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Characterization of Nano-Particles of the Barks of Vitelleria Paradoxa, Ficus Thonnigil and Hyptis Suaveolens Extract as Green Corrosion Inhibitor on Mild Steel

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ABSTRACT
In this research, nano-particles of the Barks of Vitelleria Paradoxa, Ficus Thonnigil and Hyptis Suaveolens Extract have been characterized to determine their suitability as green corrosion inhibitors for mild steel.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). The experimental results showed that, 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. The FTIR showed that, the plant materials containfunctional 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 while the SEM/EDX showedhigh amount of oxygen 64.6 Wt% in the nano particles of Vitelleria paradoxa and 73.1 Wt% in the nano particles of Ficus Thoningii. The results of the study show that Hyptis Suaveolensis (HSLP) extract blended with nanoparticles from the back of Ficus thoningii and Vitelleria Paradoxa 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.

Key words: Green corrosion Inhibitors; Nanoparticle, phytochemical, FTIR and SEM/EDX

I. INTRODUCTION

Corrosion of metals is a serious concern, it is considered an important issue for many industries such as oil and gas, construction, marine, aerospace, petrochemical and in the military (Soufeiani et al., 2020). Globally, researchers are looking for low-cost and good-performance solutions to prevent and reduce corrosion (Yang et al., 2018). 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; chromates and benzimidazole derivatives that can prevent corrosion are very toxic and cause environmental problems (Harb et al., 2020; Edraki et al., 2022). Due to changes in the laws on the use of chemicals, many of these inhibitors are no longer allowed. In recent years, there has been growing interest in exploring alternative corrosion inhibition strategies that can overcome these drawbacks and provide more sustainable and efficient solutions (Bijapur et al., 2023). One such promising approach involves the utilization of nanoparticles of plant materials as corrosion inhibitors. Nanoparticles, defined as particles with dimensions ranging from 1 to 100 nanometers, exhibit unique physical, chemical, and mechanical properties compared to their bulk counterparts. These properties, including high surface area-to-volume ratio, enhanced surface chemistry and reactivity, and quantum size effects, make nanoparticles attractive candidates for corrosion inhibition applications. Nanoparticle based corrosion inhibitors, for example, offer potential advantages over conventional inhibitors, including enhanced effectiveness, reduced toxicity, and greater versatility. Research in this area is ongoing, with efforts focused on exploring novel nanoparticle formulations, understanding their mechanisms of action, and optimizing their performance for specific applications (Elhady et al., 2023).

An inhibitor is a substance (chemical or natural) that, when added in low concentrations (1 to 15,000 ppm) in a corrosive environment, will reduce or prevent corrosion. According to Sheydaei,(2024) an ideal inhibitor should have factors such as no toxicity and pollution problems, long-term effectiveness, good protection at very low concentrations, uniform and localized corrosion suppression, protection of all materials exposed to corrosion species, efficiency under all conditions (temperature and velocity), as well as not creating deposits on the metal surface. Conventional corrosion inhibitors can be broadly classified into organic and inorganic inhibitors based on their chemical composition. Organic inhibitors are typically organic compounds containing heteroatoms such as nitrogen, sulfur, and oxygen, which form complexes with metal ions or adsorb onto metal surfaces, forming a protective film. Examples of organic inhibitors include amines, imidazoles, thioureas, and organic acids as well as π-bonds, and aromatic heterocyclic rings (Proença et al., 2022; Luo et al., 2024). Inorganic inhibitors, are often metal salts or oxides that react with metal surfaces to form passive layers, thereby reducing corrosion rates. Common inorganic inhibitors include chromates, phosphates, nitrites, and molybdates (Marzorati et al., 2018).

In recent years, there has been a paradigm shift towards the use of sustainable plant materials as green corrosion inhibitors, the use of plants extracts as corrosion inhibitors has been studied by several researchers (Marzorati et al., 2018; Elhady et al., 2023 and Sheydaei, 2024) with favorable results.Plant materials(including plant barks, extracts and fruit waste) have received much attention. Many of them have antimicrobial properties in addition to anti-corrosion properties due to being rich in bioactive compounds (Sheydaei,2024). Almost all parts of the plant such as leaves, roots, bark, flowers, fruits, wood and seeds have been used to prepare corrosion inhibitors, because they have abundance of phytochemicals (Ji et al., 2016). According to Yeganeh et al. (2020) their corrosion inhibition results are comparable to conventional organic and anodic inhibitors, and in some cases, they have been more efficient.

