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RESEARCH ARTICLE

STUDY OF CORROSION INHIBITION FOR COPPER METAL BY USING WITHANIA SOMNIFERA EXTRACT IN ACIDIC AND AMMONIACAL MEDIUM

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ABSTRACT

Chemical based corrosion inhibitors are effective in preventing corrosion however they pose significant risks to the environment. In the field of corrosion science, plant extracts and their byproducts have demonstrated considerable promise as inhibitors for protecting metals and alloys from degradation in aggressive environments. Aswag and haroot extract was used as a natural corrosion inhibitor for copper metal in acidic and ammoniacal medium. Inhibition effect of root extract of Withaniasomnifera on copper has been studied using weight loss method at temperature range 303K to 343K. Result reveal that inhibition efficiency increases with increasing concentration of Withaniasomniferaroot extract from 100ppm to 700ppm. Effect of temperature on inhibition efficiency has also been shown. Thermodynamic parameters have also been reported. The adsorption of root extract obeyed Langmuir Adsorption Isotherm. Spectral Data have been discussed.

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INTRODUCTION

Natural ingredients derived from plants have long been acknowledged for their uses and applications. These compounds often exhibit a wide range of chemical, biological, and physical properties, making them valuable in numerous industrial and scientific applications. In recent years, attention has increasingly turned toward the use of plant-based materials as environmentally friendly alternatives in various sectors, particularly in corrosion inhibition. The appeal of these natural substances lies in their abundance, affordability, renewability, and biodegradability characteristics that position them as sustainable alternatives to conventional synthetic inhibitors. Within the domain of corrosion science extracts derived from plants and their byproducts have exhibited significant potential as eco-friendly corrosion inhibitors for safeguarding metals and alloys against degradation. These natural inhibitors, often referred to as "green corrosion inhibitors," are free from toxic heavy metals and hazardous chemicals, aligning well with the principles of green chemistry and sustainable development. Numerous research efforts have confirmed the efficacy of various plant-derived compounds in mitigating corrosion in both acidic and alkaline media. The use of such substances not only ensures environmental safety but also offers a cost-effective solution to industries facing corrosion-related challenges.

Preparation of the Inhibitor Solution: The corrosion inhibition study employed a natural plant extract derived from the roots of *Withaniasomnifera* (Ashwagandha). The root extract served as the corrosion inhibitor, selected for its well-documented phytochemical constituents, such as withanolides and alkaloids, which are known to possess properties conducive to metal surface adsorption and

Collection and Drying of Plant Material: Root samples of *Withaniasomnifera* were collected from a verified botanical source to ensure authenticity. To remove any adhering soil, microorganisms, or other impurities, the roots were thoroughly washed under running tap water followed by rinsing with distilled water. Clean samples were then shade-dried for 10–12 days at ambient temperature. Shade drying was preferred over direct sun exposure to prevent thermal degradation of sensitive bioactive compounds.

Extraction Procedure: After complete drying, the roots were chopped into smaller pieces to enhance surface area and facilitate efficient extraction of phytochemicals. These root fragments were then soaked in an ethanol solution (analytical reagent grade, ~95%) for a duration of 72 hours at room temperature (298 ± 2 K). Ethanol was chosen as the solvent due to its polarity and effectiveness in extracting both polar and moderately non-polar compounds from plant matrices. Post-soaking, the ethanolic mixture was filtered using Whatman No. 1 filter paper to separate the plant residue from the liquid phase. The collected filtrate was then subjected to rotary evaporation and gentle heating to remove ethanol. This process was conducted using a water bath under reduced pressure to preserve heat-sensitive phytoconstituents. The partially concentrated extract was left in a covered container at room temperature for an additional 24 hours to allow any remaining solvent to evaporate. The resulting viscous crude extract was air-dried to obtain a semi-solid mass, which was then stored in an amber-colored airtight container to prevent degradation from light and moisture. This crude extract was used to prepare a stock solution, from which test solutions of varying concentrations—100 ppm to 700 ppm—were formulated by appropriate dilution with distilled water. These concentrations were selected to observe the dose-dependent effect of the inhibitor on

Preparation of Corrosive Medium: Two corrosive environments were prepared to evaluate the performance of the inhibitor under different conditions:

Ammoniacal Medium: Ammoniacal corrosion medium was prepared by diluting laboratory-grade (AR) concentrated aqueous ammonia to obtain a 2N ammoniacal solution. Ammonia is known to form soluble copper-ammonia complexes, which aggressively corrode copper by dissolving its oxide layer. This medium simulates industrial environments where ammonia is used in cleaning agents or refrigerants.

