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DPPH Radical Scavenging Activity and Corrosion Inhibition Activity of Nano Sized Schiff Base

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Abstract
This work investigated the effectiveness of 1-(((4-bromophenyl)imino)methyl)naphthalen-3-ol, a nanosized Schiff base, in preventing mild steel corrosion in HCl using weight loss measurements and surface characterization. Using the weight loss method, the synthesized compound's efficacy as a corrosion inhibitor in an acidic medium was assessed. Maximum inhibition efficiency was found at an ideal dose of 1×10^{-3} M for the inhibitor at 298 K. The DPPH-free radical activity's ability to scavenge radicals led to an investigation of the Schiff base's antioxidant activity. The Schiff base exhibited good antioxidant action.

Key words: Corrosion, 1M HCl, Schiff base, antioxidant, DPPH

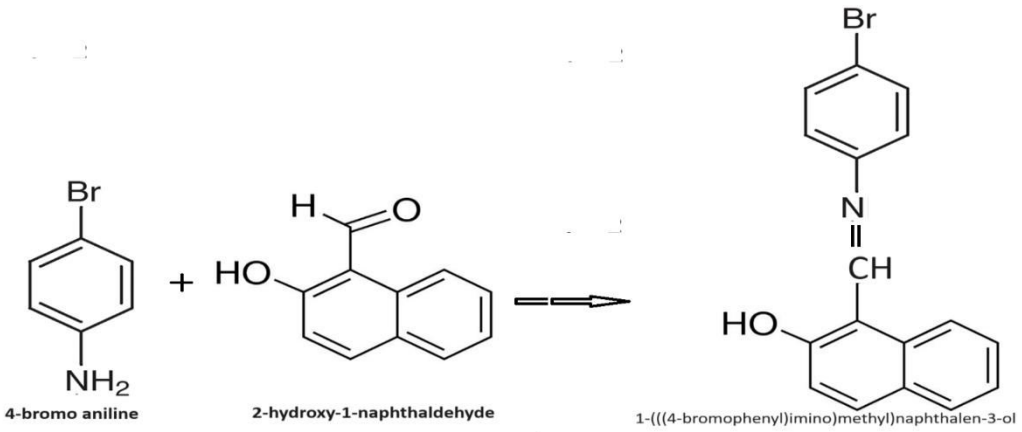
INTRODUCTION

Metallic substrate corrosion is a significant issue in many industrial processes. Because of their superior film-forming and adsorption capabilities, Schiff bases have become attractive options for corrosion inhibitors [1]. Aldehydes or ketones react with amines to form an imine or azomethine group, which is how Schiff bases are created [2,3]. An important and functional conjugated system exists in Schiff bases of aromatic aldehydes [4]. Interestingly, the range of Schiff bases has been expanded due to the flexibility of the imine group linked to this class of compounds, which permits the attachment and extension of p-donating groups. Corrosion chemists are more interested in these properties because of their potential application in protecting metal surfaces [5]. Because Schiff bases are easily synthesised, have aromatic rings that provide an electron cloud, and can include some electronegative atoms as substituents, which could determine their overall potency and aid in the development of better corrosion inhibitors technologically, they were employed as corrosion inhibitors [6]. They have a great chance of being adsorbed on metal surfaces due to the C=N group, planarity of p-bonding orbitals, and electrons from the associated electronegative atoms [7]. It is necessary to provide corrosion inhibitors with a high yield, low cost, low toxicity, and high efficiency so they can be used as additives in a variety of industrial

applications. It was demonstrated that Schiff base compounds have a track record of effectively preventing metal corrosion in acidic conditions [8-10]. Since the mechanism of action of these inhibitors is surface adsorption, the type of the metal surface, the electrochemical potential at the interface, and the environment in which the inhibitor operates all affect how effective the inhibitor is. The efficiency of the inhibitor is dependent on its chemical structure, which includes the number of adsorption active centres in the molecule and their charge densities, the size of the molecule, the mode of adsorption, and the protected area of the inhibitor on the metal surface [11,12]. After synthesising the Schiff base 1-((4-bromophenyl)imino)methyl)naphthalen-3-ol), ^{13}C NMR and ^{1}H NMR spectroscopy were used to identify the molecular structure. The weight loss method was used to assess this molecule's capacity to prevent mild steel corrosion in an acidic media (1M HCl). Based on the radical scavenging impact of DPPH-free radical activity, the antioxidant activity of the produced Schiff base was examined.