According to Sivakumar and Srikanth (2020) many plant compounds have been evaluated for corrosion inhibition, but given that there are different types of plants in the world, there are still numerous types of plants remaining. In this research nano-particles of the barks of Vitelleria Paradoxa, Ficus Thonnigil andHyptis Suaveolens extract have been characterized for use as green corrosion inhibitor on mild steel. These plant materials like other plant materials are less expensive, available, non-toxic, easy preparation method, environmentally friendly, and biodegradable (Sheydaei, 2024). Theseplant materialshave been screened using different tests to determine their potential for corrosion inhibition. By carrying out phytochemical analysis using qualitative and quantitative screening, Fourier transform infrared (FTIR) spectroscopy and scanning electron microscopy (SEM) on the nano-particles.

II. MATERIALS AND METHODS

2.1 MATERIALS

Materials used for this research includes plants materials; the leaves extract of Hyptis Seaveolen L Poit, the bark of Ficus thonningii and bark of Vitellaria paradoxa, methanol, distilled water, ethanol and Sulphuric acid.

2.2 METHODS

The following experimental procedure were followed in the research.

2.2.1 Preparation of Hyptic Suaveolen L poit extract

Freshly harvested Hyptic Suaveolen L Poit leaves were collected and prepared in line with Alan Miralrio and Araceli Espinoza Vázquez (2020). The leaves were washed under running water, air dried and pulverized. The pulverized hyptic Suaveolen L poit (leaves) of 100g was soaked in 500ml of methanol in an extraction thimble for about three days, the liquid extract was allowed to drain into an evaporation dish, and the extract rinsed finally with SME ethanol. The extracted solution was then evaporated on a hot plate. The residue was then scrapped from the dish as the extract. Concentrated Sulphuric acid (H_2SO_4) was later used to dilute/dissolve the dried highly viscous dark brown plant extract. The Hypticsuaveolen L poit extract is shown in Plate 1.

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Plate 1: Photograph of Hyptis Suaveolens L Poit Extract

2.2.2 Preparations of nanoparticles of Vitellaria paradoxa and Ficus thonningii back

50kg each of Vitellaria Paradoxa and Ficus thonningii tree barks were collected and sun dried for four weeks and then Oven dried within temperature range of 40 to 50^{o}C for four hours to eliminate all residue moisture. The Vitellarial peradoxa and Ficus thonningii tree bark were separately crushed to produce macro-fiber particle. The fibres were introduced at separate times in to the ball mill for further reduction. The charged high energy impact grinding ball milling was left to run for 48hrs non-stop for each sample. The ball milling was further carried out for 24hrs to obtain nanoscale particles. Scanning Electron Microscopy (SEM) was undertaken to determine the particle size, distribution, and shape. An average particle size of 100nm was obtained.

(a) (b)

Plate 2: (a) Bark of Vitellaria Paradoxa Nano particles(b) Back of Ficus thonningii Nano particles

2.3 Characterization of Nano-Particles and extracts of Vitellaria paradoxa and Ficus thonningii bark

2.3.1 Phytochemical screening of plant materials

The confirmatory qualitative and quantitative phytochemical screening of plant materials was performed to identify the main classes of compounds present in the extracts following standard protocols. The qualitatively and quantitatively screening was done using phytochemical analysis.

2.3.2 Fourier transforms infrared spectroscopic (FTIR) analysis

Fourier transforms infrared spectroscopic analysis was also conducted to identify the chemical bonding and functional groups present in the extracts and each of the Nano-particles. FTIR was carried out using a spectrometer which has the capability to analyze materials with the passage of infrared light through it. The beam of light from IR source was passed through a monochromatic controller with a selector which ensures that only specified wavelengths from 4000 to 400cm^{-1} are emitted. The sample was placed in a holder in the path of the IR source. A detector reads the analog signal and converts it to a spectrum. A computer was then used to analyze the signals and identify the peaks in the spectrum. The spectra produced for each extract were recorded and used in determining the functional groups present in the extract using the peaks of infrared absorption frequencies in the plot of transmittance against wavenumber.

