EVALUATION OF INHIBITIVE PROPERTIESOF ORGANIC NANO-PARTICLES OBTAINED FROM THE BARKS OF *FICUS THONNINGII* BLENDED WITH *HYPTIS SUAVEOLENS* EXTRACT AS CORROSION INHIBITOR ON MILD STEEL IN 1M H₂SO₄ SOLUTION

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Abstract: **Globally, researchers are looking for low-cost and good-performance solutions to prevent and reduce corrosion. Over the years traditional (chemical) corrosion inhibitors have been used to prevent corrosion. Although these inhibitors have good efficiency, their use has been limited due to high cost, toxicity, non-biodegradability, and health hazards. This study, evaluated the inhibitive properties of nano-particles obtained from the barks of Ficusthonningii (FT) blended with Hyptis Suaveolens L Poit (HSLP) extract as corrosion inhibitor on mild steel. Leave extracts of HSLP and nano particles prepared from the stem barks of FT were used as corrosion inhibitor for mild steel in 1m H₂SO₄ using weight loss technique. The study was conducted at room and elevated temperatures of 313, 323, 333 and 343K. The test period was varied from 48 to 336 hours at 48 hours interval, at room temperature and 3 hours at each elevated temperature.The experiments were performed with extract concentrations of 0.1, 0.2, 0.3, 0.4 and 0.5 g/L. with 0.5 g of nano particles from barks of FT blended with HSLP in each concentration. Weight loss measurements were used to compute the corrosion rate and inhibition efficiencies in the various concentrations. The results showed that the corrosion rates increased with time of immersion in the absence of inhibitor but decreased with inhibitor concentration and in the blends of the HSLP/FT nano particles at room and elevated temperatures, while inhibition efficiency increased with increase in inhibitor concentration and in the blends of the HSLP/FT nano particles at room temperature but decreased with temperature rise. Inhibition efficiencies of 93.54 and 96.01% at 0.5 g/L were obtained for HSLP and HSLP/FT respectively at room temperature. At elevated temperatures of 313K to 343K inhibition efficiencies reduced to a minimum of 70% at 0.5 g/L and temperature of 343K implying desorption of inhibitor at higher temperatures. Based on the results obtained, the study concluded that, the extract and blends with nano particles of FT acted as good inhibitors for mild steel corrosion in acidic medium with inhibition efficiencies above 70%. hence, these inhibitors can be used for corrosion control and prevention.**

Keywords- **Organic; nanoparticles; Mild Steel; Ficus Thonningii and Corrosion Inhibitor**

I. INTRODUCTION

Corrosion of mild steel has been a major challenge in various industries, leading to significant economic and environmental costs. The continuous deterioration of metallic structures, especially mild steel, due to corrosion is a concern as it results to economic losses and environmental consequences globally[1]. This deterioration not only affects the structural integrity of materials but also has a negative impact on ecosystems and human safety. The environmental impact resulting from corrosion is a multifaceted concern. The release of metal ions and rust byproducts into soil and water systems unfavorably affects biodiversity, water quality, and agricultural productivity [2].Consequently, many organic and inorganic inhibitors are developed and used [3]. However, their application is restricted due to the high cost as well as environmental and human toxicity [4]. To overcome these difficulties, a paradigm shift towards sustainable and environmental friendly corrosion inhibition strategies is necessary. These inhibitors not only mitigate corrosion but also align with the growing global consciousness towards eco-friendly practices, ensuring minimal environmental impact throughout their life cycle [5].

In recent years nanomaterials have emerged as promising candidates for this purpose due to their unique properties and environmental friendly characteristics [2]. Nanomaterials can function as inhibitors by adsorbing onto the metal surface, forming protective layers, altering the kinetics of electrochemical reactions, or hindering the transport of corrosive agents to the metal interface [6] thus extending the lifespan of the material [2].

