



OPEN Nanoengineered t-ZrO₂ coatings for superior corrosion resistance on steel surfaces

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Corrosion of steel has several catastrophic consequences in various sectors. The inorganic nanoparticle-based anticorrosive coating on steel drew important attention to its large surface-to-volume ratio. The primary aim of this study is to synthesize and characterize t-ZrO₂ nanoparticles at an optimized annealing temperature and evaluate their structural, morphological, and optical properties. Additionally, the study investigates their effectiveness as a corrosion inhibitor for carbon steel in 1 M H₂SO₄. The study explores the cheap, facile, green synthesis of t-ZrO₂ nanoparticles (NPs) through bark extract from the gum arabic plant (*Acacia nilotica*) for anticorrosive coatings on carbon steel. X-ray diffraction (XRD) analysis confirms the tetragonal phase structure and the crystallite size, calculated using Scherrer's formula, is found to be 8.1 nm. Fourier-transform infrared (FT-IR) spectroscopy reveals the presence of Zr-O bonding along with organic residues from plant extracts, confirming the formation of t-ZrO₂ NPs. Field emission scanning electron microscopy (FESEM) images confirm a rock stone-like structure, while energy dispersive X-ray (EDX) spectroscopy verifies the presence of Zr and O elements. The study further investigates the corrosion inhibition efficiency of t-ZrO₂ NPs on carbon steel in 1 M H₂SO₄. The atomic force microscopy (AFM) analyses reveal a smoother surface with reduced roughness in the presence of the inhibitor. Electrochemical measurements, including weight loss, potentiodynamic polarization, and electrochemical impedance spectroscopy (EIS), confirm a significant reduction in corrosion rate. The inhibition efficiency reaches 95.2% at 200 ppm of 0.2 M t-ZrO₂ NPs, with an increased charge transfer resistance (R_{ct}) of 14,715 Ω cm² and a reduced double-layer capacitance (C_{dl}) of 0.631 $\times 10^8$ F/cm². These findings demonstrate that t-ZrO₂ NPs act as an effective corrosion inhibitor for carbon steel in acidic environments.

Keywords Green synthesis, *Acacia nilotica*, t-ZrO₂ NPs, Anticorrosion study

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Nanotechnology, which involves deploying substances at the atomic and molecular scale, has introduced a new age of materials with unique properties that differ significantly from their bulk counterparts. Among these materials, nanoparticles stand out due to their exceptional chemical, mechanical, thermal, magnetic, and electrical characteristics, attributed to their quantum effects with high surface area-to-volume ratio^{1,2}. These distinctive features make nanoparticles a subject of intense study and a cornerstone of various technological advancements^{3,4}.

The study of metal oxide nanoparticles, particularly those involving the combination of two or more metals, has garnered significant interest⁵. These nanoparticles exhibit a broad spectrum of physical and chemical properties, making them suitable for a variety of uses, from catalysis to electronics and biomedical fields⁶. Zirconium dioxide (ZrO₂), known as zirconia, is a ceramic material with excellent mechanical properties, chemical stability, and biocompatibility⁷. Its nanoparticle form further enhances these properties, making it a valuable material in numerous high-tech applications, such as oxygen sensors, fuel cells and dental implants^{8–10}.

Zirconium, a transition metal similar to titanium, is characterized by its remarkable resistance to corrosion, making it an ideal material for applications in harsh environments¹¹. Naturally occurring zirconium can be processed into nanoparticles using various methods. These nanoparticles are noted for their high fracture toughness, tensile strength, and hardness^{12,13}. The metal's natural isotopic forms, particularly the dominant 90Zr, play a crucial role in its industrial applications¹⁴. The synthesis of zirconium nanoparticles can be achieved through multiple techniques, including the Kroll process, which involves reducing zirconium tetrachloride with magnesium at high temperatures¹⁵. Other methods, such as sol-gel, hydrothermal, and laser ablation, are also utilized to produce ZrO₂ NPs in various phases, including cubic and monoclinic forms¹⁶.

Nanotechnology faces several challenges, including precise control over nanoparticle size, shape, and distribution and ensuring the reproducibility and scalability of synthesis methods^{17,18}. The unique properties of nanoparticles can lead to new functionalities, but they also pose potential risks, such as toxicity and environmental impact. Therefore, understanding and controlling these properties are crucial for the safe and effective use of nanomaterials¹⁹. This research seeks to advance the understanding of zirconium nanoparticles by developing eco-friendly synthesis methods and evaluating their anticorrosive properties on carbon steel in acidic conditions²⁰. The use of t-ZrO₂ NPs in anticorrosion coatings is significant due to their excellent chemical stability, high thermal resistance, and ability to form a dense, protective barrier on metal surfaces. These nanoparticles reduce corrosion rates by preventing direct contact between aggressive electrolytes and the metal substrate. Additionally, their high surface area enhances adhesion properties, ensuring a uniform and defect-free coating. The incorporation of t-ZrO₂ NPs in protective coatings offers an eco-friendly and efficient approach to mitigating corrosion in industrial applications, extending the lifespan of metal components, and reducing maintenance costs. This study aims to refine the production method to achieve higher synthesis efficiency, better structural and functional properties of the nanoparticles, and reduced environmental impact. In addition, it explores new application areas for zirconium nanoparticles, with a special emphasis on high-performance anticorrosive uses^{21,22}.

Experimental methods

Chemicals used

Zirconium Dichloride Oxide (ZrOCl₂·8H₂O) analytical grade purchased from Sigma–Aldrich (99% purity by weight) is used without further purification.

Plant material collection

The *Acacia nilotica* bark extract powder used in this study was obtained from Dr. A. Aslam (Fellow of the Indian Association for Angiosperm Taxonomy), Associate Professor of Botany, Jamal Mohamed College (Autonomous), Tiruchirappalli, Tamil Nadu, India.

Preparation of t-ZrO₂ NPs by means of *Acacia nilotica* bark extract

To prepare the aqueous bark extract, 100 mL of double-distilled water and 10 g of finely chopped *Acacia nilotica* bark were heated at 60 °C for 30 min to extract the bioactive compounds, followed by filtration through Whatman No. 1 filter paper to obtain the aqueous extract used for the nanoparticle synthesis. A 0.2 M solution of zirconium dichloride oxide (ZrOCl₂·8H₂O) was prepared and gradually added to the *Acacia nilotica* bark extract under constant stirring to facilitate the green synthesis of t-ZrO₂ nanoparticles. After four hours of stirring at 65 °C, a white precipitate formed from this combination. To avoid agglomeration, the precipitate was separated using filtration, cleaned with distilled water, and dried for four hours at 100 °C. At 600 °C, the dried powder was finally calcined. Figure 1 displays the schematic representations. In this study, a 0.2 M precursor concentration of ZrOCl₂·8H₂O was selected based on preliminary synthesis trials and previous reports indicating that this concentration yields zirconia nanoparticles with optimal crystallinity and phase stability for t-ZrO₂ systems^{23–29}.

Specimen preparation for carbon steel

Carbon steel specimens were obtained from a single sheet with the following composition: 2% carbon, 0.06% phosphorus, 0.026% sulfur, and 0.4% manganese, with the remainder being iron. Each specimen, measuring 1.0 × 4.0 × 0.2 cm, was polished to a mirror-like finish and cleaned with trichloroethylene before use in surface analysis and weight-loss corrosion testing. For potentiodynamic polarization experiments, a carbon steel specimen was mounted in a Teflon holder, exposing a 1 cm² cross-sectional area as the working electrode, while a platinum electrode was used as the counter electrode. Both electrodes were degreased with trichloroethylene and polished to a mirror finish. Analytical-grade sulfuric acid was diluted using double-distilled water to create a 1 M H₂SO₄ solution.