2.3.3 Scanning electron microscopy (SEM) on nano-particles

Scanning electron Microscopy (SEM) gives morphological examination with direct visualization. The dry Nano particle for each sample of Vitellaria Paradoxa and Ficus thonningill tree barks were dropped on a sticky surface of 1cm square section and spread evenly across the surface to ensure even distribution. The 2 samples were fixed on the base of an aluminium sample holder and subjected to carbon coating. The coated samples were then introduced into SEM machine for imaging. In the machine, the samples were then scanned with a focused fine beam of electrons and the particle images were transmitted to the desktop and printer network. The surface characteristics of the samples were obtained from the secondary electron emitted from the sample surface.

III. RESULTS AND DISCUSSION

3.1 Phytochemical Analysis

The results of the phytochemical analysis of the plant materials are presented in Tables 4 and 5. Table 4 shows the result of qualitative screening while Table 5 gives the quantitative analysis of the main bioactive elements found in these materials.Qualitative screening showed that, these materials have active biocomponents; glycosides, saponins, tannins, flavonoids, alkaloids, steroids and phenols that could be responsible for corrosion inhibition of mild steel.

Table 4: Phytochemical Constituents of Hyptis Suaveolens Extract, nano particles of Vitellaria Paradoxa and Ficus Thoningiiusing Qualitative Screening

S/N	Constituents	Test carried out	Hyptis Suaveolens (Ext)	Vielleria Paradoxa (Vp)	Ficus Thoningii (Ft)
1	Glycosides	Fehling test	+	+	+
2	Glycosides	Killer killiani test	+	+	+
3	Saponins	Frothing test	+	+	+
4	Terpenoids	Salkowski test	+	+	+
5	Tannins	Ferric test	+	+	+
6	Flavonoids	Shinoda test	+	+	+
7	Alkaloids	Dragendorff test	+	+	+
8	Phenols	Ferric chloride test	+	+	+
9	Steroids	Liebermann–Burchard	+	+	+

Keys: + Present, and – Absent

It can be observed from the quantitative analysis result that all the three materials contain varying amounts of phytochemicals. It is observed that the amounts of glycosides, terpenoids, alkaloids, and flavonoids were low in Hyptis Saveolens Extract, Ficus Thonningii bark and Vitelleria Paradixa bark nanoparticles; while steroids, saponins, tannins and phenols were found to be in higher quantities.

This shows that the main inhibitory effect of these materials could be due to the presence of steroids, saponins, tannins and phenols. It is observed that Ficus Thonningii has higher phenols and tannins compared to Hyptis Saveolens extract and Vitelleria paradixa. The phenol content was found to be the highest in all the three materials with HSLP (4.285 GAE), VP (5.13 GAE) and FT (6.25 GAE) respectively.

Table 5: Phytochemical Constituents of Hyptis Suaveolens Extract, nano particles of Vielleria Paradoxa and Ficus Thoningii using Quantitative Screening

S/N	Constituents	Hyptis Suaveolens (Ext)	Vielleria Paradoxa (Vp)	Ficus Thoningii (Ft)
1	Glycosides	0.230 g	0.17 g	0.71 g
2	Saponins	1.33 g	0.945 g	2.2 g
3	Terpenoids	0.6 g	0.83 g	1.33 g
4	Tannins	3.595 g	3.845 g	5.535 g
5	Flavonoids	0.555 g	0.835 g	0.82 g
6	Alkaloids	0.91 %	0.61 %	1.535 %
7	Phenols	4.285 g	5.13 g	6.25 g
8	Steroids	1.095 g	1.275 g	1.625 g

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The presence of tannins in higher quantity shows that these materials are not harmful as reported by several researches (Jiet al., 2016; Proençaet al.,2022 and Luo et al., 2024). The inhibitive action of plant extracts could be attributed to the presence of phytochemical constituents found in them (Sheydaei,2024). These constituents usually contain significant number of oxygen, sulphur and nitrogen atoms incorporated in their structure (Ji et al., 2016). The phytochemical constituents adsorb on the metal surface through the lone pairs of electrons present on the oxygen, sulphur and nitrogen atoms. The adsorption of such compounds on metal surface produces a barrier for charge and mass transfer between the metal and corrodent leading to retardation of the corrosion process.