Hyptis Suaveolens(L) Poit Extract and nano-particles of the Barks of Ficus Thonnigil have been characterized to determine their suitability as green corrosion inhibitors for mild steel by[7]. Characterizations of the plant materials was done by qualitative and quantitative analyses, Fourier transforms infrared (FT-IR) spectroscopy and scanning electron microscopy attached with energy dispersive x-ray (SEM/EDX) was done. The phytochemical analysis revealed that, these materials have active biocomponents; glycosides, saponins, tannins, flavonoids, alkaloids, steroids and phenols that could be responsible for corrosion inhibition of mild steel [7-8]. The FTIR showed that, the plant materials contain functional groups with C-H stretch, O-H stretch, C=C stretch, S=O stretch, C-O stretch, C≡C triple bond and C–H bend at various frequencies which shows the presence of aromatic groups that could be responsible for the inhibitive properties of these materials [9-10]. The SEM/EDX showed high amount of oxygen 73.1 Wt% in the nano particles of Ficus Thoningii. The results of the study established that Hyptis Suaveolens(HSLP) extract blended with nanoparticles from the barks of Ficusthoningii have the potential to be utilized as green corrosion inhibitors on mild steel and could be an alternative source of green corrosion inhibitors which could serve as an alternative to synthetic inhibitors which are expensive, cancerous and environmentally unfriendly. This research therefore, aims to evaluate the inhibitivepotential of nanoparticles from the Barks of Ficus Thonnigii blended with Hyptis Suaveolens(L) Poit Extract as green corrosion inhibitors for mild steel using Weight loss method.

II. MATERIALS AND METHODS

A. Materials
Materials used for this research includes; mild steel coupons, plants materials; the leaves extract of Hyptis Seaveolen L Poit, the barks of Ficusthonningii, methanol, distilled water, ethanol, hydrochloric acid and acetone.

B. Methods
Material Collection and Preparation
The mild steel sheet of composition (wt %): C (0.04), Si (0.004), Mn (0.225), P (0.004), Cu (0.002), Ti (0.007) and the rest Fe was mechanically cut into coupons of $40 \times 30 \times 0.5$ mm. A small hole of 3 mm was drilled at the end of each coupon for its suspension in H_2SO_4 solution with the help of flexible copper wire. The coupons were degreased, polished using different grades of silicon carbide impregnated with the emery paper of 120, 400, 800 and 1000 grits; cleaned with acetone, dried and stored in a desiccator.

Hyptis Suaveolens Extract (HSPL) for the research work were extracted and prepared in accordance with standard procedures as reported by [7].

Ficus Thonningii(FT) tree barks for the research work were processed into nano particles in accordance with standard procedures as reported by [7].

Preparation of Inhibitor Concentrations and Coding
1M H_2SO_4acid solution which is the corrosion media for this study was prepared by diluting in distilled water. The H_2SO_4 had a concentration of 11.96 M from which 1M acidic solution is prepared by diluting 83.61ml of the 11.96 M H_2SO_4 with 900ml of distilled water and making it up to 1000ml of H_2SO_4 solution in line with[11]. Concentrations of 0.1g/L, 0.2g/L, 0.3g/L, 0.4g/L and 0.5g/L were prepared using equation (1). Five beakers contained acid solution with extract; another five beakers contain acid/extract and Ft; as tests media while the 11th beaker contained 1 M H_2SO_4 solution without inhibitor as a control (blank).

$$C_1V_1 = C_2V_2 \qquad (1)$$

$$V_1 = \frac{C_2V_2}{C_1}$$

Where: C_1 is the initial concentration of the extract; V_1 is the initial volume needed to obtain desired concentration; C_2 is the final concentration (working concentration) and V_2 is the final volume (working volume).

The concentrations of the HSLP extract and nanoparticles from barks of Ft used in this study and their combinations are coded and presented in Table 1.

Table 1: Coding for corrosion containers

BLANK (0.0)				
HSLP 1 (0.1)	HSLP 2 (0.2)	HSLP 3 (0.3)	HSLP 4 (0.4)	HSLP 5 (0.5)
HSLP/Ft1(0.1/0.5)	HSLP/Ft2(0.2/0.5)	HSLP/Ft3(0.3/0.5)	HSLP/Ft4(0.4/0.5)	HSLP/Ft5(0.5/0.5)

These labels were used for the beakers and the coupons immersed in them for easy identification and recording.
Where;
HSLP – Hyptis Suaveolens L Poit leaves Extract
FT- Ficusthonningii barks nanoparticles
HSLP/FT- Hyptis Suaveolens L Poit leaves Extract and Ficusthonningii barks nanoparticles

Corrosion Measurement Tests
Weight loss corrosion measurement at room and elevated temperature was employed to determine the corrosion inhibition of Hyptis Suaveolens L Poit leaves Extract and effect of nano-particles of Ficusthonningii barks blended with Hyptic Suaveolen L Poit leaves Extract on corrosion inhibition of mild steel in line with [11].