MATERIALS AND METHODS

Sigma Aldrich provided the 2-hydroxy-1-naphthaldehyde and 4-bromo aniline. In an alcoholic medium, 4-bromo aniline (0.01 mol) and 2-hydroxy-1-naphthaldehyde (0.01 mol) are condensed to form Schiff base ligand. The combination is then refluxed for four to five hours at 40 to 50. The yellow solid product formed is dried at room temperature and cleaned with ethanol. Scheme I illustrate the synthesis of the Schiff base ligand. The Schiff base ligand's ^{13}C and ^{1}H spectra are displayed in Figures 1(a) and 1(b), respectively.



Scheme 1: Synthesis of Schiff base

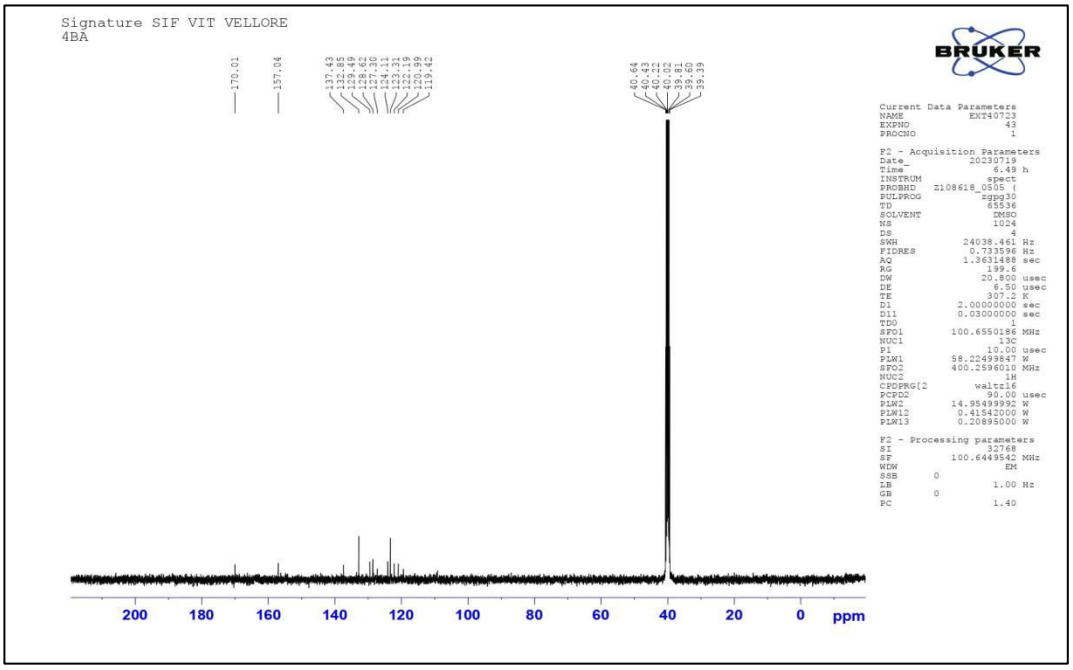


Fig.1 (a): ^{13}C spectrum of schiff base ligand

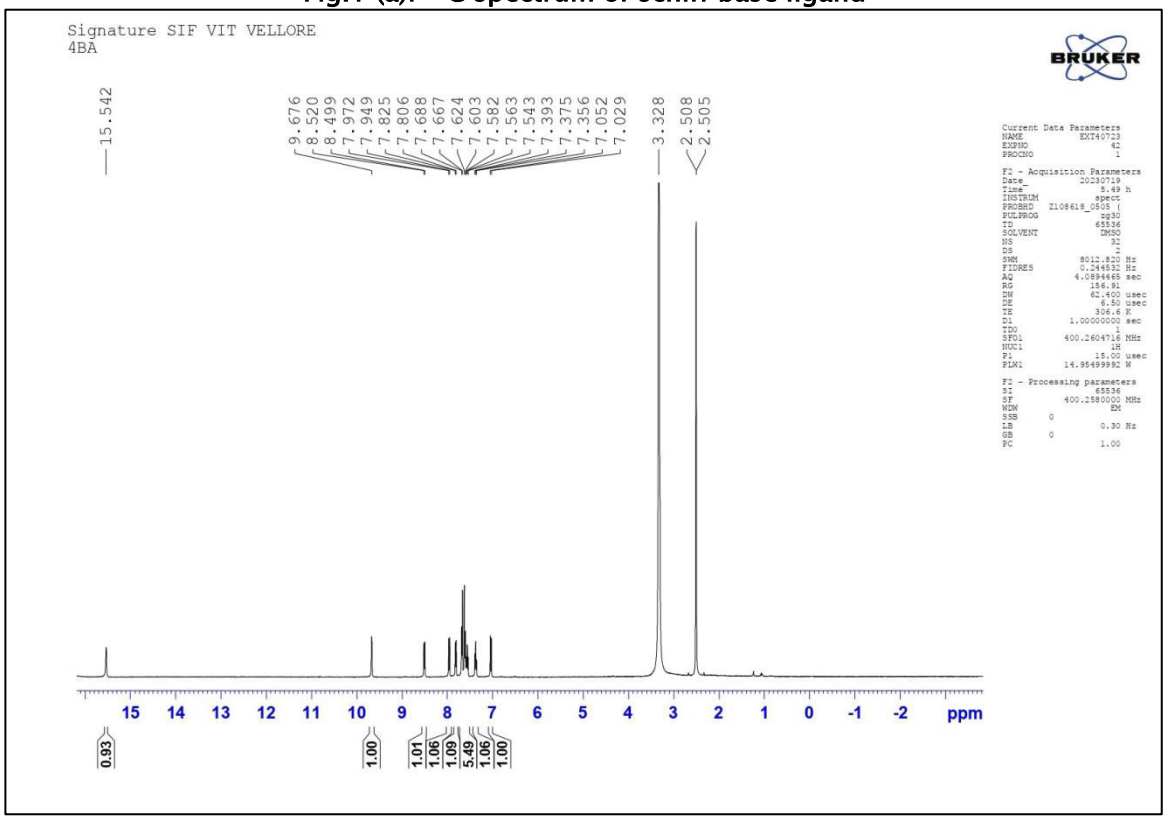


Fig.1(b): ^{1}H spectrum of Schiff base ligand

Weight loss measurements :

Coupons made of mild steel were used for corrosion testing. Hydrochloric acid was used to prepare the corrosive acid solutions at a concentration of 1M. The necessary acid concentration (1M HCl) was made with deionized water. The tests were conducted with a range of immersion times (1–5) hours, both in the presence and absence of inhibitor concentrations (1×10^{-3}M – 1×10^{-6}M). Equation (1) was used to calculate the corrosion rate (W) [13].

$$W = \frac{WL.K}{A.t.D} \qquad [1]$$

Where W is the corrosion rate, A is the specimen's area (inch^2), D is the mild steel density (g/cm^3), K is a constant (543) (giving rate in mpy), and t is the immersion period (in hours). Using Equation (2), the inhibition efficiency (IE%) was calculated [14]:

$$IE\% = \frac{Wa - Wb}{Wa} X\ 100\% \qquad [2]$$

where the corrosion rates in the presence and absence of the inhibitor, respectively, are represented by Wb and Wa.