Acidic Medium: A 0.5N hydrochloric acid (HCl) solution was prepared by diluting concentrated AR-grade HCl with distilled water. The acidic medium simulates pickling, descaling, and industrial waste conditions, where copper is frequently exposed to acidic solutions. The corrosive nature of HCl toward copper makes it suitable for studying the inhibitor's efficacy under strongly aggressive conditions. All solutions were freshly prepared before each experimental cycle and stored in sealed containers to avoid atmospheric contamination and concentration changes due to evaporation.

Preparation of Copper Specimens: High-purity copper metal sheets were used to fabricate corrosion specimens. Each sheet was cut into rectangular pieces with precise dimensions: 2.5 cm × 2.5 cm × 0.01 cm. A small hole was drilled near one edge of each specimen to facilitate suspension in the test solutions using non-reactive threads.

Surface Preparation: Prior to immersion, each copper specimen underwent meticulous surface treatment to ensure uniformity in exposure and reproducibility of results:

- Polishing: Specimens were polished using a sequence of emery papers (grades 400 to 1200) to obtain a smooth and even surface.
- Degreasing: After polishing, the metal surfaces were cleaned with acetone to remove oil, grease, or organic contaminants.
- Rinsing: Samples were then rinsed thoroughly with distilled water.
- Drying: Finally, the specimens were oven-dried and cooled in a desiccator.
- Weighing: Each dry specimen was weighed on an analytical balance with a precision of ±0.0001 g to record the initial mass (W_1).

METHODOLOGY: WEIGHT LOSS METHOD

The weight loss method was adopted to quantify the corrosion rate and inhibition efficiency due to its simplicity and reliability for long-duration exposure studies.

Experimental Setup: Each cleaned and pre-weighed copper specimen was suspended vertically in a 100 mL V-shaped capillary beaker containing the test solution (acidic or ammoniacal), with or without the inhibitor. The immersion was done in duplicate for each condition to ensure accuracy and reproducibility. Beakers were then placed in a thermostatically controlled water bath to maintain constant

Weight Loss Measurement- Weight loss analysis is one of the simplest and most reliable methods for determining the corrosion rate. In this method, a pre-weighed metal or alloy specimen is immersed in a corrosive medium for a specific duration. After the exposure period, the specimen is cleaned to remove corrosion products and reweighed. The difference in weight (before and after exposure) is used to assess the corrosion behaviour. For this study, the weight loss of copper specimens immersed in test solutions both with and without various

concentrations of the extract was recorded. The corrosion rate (CR) was calculated using the formula:

$$CR = \Delta W / (A \times t) \quad (1)$$

Where:

ΔW is the average weight loss of the copper specimen (in milligrams), A is the total surface area of the specimen and t is the immersion time, CR is corrosion rate expressed in $\text{mg}\cdot\text{cm}^{-2}\cdot\text{h}^{-1}$.

This method is most suitable when corrosion proceeds at a moderate and steady rate. Among various techniques, weight loss measurement is often preferred due to its simplicity and reliability. The inhibition efficiency (IE%) of the extract was calculated using the formula:

$$IE (\%) = [(W_0 - W) / W_0] \times 100 \quad (2)$$

Where:

I.E. = Inhibition Efficiency

W_0 = weight loss without the inhibitor

W = weight loss with the inhibitor

Surface coverage (θ) was calculated using:

$$\theta = (W_0 - W) / W_0 \quad (3)$$

These formulas help quantify the effectiveness of the inhibitor in reducing corrosion. Corrosion through weight loss can be accurately measured when specimens of identical size are used, with results expressed as weight loss per unit area per unit time. Before immersion, each specimen initial weight was recorded using an analytical balance. The specimens were then placed in 150 mL beakers containing 100 mL of the corrosive medium, both in the absence and presence of varying concentrations of *Withaniasomnifera* root extract. After a 24-hour immersion period, the specimens were removed, thoroughly washed, dried, and reweighed. The average weight loss was calculated from three parallel copper specimens to ensure accuracy.

