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2-{(3-nitrobenzylidene)amino}phenol (SBNAP) as Inhibitor of Aluminium and Mild Steel Corrosion in Sulphuric acid media

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Abstract
Corrosion inhibitory effect of 2-{(3-nitrobenzylidene)amino}phenol (SBNAP) Schiff base on mild steel and aluminium in Sulphuric acid media were investigated by weight loss and gasometric methods at a temperature range of 30 °C – 50 °C. The results showed that the inhibition efficiency of SBNAP for mild steel increased with increase in temperature while that of aluminium decreased as the temperature increased. The plots of log (wi - Δw) against time gave linear graphs which confirmed a first order reaction mechanism for both metals. The inhibition efficiency of 34.98% at 1.0×10^{-2} M concentration for mild steel was obtained while that obtained for aluminium was 63.81% at 30 °C. The positive values of the heat of adsorption, entropy of adsorption and the negative free energies of adsorption suggest an endothermic reaction, a disordered system and spontaneous adsorption of the studied Schiff base on the metals surface respectively. The values of the average activation energies and free energies of adsorption support physisorption reaction mechanism.

Keywords: Inhibitor, Aluminium, Sulphuric acid, Temperature, Schiff base

1. Introduction
Use of corrosion inhibitors is one of the most popular and efficient methods of protecting metals from corrosion. This is because corrosion inhibition is a convenient and relatively inexpensive method to reduce corrosion, (Paulina *et al.*, 2014) [19].

Generally, the heterogeneous organic compounds having electron density on heteroatoms like O, N, S, etc. have the tendency to resist corrosion. The heteroatoms are the active center for the process of adsorption on metal surfaces, (Upadayay and Mathur, 2007) [21]. In addition, compounds that contain electronegative groups, π-bonds, conjugated double or triple bonds, etc. normally exhibit good inhibitive property. They supply electrons from the electron-rich bonds to the metals which they are effectively adsorbed, (Ashish and Quraishi, 2012) [4].

Schiff base compound: 2-{(3-nitrobenzylidene)amino}phenol (SBNAP) used in this research is condensation product of a primary amine and an aldehyde. Recent publications show increased attention to Schiff base compounds as corrosion inhibitors because of their effectiveness in reducing corrosion of various metals such as steel, copper and aluminum.

According to the research findings of Chitra *et al.* (2010) [6, 7], the ability of the inhibitor to form a long lasting film depends upon its chemical structure and interaction with the corrosive environment. Chitra *et al.* (2010) [6, 7] has reported that the presence of unoccupied π* orbitals in the Schiff base molecules, which enable electron back donation from the metal d- orbitals is partly responsible for the stronger inhibition efficiencies shown by some Schiff bases.

Metals are chemical elements that are typically lustrous solids (except mercury) that are good conductors of heat and electricity. Metals form coordination compounds when they react with ligands. The metals of interest in this study are mild steel and aluminium.

Steel is categorized based on the percentage of carbon it contains; mild steel contains between 0.16% to 0.29% carbon. Mild steel is one of the highest volume of steel in production, it is very reactive compared to other categories of steel. It is not a very hard material, not brittle nor ductile but malleable. Corrosion of mild steel occurs due to the

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reaction of Fe with oxygen and water. It can easily revert to rust in the presence of water and oxygen (Erma 2010, Mohammed 2011, Gopi *et al.*, 2010) [12, 17, 13].

Aluminium and its alloys find a wide variety of technological applications owing to the balanced combination of mechanical, chemical and physical properties such as stability for surface treatment, high ductility, thermal and electrical conductivity, low density, and recycling ability. Aluminum exhibits passive behavior in aqueous medium due to the formation of a strong passive oxide film on the surface. The adhesive passivating surface was meant to protect the metal but due to its amphoteric nature cannot prevent corrosion of the metal by acids and bases as it dissolves readily on exposure to strong acids and alkaline solutions, (Paulina *et al.*, 2014) [19].

Sulphuric acid and other acids especially at high temperature therefore, present a huge threat to the metal and its finished products as a major cause of corrosion. Sulphuric acid, with a historical name oil of vitriol, is a highly corrosive strong mineral acid with the molecular formula H_2SO_4. It is a pungent-ethereal, colourless to lightly yellow viscous liquid which is soluble in water at all concentrations. It is dyed dark brown during production to alert people to its hazards.