Weight loss corrosion measurement at room and elevated temperature
Weight loss measurements at room and elevated temperature were conducted according to [12] standard procedure in line with [11]. Also, the influence of temperature on the corrosion behaviour of mild steel in 1 M H_2SO_4 in the absence and presence of the inhibitor extracts and nano particles was investigated by weight loss method at 313K, 323K, 333K and 343K. The difference in weight of the coupons before and after exposure in acidic solution was recorded as weight loss (W) of the metal as presented in equation 2.

$$W = W_o - W_i \qquad (2)$$

Where, W = weight loss in grams (g), Wo = weight before immersion in (g) and Wi = weight after immersion in (g). The corrosion rate was determined using equation 3.

$$C.R. \,(mm/yr) = \frac{87.6\Delta W}{DAT} \qquad (3)$$

Where: ΔW= weight loss in (mg),
D = density of the metal, (g/cm^3),
A = total surface area of the coupon (cm^2),
87.6 is the unit conversion constant, and,
T = time of exposure (hours).
The inhibition efficiency of the samples was determined using equation 4 [13]

$$I.E = \frac{CR_0 - CR_1}{CR_0} \times 100\,\% \qquad (4)$$

Where, IE is inhibition efficiency (%), CR_0 and CR_1 are corrosion rates in the absence and presence of inhibitor respectively.

III.   RESULTS AND DISCUSSION

Effect of Immersion Time on Corrosion Rate at Room Temperature
Weight loss data was used in computing the corrosion rates and the results are presented in Figures 1 – 9. It was observed in Figure 1 that corrosion rates in the blank were higher than in the presence of the various inhibitor concentrations. The corrosion rate decreased from 0.0339 mm/year in the blank to 0.0154mm/year at inhibitor concentration of 0.1 g/L and at 0.5 g/L, it decreased to 0.0035 mm/year. This clearly shows that the HSLP inhibits the corrosion of mild steel in the acidic solution. According to[7] the reduction in corrosion rate in the presence of the inhibitor could be attributed to phytochemical constituents of Hyptis Suaveolens L Poit leaves Extract which contain sulphur, nitrogen and oxygen atoms that act as centers of adsorption on the metal surface. This result is in agreement with the work of [11]who worked on Inhibitive Properties of Acanthospermumhispidum Dc Leaves Extracts on Corrosion of Mild Steel in 1M HCl solution.
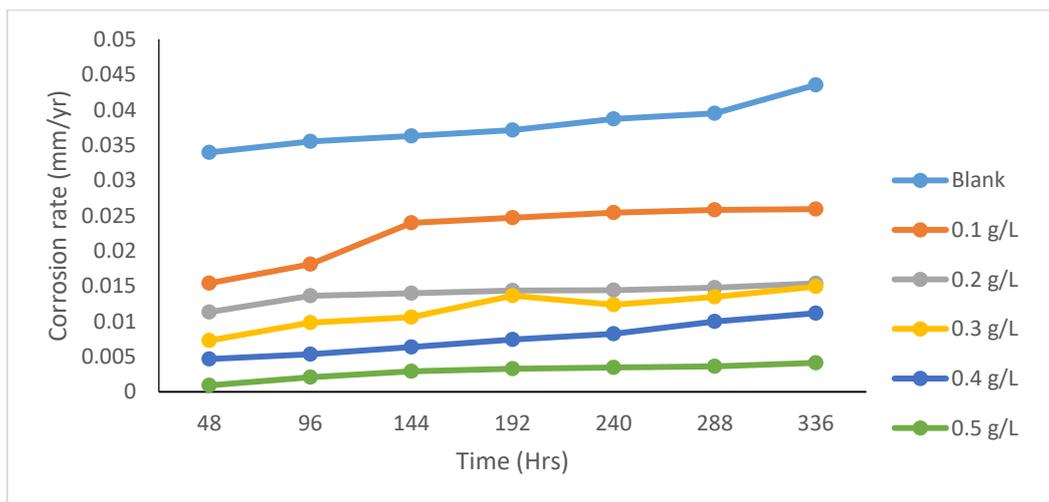

Figure 1: Variation of Corrosion Rate against exposed time in the absence and presence of Hyptis Suaveolens extract in 1M H_2SO_4 Solution.