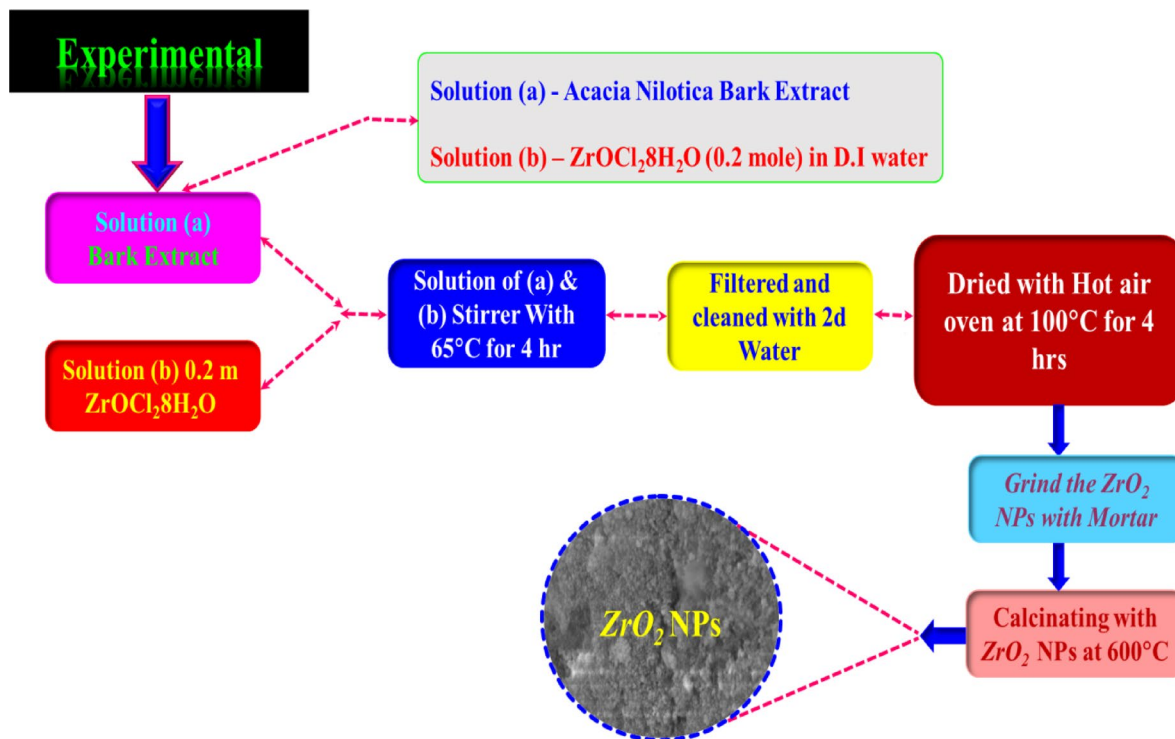


Fig. 1. A structured method for the process of chemical reduction root of a t-ZrO₂ NPs for 0.2 mol concentrations.

Formulating stock solutions

All solutions are made using deionized water and premium H₂SO₄ is diluted to the required concentration. The inhibitor is dissolved directly in this acidic medium, and the solution is then adjusted to reach the target concentration.

Characterization techniques

Before and after being submerged in 1 M H₂SO₄ for three hours, three polished carbon steel (CS) specimens were weighed with different extract quantities to determine weight loss.

$$\text{Inhibition Efficiency } IE(\%) = \left(\frac{W_o - W_1}{W_o} \right) \times 100\% \quad (1)$$

Weight loss with the inhibitor is represented by W₁, and weight loss without the inhibitor is represented by W_o. Using the formula, the corrosion rate is calculated in mmd,

$$\text{Corrosion Rate (mdd)} = \frac{53.5 \times W}{a \times t} \quad (2)$$

where W stands for weight loss (g), *a* for specimen area (cm²), and *t* for exposure time (h).

Electrochemical Impedance Spectroscopy (EIS) measurements were carried out in a frequency range spanning from 1 Hz to 100 kHz (10⁵ Hz) with an applied AC amplitude of 0.005 V, a starting voltage of 0 V, and a 2-s quiet period before measurement, using a CHI 660 A electrochemical workstation. The experiment used a three-electrode cell setup, as shown in Fig. 2. The saturated calomel electrode (SCE) act as a reference electrode, and platinum electrode was used as the counter electrode. Carbon-based steel was used as an electrode. LPR values were derived from the polarization investigation, and the polarization analysis under consideration was able to compute corrosion parameters suggestive of rust current (I_{corr}), rust potential (E_{corr}), and Tafel slopes (anode = b_a and cathode = b_c). Using a polarization analyzer, the AC impedance spectra were evaluated.

In order to describe a system's resistance to AC flow, Measurements of impedance are taken at every frequency within the specified interval. The following formula is employed to determine the value of the double-layer capacitance.

$$C_{dl} = \frac{1}{(2\pi \times f_{\max} \times R_{ct})} \quad (3)$$

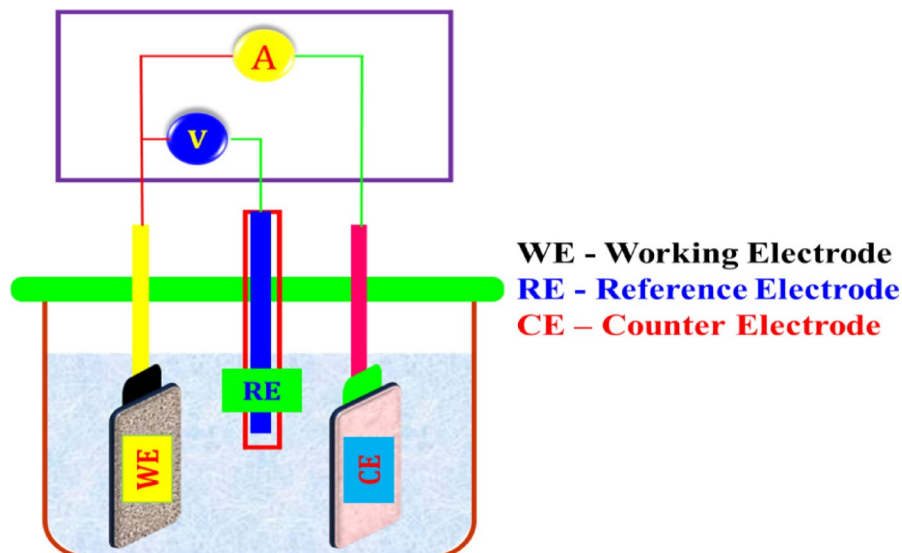


Fig. 2. Schematic diagram of a three-electrode cell assembly.

where, C_{dl} is the double-layer capacitance (F/cm^2), R_t (more appropriately denoted as R_{ct}) is the charge transfer resistance (Ωcm^2) and f_{max} is the frequency at which the imaginary component of impedance (Z'') reaches a maximum. The real (Z') part and imaginary (Z'') parts, given in ohms, were taken into consideration when analyzing the cell's resistance at different frequencies. The values of charge transfer resistance (R_{ct}) and double-layer capacitance (C_{dl}) were calculated using the Nyquist plot. Using Bode graphs, the logarithmic (\log) impedance value (z/ohm) was determined.

The inhibition efficiency EIS studies are calculated using following formulas

$$IE(\%) = \frac{i_{Corr} - i_{inh}}{i_{Corr}} \times 100 \quad (4)$$

where, i_{corr} -corrosion current density without inhibitor and i_{inh} -corrosion current density with inhibitor

$$IE(\%) = \frac{R_{ct,inh} - R_{ct}}{R_{ct,inh}} \times 100 \quad (5)$$

where, $R_{ct,inh}$ —charge transfer resistance with inhibitor and R_{ct} —charge transfer resistance without inhibitor.

FTIR spectroscopy was conducted Utilizing a Perkin Elmer spectrophotometer within the $400\text{--}4000\text{ cm}^{-1}$ range. After removing the protective coating, the CS samples were exposed to the plant leaf extracts and let soak in $1\text{ M H}_2\text{SO}_4$ at RT for three hours. Scanning electron microscopy (SEM) studies were performed before and after corrosion to evaluate how the inhibitor affects the CS surface. The surface topography of the CS was analyzed using SEM both with and without the inhibitor, with images acquired using a JEOL JSM 6390. After corrosion, the CS samples were dried after being cleaned with double-distilled water. Three hours later, we tested in both inhibitor and blank solutions.

