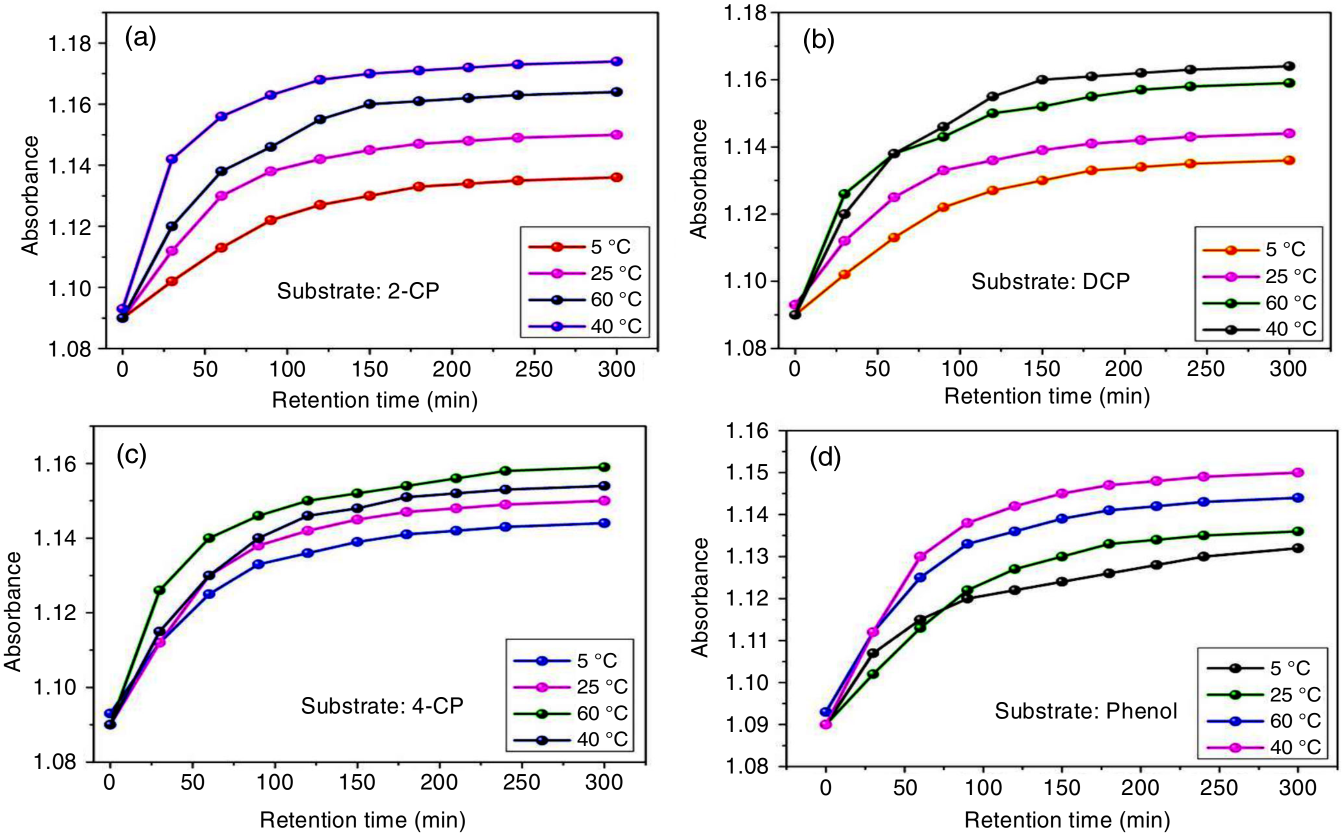
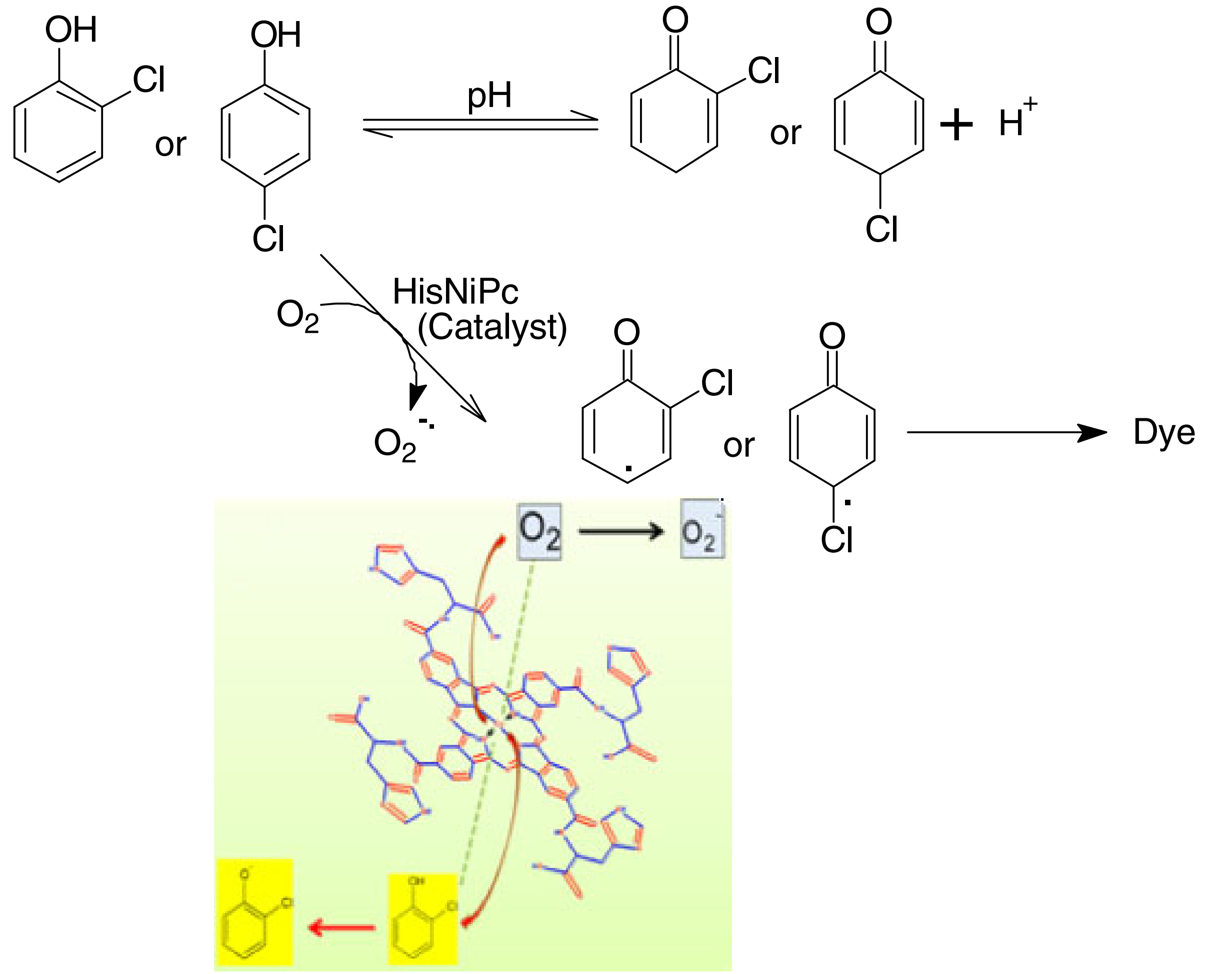