In the present research work the effect of SBNAP as corrosion inhibitor for mild steel and aluminum in 2M Sulphuric acid solution is investigated.

The aim of this study is to evaluate the corrosion inhibition potential of 2-[(3-nitrobenzylidene)amino] phenol (SBNAP) and to Carry out kinetic studies on the corrosion inhibition of mild steel and aluminium in sulphuric acid solutions.

The equations used for calculating the various parameters are presented below:

i. Inhibition efficiency at 30°C, 40°C and 50°C

% Inhibition Efficiency =

$$\frac{\Delta W_B - \Delta W_i}{\Delta W_B} X \frac{100}{1} \quad \text{...........................(1)}$$

Where $\Delta W_B \, and \Delta W_i$ are the weight loss of metal coupons without and with inhibitor respectively.

ii. Corrosion rate at 30 °C, 40 °C and 50 °C
Corrosion rate (g/cm²/day) =

$$\frac{\Delta w}{Area \, x \, day} \quad \text{...................(2)}$$

$$(cm^2)$$

The order of reaction was determined from the rate plots and the rate constants were calculated.

iii. Rate constant (k) at 30 °C, 40 °C and 50 °C for first order reaction

$$k = \frac{2.303}{Time} \log \frac{wi}{wf} \quad \text{...........................(3)}$$

The activation energies, Ea of the metals in acids solutions containing SBNAP, were calculated by making use of the integrated form of the Arrhenius equation as

iv. Activation energy (kJmol⁻¹)

$$Ea = \frac{2.303 R T_1 T_2 (\log K_2 / K_1)}{T_2 - T_1} = \frac{Jmol^{-1}}{1000} = kJmol^{-1} \text{.................(4)}$$

Where R= is the gas constant in Joule (J), T= Absolute temperature in Kelvin
R= 8.314 Jmol⁻¹ K⁻¹ or 8.314 x 10⁻³ kJmol⁻¹K⁻¹
K_1 and K_2 are rate constants at T_1 (30 °C) and T_2 (40 °C) respectively or T_1 (40 °C) and T_2 (50 °C)

v. Surface coverage (θ) at 30 °C, 40 °C and 50°C
The surface coverage θ of the inhibitor at different concentrations and different temperatures was calculated using the following equation according to Chitra *et al.* (2010) [6, 7]

$$1 - \theta = \frac{b}{b_0} \quad \text{..(5)}$$

Where θ= surface coverage, b_i = the inhibited corrosion rate or weight loss with inhibitor b_0 = the uninhibited corrosion rate or weight loss of blank.

vi. Standard free energy change (ΔG^O_{ads}) for the adsorption process (kJmol⁻¹) at 30 °C, 40 °C and 50 °C
According to Abiola and Tobun (2010) [2] one could calculate the values of the standard free energy change for the adsorption process (ΔG_{ads}) of the inhibitor from θ values with the help of the equation C/θ = 1/K + C ………………………………………………..(6)
Where K is the equilibrium constant for the adsorption/desorption process of the inhibitor molecules on the metal surface and C is the inhibitor concentration.
From the plot of C/θ against C, a line of best fit gives a value for the gradient of the line; K is calculated from the relationship: 1/K = Gradient of the line.
The values of the standard free energy of adsorption was calculated using the equation:

ΔG_{ads}= -2.303RT log (55.5K)……………………………. (7)

The value of 55.5 is the concentration of water in solution.

vii. Entropy of adsorption, ΔS_{ads} (Jmol⁻¹ K⁻¹) were calculated from the equation:

$\Delta G_{ads} = \Delta H_{ads} - T \Delta S_{ads}$…………………………………….. (8)