Results and discussion

XRD analysis

Figure 3 presents the XRD pattern of the t-ZrO₂ NPs at 0.2 mol concentration. The diffraction peaks get more intense and the maximum intensity observed in 0.2-mol concentration at an optimized annealing temperature of 600 °C. The XRD comparison is made against standard bulk data (JCPDS card no. 88-1007). However, the nanoscale nature of the synthesized t-ZrO₂ NPs leads to size- and surface-induced broadening and minor peak shifts. These deviations are accounted for by the calculated crystallite size (8.1 nm), and the presence of strain and stacking faults further confirms the influence of nanoscale effects on structure. The composition of t-ZrO₂ is established by the diffraction angle of the peaks at 30.52, 35.19, 50.65, and 60.60° and their corresponding ($h\ k\ l$) planes (101), (110), (200) and (211) respectively. No phase change was observed in t-ZrO₂ NPs with 0.2 mol concentration. Changes in the crystallite size and lattice parameter, however, are responsible for a minor deformation in the NPs' structure²³⁻²⁵. At 0.2-mol concentration, a preferred orientation along the (101) plane is seen. As shown in Fig. 3, the prominent peak intensity increases progressively. These findings suggest that NP production in the (101) direction is significantly influenced by mole concentration²⁶⁻²⁹. Nonetheless, peak height in some planes may be influenced by the crystallites' preferred orientation. The matching peak height will often rise if additional crystallites align themselves in a single direction. Scherrer's formula, which is provided in Eq. (6), is utilized to calculate the crystallite size (D) of the t-ZrO₂ NPs. The dislocation density (δ), microstrain (ϵ), and stacking fault (SF) are attained by using the following Eq. (7-9)³⁰.

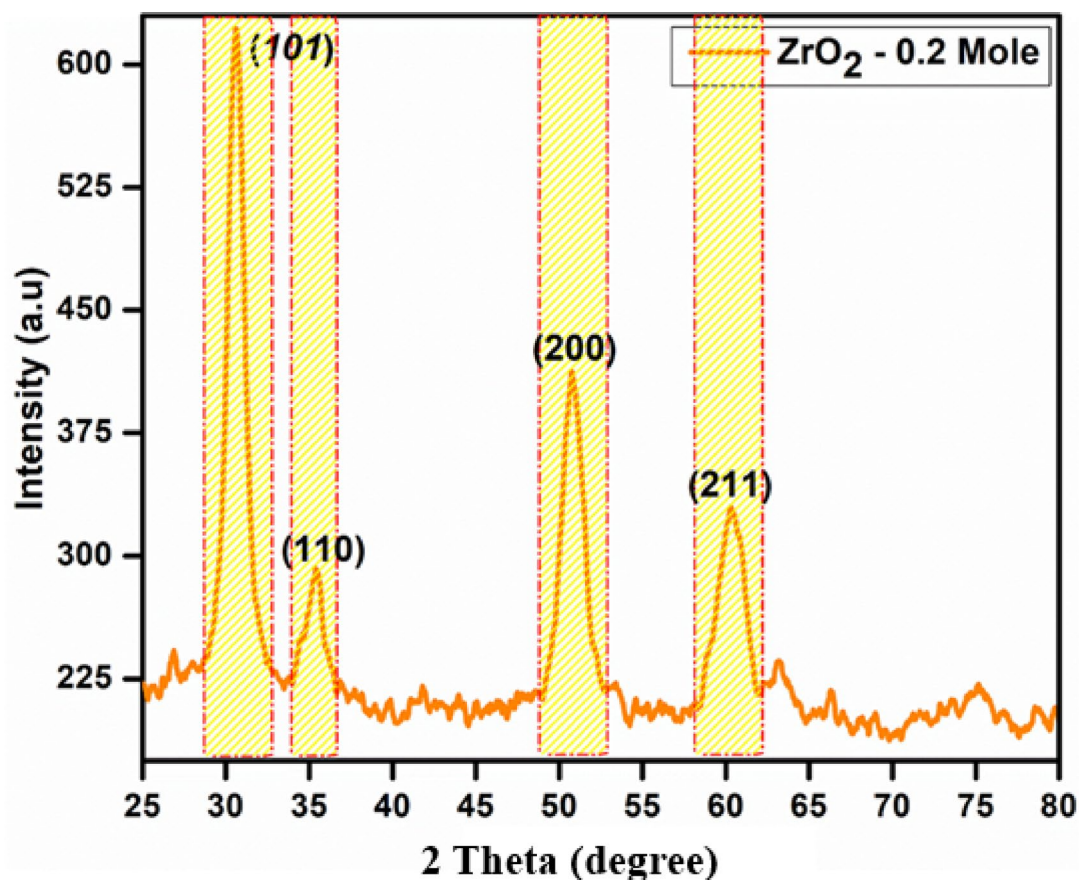


Fig. 3. XRD pattern of the t-ZrO₂ NPs for 0.2 mol concentrations.

$$\text{Crystallite Size } D = \frac{(0.89) \lambda}{\beta \cos \theta} \quad (6)$$

$$\text{Dislocation Density } \delta = \frac{1}{D^2} \quad (7)$$

$$\text{MicroStrain } \varepsilon = \frac{\beta \cos \theta}{4} \quad (8)$$

$$\text{Stacking Fault } SF = \left[\frac{2\pi^2}{45 (3 \tan \theta)^{\frac{1}{2}}} \right] \beta \quad (9)$$

Here, D represents the crystallite size, λ is the wavelength (0.15418 nm) for CuK α , β denotes the full width at half maximum of the diffraction peak, and θ , the diffraction angle in degrees. The XRD pattern of the 0.2 M t-ZrO₂ sample confirms the formation of a well-crystallized tetragonal (t-ZrO₂) phase. The diffraction peaks are sharp and well-defined, indicating good crystalline quality and minimal structural defects. The average crystallite size, calculated using the Debye–Scherrer equation, is approximately 8.1 nm. The corresponding microstructural parameters—dislocation density ($\delta = 1.09 \times 10^{16}$), microstrain ($\varepsilon = 0.0316$), and stacking fault ($SF = 0.0113$)—suggest a low level of lattice imperfections and confirm the high degree of crystallinity of the synthesized nanoparticles. Overall, the XRD analysis provides clear evidence for the successful synthesis of high-quality tetragonal ZrO₂ nanoparticles at the optimized 0.2 M concentration.

Functional group analysis

The FTIR spectrum of Acacia plant bark extract shows (Fig. 4a) key absorption bands: 3286 cm⁻¹ (O–H/N–H stretching – alcohols, phenols, or amines), 1610 cm⁻¹ (C=O or C=C stretching—carbonyl or aromatic/aliphatic double bonds), 1442 cm⁻¹ (C–H bending—CH₂/CH₃ scissoring), 1031.33 cm⁻¹ (C–O–C or C–N stretching—ethers, esters, or amines), and 605 cm⁻¹ (aromatic C–H bending or halogen-related C–Cl/C–Br stretch). These indicate the presence of phenolic aromatic structures and possibly halogenated compounds. Figure 4b shows the FT-IR spectrum for the synthesized ZrO₂ NPs. The band observed at 605.28 cm⁻¹ indicates the Zr–O bond stretching vibration in zirconium dioxide. The peak at 1123.40 cm⁻¹ suggests C–O stretching vibrations, likely due to residual plant extracts associated with the nanoparticles. The band 1382.49 cm⁻¹ resembles the symmetric

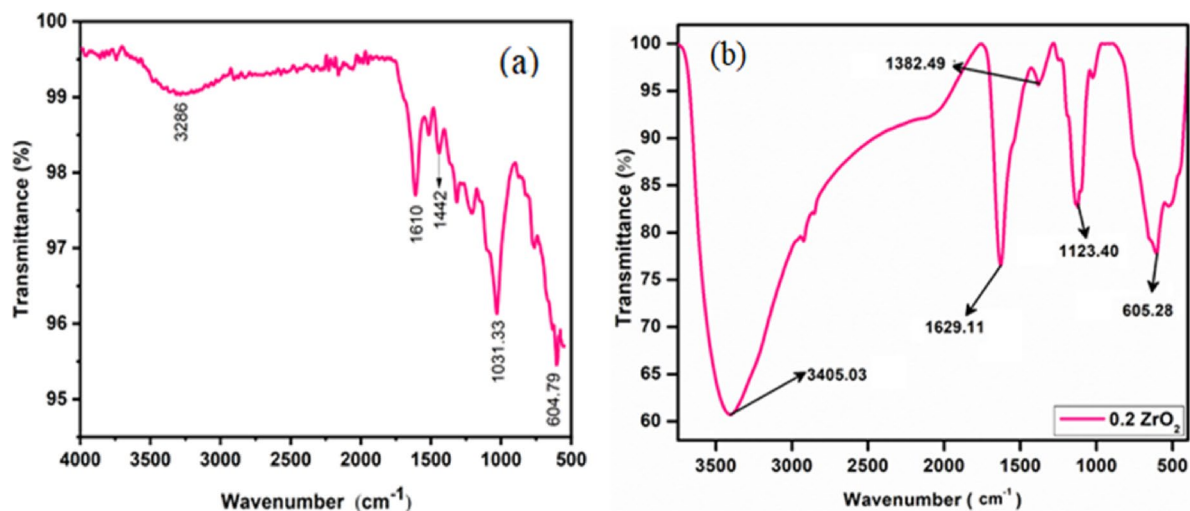


Fig. 4. FTIR spectrum of (a) Acacia plant bark extract (b) t-ZrO₂ NPs for 0.2 mol concentrations.

stretching vibration of the C–H bond in –CH₃ groups, possibly from organic residues of the plant extract. The peak 1629.11 cm⁻¹ is typically related with C=O stretching vibrations in amides or carbonyl groups, likely originating from organic compounds in the plant extract³¹.

