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Gravimetric Performance Evaluation of Wood Vinegar Liquor and Wood Vinegar Liquor Nano-fluid Corrosion Inhibitors

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ABSTRACT

This research investigated the performances of Wood Vinegar Liquor (WVL) and Wood Vinegar Liquor Nanofluid (WVL+NP) as corrosion inhibitors on micro-alloyed steel immersed in 1.0 M HCl solution. Gravimetric method was used to determine effectiveness of the inhibitors by monitoring corrosion rates and evaluating inhibition efficiencies of inhibited and control (1.0 M HCl) solutions. The micro-alloyed steel samples were immersed in 1.0 M HCl, WVL-inhibited 1.0 M HCl and WVL+NP-inhibited 1.0 M HCl solutions respectively, for periods ranging 24 – 168 hours at a step of 24 hours. Results obtained indicates that from 24 - 72 hours, samples immersed in 1.0 M HCl, WVL-inhibited 1.0 M HCl and WVL+NP-inhibited 1.0 M HCl solutions experienced rises in corrosion rates, for which samples immersed in 1.0 M HCl recorded highest corrosion rate values within these periods. However, samples immersed in 1.0 M HCl showed decline in corrosion rate after reaching a maximum of 36.19 mmpy at 72 hours, to 12.29 mmpy at 168 hours. Whereas, Samples immersed in WVL-inhibited 1.0 M HCl and WVL+NP-inhibited 1.0 M HCl solutions continued to experience increased corrosion rates beyond 72 hours, attaining their maximum values of 30.44 and 30.79 mmpy respectively at 96 hours. Corrosion inhibition efficiencies evaluated showed that WVL inhibited corrosion of micro-alloyed steel in 1.0 M HCl by recording higher values in the range of 53.85 – 12.47% between 24 – 96 hours of immersion. When these inhibitors are used for period beyond 96 hours, they are like to worsen corrosive effect of 1.0 M HCl.

Keywords: *Nanofluid, Wood vinegar Liquor, Corrosion Inhibition, Weight Loss, Gasification Bye-product*

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INTRODUCTION

There is a trend towards using green inhibitors for the control of corrosion focusing on the demand for eco-friendly solutions that handle corrosion issues while protecting the eco system. Corrosion is a significant threat in underground pipelines during drilling and refinery (Okonkwo *et al.*, 2024). When metals and alloys corrode it react with the environment which result to expensive repairs and raises serious environmental concerns (Lenasi, 2023). The current solution in the market is based on heavy and harmful chemicals which causes deforestation, loss of aquatic lives, health risk in humans and as well environmental pollution.

The greatest fear in engineering industries is corrosion especially in carbon steel. Rusting of carbon steel makes it weaker thereby reducing its lifespan (Dwivedi *et al.*, 2017). Carbon steel is common as it is resistant, easy to avail and cheap but is quickly degraded by substances like salt, acidic solutions or air pollution (Verma *et al.*, 2021). Alloyed carbon steel is a category of steel that contains carbon, iron and small quantities of elements such as manganese, chromium or nickel (Sha *et al.*, 2024). Carbon steel is considered eco-friendly and affordable (Tong, 2022). When low carbon steel is recycled, they are used to make steel pipes, heat exchangers, boilers, pressure tanks which are essential in petroleum, marine and manufacturing industries (Adesusi *et al.*, 2022). The use of environmental substitutes like plant extracts and biodegradable mixtures are potent corrosion inhibitors (Korniy *et al.*, 2023). Iron oxide nanoparticles include compounds like magnetite and hematite are used in corrosion prevention because of their sizes and broad surface area. They help in the breakdown of carbon steel with the properties that make the buildings durable (Vallabani and Singh, 2018). The practice of iron oxide nano particle has advanced significantly from Lefort's early co-precipitation method from the 19th century till present day. Co-precipitation method remained foundational while techniques like hydrothermal, sol-gel, microemulsion and thermal decomposition broaden the particle characteristics (Cristina *et al.*, 2012).