A plot of ΔG_{ads} versus T (K) gave a straight line with a gradient (slope) of (-ΔS_{ads}). From the values of ΔG_{ads} and ΔS_{ads}, the adsorption enthalpy ΔH_{ads} was calculated.
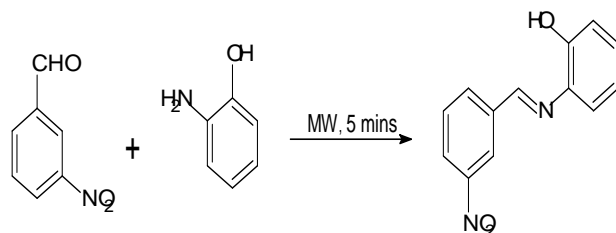**ix.** Langmuir isotherm is given by the plot of:C/θ versus C (Abiola and Tobun 2010 [2]; Kumar *et al.*, 2010) [16]

% inhibitor efficiency (H_2 gas evolution)

$$= \frac{V_b - V_i}{Vb} \, x \, 100\% \quad \text{……………………………… (9)}$$

2. Experimental
2.1. Synthesis of 2-[(3-nitrobenzylidene) amino] Phenol (SBNAP).
SBNAP was prepared by the condensation reaction of 3-nitrobenzaldehyde and 2-aminophenol as illustrated in scheme 1.

Scheme 1: Synthetic route to SBNAP

4.5336 g (0.030 mol) of 3-nitrobenzaldehyde was mixed with 3.2736 g 2-aminophenol (0.030 mol) in a conical flask. The mixture was stirred thoroughly and allowed to heat in a microwave oven; after 3minutes of heating, there were foams in form of smoke coming out of the microwave. The reaction was completed after 5 minutes of heating. The mixture was brought out, allowed to cool, then, ethanol was added to it and heated on a heating mantle, and this was allowed to cool and filtered. The filtrate was recrystallized with water to obtain SBNAP yellow crystalline compound (melting point: 98-100°C; yield: 71.00%).

2.2. Preparation of test solutions
Test solutions were prepared using distilled water.

2.2.1. Preparation of Sulphuric acid Solution.
2 M stock solution of 98.07% sulphuric acid was prepared by diluting 111 cm^3 of the raw acid with distilled water in 1000 ml volumetric flask and made up to the mark.

2.2.2 Preparation of 2-[(3-nitrobenzylidene)amino] phenol (SBNAP)
250 ml solution of 1.0×10^{-2} M SBNAP was prepared by dissolving 0.610 g of SBNAP in 2 M H_2SO_4 solution in 250 ml volumetric flask. Other concentrations (1.0×10^{-3} M, 1.0×10^{-4} M, 1.0×10^{-5} M and 1.0×10^{-6} M) were prepared from the stock solution by diluting with calculated volumes of 2 M H_2SO_4 solution.

2.3. Preparation of Metal Coupons
The 3SR aluminium alloy sheet of 0.052 cm thickness, 98.5076% purity and mild steel of 0.1 cm thickness used for the study were cut into 4cm by 5 cm coupon size for weight loss and 2 cm by 5 cm for hydrogen gas evolution measurements. The coupons were polished using sand-paper and a hole of 2mm was opened at one edge of each coupon. A rubber thread of 25 cm was passed through the opened hole of each coupon. The coupons were soaked in ethanol solution for 10 minutes for de-greasing, rinsed with distilled water, dried in acetone, then heated to Constant weight and finally kept in a desicator containing calcium chloride.

2.4. Temperature of Corrosion Medium
The temperatures under which this study was carried out were 30 °C, 40 °C and 50 °C. The thermostated water bath was used to maintain the 40 °C and 50 °C higher temperatures required over the given period of the experiment.

2.5. Weight loss Determination
The dried coupons of mild steel were carefully weighed, each placed in different 250 ml beakers containing 2 M concentrations of sulphuric acid, at 30 °C, 40 °C and 50 °C; they were retrieved at 6 hours interval progressively for 48 hours. The same experiment was carried out with aluminium

alloy, however, unlike mild steel, the aluminium coupons were retrieved at 30 minutes intervals progressively for 240 minutes (4 hours).

The mild steel coupon retrieved were immediately brushed with iron brush, washed in water, rinsed in ethanol and acetone, then dried inside a 1000 ml beaker place in a thermostat heating mantle to Constant weight before final weighing. The weight loss was calculated in grams as the difference between the initial weight prior to immersion, and weight after removal of the corrosion product. Each reading reported was an average of two readings recorded to the 0.0l mg. The above procedure was also used for the aluminium coupons (Toliwal *et al.*, 2010) [20].