Corrosion rates for the copper samples were determined across a temperature range of 303 K to 333 K using Equation 1, and the percentage inhibition efficiency was calculated using Equation 2. The resulting data including corrosion rates and inhibition efficiencies at different concentrations of *Withaniasomnifera* root extract in ammoniacal solution and acidic solution across the specified temperature range are summarized in the accompanying table 1.

RESULTS AND DISCUSSION

Effect of Concentration of Extract on Inhibition Efficiency- The effectiveness of *Withaniasomnifera* root extract as a corrosion inhibitor was assessed through weight loss analysis after 24 hours of metal immersion at temperatures ranging from 303 K to 333 K. A control sample without any extract was used for comparison, while the test solutions contained varying concentrations of the extract from 100 ppm to 700 ppm. Table 1 provides the corrosion rate and inhibition efficiency values obtained in both hydrochloric acid and ammoniacal solutions. The data reveal that inhibition efficiency improves as the extract concentration increases. At a concentration of 700 ppm, the extract demonstrates an efficiency of approximately 85% in both corrosive media. This corresponds to a noticeable reduction in the corrosion rate, indicating that the enhanced inhibition is directly linked to the suppressive effect of the extract on metal degradation.

The increasing efficiency suggests that the phytochemicals present in the extract adsorb onto the metal surface, creating a protective layer that slows down the corrosion process. Effect of Solution Temperature on Inhibition Efficiency- Corrosion inhibition experiments were conducted over a temperature range of 303 K to 333 K, with the corresponding results presented in Table 1.

Table 1. Inhibition efficacy and corrosion rate of Withania Somnifera extract for copper

| Medium | Temp (K) | 303 | | | 313 | | | 323 | | | 333 | | | |
|-----------------|----------|------------------------|----------|------------|-------|----------|------------|-------|----------|------------|-------|----------|------------|-------|
| | | Conc. Of extract (ppm) | wt. loss | corr. rate | I.E |
| HCl | Blank | | 0.0248 | 0.165 | - | 0.218 | 0.211 | - | 0.0433 | 0.289 | - | 0.0726 | 0.484 | - |
| | 100 | | 0.0155 | 0.103 | 36.50 | 0.130 | 0.140 | 34.12 | 0.0306 | 0.204 | 29.66 | 0.0553 | 0.369 | 24 |
| | 300 | | 0.0127 | 0.085 | 45.99 | 0.175 | 0.116 | 45.08 | 0.0251 | 0.167 | 42.10 | 0.0440 | 0.293 | 39.02 |
| | 500 | | 0.0102 | 0.069 | 57.10 | 0.142 | 0.095 | 54.05 | 0.0212 | 0.139 | 51.11 | 0.0387 | 0.256 | 47.02 |
| | 700 | | 0.0045 | 0.030 | 80.00 | 0.071 | 0.096 | 76.01 | 0.0105 | 0.073 | 75.08 | 0.0232 | 0.154 | 68.02 |
| NH ₃ | Blank | | 0.168 | 0.112 | - | 0.218 | 0.211 | - | 0.0494 | 0.333 | - | 0.071 | 0.446 | - |
| | 100 | | 0.0072 | 0.048 | 56.31 | 0.143 | 0.096 | 55.00 | 0.0231 | 0.155 | 53.00 | 0.0342 | 0.228 | 49.03 |
| | 300 | | 0.0035 | 0.024 | 76.39 | 0.072 | 0.047 | 76.52 | 0.0127 | 0.085 | 74.02 | 0.0200 | 0.133 | 70.05 |
| | 500 | | 0.0022 | 0.015 | 85.09 | 0.057 | 0.037 | 82.99 | 0.0096 | 0.065 | 80.31 | 0.0154 | 0.102 | 77.13 |
| | 700 | | 0.0005 | 0.004 | 94.29 | 0.011 | 0.065 | 94.00 | 0.0032 | 0.021 | 93.11 | 0.0064 | 0.043 | 91.09 |