Green corrosion inhibitors are environmentally friendly substances which are made from natural, non-toxic and renewable resources which reduces environmental pollution and the risk involved. They are easy to access and cost effective (Okonkwo *et al.*, 2024). Conventional Corrosion Inhibitors produce protective films that reduce corrosion especially those that contain hexavalent chromium (Cr VI) which are very toxic and harmful to the environment (Sneddon, 2012). The use of natural plant extract combined with nanoparticle will aid in spreading the nanoparticle and making them strong enough to preventing corrosion (Antunes et al., 2023). Plant extracts contain many bioactive elements which provide effective protection. Whenever these compounds settle on metallic surfaces, they form a physico-chemical barrier on the metallic surface retarding the kinetics of the corrosive reactions. Simultaneous antioxidant functions also reduce the negative redox effects that are present along with corrosion (Gabsi *et al.*, 2023). Wood vinegar liquor has been in use for over 2000 years ago in countries such as China, Greece, Japan and India. These countries used it for agricultural, medicinal and ceremonial purposes. Wood vinegar liquor is a dark liquid that is produced during destructive distillation of wood. It is produced by the heating of wood with oxygen hence affordable, biodegradable renewable and eco-friendly (Bouket *et al.*, 2022). It is rich in organic compounds like phenolics, flavonoids, polyphenolics. Heating occurs in the absence of oxygen at a temperature of 300^{o}C and 500^{o}C. In this process vaporization occurs. After the vapors it condenses in form of a liquid which separates in several layers with wood vinegar constituting one of them and solid impurities is removed. The liquid is frequently distilled further to increase the purity of the liquid. The purified wood vinegar is used in agricultural purposes and corrosion prevention (Iacomino *et al.*, 2024). Pyrolysis is carried out at atmospheric pressure, high pressure where uncontrolled combustion is avoided. In wood vinegar liquor Phenolic compounds are classes of aromatic organic compounds that contain one or more hydroxyl group attached to a benzene ring. These compounds are formed by the thermal degradation of liglin a complex polymer (Theapparat *et al.*, 2014). Phenols are effective at neutralizing free radicals. Their hydroxyl group releases hydrogen atoms that aid to stabilize these reactive molecules contributing to oxidative stress. The antioxidant strength is enhanced by the number and location of hydroxyl and methoxy groups on the aromatic ring. This property is important in food preservation and inflammation (Liu *et al.*, 2020). Phenolic compounds are interactive with metals, proteins, and biological tissues because of the aromatic rings and hydroxyl groups. The interaction helps them to bind with other molecules, function as chelators, and in redox processes. In materials science, phenolic acids are added to biomaterials to improve their healing capabilities and antimicrobial effects (Lobiuc *et al.*, 2023). Long fatty acid aid corrosion prevention, such fatty acids form a hydrophobic film on metal surfaces. The layer keeps out moisture, oxygen, acidic elements and the metal is not able to corrode or perish. The greater the length of the chain of the fatty acids, the more effective it is in corrosion prevention. Fatty acid coatings are used in industries to increase the life span of pipelines during harsh conditions (Esm *et al.*, 2024). With this understanding, this research is focused on studying the potential of wood vinegar liquor and wood vinegar liquor nanofluid as corrosion inhibitors.

MATERIALS AND METHODS

Material

Micro-alloyed carbon steel was purchased in Abeokuta Ogun state, Nigeria. The Hydrochloric acid was obtained from Benin City, Edo state, Nigeria. Wood Vinegar Liquor (WVL) was obtained as bye-product from wood gasification. Iron-oxide nanoparticle was obtained by allowing iron to self-corrode under atmospheric condition and latter sieved to obtain required nano-size. WVL nanofluid (WVL+NP) was obtained by mixing 1.0 g of iron-oxide nanoparticle with 100 mL of WVL using magnetic stirrer operated at 800 rpm for 1 hour. The equipment used include hack saw for cutting the micro alloyed carbon steel into required sizes, weighing balance was used to determine the weight loss, magnetic stirrer and beakers. Wood vinegar liquor obtained as bye-product wood gasification, iron-oxide nanoparticle.