Experiments were repeated with the introduction of five different concentrations (1.0×10^{-2} M, 1.0×10^{-3} M, 1.0×10^{-4} M, 1.0×10^{-5} M, and 1.0×10^{-6} M) of SBNAP in 2 M H_2SO_4 solutions at 30°C, 40°C and 50 °C.

2.6. Gasometric Measurements
Eighteen aluminium coupons (2 cm x 5 cm x 0.052 cm) were used in the experiments for test solutions containing 8 M H_2SO_4 with the five different concentrations of the SBNAP additives. Also, corrosion inhibition experiments involving eighteen mild steel coupons of 2 cm x 5 cm x 0.1 cm in 8 M H_2SO_4 acid solutions with the additives were carried out. The two faces of each of the metal coupons had 20.0 cm^2 total surface area.

The volume of the test solutions used in each experiment was 50 ml. A 50 ml of each test solution was introduced into the reaction vessel connected to a burette through a delivery tube. The initial volume of air in the burette was recorded. One metal coupon was dropped into the test solution and the reaction vessel quickly closed. The various possible outlets where the expected hydrogen gas from the reaction cell would likely escape were properly sealed with a masking tape. The volume of hydrogen gas evolved was recorded every 1 minute for 30 minutes for Al in 8 M H_2SO_4 test solutions. Each experiment was conducted on a fresh specimen of metal coupon. The hydrogen gas evolved displaced the fluid in the gasometric set - up, which was read directly. The experiment was repeated in the presence of the five different concentrations of 1.0×10^{-2} M to 1.0×10^{-6} M for the studied Schiff base.
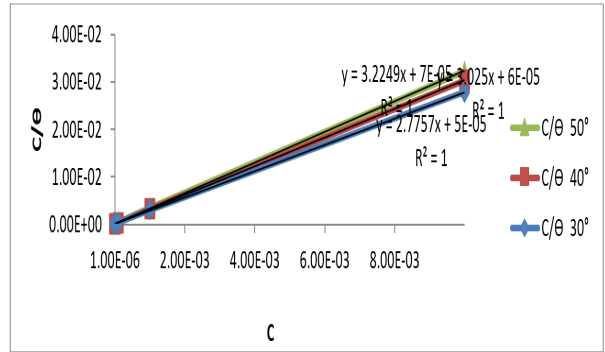
3. Results and Discussion
### 3.1. Data presentation in Figures
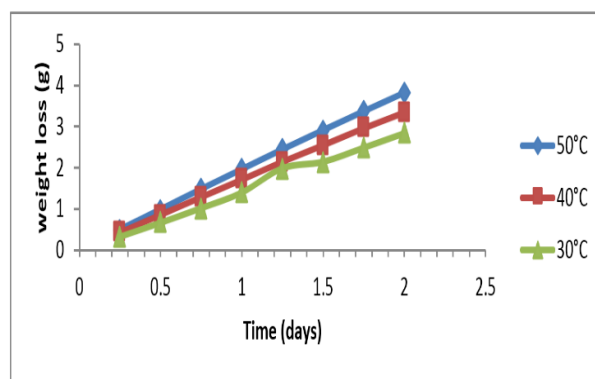
Fig 1: Variation of Weight loss (g) with time (days) for mild steel in 2 M H_2SO_4 without inhibitor at different temperatures.
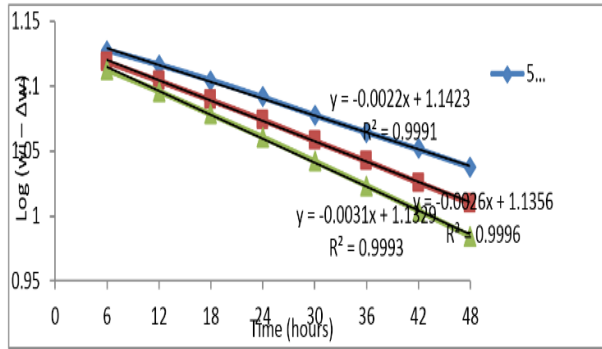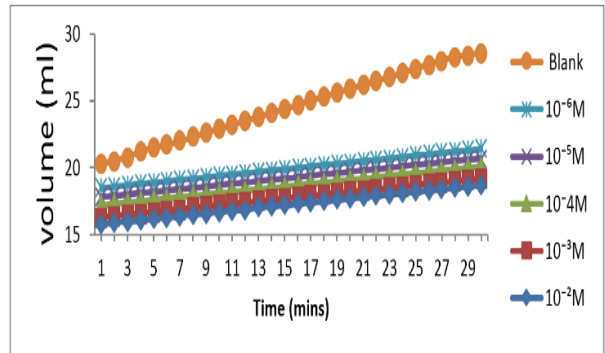
Fig 2: Variation of Log (wi – Δw) with time (hours) for mild steel in 2 M H₂SO₄ without inhibitor at different temperatures.
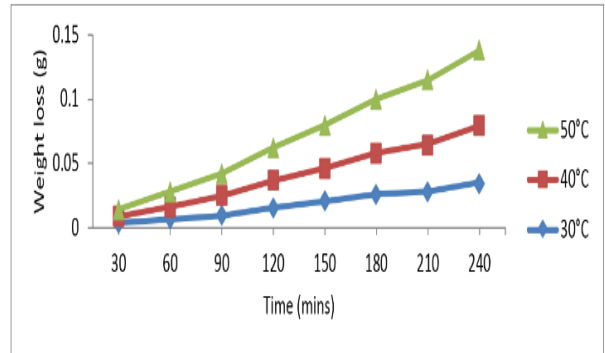