Effect of Inhibitor Concentration on corrosion Rate

The results of corrosion rate against inhibitor concentration are presented in Figures 2 and 3. It was observed that corrosion rates decreased with increase in inhibitor concentration. The reduction in corrosion rate could be attributed to the fact that at higher concentration, more of the inhibitor species are available to block corrosion sites by forming protective film on the surface of the metal, hence reducing corrosion rate. The values of corrosion rate presented in Figure 2 show that corrosion rates in the presence of HSLP were lower than that of the blank solution. The corrosion rate decreased from 0.0339mm/year in the blank to 0.0035 mm/year in the presence of HSLP at 0.5 g/L concentration.

In the presence of the blends of HSLP and F_T Nano particles (Figure 3) corrosion rate was observed to have decreased from 0.153 mm/year in the blank to 0.00014 mm/year at 0.5 g/L of the blend after 48 hours of immersion. There was a gradual reduction in corrosion rate as the inhibitor concentration increased. This is an indication that at higher concentration the inhibitor blend is able to form a protective film on the mild steel surface, hence reducing the rate of corrosion.The use of nanoparticles enhances the adsorption of the inhibitor with increased surface energy as a result of increased number of surface atoms.This finding is in line with those of [8-14] who worked on different inhibitors and their blends. They concluded that the rate of corrosion was higher in the medium without inhibitor compared to that with inhibitor.
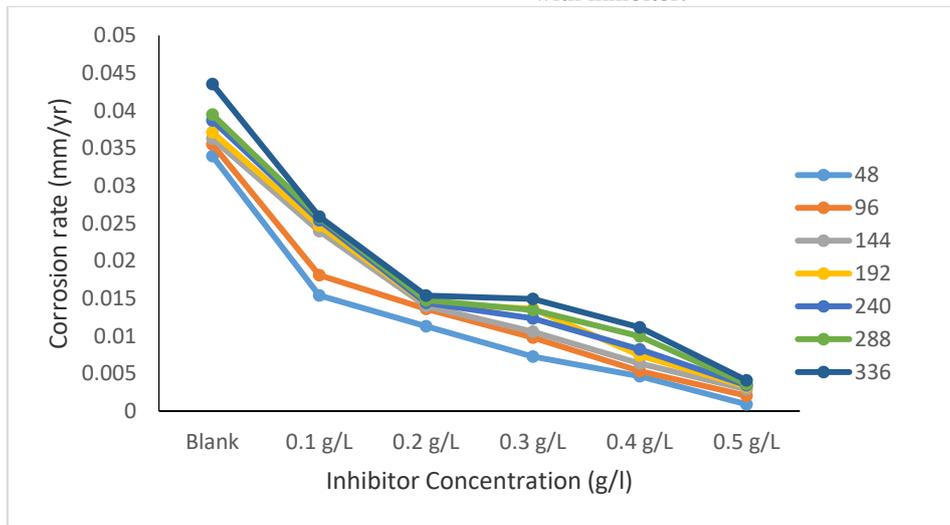

Figure 2: Variation of Corrosion Rate against Inhibitor Concentration in the absence and presence of Hyptis Suaveolens extract in 1M H_2SO_4 Solution.
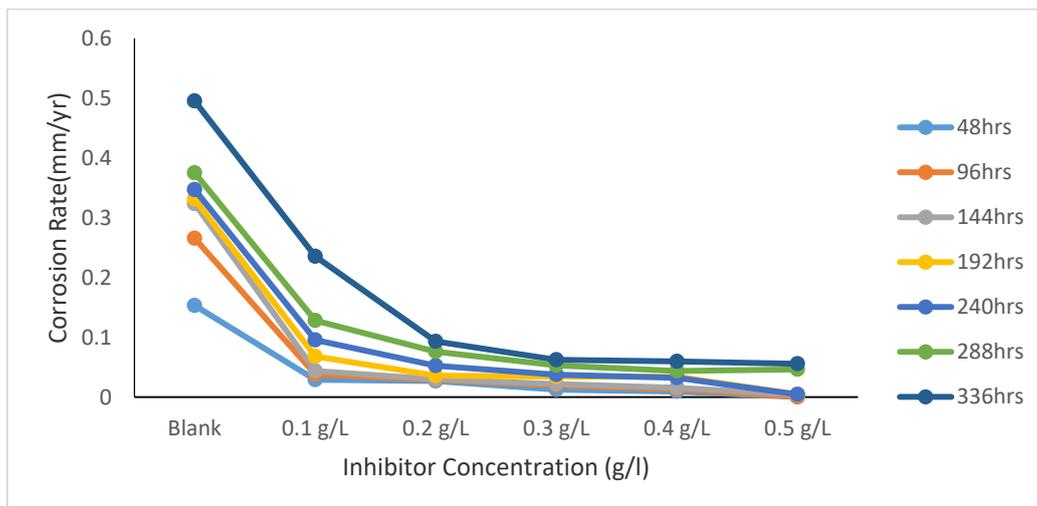