3.2 Fourier Transform Infrared Spectroscopic (FTIR) analysis

Results of FTIR spectral for the plant materials are shown in Figure2 2-4. FTIR was used to identify the organic compounds present in the studied materials as presented in Table 6. It is observed that the 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 bendat various frequencies which shows the presence of aromatic groups that could be responsible for the inhibitive properties of these materials (Sheydaei,2024). Okewale and Adesina (2020) reported that,these functional groups are responsible for inhibitive effect on mild steel corrosion.

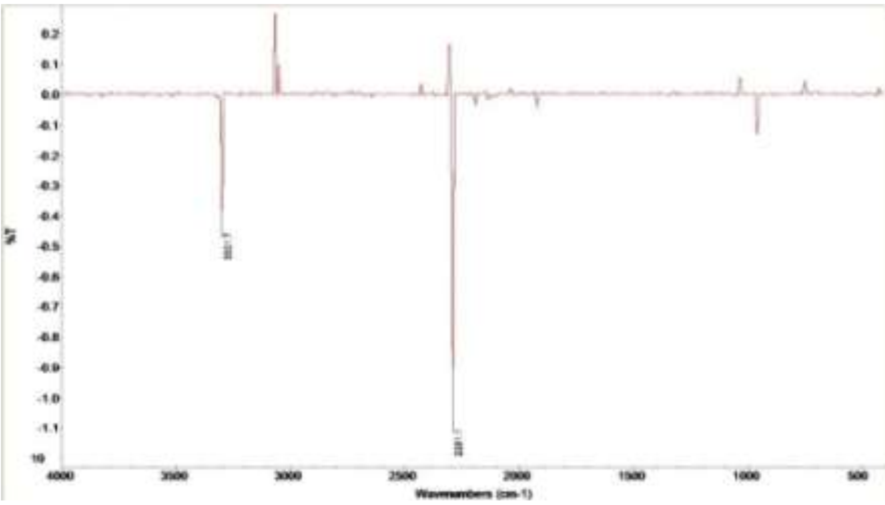

Figure 2: Fourier Transform Infrared Spectral for Hyptis Suaveolensis (HSLP) Extract

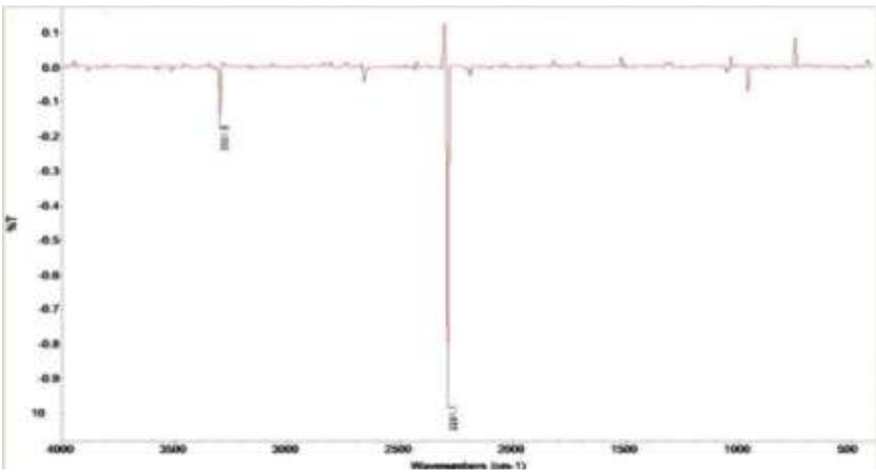

Figure 3:Fourier Transform Infrared Spectral for Nano particles of Vitellaria Paradoxa