The band at 3405.03 cm⁻¹ is ascribed to O–H stretching vibrations, signifying the occurrence of hydroxyl groups, which may come from water molecules, alcohols in the plant extract, or surface hydroxyl groups on the nanoparticles. This analysis confirms the formation of ZrO₂, with the broad peak suggesting the nanoparticles possess a nanostructure³².

UV–Vis spectroscopy analysis

Utilizing UV–Vis absorption spectroscopy, the optical characteristics of t-ZrO₂ NPs were examined. Figure 5a displays the room temperature UV–Vis absorption spectrum of the t-ZrO₂ NPs. This spectrum corresponds to t-ZrO₂ NPs at a concentration of 0.2 mol, optimized at an annealing temperature of 600 °C. The UV–Vis absorption band at 317 nm corresponds to the characteristic absorption of t-ZrO₂ nanograins, influenced by quantum size effects, in contrast to the bulk exciton absorption. This redshift is attributed to specific experimental conditions and defect states introduced during green synthesis³³. Perhaps as a result of the particular experimental synthesis conditions, the absorption in the visible range suggests that the generated t-ZrO₂ NPs have some defect energy levels³⁴. The Tauc plot determines prepared structures' optical band gap Eg (Fig. 5b).

According to the following connection, the absorption coefficient in this case depends on the photon energy and band gap (10):

$$\text{Band gap } (\alpha h\nu) = A(h\nu - E)^n \quad (10)$$

where A is the proportionality constant, ν is the vibration frequency, Eg, the band gap, h is the Planck constant, and α is the absorption coefficient. When $n=2$, it specifies directly allowed transitions and intercepts on the E-axis, giving the values of E_g. Because of the size effect, the intercept of the curve's extrapolated linear component has yielded an energy band gap Eg of roughly 3 eV³⁵. The green synthesis method minimizes the band gap value of t-ZrO₂ NPs.

Surface morphology

FESEM analysis of t-ZrO₂ NPs

FESEM micrographic images of t-ZrO₂ nanoparticles for 0.2-mol concentration is shown in Fig. 6a,b, displays the t-ZrO₂ grains that are micrometer and nanometer in size. The surface morphology exhibits continuous variation in mole concentration on the t-ZrO₂ nanoparticle surface. FESEM images of t-ZrO₂ nanoparticles reveal no cracks observed, even in the large area scanning. For the t-ZrO₂ NPs, the randomly oriented rock stone-like structure is detected. Due to the Zr atoms oxidation, the mole concentration favors quick and defects-free growth crystallites^{36–38}. The surface morphology of Zirconium Oxide nanoparticles is studied, and their large surface area makes an increased quantity of atoms on the crystal compound's surface compared to the atoms inside the crystal. The bulk of t-ZrO₂ particles is formed by the interaction of these atoms with other contiguous surface atoms or by the absorption of other species. These atoms on the surface of the zirconium oxide nanoparticles have more dangling bonds and free valance bonds. The annealing temperature support to minimize the crystalline size values follows the XRD results³⁹.

SEM analysis of carbon steel with inhibitor

Scanning Electron Microscopy (SEM) provides detailed images of the carbon steel, offering insight into the surface morphology. The study examined the SEM morphology of carbon steel in 1 M H₂SO₄ solution, both

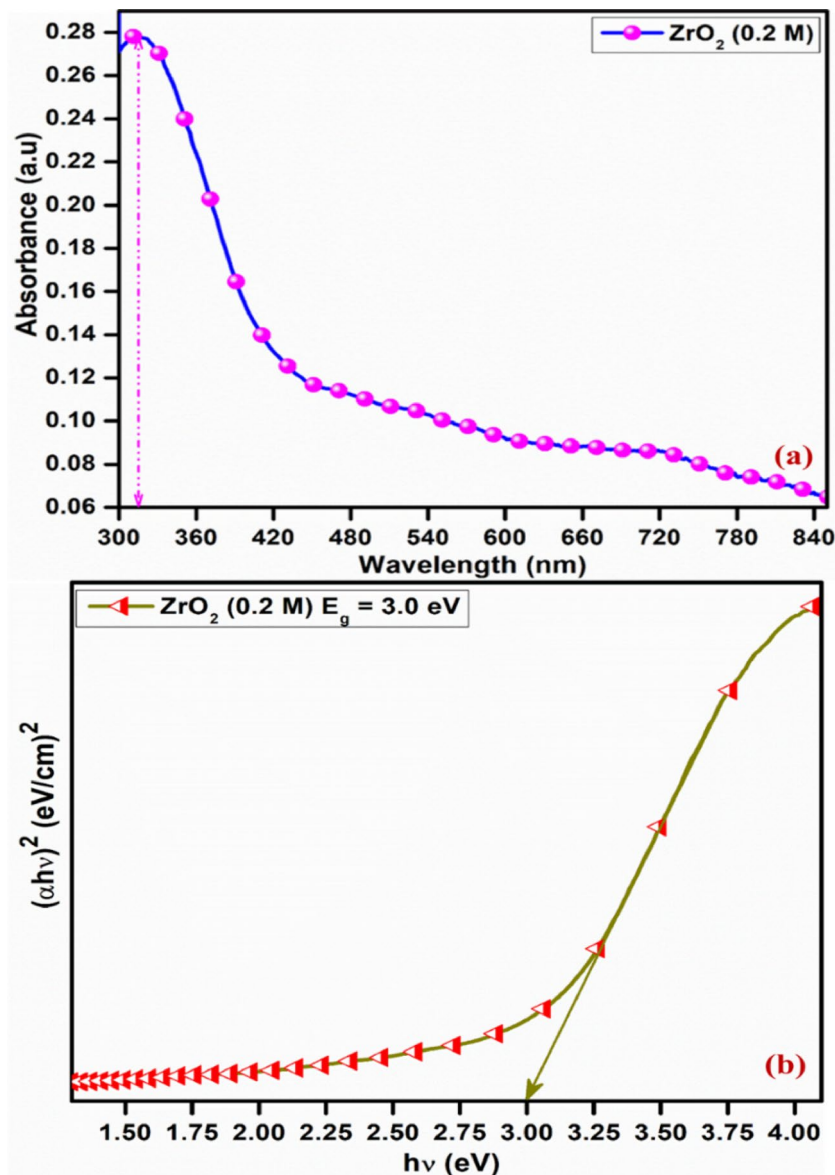


Fig. 5. UV-Vis analysis of t-ZrO₂ NPs for 0.2 mol concentrations (a) Absorption (b) plots of $(\alpha h\nu)^2$ Vs $h\nu$.

with and without an inhibitor system. Figure 7a,b represent the polished carbon steel surface, which appears smooth and free from any corrosion features. No pits or deposits are visible, confirming the unexposed, pristine condition of the metal. While Fig. 7c,d corresponds to the blank (uninhibited) carbon steel sample immersed in 1 M H₂SO₄. The surface is rough, uneven, and covered with irregular corrosion products. Distinct corrosion pits can be observed as dark, circular depressions, indicating localized attack due to acid exposure. Figure 7e,f show the inhibited carbon steel sample exposed to 1 M H₂SO₄ + 200 ppm of 0.2 M t-ZrO₂ nanoparticles. The surface is comparatively smoother with fewer visible defects or pits, suggesting that the inhibitor effectively suppressed corrosion and promoted the formation of a protective film on the steel surface²¹. Hence, the morphology and size of t-ZrO₂ NPs significantly influence their anticorrosion properties by affecting surface coverage, adhesion, and barrier effectiveness. The observed higher surface area ensures uniform dispersion and reduces coating porosity, which minimizes the penetration of corrosive agents. Additionally, optimized nanoparticle size improves charge transfer resistance, reducing the corrosion rate and enhancing overall coating performance.