The findings indicate that inhibition efficiency declines as the temperature increases. This reduction is attributed to the desorption of inhibitor molecules from the metal surface at elevated temperatures. The highest inhibition efficiency was observed at 303 K. As the temperature rises from 303 K to 333 K, a gradual decrease in efficiency is noted. The relationship between temperature and inhibition efficiency at different extract concentrations is illustrated in the graph shown in the figure.

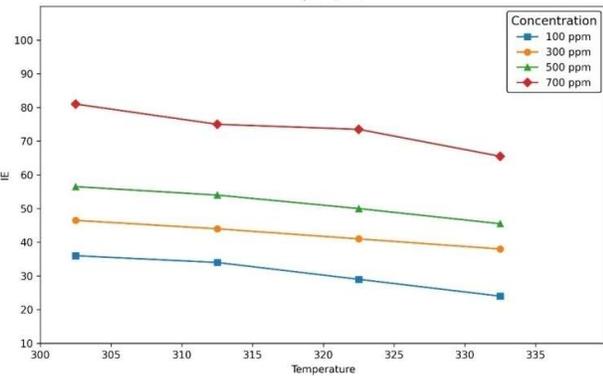


Figure 2 I.E vs Temperature graph in Acidic Medium

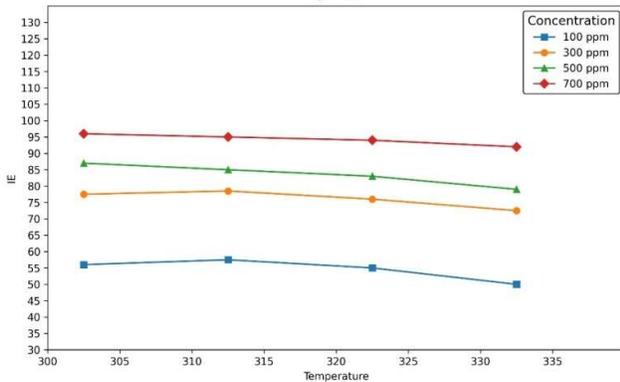


Figure 3 I.E vs Temperature graph in Ammoniacal Medium

Determination of Thermodynamic Parameters

Activation Energy-Thermodynamic parameters are crucial for understanding the mechanism of corrosion inhibition. The Arrhenius equation describes how corrosion rate varies with temperature and is given by:

$$\log(CR) = A + (-E_a / RT)$$

where CR represents the corrosion rate, A is the frequency factor, E_a is the apparent activation energy, R is the molar gas constant (8.314 K⁻¹mol⁻¹) and T is the absolute temperature in Kelvin.

The corresponding Arrhenius plots of log (CR) versus 1/T yield straight lines, as shown in the figure. The slope of these lines, which is equal to -E_a/R, was used to calculate the activation energy for *Withaniasomnifera* root extract, and the values are summarized in Table 2. In the absence of the extract, the activation energy is comparatively low. However, as the concentration of the root extract increases, the activation energy also rises. This increase in activation energy suggests that the extract molecules adsorb onto the copper surface, thereby inhibiting corrosion by blocking active sites. Additionally, the corrosion rate decreases with increasing extract concentration. However, at elevated temperatures, the corrosion rate increases again due to desorption of the inhibitor molecules. The observed decline in inhibition efficiency with rising temperature indicates that the adsorption of the root extract on the copper surface is predominantly governed by physisorption.