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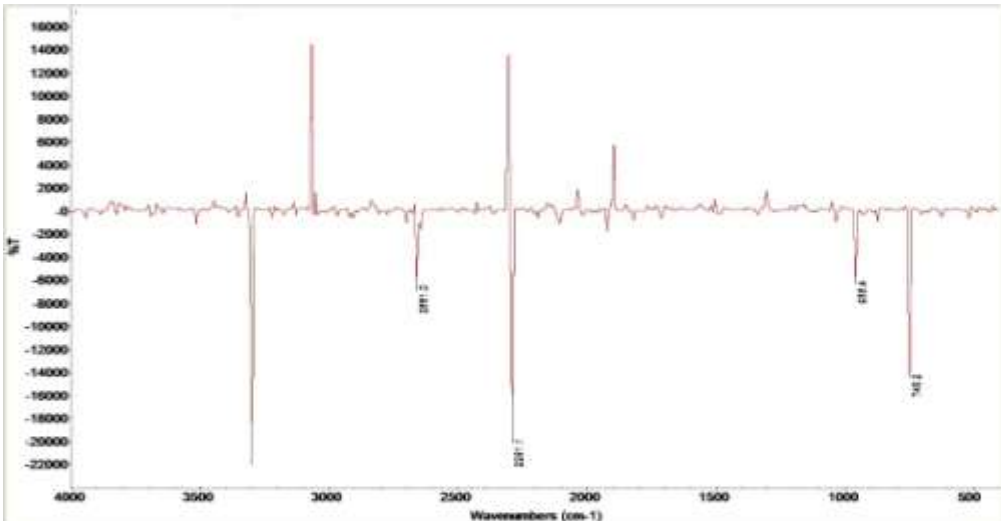

Figure 4: Fourier Transform Infrared Spectral for Nano particles of Ficus Thoningii

These functional groups were observed with peak frequencies in the ranges of 1021.3 - 3301.7 cm^{-1} for HSLP; 1017.6 – 3310.4 cm^{-1} for FT and 1021.3 – 3301.6 cm^{-1} for VP respectively as presented in Table 6. These results also confirm that, the plant materials have anticorrosion potential.

Table 6: Summary of Peaks obtained from FTIR Spectroscopy Analysis for Hyptis Suaveolensis Extract, (HSLP), Nano particles of Vitellaria Paradoxa (Vp) and Ficus Thoningii (Ft)

S/N	HSLP		Vp		Ft	
	Frequency (cm^{-1})	**Functional group**	**Frequency (cm^{-1})**	**Functional group**	**Frequency (cm^{-1})**	**Functional group**
1	3301.7	O–H stretch	3301.6	O–H stretch	3310.4	O-H Stretch
2	2944.6	C–H stretch	2944.6	C–H stretch	2.6	C–H stretch
3	2832.8	C–H stretch	2691.1	C–H stretch	2691.8	C–H stretch
4	2291.1	C≡C triple bond	2291.1	C≡C triple bond	2299.9	C≡C triple bond
5	1654.9	C=C stretch	1654.9	C=C stretch	1650.0	C=C stretch
6	1449.9	C–H bend	1449.9	C–H bend	1449.9	C–H bend
7	1408.9	S = O stretch	1408.9	S = O stretch	1408.9	S = O stretch
8	1110.7	C – O stretch	1110.7	C – O stretch	1114.5	C – O stretch
9	1021.3	C – O stretch	1021.3	C – O stretch	1017.6	C – O stretch

3.3 Scanning Electron Microscopy (SEM/EDX) on Nano-Particles

Results of the scanning electron microscopy (SEM) on the nano-particles of plant materials are shown in Figures 5 and 6.The results shows high amount of oxygen 64.6 Wt% in the nano particles of Vitelleria paradoxa and 73.1 Wt%in the nano particles ofFicus Thoningii. According to Luo et al. (2024) organic inhibitors contain heteroatoms such as oxygen, sulfur, and nitrogen which form complexes with metal ions or adsorb onto metal surfaces, forming a protective film.