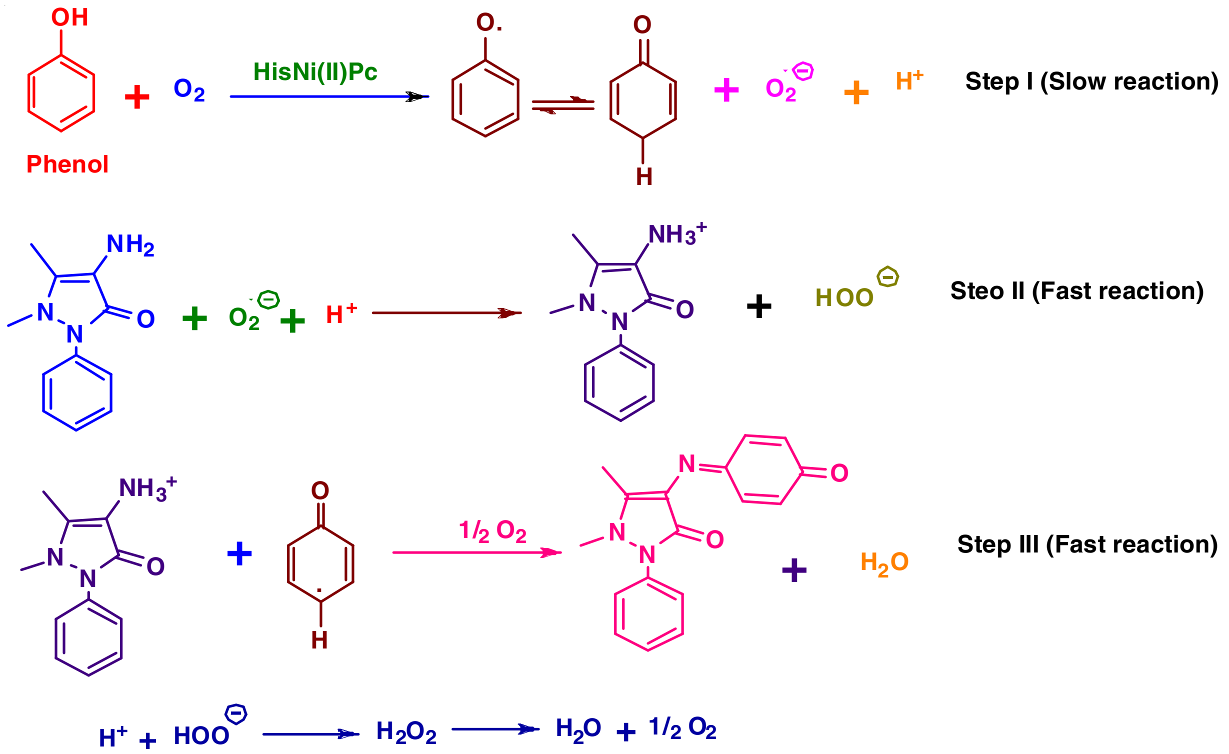