Methods

The samples were prepared for the gravimetric experiment work by machining on center lathe machine to a diameter of 23 mm and cut into plate of 7 mm thickness as:

Sample A: immersed in 1.0 M HCl blank
Sample B: immersed in 1.0 M HCl inhibited by WVL
Sample C: immersed in 1.0 M HCl inhibited by WVL+NP

Figure 1: Immersion test samples setup

The machined samples were subsequently polished on surface grinding machine and abraded using grades of emery clothes to achieve mirror-like surface. Each sample prepared were provided with a hole close to one end of its circumference for anchoring, by boring on a drilling machine. Samples were degreased in ethanol and acetone, and rinsed with double distilled water. The samples were dried in oven before being stored in a desiccator prior to immersion in corrosive medium for gravimetric experimental work. It was weighed to obtain initial weight (W_i) before being anchored to polymer-made rope, and suspended over beaker containing 200 mL of 1.0 M HCl in which each sample was immersed, Figure 1 shows setup of the immersion process. The immersion period covered 24 – 168 hours at a step of 24 hours at room temperature. After each immersion, samples were withdrawn from the corrosive solution, degreased in ethanol and acetone, and washed in double distilled water using soft polymer-made brush in order to remove all corrosion products from sample surface. Each sample was dried in oven after corrosion product removal and reweighed to obtain a new weight (W_f). The weight loss was evaluated by subtracting W_f from W_i ($\Delta W = W_i - W_f$). Therefore, Corrosion rate (CR) in mmpy was calculated according to Equation 1 (Malaret, 2022), such that parameter A in the equation was evaluated according to Equation 2.

$$\mathrm{CR} = \frac{\mathrm{k}\Delta \mathrm{W}}{\mathrm{AT}\rho} \quad (1)$$

$$\mathrm{A} = \pi \mathrm{D}\left(\frac{\mathrm{D}}{2} + \mathrm{h}\right) \quad (2)$$

where k is a constant (8.76×10^4); A is total surface area of immersed sample; ρ is density of sample (7.87 g/cm^3); T is time of immersion; D is diameter of sample; h is height of sample.
Procedure was repeated for 1.0M HCl containing WVL and WVL+NP of concentration 0.5 g/L as inhibitors and inhibition efficiencies measured in percentage, were obtained according to Equation 3.

$$\mathrm{IE}_{WL} = \frac{\mathrm{CR}_{unin\,hi} - \mathrm{CR}_{inhi}}{\mathrm{CR}_{unin\,hi}} \times 100 \quad (3)$$

where CR_{uninhi} is corrosion rate of steel in uninhibited solution; CR_{inhi} is corrosion rate of steel in uninhibited solution.

RESULTS AND DISCUSSION

Presented in Figure 2 are the contact angle of Wood Vinegar Liquor and wood vinegar liquor nanofluid obtained from drop characteristics test. WVL has a higher contact angle of 57.25°, an indication of lower wettability, compared to WVL+NP that recorded 47.98° contacted angle. WVL+NP as corrosion inhibitor is poised to be more readily adsorbed on steel surface when it contained in corrosive medium in which is immersed, owing to it higher wettability inferred from lower contact angle 47.98° obtained (Hirano *et al.*, 2021). This ability of WVL+NP to more readily adsorb is an indication that it will reduce corrosion rate of experimental steel in 1.0 M HCl by forming a more stable protective film which limits HCl ingress.

The addition of iron-oxide nanoparticle to WVL to form WVL nanofluid caused decrease in contact angle of WVL thereby increasing its wettability for corrosion inhibition of micro-alloyed steel in corrosive solution. Lower contact angle observed with WVL+NP confirm the synergistic effect between the organic extract and nanoparticles. Figures 3-5 show the plot of weight loss, corrosion rate and inhibition efficiency against exposure time for the period ranging from 24 – 168 hours. Figure 3 displays the weight losses of micro-alloyed steel samples immersed in 1.0 M HCl solution over 168 hours for three categories of samples which are control, WVL Inhibited 1.0 M HCl and WVL+NP Inhibited 1.0 M HCl solutions. It indicates that from 24 - 96 hours, the micro-alloyed samples immersed experienced rise in weight in the respective solution such that samples in control solution recorded highest weight losses at each instance reaching maximum of 4.01 g at 96 hours. After 96 hours of immersion, samples immersed in