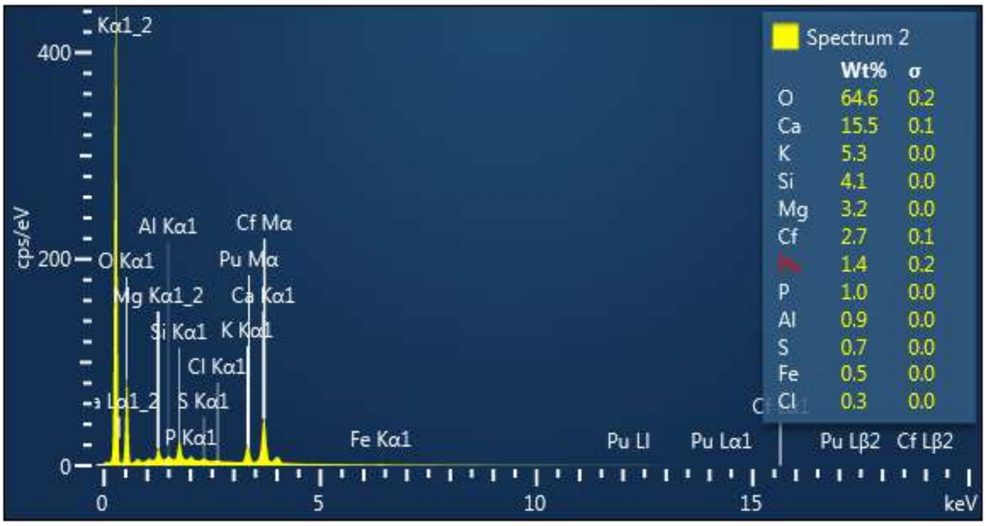

Figure 4: SEM/EDX Images of Vitelleria paradoxa nanoparticles @ 10µ

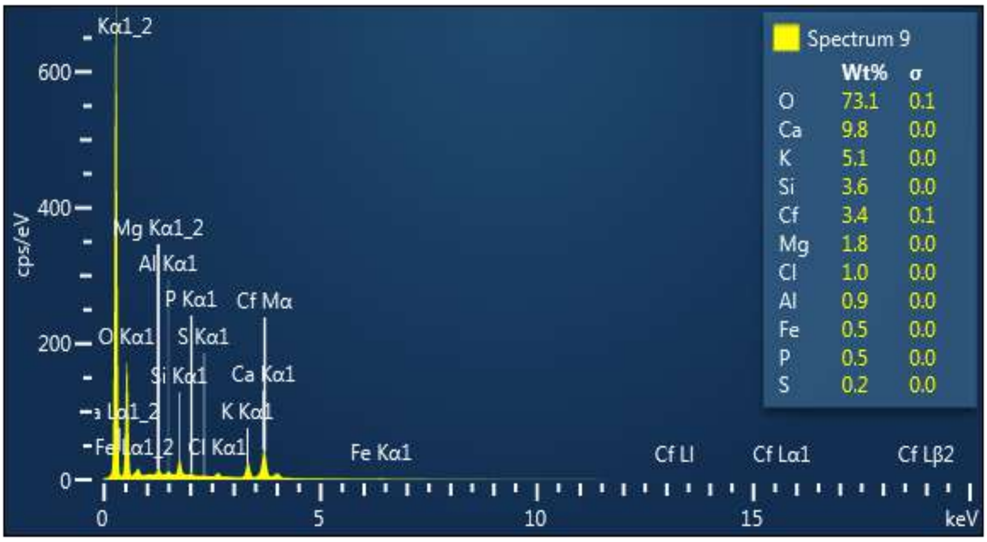

Figure 5: SEM/EDX Images of Ficus Thoningii nanoparticles @ 10µ

IV. CONCLUSION

Corrosion inhibitors are rarely used as a single compound. The formulation can be composed of two or more inhibitors which may have different characteristics. This is due to some factors: A single inhibitor can only inhibit the corrosion of few metals, when the environment involves multi-metal systems, the inhibitive action may sometimes cause jeopardizing effects to other metals. Advantages from anodic and cathodic inhibitors can be combined and optimized for best performance. Hence, (Hyptissuaveolens L Poit), bark of Vitellaria Paradoxa nano- particles and bark of Ficus thonningiinanoparticles and their blend may be found veritable as a synergy in inhibiting corrosion on mild steel.

Phytochemical screening of HSLP extract, FT and VP Nano particles revealed that they containbioactive constituents such as alkaloids, tannins, flavonoids, saponins, terpenoids, phenols, glycosides and steroids which have inhibitive properties. SEM on the nano-particles of the plant materials showed high percentage of oxygen. The results of the study show that Hyptis Suaveolensis (HSLP) extract blended with nanoparticles from the back of Ficus thoningii and Vitelleria Paradoxa have the potential to be utilized as green corrosion inhibitors on mild steel and could be an alternative source of green corrosion inhibitors which are non-toxic and easy to prepare.

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