Fig 3: Variation of Weight loss (g) with time (mins) for the corrosion of mild steel coupons in 2 M H₂SO₄ solution containing different concentrations of SBNAP at 30 °C

Fig 4: Variation of Log (wi-Δw) with time (hours) for mild steel coupon dissolution in 2 M H₂SO₄ at different concentrations of SBNAP at 30 °C

Fig 5: Variation of Inhibitor Efficiency with inhibitor concentration (M) for the corrosion of mild steel coupons in 2 M H₂SO₄ solution containing SBNAP at different temperatures.

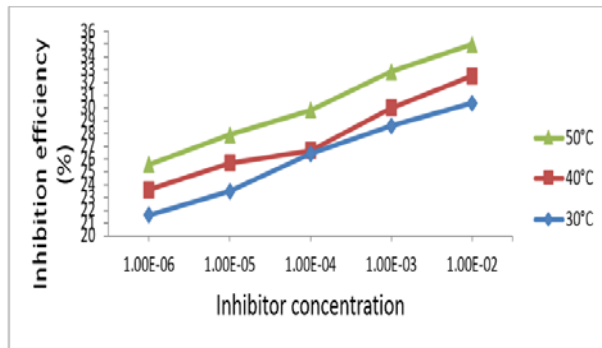**Fig 6:** Langmuir adsorption isotherm (plotted as [C/θ]) versus C for the inhibition of mild steel corrosion in 2 M H₂SO₄ solution by SBNAP
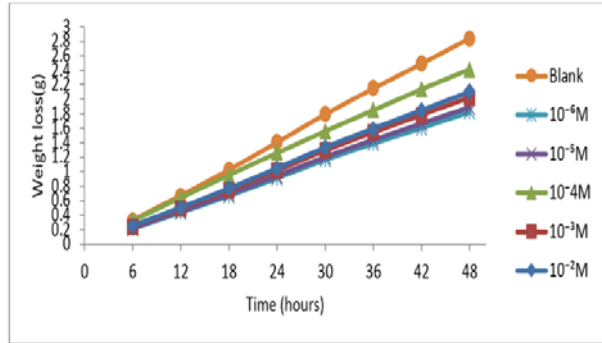
Fig 7: Variation of Volume of hydrogen gas evolved with time (minutes) for the inhibition of mild steel corrosion in 8M H₂SO₄ solution by SBNAP at 30 °C
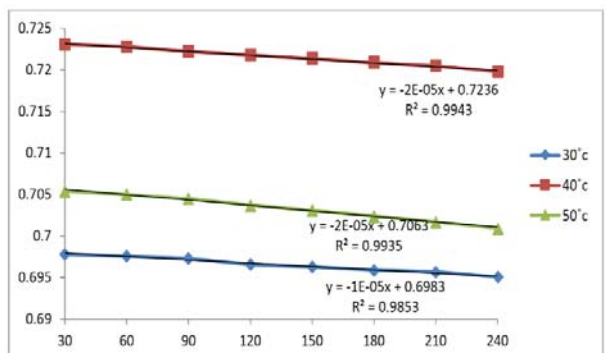