Fig. 9. Catalytic oxidation of phenol substrate by UV-vis absorption method by variation of temperature of (a) *o*-CP (b) DCP (c) *p*-CP and (d) phenol



Scheme-IV: The effect of dissolved O_2 on the catalytic-oxidation of phenol substrate by UV-vis absorption method.



Scheme-V: The mechanism involved in the oxidation of phenol substrate (the same mechanism applicable for *o*-CP, *p*-CP and DCP)

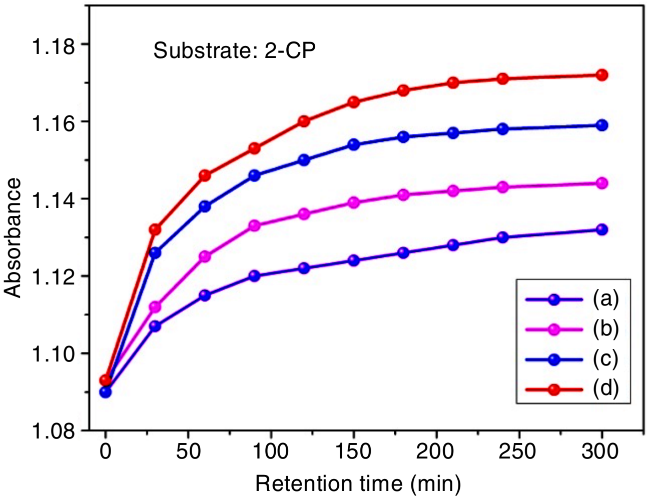


Fig. 10. Reaction curve in presence of (a) under N_2 environment with catalyst (b) dark condition with catalyst (c) sunlight (d) excess of oxygen

of *p*-quinoid radical by treating with phenol and oxygen; step-II: formation of antipyrine-NH_3^+; and step-III: formation of chloro substituted antipyrilquinoneimine dye.

Conclusion

A novel histidine substituted nickel(II) phthalocyanine (HisNiPc) complex was synthesized and chacterized by mass, FT-IR, UV-visible, elemental analysis, X-Ray diffraction and Thermogravimetric techniques. Furthemore, the catalytic oxidation behaviour of new complex against phenols and chloro phenols in water was also carried out reported. The results indicated that several phenolic compounds like *p*-chlorophenol (*p*-CP), *o*-chlorophenol (*o*-CP), 2,4-dichlorophenol (DCP) and phenol were easily oxidized in the presence of HisNiPc catalyst and rapidly reacts with 4-aminoantipyrine to produce pink dye. The formation of the pink dye involved the transfer of a single electron to dioxygen using the HisNiPc catalyst.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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