Antioxidant activity: Based on the radical scavenging ability of 1-diphenyl-2-picrylhydrazyl (DPPH)-free radical activity, the antioxidant activity of Schiff base and the standard were evaluated. A freshly made DPPH solution had a maximum absorbance at 517 nm and had a deep purple colour. When there is an antioxidant in the medium, the purple colour fades. Antioxidant molecules can therefore neutralise DPPH free radicals and transform them into a colourless byproduct. The test sample's diluted solutions were meticulously made in methanol. A standard solution was ascorbic acid. A 0.002% DPPH solution was made in methanol.

This solution was combined with 1 mL each of the sample and standard solutions in a volume of 2 mL. After 30 minutes of darkness, the absorbance of these solution was determined using a UV-Visible spectrophotometer at 517 nm. As a blank, 1 millilitre of methanol and 0.002% DPPH solution were employed. The ratio of the sample absorbance reduction to the tested compound's absorbance at 517 nm is used to express the free radical scavenging abilities. Equation (3) was used to calculate the percentage inhibition after the absorbance was measured [15].

$$I\% = \frac{Abs_{Blank} - Abs_{Sample}}{Abs_{Blank}} X\ 100\% \qquad [3]$$

Where Abs $_{Blank}$ = Absorbance of the blank and Abs $_{Sample}$ = Absorbance of the sample. The percentage of DPPH free radical scavenging activity was directly correlated with the compound's concentration.

RESULTS AND DISCUSSION

^{1}H-NMR spectrum analysis : ^{1}H NMR spectra of the synthesized compound were recorded in DMSO-d6 (500MHz) as the solvent) in Fig.1b. The region at δ 7.54-7.65 ppm (s, 6H, aromatic rings) is due to aromatic protons [16, 17]. δ at 9.67ppm (s, H, CH=N) [26,27]. ^{1}H NMR spectra of the synthesized compound shows a signal at δ 9.67 ppm [18] due to phenolic OH. In ^{13}C NMR spectrum, 119.42 to 137.43 (C1 -C10) chemicalshiftsareidentified as naphthalene ring, 157.0 due to carbon assigned at deshielding CH=N .

SEM studies : Scanning electron microscope (SEM) has been actually used in order to determine the particle size and the morphology of the prepared compounds. So, SEM micrograph of the Schiff base was shown in Figure 5 . From the data, it was indicated the successful formation of nanosized particles. The particle size of ligand was in between 44 to 94 nm.