Figure 3: Variation of Corrosion Rate against Inhibitor Concentration in the absence and presence of Hyptis Suaveolens extract and nanoparticles obtained from the barks of Ficus Thonnigii in 1M H_2SO_4 Solution.

Effect of Inhibitor Concentration on Corrosion Inhibition Efficiency

The effect of inhibitor concentration on inhibition efficiency of HSLP and the blends of HSLP/FT are presented in Figures 4 and 5. It was observed from the plots of inhibition efficiency against inhibitor concentration for the extract and blends with nanoparticles that inhibition efficiencies increased with increase in concentration from 0.1 g/L to 0.5 g/L. In the presence of HSLP, the inhibition efficiency increased from 78.13% at 0.1g/L to 93.54% at 0.5 g/L.
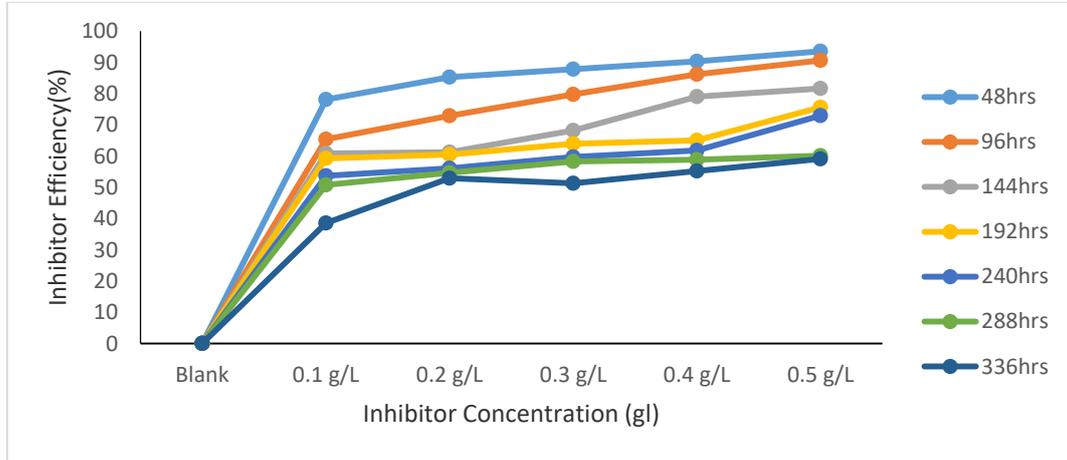
Figure 4: Inhibition Efficiency against Inhibitor Concentration in the Absence and Presence of blends of Hyptis suaveolens extract as inhibitor at room temperature (30±2)

In the presence of the blend of HSLP and FT nano particles the efficiency increased from 78.40% at 0.1g/L to 96.01% at 0.5g/L. This increasing trend of inhibition efficiency with inhibitor concentration could be attributed to the formation of a protective film which constituted a barrier on the metal. This effectively separated the acidic solution from getting in contact with the metallic surface thereby reducing the corrosion reaction rate and enhancing efficiency [14].
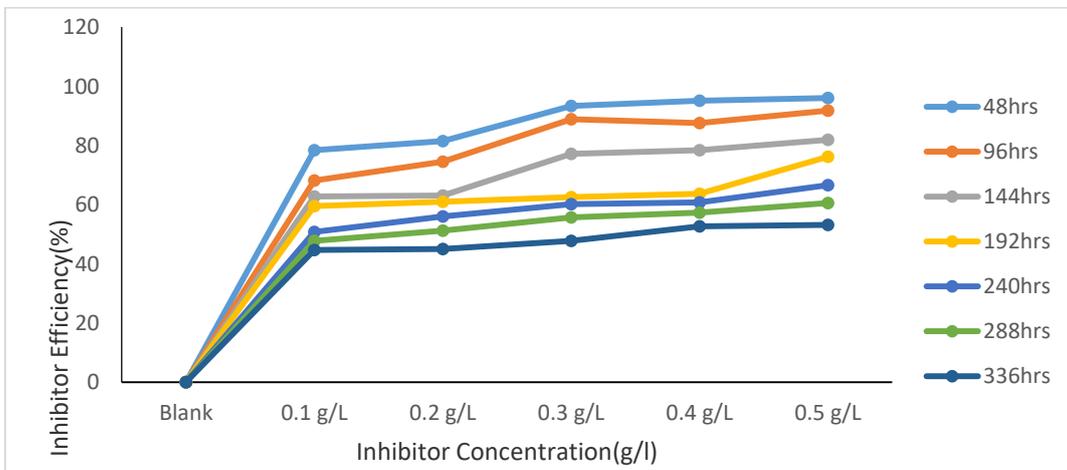