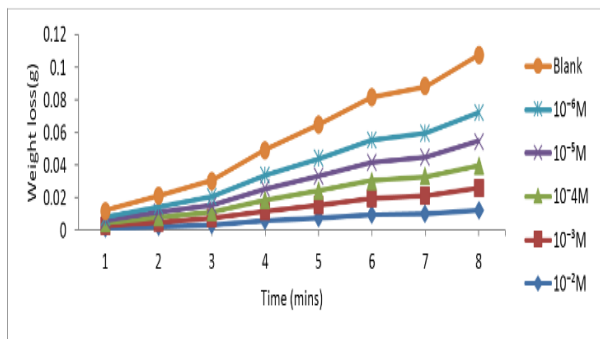**Fig 8:** Variation of Weight loss (g) with time (mins) for aluminium in 2 M H₂SO₄ without inhibitor at different temperatures
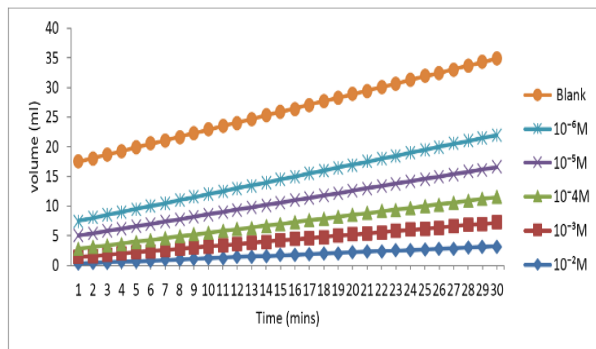

Fig 9: Variation of Log (wi – Δw) with time (mins) for aluminium in 2 M H₂SO₄ without inhibitor at different temperatures.

Fig 10: Variation of weight loss (g) with time (mins) for the corrosion of aluminium coupons in 2M H_2SO_4 solution containing different concentrations of SBNAP at 30 °C

Fig 11: Variation of Log (wi- Δw) with time (mins) for the dissolution of aluminium coupons in 2M H_2SO_4 solution containing different concentrations of SBNAP at 30 °C

Fig 12: Variation of inhibitor efficiency with inhibitor concentration for the corrosion of aluminium coupons in 2 M H_2SO_4 solution containing SBNAP at different temperatures

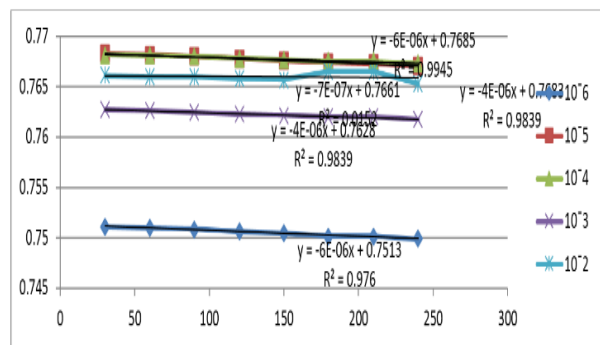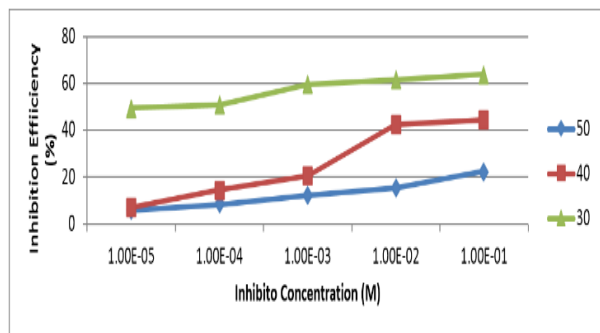**Fig 13:** Langmuir adsorption isotherm (plotted as C/θ versus C) for the inhibition of aluminium corrosion in 2 M H_2SO_4 solution by SBNAP
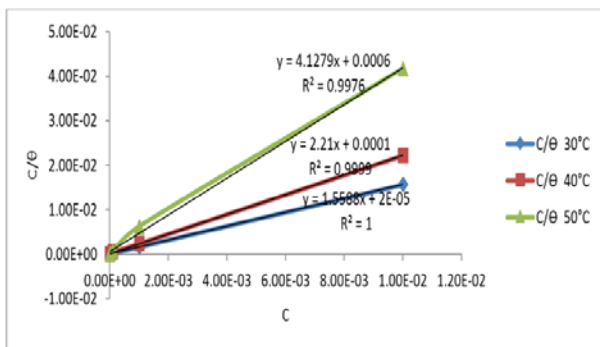