EDX analysis

The elements in the produced samples were examined using energy dispersive spectroscopy. Figure 8 displays the EDS spectrum of the prepared sample. From the figure it can be understood that the sample contains only Zr, O NPs with 0.2 mol concentration. The atomic % of Zr and O are 17.68 and 63.78 t-ZrO₂ with 0.2-mol concentration. The presence of t-ZrO₂ NPs in the sample is confirmed by the strong peaks that correspond to Zr and O. The added peaks in EDS spectra are associated with the annealing temperature of 500 °C, C, Cl, and Cu used for sample preparation. The annealing temperature causes the Zr content to decrease as the oxygen level

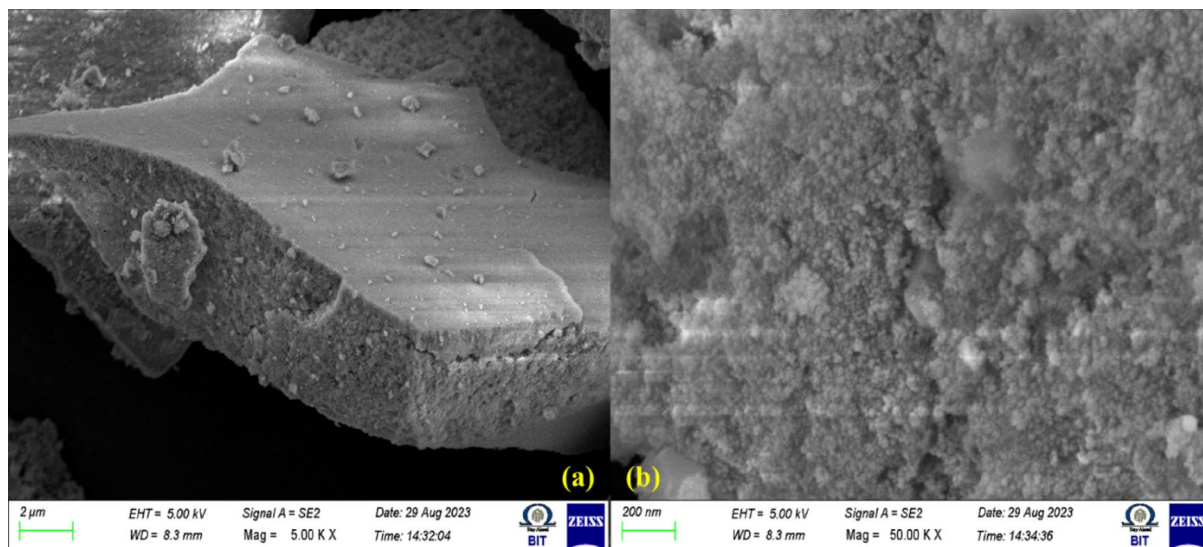


Fig. 6. (a,b) FESEM images of t-ZrO₂ NPs for 0.2 mol concentrations.

increases. From the EDX results, the t-ZrO₂ NPs specify the strong impact of the Zr lattice at mole concentration. This result discloses a suitable chemical composition with stoichiometry of chemical elements was achieved for the zirconium oxide nanoparticle¹⁷.

Atomic force microscopic studies

Figure 9a–c shows the two-dimensional AFM images of the film-protected metal (1 M H₂SO₄ + inhibitor), the corroded surface (immersed in 1 M H₂SO₄), and the polished metal surface. In Fig. 9a–c, the equivalent three-dimensional images are also displayed. Table 1 lists the AFM parameters for refined carbon steel, along with carbon steel submerged in 1 M H₂SO₄, and in the inhibitor system⁴⁰. To ensure clarity in interpreting the AFM data, the physical meanings of the symbols in Table 1 are defined as follows. The arithmetic mean roughness (S_a) represents the average absolute deviation of surface heights from the mean plane, providing an overall measure of surface texture. The root mean square roughness (S_q) quantifies the standard deviation of height variations and is more sensitive to extreme peaks and valleys than S_a . The maximum peak height (S_p) corresponds to the height of the highest asperity from the mean plane, while the maximum peak-to-valley height (S_y) denotes the total vertical distance between the highest peak and the deepest valley within the scanned area. Collectively, these parameters describe the surface topography and enable a quantitative assessment of the inhibitor's ability to smooth and protect the carbon steel surface.

In the corrosive media (blank), carbon steel's average surface roughness is found to be noticeably high. However, 200 ppm of 0.2 M t-ZrO₂ NPs inhibitor reduces this value. While it remains higher compared to the carbon steel surface which is polished, it is lower than the roughness in the corrosive medium (blank). This reduction is attributed to forming a protective layer on the carbon steel surface in the presence of the inhibitor, which appears smooth. The same trend is observed for the other three parameters such as root mean square roughness, maximum peak-to-valley height, and maximum peak height⁴¹.

Electrochemical analysis

Weight loss method

The carbon steel's corrosion rates (CR) when submerged in a 1 M H₂SO₄ solution, as well as the inhibition efficiencies (IE) with and without the presence of the 200 ppm 0.2 M t-ZrO₂ NPs inhibitor, obtained via the weight loss method, are given in Table 2. It is noted that the 200 ppm 0.2 M t-ZrO₂ NPs provide an inhibition efficiency of 95.2%. The inhibitory efficiency also improves with an increase in the 0.2 M t-ZrO₂ NPs concentration⁴². This enhancement is attributed to increased surface coverage at higher inhibitor concentrations, which slows the dissolution of carbon steel. The electron-donating characteristics of the oxygen atoms in conjunction with the delocalized π -electrons, are responsible for the increased inhibitory efficiencies. This observation aligns well with findings reported by various researchers⁴³.

Potentiodynamic polarization analysis

The Tafel plots illustrating the inhibition of corrosion upon the addition of 200 ppm of 0.2 M t-ZrO₂ NPs are presented in Fig. 10. Critical parameters such as corrosion current density (I_{corr}), corrosion potential (E_{corr}), cathodic (b_c) and anodic (b_a) Tafel slopes, and linear polarization resistance (R_p) are summarized in Table 3. Adding NPs to the acidic solution decreased I_{corr} values for the system containing 200 ppm of 0.2 M t-ZrO₂ NPs (1.987×10^{-6} $\mu\text{A}/\text{cm}^2$) compared to the solutions without the inhibitor (0.224×10^{-6} $\mu\text{A}/\text{cm}^2$). This decrease in corrosion When the inhibitor is present, the current density shows that the inhibitors are adsorbed onto the metal surface, preventing corrosion⁴⁴. The measured corrosion potential value for the blank system was -0.241