Figure 5: Inhibition Efficiency against blends of Hyptissuaveolens extract in 1M H_2SO_4 and nanoparticles obtained from the barks of Ficusthonningii inhibitor concentration at room temperature (30±2)

Effect of Elevated Temperature on Inhibitor performance
Effect of elevated temperature on weight loss and corrosion rate
The effect of elevated temperatures in the range of 313 K to 343 K on corrosion rate is presented in Figures 6 – 9 for HSLP and HSLP/FT blends respectively. It was observed in Figure 6 that there was increase in the corrosion rate as the temperature was increased at the various inhibitor concentrations. This is expected as chemical reactions usually increase with rise in temperature. It was also observed that the increase in corrosion rate is higher in the control (blank) than in the solution containing inhibitor. This finding is in agreement with that of [15-11].
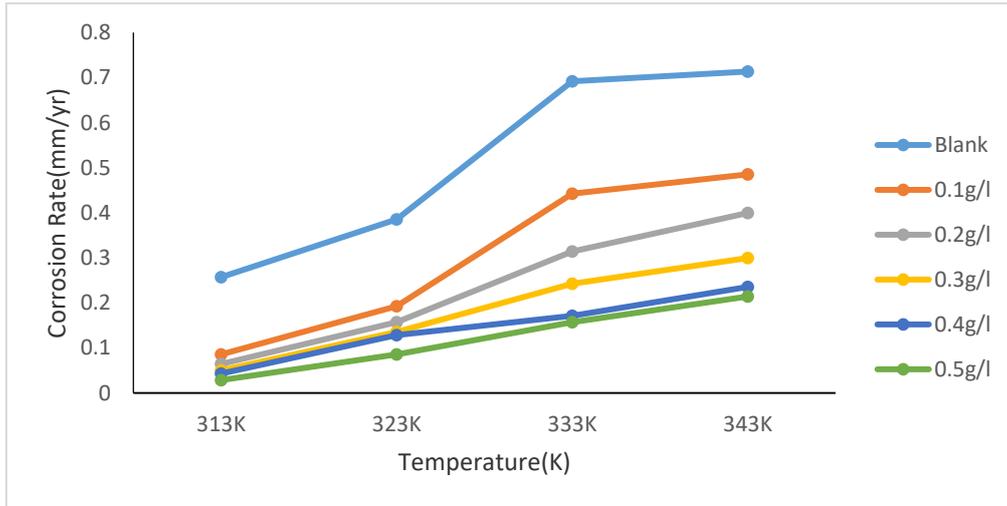
Figure 6: Corrosion Rate against Temperature of Hyptis Suaveolens Extract at Elevated Temperature