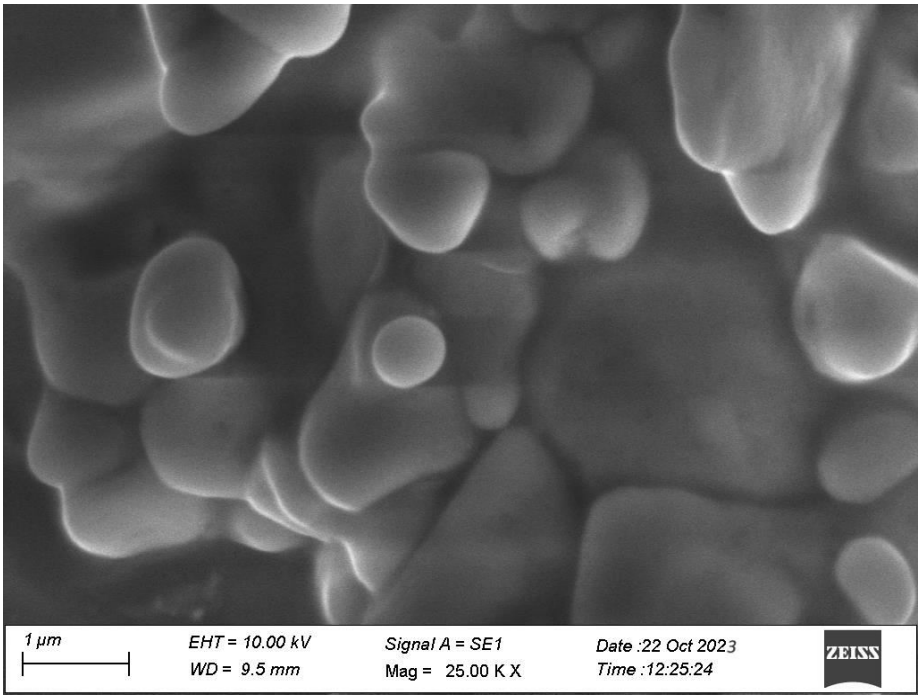


Figure 2; SEM image of Schiff base ligand showing nano sized granules

Weight loss measurements

Effect of exposure time on the corrosion process

The weight loss method was used to examine the impact of the inhibitor's addition on the corrosion of mild steel in 1M HCl solution at a temperature of 298K after exposure times ranging from 1 to 5 hours, both in the presence and absence of 10^{-3}M of the produced Schiff base as a corrosion inhibitor. Fig. 3 presents the findings. In comparison to the hydrochloric solutions alone, the figure clearly demonstrates a significant decrease in the IE% with an increase in immersion time of the metal coupons from 1 hour to 5 hours in the presence of the inhibitor. This is because prolonged exposure to electrolyte causes a robust passive protective layer to grow on the metal's surface [19]. Table 1 summarises the weight loss (gm) and corrosion rate (mpy) values.

Table 1: Weight loss and corrosion rate for mild steel in 1M HCl alone and in presence of inhibitor (C= 1×10^{-3} M) at different exposure time

The Variable	Corrosion medium	Exposure time (hours)				
		1	2	3	4	5
Weight loss (gm)	HCl only	0.0041	0.0039	0.0028	0.0025	0.0015
Corrosion rate (mpy)	HCl only	67.52	65.05	47.60	42.66	26.19
Weight loss (gm)	HCl + inhibitor	0.0025	0.0022	0.0016	0.0012	0.0011
Corrosion rate (mpy)	HCl + inhibitor	41.34	36.40	33.90	20.59	18.45

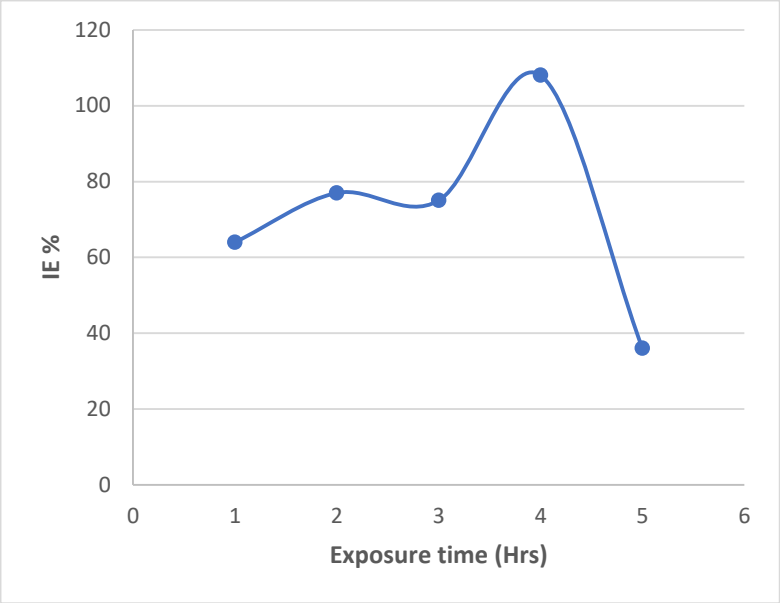


Fig.3: Inhibition efficiency (IE%) Vs Exposure time (In hrs)

Effect of inhibitor concentration on the corrosion process

Table 2 presents the weight loss data and the corrosion rate of mild steel in 1MHCl, both in the presence and absence of different inhibitor concentrations, at 298 K, following a 3-hour exposure. The findings demonstrate that as inhibitor concentration increases, both the weight loss data and the rate of corrosion decrease [20-22].