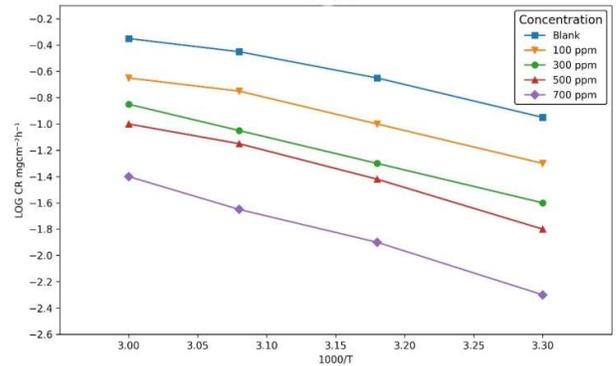


Figure 4 Arrhenius plot of Withaniasomnifera in Ammoniacal Medium

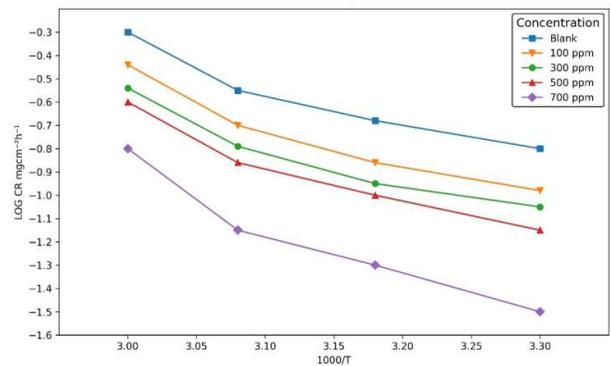


Figure 5. Arrhenius plot of Withaniasomnifera in Acidic Medium

Determination of enthalpy and entropy- Kinetic parameters such as enthalpy (ΔH) and entropy (ΔS) of activation were determined using the Transition State Theory equation expressed as

$$CR = (RT / Nh) \times \exp(\Delta S / R) \times \exp(-\Delta H / RT)$$

In this equation,

CR represents the corrosion rate, R is the universal gas constant, T is the absolute temperature in Kelvin, N is Avogadro's number, and h is Planck's constant.

The activation enthalpy (ΔH) was calculated from the slope of the plot using the relation:

$$\Delta H = \text{slope} \times 2.303 \times R$$

The activation entropy (ΔS) was derived from the intercept of the plot using the equation:

$$\Delta S = 2.303 \times R \times (\text{intercept} - \log(R / Nh))$$

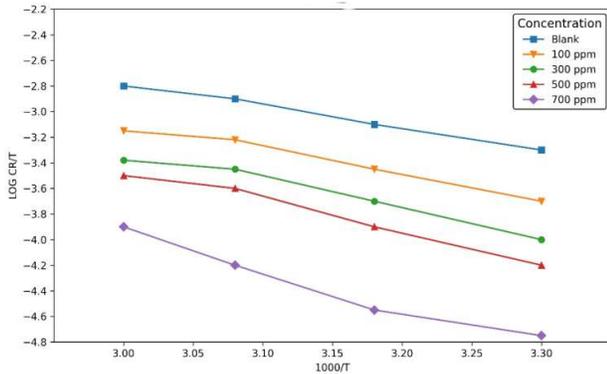


Figure 6 Transition State Plot of *Withaniasomnifera* extract in Ammoniacal medium

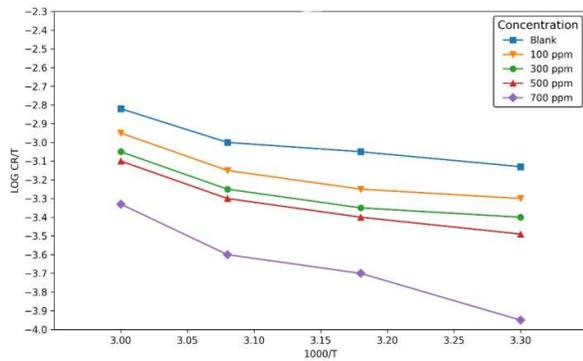


Figure 7. Transition State Plot of *Withaniasomnifera* extract in Acidic medium

These kinetic parameters provide insight into the nature of the corrosion inhibition process, particularly the energy barrier and molecular arrangement during the metal-inhibitor interaction. The figure displays the transition state plots for copper corrosion in both the absence and presence of varying concentrations of *Withaniasomnifera* root extract. The plots of $\log(CR)$ versus $1/T$ yield straight lines, from which the enthalpy (ΔH) and entropy (ΔS) of activation were calculated using the slope and intercept, respectively. The corresponding values are presented in Table 2. As shown in Table 2, the ΔH values are positive and increase with higher concentrations of the extract, indicating improved inhibition efficiency. This rise in enthalpy suggests that more energy is required for the corrosion process as the inhibitor concentration increases, reflecting stronger interaction between the inhibitor molecules and the metal surface.