The same observations were made in the blends of HSLP and FT nano particles showing that corrosion rate increased with increase in temperature (Figure 7) but decreased with increase in inhibitor concentration. Corrosion rate increased from 0.0125 mm/year at 313K to 0.0189 mm/year at 343 K in the uninhibited solution, but in the inhibited solution containing 0.1 g/L, it decreased from 0.0125 mm/year in the blank to 0.0020 mm/year at 0.1g/L and at the highest concentration of 0.5g/L, corrosion rate reduced from 0.0125 mm/year to 0.0004 mm/year at 313K.
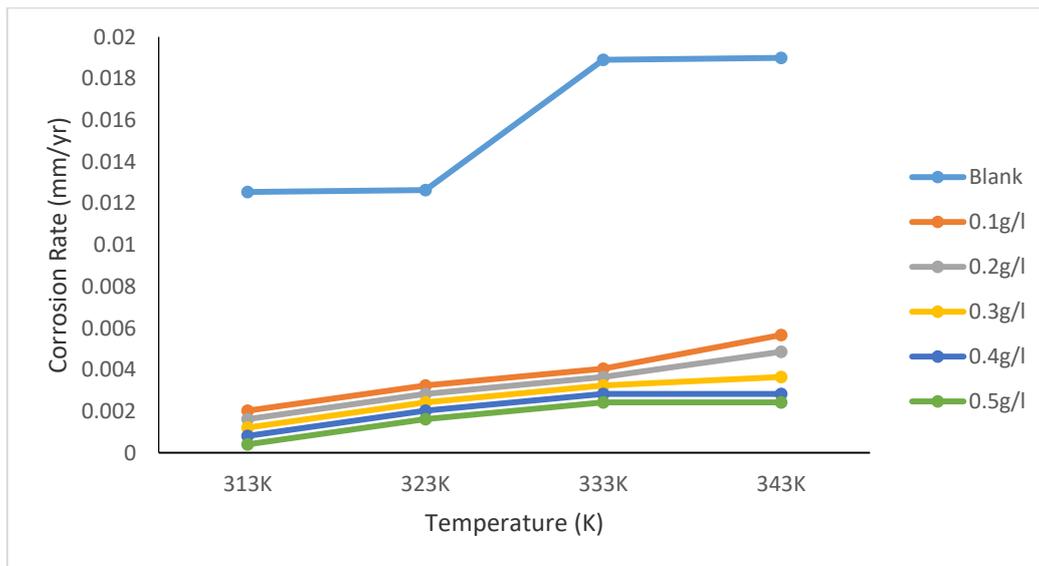

Figure 7: Corrosion Rate against Inhibition Concentration of Hyptis Suaveolens Extract Blend with Bark of Ficus Thonningii (FT) at Elevated Temperatures