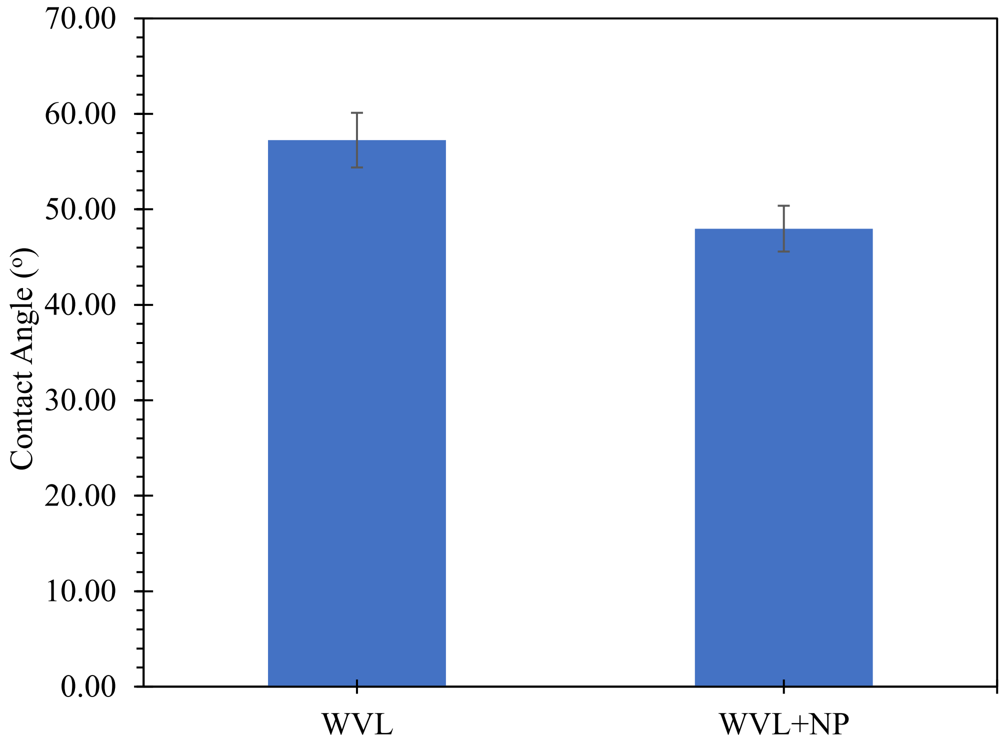

Figure 2: Contact Angle of Wood Vinegar Liquor and Wood Vinegar Liquor Nanofluid

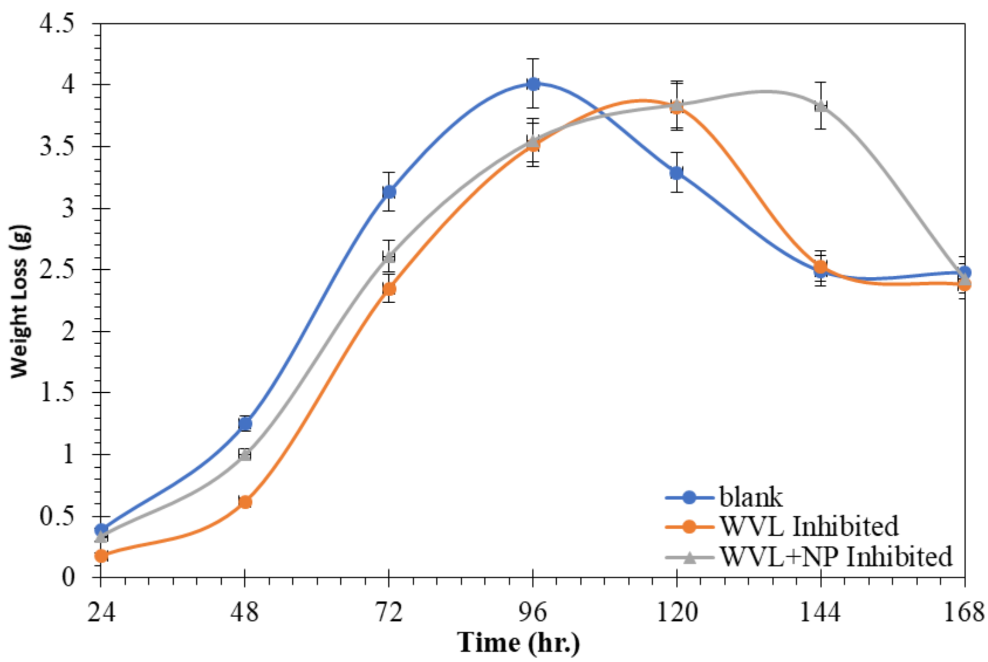

Figure 3: Graphical Plot of Weight loss (g) against Time (T)