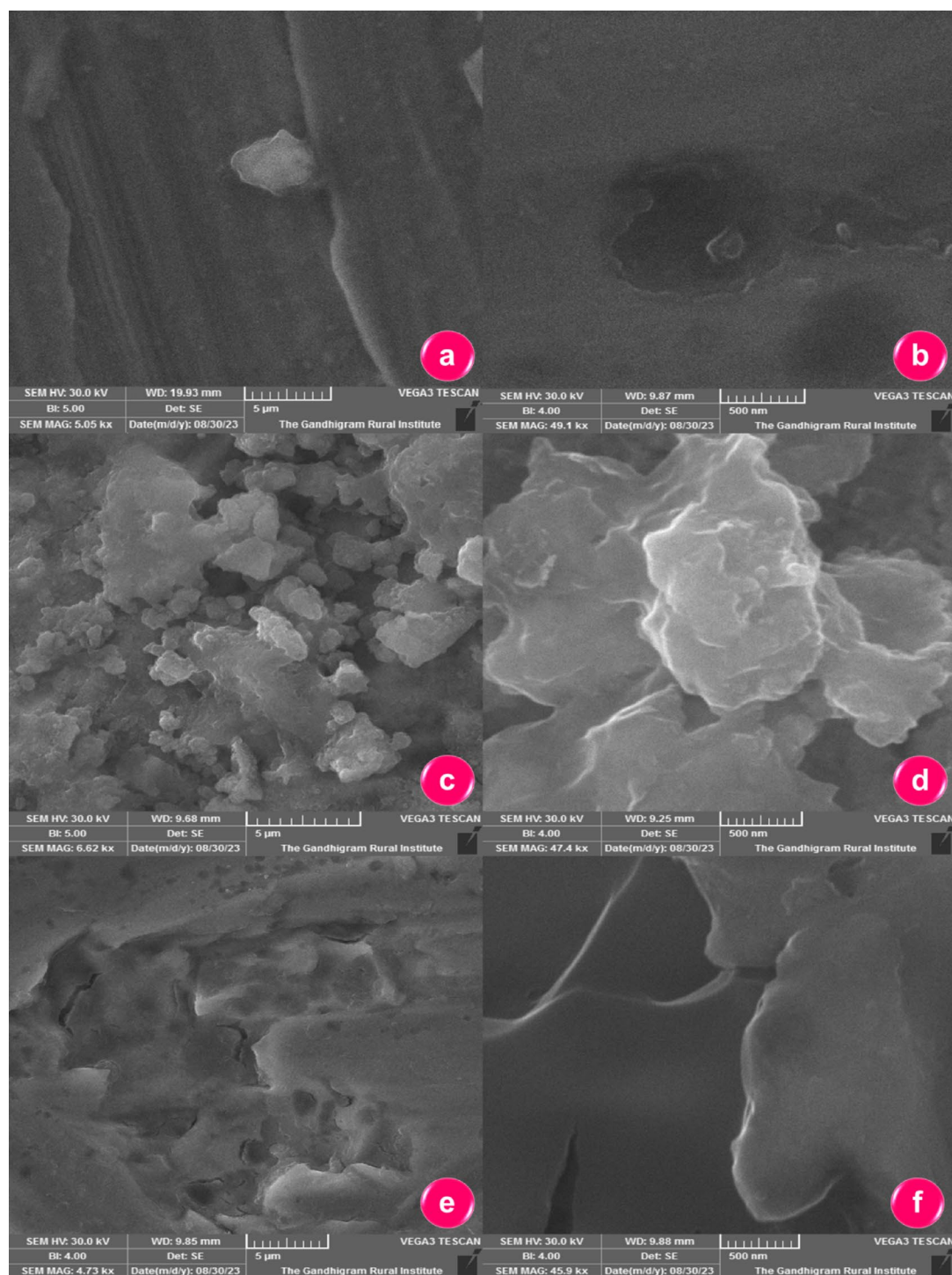


Fig. 7. SEM micrographs of (a,b) polished carbon steel, (c,d) carbon steel exposed to 1 M H_2SO_4 , and (e,f) carbon steel exposed to 1 M H_2SO_4 containing 200 ppm of 0.2 M t-ZrO₂ nanoparticles.

(mV vs. SCE) which increased to 0.015 (mV vs. SCE) with the inhibitor. Additionally, the linear polarization resistance (R_p) was lower for the blank system at 14,240 $\Omega \text{ cm}^2$, compared to 24,690 $\Omega \text{ cm}^2$ for the system with 200 ppm of 0.2 M t-ZrO₂ NPs, suggesting the development of a shielding layer on the metal surface⁴⁰.

Electrochemical impedance spectroscopy analysis

The development of a protective layer on the surface of carbon steel was verified by AC impedance spectra. The reduction in double-layer capacitance (C_{dl}) observed in EIS studies upon the addition of t-ZrO₂ NPs is attributed to the formation of a compact, insulating nanoparticle layer on the carbon steel surface. This layer reduces the effective area of the double layer at the metal/electrolyte interface, thereby decreasing the capacitance. The high

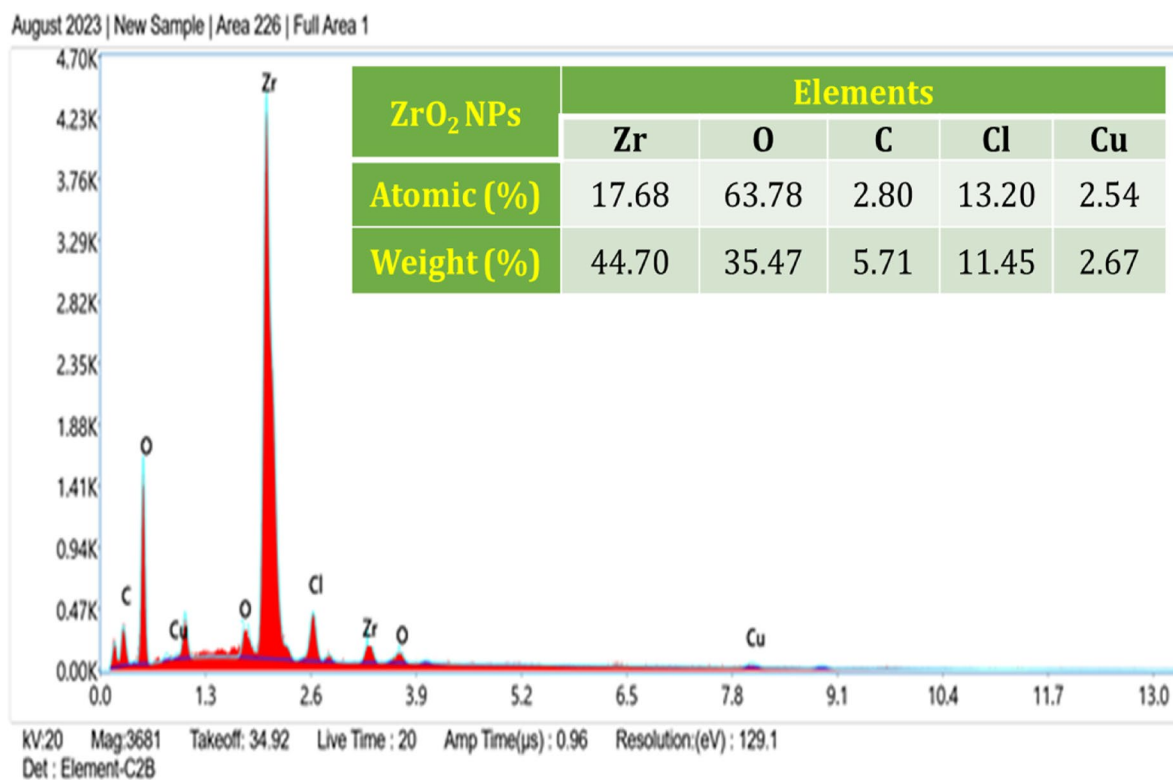


Fig. 8. EDX spectrum of t-ZrO₂ NPs for 0.2 mol concentration.

surface area and chemical stability of t-ZrO₂ NPs promote uniform adsorption, minimizing electrolyte access to the metal surface and enhancing the corrosion protection mechanism⁴¹. Figure 11a–c (Nyquist plots) displays the AC impedance spectra of submerged carbon steel in an aqueous solution containing 1 M H₂SO₄ both with and without the 200 ppm 0.2 M t-ZrO₂ NPs inhibitor with electrical circuit equivalent. Table 4 displays the AC impedance parameters that were obtained from the Nyquist plots, including the double-layer capacitance (C_{dl}) and charge transfer resistance (R_{ct}). The charge transfer resistance (R_{ct}) rose from 1,769 Ω cm² to 14,715 Ω cm² with the addition of 200 ppm of 0.2 M t-ZrO₂ NPs, whereas the C_{dl} value dropped from 2.090×10^7 F/cm² to 0.631×10^7 F/cm². From 713.60 to 17,729.02, the impedance value [$\log(z/\Omega)$] increased. Table 5 shows the key findings of different coating materials previously reported and our current material under high temperature, humidity and salt water. The conclusion that a protective coating forms on the carbon steel surface is supported by these data^{39,45,46}.

Cost–benefit and environmental compatibility

The green synthesis of t-ZrO₂ NPs using *Acacia nilotica* bark extract is both cost-effective and environmentally friendly compared to traditional chemical synthesis methods. The use of plant extracts eliminates the need for toxic reducing agents and solvents, lowering production cost and toxicity. The process operates at relatively low temperatures and is scalable for industrial applications. The high inhibition efficiency (95.2% at 200 ppm) demonstrates excellent performance, justifying the cost of nanoparticle synthesis. Environmentally, the use of biomass byproducts promotes sustainability, and t-ZrO₂ is chemically inert and non-toxic, further enhancing ecological compatibility. These features contribute to industrial relevance, especially in sectors requiring durable, eco-friendly corrosion inhibitors.