Fig 14: Variation of Volume of hydrogen gas evolved with time (minutes) for the inhibition of aluminium corrosion in 8M H_2SO_4 solution by SBNAP at 30 °C

3.2. Discussion

3.2.1 Effect of temperature on mild steel and aluminium corrosion by H_2SO_4 solutions

The effect of temperature on the corrosion of mild steel and aluminium was carried out and the results presented in Figures 1 and 8 respectively. As the temperature increased from 30 °C to 50 °C the weight loss also increased. It was observed that mild steel had higher corrosion rate than aluminium as the temperature increased from 30 °C to 50 °C, similar observation was made by Orubite and Oforka (2004) [18]. This observation was due to the fact that chemical reaction rates generally increase with rising temperature (Devender, 2010) [9]. Increase in temperature leads to increase in the kinetic energy possessed by the reacting molecules thereby making the molecules to overcome the energy barrier faster. Other factors that may be responsible for the increase in weight loss with increasing temperature were increase in solubility of the protective films on the metals surface, increase in the rate of diffusion and ionization of the reacting species in the corrosion process.

Figures 2 and 9 showed a linear relationship between the plot of log (wi-Δw) against time for mild steel and aluminium respectively in H_2SO_4 without inhibitor at different temperatures. These indicate a first order reaction kinetics with respect to the concentration of H_2SO_4 solution, (Toliwal et al., 2010) [20].

Figures 3 and 10 showed that SBNAP inhibited the corrosion of mild steel and aluminium coupons in H_2SO_4 solution as evidenced by a general decrease in weight loss of the mild steel at the end of the corrosion process. This is in agreement with the findings by James (2006) [15], and Ekpe et al., (2001).

The decrease in the volume of hydrogen gas evolved as the concentration of the inhibitors increased as shown in Figures 7 and 14, implies that the synthesized Schiff base SBNAP inhibits corrosion. This confirms the assertion that increase in concentration of inhibitors increases inhibition efficiency (Ekpe et al., 1995) [11].

3.2.2. Effect of Schiff base concentration on inhibition efficiency

Also, Figures 5 and 12 graphically demonstrated that inhibition efficiency increases with increase in concentration of the inhibitors. This further revealed that the volume of hydrogen gas evolved with time decreases as the concentrations of the Schiff base increased from 1.0 x 10^{-6} M to 1.0 x 10^{-2} M. The highest hydrogen gas evolution was obtained with the uninhibited 2M H_2SO_4 solutions (Figures 7

and 14); (Bansiwal *et al.*, 2000 and Ebenso 1998) [5, 10]. However, the inhibition efficiency of SBNAP increased with increase in temperature in mild steel unlike in aluminium where it decreased.

The plot of log (wi-Δw) against time (hours) at 30 °C, 40 °C and 50 °C temperature levels showed linear graphs, the lines of best fit have an average value of R^2= 0.999; this confirms a first order reaction kinetics for mild steel corrosion in H_2SO_4 solution containing different concentrations of SBNAP as shown in Figures 4 and 11.

3.2.3. Adsorption Isotherms of Schiff base on mild steel
Values of θ were tested graphically for fit to Langmuir adsorption isotherm plots of C/θ versus C were linear as presented in Figures 6 and 13 for the inhibition of the corrosion of mild steel and aluminium in H_2SO_4 acid respectively. The linear correlation coefficient of the fitted data is good (> 0.997). These results confirm that the inhibition is due to the adsorption of the inhibitors onto the metal surface and the adsorption obeys the Langmuir isotherm, (Abiola and Tobun 2010) [2].