control solution recorded decline in weight lose that reached a minimum of 2.48 g at 168 hours of immersion. Beyond the 96 hours of immersion, samples immersed in WVL Inhibited 1.0 M HCl and WVL+NP Inhibited 1.0 M HCl solutions continued to experience increases in weight losses to reach maximum of 3.82 and 3.84 g respectively, at 120 hours. However, samples immersed in WVL inhibited 1.0 M HCl solution recorded lower weight loss

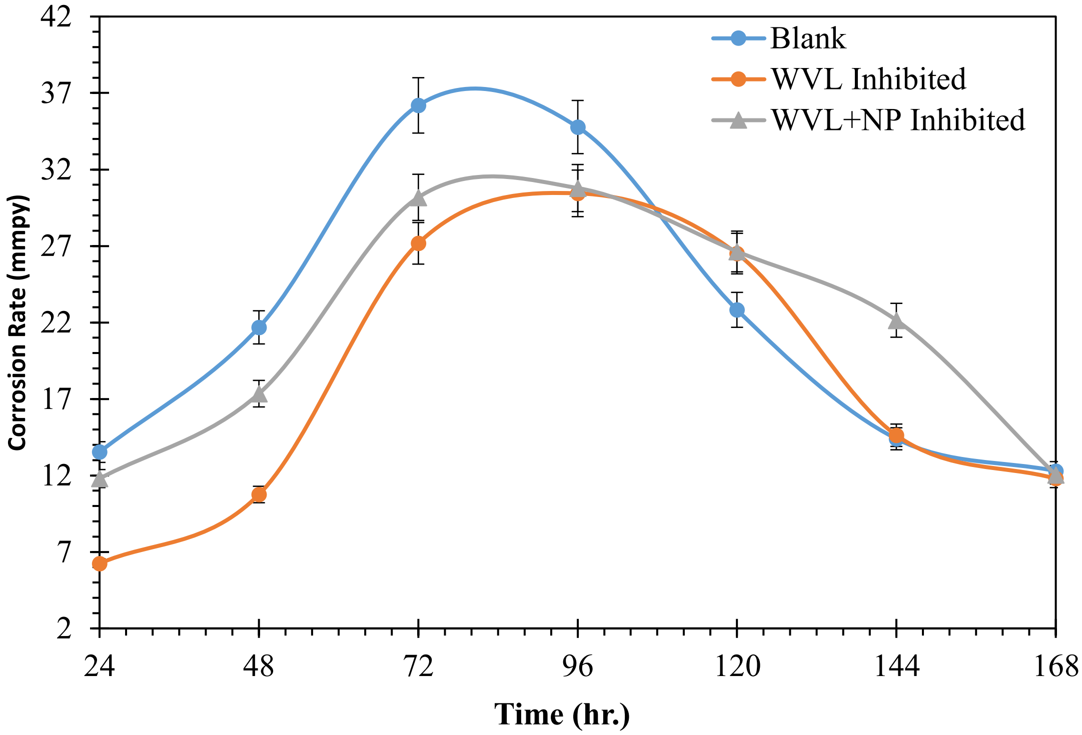

Figure 4: Graphical plot of Corrosion rate (mmpy) against time.

than those in WVL+NP Inhibited 1.0 M HCl solution within immersion period of 24 – 120 hours. After 120 hours of immersion, decreases in weight losses of samples immersed in WVL Inhibited 1.0 M HCl and WVL+NP Inhibited 1.0 M HCl solutions was recorded up till 168 hours of immersion, reaching minimum of 2.38 and 2.43 g respectively.

Compared to samples immersed in control solution, samples immersed in WVL Inhibited 1.0 M HCl and WVL+NP Inhibited 1.0 M HCl solutions experienced lower decline in weight losses, which began later than the samples in control solution. These may be attributed to probable decreases in pH of WVL Inhibited 1.0 M HCl and WVL+NP Inhibited 1.0 M HCl solutions over time more than control solution between 96 – 168 hours of immersion.