Conclusion

0.2 M ZrO₂ NPs were successfully synthesized using a green synthesis method with *Acacia nilotica* extract as the reducing agent, as confirmed by XRD studies. The XRD analysis showed that the 0.2 M ZrO₂ NPs possess a tetragonal structure. FTIR analysis identified the existence of all relevant functional groups. Scanning electron microscopy (SEM) analysis showed a rock stone-like structure for the 0.2 M ZrO₂ NPs. Furthermore, t-ZrO₂, after being coated in carbon steel, shows a much smoother surface with reduced corrosion. EDAX confirms the existence of zirconium (Zr) and oxygen (O) in the synthesized ZrO₂ NPs. The 0.2 M ZrO₂ NPs exhibited significant effectiveness in inhibiting the carbon steel corrosion in an aqueous 1 M H₂SO₄ solution. Polarization studies revealed that these nanoparticles primarily function as anodic inhibitors, mainly influencing the anodic reaction. Weight loss experiments demonstrated that a formulation containing 200 ppm of 0.2 M ZrO₂ NPs achieved an impressive 95.2% inhibition efficiency in mitigating the corrosion of carbon steel in the same acidic environment. Electrochemical impedance spectroscopy (EIS) measurements indicated a rise in the resistance to charge transfer (R_{ct}) and a reduction in corrosion current (I_{corr}) and double-layer capacitance (C_{dl}), which

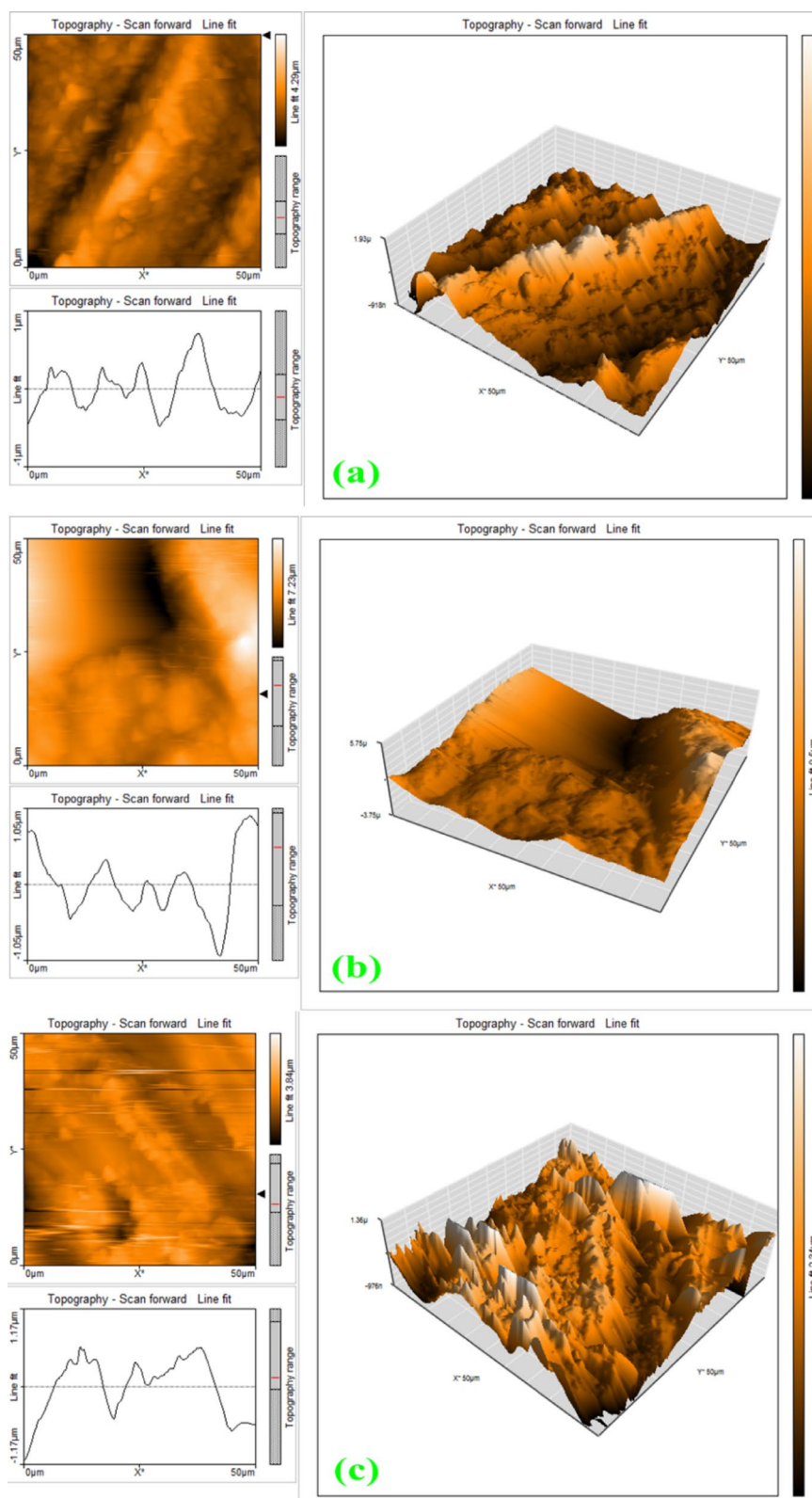


Fig. 9. AFM images of (a) Polished Carbon Steel (b) CS immersed in the 1 M H_2SO_4 solution (c) Carbon steel + 1 M H_2SO_4 + 200 ppm of 0.2 M t- ZrO_2 NPs.

Samples	Values in (nm)			
	Maximum Peak Height (S_p)	Root Mean Square Roughness (S_q)	Average Roughness (S_a)	Maximum peak-to-valley height (S_y)
Carbon Steel Surface	3109.30	503.60	370.57	1653.30
1 M H_2SO_4 + Carbon Steel	2648.30	370.13	285.72	257.70
1 M H_2SO_4 + Carbon Steel + Inhibitor (200 ppm)	2472.30	340.42	265.73	228.50

Table 1. AFM data for carbon steel immersed in the inhibitor solution.

0.2 M ZrO_2 NPs (ppm)	CR (mpy)	IE (%)
0	5.478	-
40	2.182	62.3
80	2.105	76.2
120	2.032	81.5
160	2.001	90.7
200	1.934	95.2

Table 2. Inhibition efficiency (IE %) obtained by weight loss method.

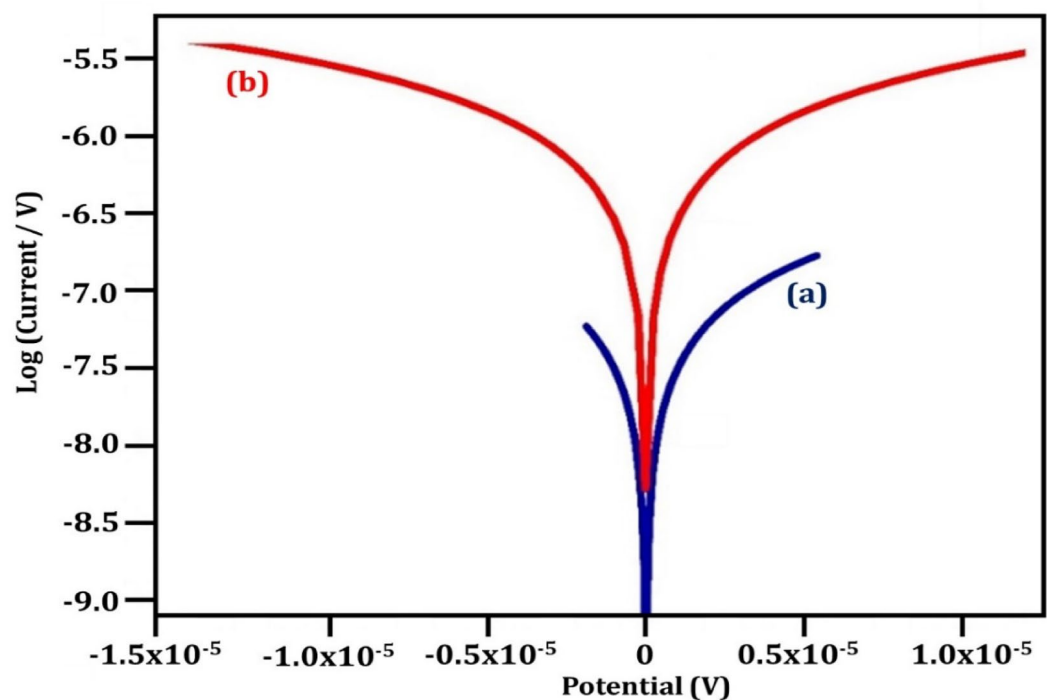


Fig. 10. PDP Curves corrosion of CS (a) 1 M H_2SO_4 (blank) (b) 200 ppm of 0.2 M t- ZrO_2 NPs.