Effect of elevated temperature on inhibition efficiency
The result of how temperature change affects inhibition efficiency in the presence of the inhibitors is presented in Figures 8 and 9. It was observed that inhibition efficiency generally decreased with increase in temperature but increased with increasing concentration of inhibitors. This could be attributed to increase in corrosion activation energy in the presence of inhibitor compared to the blank as a result of the formation of an adsorption surface film which implies physical adsorption process taking place [16]. Inhibition efficiency values in the presence of HSLP decreased from 96.8 to 70 %; those of HSLP plus Nano blend were found to reduce from 96.8 to 70 %; at elevated temperatures of 313K to 343K at the highest inhibitor concentration of 0.5 g/L.
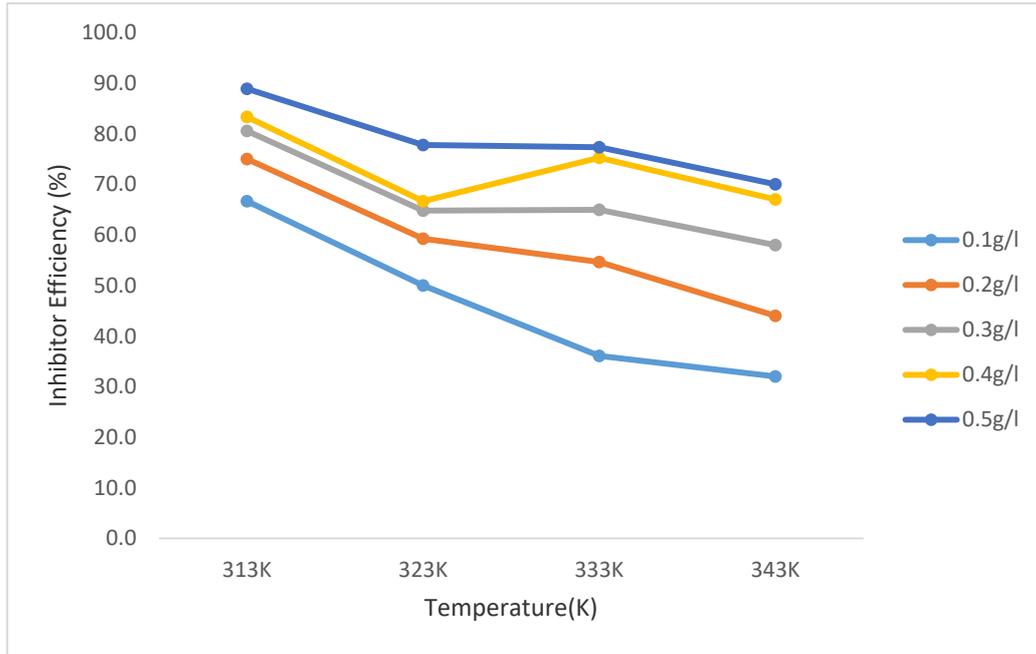
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Figure 8: Inhibitor Efficiency against Temperature of HyptisSuaveolens Extract at Elevated Temperature.
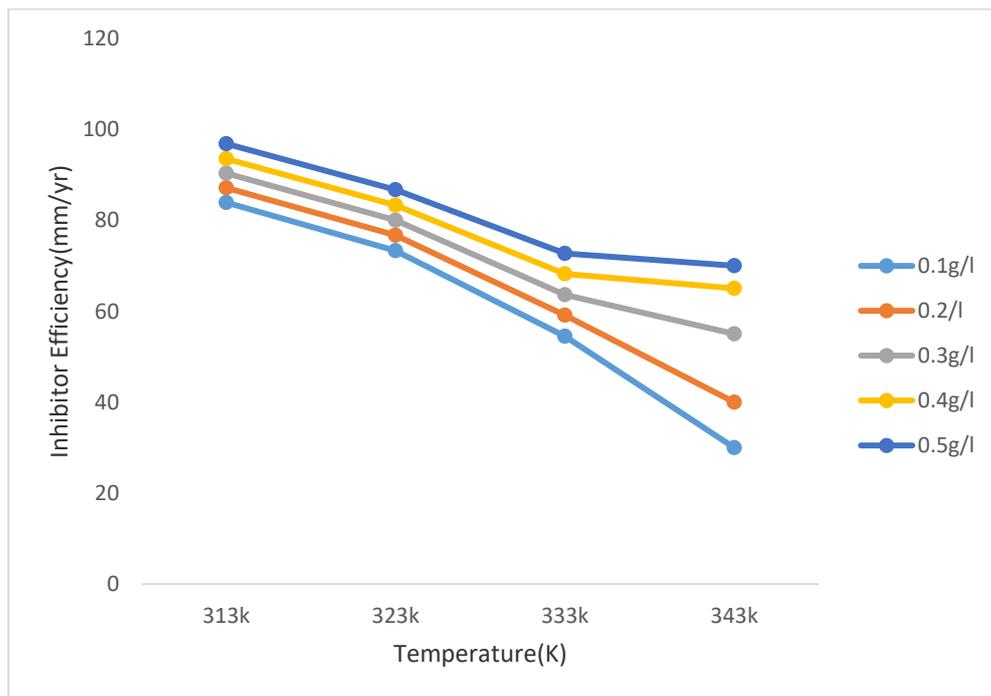

Figure 9: Inhibition Efficiency against Temperature of Hyptis Suaveolens Extract   Blend with nano particles of the Barks of Ficus Thonningii (FT) at Elevated

Temperature

The decrease in inhibition efficiency with rise in temperature confirms that at higher temperatures dissolution of mild steel predominates on the surface. This means that the inhibitor loses strength at higher temperatures suggesting physical adsorption process.

This result shows that the blends of HSLP extract and nanoparticles gave good efficiency at elevated temperature than the extract only. This could be attributed to the presence of nanoparticles which provided increased surface –to-volume ratio with complex compounds to reduce the reaction at the surface and control the corrosion rate by

blocking active sites on the metal surface and provide thermal stability [16].

IV. CONCLUSION

The results of this study show that Hyptis Suaveolens (HSLP) extract blended with nanoparticles from the bark of Ficusthoningii has the potential to inhibit the corrosion of mild steel in H_2SO_4 solution. The corrosion rate increased with immersion time but decreased with increase in inhibitor concentration both at room and elevated temperatures. Inhibition efficiency increased generally with increase in inhibitor concentration. The blend of Hyptis Suaveolens and Ficusthoningii gave good inhibition efficiency at room and elevated temperature. Based on the results obtained, the study recommends that, Hyptis Suaveolens extract blended with nanoparticles from bark of Ficusthoningii can be used as corrosion inhibitors for mild steel at room and at elevated temperatures.

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