Shown in (Figure 4) are plots of corrosion rate (mmpy) against time for steel sample immersed in control, WVL Inhibited 1.0 M HCl and WVL+NP Inhibited 1.0 M HCl solutions. Between 24 – 72 hours of immersion, corrosion rate for samples immersed in all solutions increased with control solution recording the highest value of 36.19 mmpy. Beyond 72 hours of immersion, samples immersed in WVL Inhibited 1.0 M HCl and WVL+NP Inhibited 1.0 M HCl solutions experienced further increase in corrosion rates reaching values of 30.44 and 30.79 mmpy respectively, at 96 hours of immersion. Meanwhile, the corrosion rate of samples immersed in control solution continued to decline after 72 hours of immersion to attain a value of 12.29 mmpy at 168 hours. The decline in corrosion rate of samples immersed in WVL Inhibited 1.0 M HCl and WVL+NP Inhibited 1.0 M HCl solutions started after 96 hours of immersion from 30.44 to 11.79 mmpy and 30.79 to 12.04 mmpy respectively. Below 72 hours of immersion, the corrosion inhibitors experimented prevented aggressively of chloride ions on the steel sample.

This shows that the respective corrosion inhibitor are better put to effective use within 24 – 72 hours of immersion as they lost ability to retard corrosion effectively beyond 72 hours of immersion. Consequently, corrosion product developed from control solution's action on steel sample retarded the ingress of chloride ions more, by forming a more compact protective film (Orhorhoro *et al.*, 2018) as the immersion time extended beyond 72 hours. The intersection of error-bars between 72 – 120 hours of immersion for WVL Inhibited 1.0 M HCl and WVL+NP Inhibited 1.0 M HCl solutions indicate that corrosion rates were not significantly different but below this time period, significant differences in corrosion rate were recorded.

Figure 5 shows WVL and WVL+NP variation of inhibition efficiencies with respect to passage of immersion time for carbon steel samples in inhibited solutions of 1.0 M HCl. WVL Inhibitor has significantly higher inhibition efficiency than WVL+NP which may suggest stronger surface adsorption and protective film formation, between 24 – 48 hours of immersion. However, inhibition efficiencies in both scenarios declined as immersion time increased. Decline