For linear and unity gradient, it is assumed there is no interaction between the molecules forming the uniform monolayer on the metal surface with each other. Upadhyay and Mathur (2007) [21] used Langmuir adsorption isotherm to study the characteristics of inhibitors. All the Langmuir adsorption isotherm plots shown suggest physisorption adsorption reaction mechanism, (Abdel-Aal and Morad 2001) [1].

3.2.4. Corrosion Inhibition and adsorption behavior of SBNAP on metal surface
The results obtained from this study reveal that all investigated five Schiff bases inhibit the corrosion of mild steel in H_2SO_4 solutions. The size, shape and orientation of the molecules and the electronic charge on the molecules determine the degree of adsorption as well as the adsorption bond strength and hence the effectiveness of the inhibitors (Chitra *et al.*, 2010, Hosseini *et al.*, 2010) [6, 7]. The investigated Schiff base has – CH = N functional group, two aromatic rings and different substituent groups (scheme 1).

The low inhibition efficiency of SBNAP is attributed to presence of hydroxyl group. Compounds with the electron donating groups are highly soluble in aqueous acid medium leading to the dissolution of the inhibitors' film from the metal surface. The Langmuir adsorption isotherm is obeyed as shown from the plots of C/θ against C for both metals in the studied Schiff base, the plots gave straight lines and the linear correlation coefficient of the fitted data were good (> 0.997). These results confirmed that the inhibition is due to monolayer adsorption of the inhibitors onto the metal surface (Toliwal *et al.*, 2010, Abiola *et al.*, 2011) [20, 2].

3.2.5. Proposed adsorption mechanism of SBNAP on metal surface
The inhibition efficiencies of SBNAP is higher at 30 °C than at 50 °C, suggesting that these Schiff bases are physically adsorbed on the metal surfaces for inhibition to take place causing the blockage of the active corrosion sites. This agrees with the reports of Toliwal *et al.* (2010) [20], Chitra *et al.* (2010) [6, 7], Hosseini *et al.* (2010) and Desai *et al.* (2008). The average activation energy, Ea of SBNAP is 10.88 kJ mol^{-1} and 33.38 kJmol^{-1} for mild steel and aluminium respectively. These values support physical adsorption

mechanism, since it is not above 80 kJmol^{-1} which is expected for chemisorption. This is in agreement with the earlier works of James (2006) [15] and Toliwal *et al.* (2010) [20].

The calculated free energies of adsorption of -7.54 kJ mol^{-1} and -9.00 kJ mol^{-1}, favour the assumption that the inhibition of the corrosion of mild steel is by physical adsorption mechanism, this is because all the calculated values are less than -40 kJmol^{-1} expected for physisorption mechanism. This is supported by the works of Toliwal *et al.* (2010) [20]. The positive values of heat of adsorption 7.57 and 22.91 kJ mol^{-1} for mild steel and aluminium respectively, indicate endothermic reaction.

3.2.6. Proposed adsorption model on mild steel surface for Schiff base
The proposed adsorption model according to Chitra *et al.* (2010) [6, 7] consists of
- electrostatic interaction between the charged molecules and the charged metal surface (favoured by protonation of N atom of –CH=N group),
- Interaction of unshared electron pairs on the hereroatom in the molecule with the metal surface,
- Interaction of π-electrons of aromatic ring and CH=N-groups with the metal surface. These interactions are facilitated by the flat orientation of the molecule with respect to the metal.

4. Conclusion
The two methods used for this study confirm that SBNAP inhibited the corrosion of mild steel and aluminium in 2 M H_2SO_4 solutions.

The corrosion rates of mild steel and aluminium in 2 M H_2SO_4 acid solutions without inhibitor increased with increase in temperature within 30 °C – 50 °C.

The corrosion rates of mild steel and aluminium in 2 M H_2SO_4 acid solutions in the presence of studied Schiff base decreased with increase in surface coverage θ.

The Schiff base SBNAP is found to inhibit acid corrosion at higher temperature for mild steel but fails with aluminium.

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