Inhibitor (%)	I_{corr} ($\mu A/cm^2$)	E_{corr} (mV vs. SCE)	b_a (mV/dec)	b_c (mV/dec)	LPR ($\Omega \cdot cm^2$)	IE (%)
MS + H_2SO_4	1.987×10^{-6}	-0.241	0.067	0.106	14,240	-
MS + H_2SO_4 + 0.2 M ZrO_2 NPs	0.224×10^{-6}	0.015	0.13	0.134	24,690	88.7

Table 3. Potentiodynamic polarization analysis.

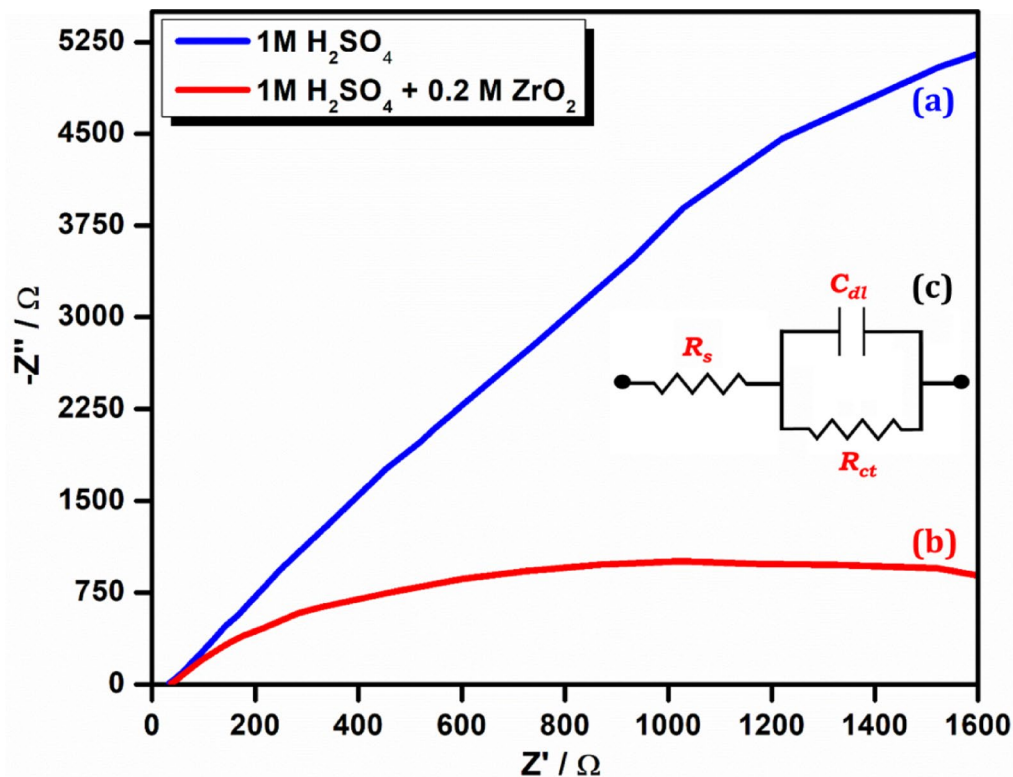


Fig. 11. Nyquist plot for the corrosion of carbon steel (a) 1 M H_2SO_4 (blank) (b) 200 ppm of 0.2 M t- ZrO_2 NPs (c) Electrical circuit equivalent (solution resistance: R_s ; charge transfer: R_{ct}).

System	Nyquist plot			IE (%)
	R_{ct} ($\Omega\text{ cm}^2$)	C_{dl} (F/cm 2)	Impedance log (Z/Ω)	
MS + H_2SO_4	1769	2.090×10^{-8}	713.6	-
MS + H_2SO_4 + 0.2 M ZrO_2 NPs	14,715	0.631×10^{-8}	17,729.02	88.2

Table 4. Electrochemical impedance parameters.

References	Coating material	External factors studied	Effect	Key findings
4	Sol-gel derived ZrO_2 NPs for thermal barrier coatings	High Temperature	Phase transformation (tetragonal \rightarrow monoclinic)	Reduced thermal stability and increased surface roughness
10	Synthesis and characterization of ZrO_2 NPs via a simple solution route	Temperature, Humidity, Saltwater	Decreased under harsh conditions	Silver oxidation and magnetite degradation impact performance
13	Natural product-based inhibitors	Temperature, Humidity, Saltwater	Significant reduction in corrosion	Comprehensive overview of green inhibitors showing sustainable corrosion protection
15	ZrO_2 NPs by a novel solvothermal method	Temperature Effect	Controlled phase transformation at 150–200 $^{\circ}C$	Enhanced degradation efficiency under visible light
21	Green eco-friendly inhibitor for carbon steel	HCl solution, Temperature	Strong inhibition of carbon steel corrosion	Macro/micro-scale experimental and computational studies demonstrated high inhibition efficiency
24	anti-corrosive efficiency of NiO NPs	Temperature, Humidity, Saltwater	Significant inhibition of microbial growth	Effective in reducing corrosion rate in saltwater
27	Green synthesized ZrO_2 NPs using plant extracts	Acidic medium (HCl, H_2SO_4), Temperature	Improved corrosion inhibition	Plant-extract-based green synthesis offers eco-friendly corrosion protection in acidic environments
31	Zirconium-based nanostructures	Corrosion, Biocompatibility	High chemical stability	Advanced ZrO_2 nanostructures provide multifunctional protection including corrosion resistance in harsh environments
17	ZrO_2 nanostructures for catalysis and sensing	Temperature, Humidity, Saltwater	Enhanced stability under harsh conditions	Demonstrated stability and catalytic activity in challenging environments
Current Work	t- ZrO_2 NPs	Temperature, Humidity, Saltwater	Decreased with extreme conditions	Effective under controlled conditions but degrades in prolonged saltwater exposure

Table 5. Comparison of previously reported coating materials with the current material.

is due to enhanced thickness of the adsorbed layer. SEM and AFM analysis revealed a smoother surface on the inhibited carbon steel in contrast to the uncontrolled samples.

Data availability

The datasets during and/or analyzed during the current study are available from the corresponding author upon reasonable request.

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Author contributions

Conceptualization, methodology, data curation, writing—original draft: HMKS, KSM, NG writing—review and editing, Project Administration, supervision, Validation—RL, KK Former Analysis, Validation, writing—review, and editing—SR, MS, SS Software, resources, supervision—PP, AM writing—review and editing, resources, methodology -SEM, MA.

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Declarations

Competing interests

The authors declare no competing interests.

Ethical approval and consent to participate

The *Acacia nilotica* bark extract powder used in this study was obtained from Dr. A. Aslam, (Fellow of Indian Association for Angiosperm Taxonomy), Associate Professor of Botany, Jamal Mohamed College (Autonomous), Tiruchirappalli, Tamil Nadu, India. This Herbarium provides and fulfils the specialized demands of bio scientists from breeding program which is duly permitted and approved by Department of botany, Jamal Mohamed College (Autonomous), Tiruchirappalli. The samples were collected and studied with local or national guidelines with no need for further affirmation. This Herbarium provides the samples for the benefit of the scholar society and hence the findings, conclusions and opinions in this publication are of the authors alone. The collection of the plant, analysis and documentation were followed guidelines provided by IUCN Policy Statement. The Herbarium specimen of *Aicacia nilotice* has been deposited in the Herbarium of the Department of Botany, Jamal Mohamed College, with reference number JMCH2025-26/1.

Additional information

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