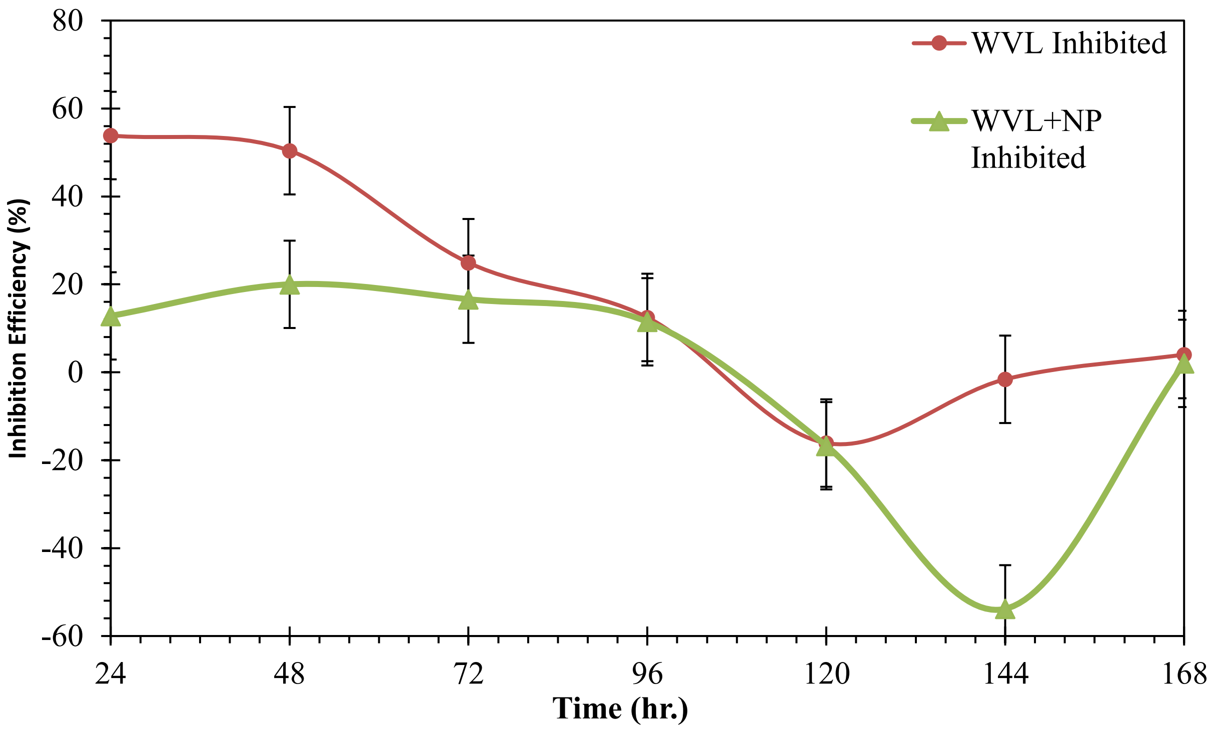

Figure 5: Graphical plot of Inhibition efficiency (%) against Time.

Figure 6: Visual representation of samples at 120 hours of immersion.

in inhibition efficiency was more pronounced for WVL inhibited HCl solution from 53.85 to 12.47% between 24 – 96 hours of immersion. On the other hand, WVL+NP inhibited solution maintained a marginal change in inhibition efficiency between 24 – 96 hours of immersion, which range between 11.47 – 20%.

Higher inhibition efficiencies recorded for WVL inhibited HCl solution between 24 – 72 hours of immersion negates indications from contact angle values which suggest that WVL+NP is better adsorbed due to enhanced wettability offered by addition of iron-oxide nanoparticle to WVL. This suggests that despite increase in wettability of WVL as a result of nanoparticle addition to form WVL+NP, decrease in pH of the solution is a likely factor which caused WVL+NP inhibited HCl solution to become more aggressive as the number of immersion hours increased. Beyond 96 hours of immersion, both inhibitors lost their inhibition efficiencies which recorded negative values, with WVL+NP inhibited HCl solution recording more negative values that reached up to -53.82% at 144 hours of immersion. This is a clear indication of possible decrease in pH of both solutions and less compact corrosion products formation, leading to loss in inhibition efficiencies (Ibrahimi, 2020).

Presented in (Figure 6) are the visual observation of corroded surfaces of samples where severe pitting can be seen on samples immersed in WVL inhibited HCl and WVL+NP inhibited HCl solutions after 144 hours of immersion, compared with sample immersed in control solution.

Conclusion

This study comprehensively evaluated the corrosion inhibition performances of wood vinegar liquor (WVL) and its nanoparticle-modified form (WVL+NP) on micro-alloyed steel in a 1.0 M HCl environment, providing insights into the synergistic and time-dependent behaviour of bio-based inhibitors. The incorporation of nanoparticles reduced the contact angle by 9.27°, enhancing surface wettability and facilitating the formation of a more adherent and uniform protective nanofluid film. Weight-loss analysis revealed progressive increases across all systems with immersion time, peaking at 4.01 g (control), 3.82 g (WVL), and 3.84 g (WVL+NP), after which reductions were observed, indicating film deterioration and possible desorption of inhibitor molecules.

Corrosion rate analysis further confirmed the protective role of both inhibitors, with maximum rates of 36.19 mmpy (control), 30.44 mmpy (WVL), and 30.79 mmpy (WVL+NP), underscoring the capacity of WVL to slightly outperform WVL+NP in mitigating degradation under prolonged exposure. Inhibition efficiencies declined progressively with immersion time, transitioning to negative values, signifying loss of protective potency and potential film breakdown due to aggressive chloride ion penetration and surface saturation. Overall, the findings establish WVL as an effective short-term corrosion inhibitor, with nanoparticle modification enhancing initial wettability but not sustaining superior long-term performance. Future work should focus on surface characterisation using electrochemical impedance spectroscopy (EIS), scanning electron microscopy (SEM), and Fourier-transform infrared spectroscopy (FTIR) to elucidate adsorption mechanisms, film composition, and degradation pathways. Additionally, optimising nanoparticle loading and integrating synergistic eco-friendly additives may yield more durable bio-inhibitor formulations for industrial-scale corrosion control.

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