Table 2 : Weight loss and corrosion rate for mild steel in 1M HCl absence and in presence of various concentrations of inhibitor

Inhibitor conc. (M)	Weight loss (gm)	Corrosion rate (mpy)
0	0.0043	70.98
$1 X 10^{-6}$	0.0034	56.00
$1 X 10^{-5}$	0.0033	54.68
$1 X 10^{-4}$	0.0021	34.42
$1 X 10^{-3}$	0.0016	33.90

Surface morphology: The donor- and electron-withdrawing sites, along with their position, determine how much organic corrosion inhibitors interact with metallic surfaces [23]. The presence of oxygen, nitrogen, and pi-electrons (double bonds) affects the inhibitor's ability to adsorb to the steel surface [24]. Figs. 4a and 4b display SEM images of the metallic substrate surface strip after five hours of exposure in an uninhibited and an inhibited hydrochloric acid solution containing Schiff base. The surface was topped and sagging, as shown in Fig. 4a, indicating significant corrosion of the metallic substrate surface. The surface properties with significantly reduced corrosion are shown in Figure 4b.

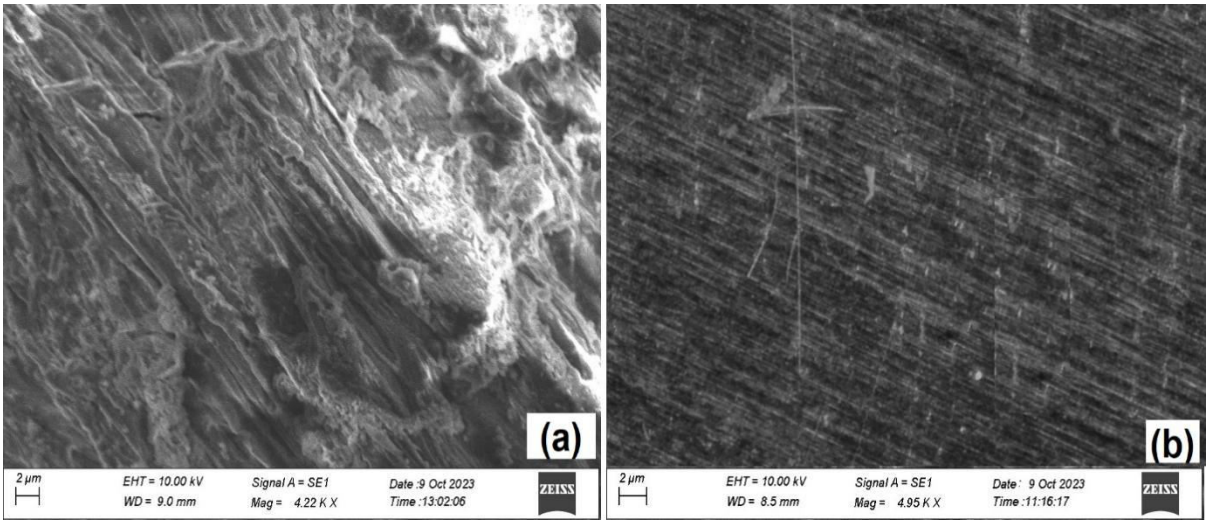


Fig.4: SEM images of Mild steel (a) mild steel with 1M HCl solution (c) mild steel with 1M HCl and inhibitor

Antioxidant activity of the Schiff base: The DPPH radical assay provides a quick and simple way to assess the antioxidant activity of the Schiff base. The DPPH is typically used in chemical analysis to determine the radical scavenging activity since it is a purple-colored, persistent free radical with an odd electron in its structure. When the antioxidants react with the DPPH, it turns colourless. The level of discolouration indicates how well the antioxidant can scavenge radicals. The reduction in absorbance at 517 nm caused by antioxidants was used to gauge the DPPH radicals' capacity for reduction. The information was derived from a plot of an antioxidant's % scavenging ability vs concentration [25-27]. Using ascorbic acid as the standard, various amounts of the Schiff base with DPPH radicals were utilised in the antioxidant assay investigation. The stable DPPH free radical activity's ability to scavenge free radicals was used to evaluate the antioxidant activity of Schiff base using the standard. They looked at variations in the test samples' capacity to scavenge free radicals; the resulting percent inhibition is displayed in Fig. 5. When compared to ascorbic acid as the standard (IC_{50} = 1.73), the DPPH scavenging the activity of the ligand (IC_{50} = 2.74) is lower, indicating that this is a significantly stronger free radical scavenger and antioxidant. The antioxidant capacity of Schiff base increased noticeably with increasing concentration, as did the activity of radical scavenging in both the standard and Schiff base in a dose-dependent manner. The Schiff base exhibited good free-radical scavenging action, according to the data.

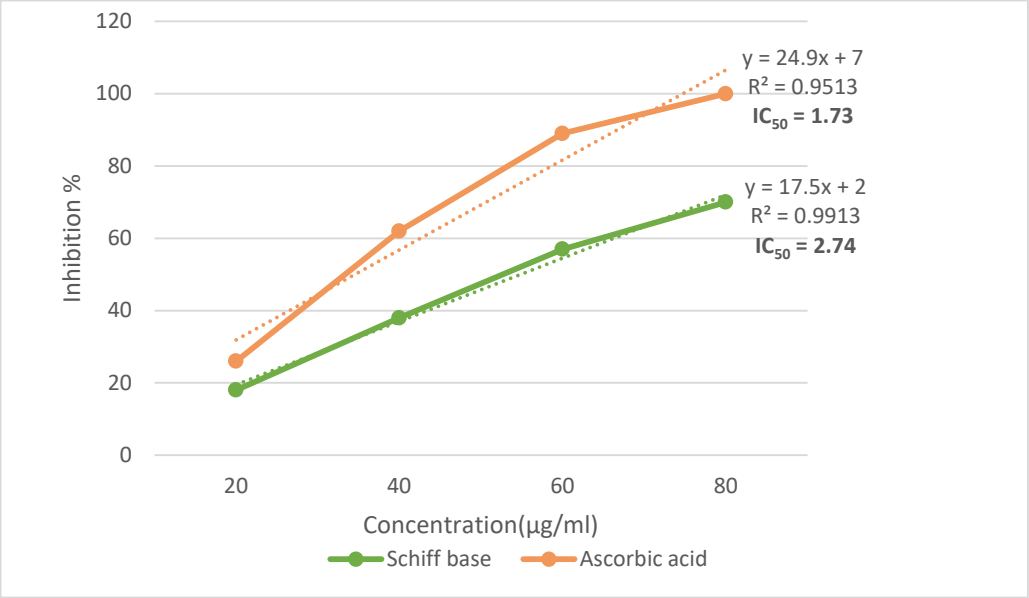


Fig.5: DPPH radical scavenging activity of Schiff base compared with Ascorbic acid

CONCLUSION

The present study reports that in a 1M HCl solution, the synthetic Schiff base 1-(((4-bromophenyl)imino)methyl)naphthalen-3-ol) exhibits good inhibitive capabilities for mild steel. The findings showed that as inhibitor concentration increases, both the weight loss data and the rate of corrosion decrease. The Schiff base's antioxidant activity was investigated based on the 1,1-diphenyl-2-picryl-hydrazyl (DPPH) free radical activity's capacity to scavenge radicals. The activity of the Schiff base was excellent.

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