Table 2. Thermodynamics parameter at different concentration of *Withaniasomnifera* extracts

| Medium | Concentration (ppm) | Ea (kJ/mol) | ΔH (kJ/mol) | ΔS (J/mol·K) |
|-----------------|---------------------|-------------|---------------------|----------------------|
| HCl | Blank | 29.24 | 26.55 | -170.80 |
| | 100 | 32.00 | 30.52 | -164.43 |
| | 300 | 33.85 | 30.02 | -163.79 |
| | 500 | 34.79 | 31.10 | -159.43 |
| | 700 | 42.00 | 39.88 | -140.76 |
| NH ₃ | Blank | 36.12 | 34.34 | -144.00 |
| | 100 | 42.17 | 38.00 | -136.98 |
| | 300 | 43.75 | 41.17 | -132.80 |
| | 500 | 50.94 | 48.44 | -117.02 |
| | 700 | 56.80 | 54.70 | -106.95 |

Adsorption Isotherm - The entropy of activation (ΔS) is negative in both inhibited and uninhibited systems, with increasingly negative values observed at higher extract concentrations. This suggests that the transition state represents an association process rather than dissociation, leading to a decrease in disorder during the formation of the activated complex. In acidic media, the primary mechanism by which corrosion inhibitors function is widely accepted to involve adsorption onto the metal surface. This process assumes that the inhibitor molecules occupy active sites on the surface, thereby preventing corrosion reactions in those areas. Conversely, corrosion continues on the uncovered regions of the metal surface.

The extent of surface coverage by the inhibitor, denoted as θ (theta), is calculated using the equation:

$$\theta = (IE / 100)$$

where IE is the inhibition efficiency (%). The values of θ are important for understanding the adsorption behaviour of the inhibitor. To evaluate the adsorption characteristics at equilibrium, the variation of surface coverage with inhibitor concentration at a constant temperature was analyzed. The data were tested for compatibility with the Langmuir adsorption isotherm, which is expressed as:

$$C / \theta = 1 / K + C$$

where C is the inhibitor concentration and K is the adsorption equilibrium constant.

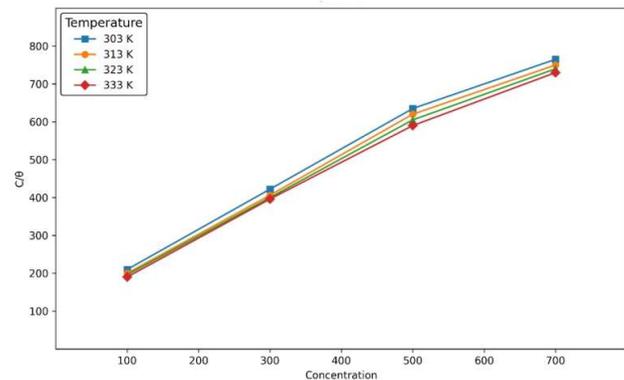


Figure 8. Langmuir adsorption isotherm of extracts of *Withaniasomnifera* in Ammoniacal Medium

A graphical plot of C / θ versus C was generated at various temperatures and is shown in the figure. The resulting straight lines, with slopes close to unity and correlation coefficients near 1, confirm that the adsorption of *Withaniasomnifera* root extract on the metal surface follows the Langmuir adsorption isotherm model. In the Langmuir adsorption model, the constant K represents the adsorption equilibrium constant, which can be determined from the intercept of the linear plot of C/θ versus C on the C/θ axis. This constant is

directly related to the standard free energy of adsorption (ΔG°_{ads}) through the equation:

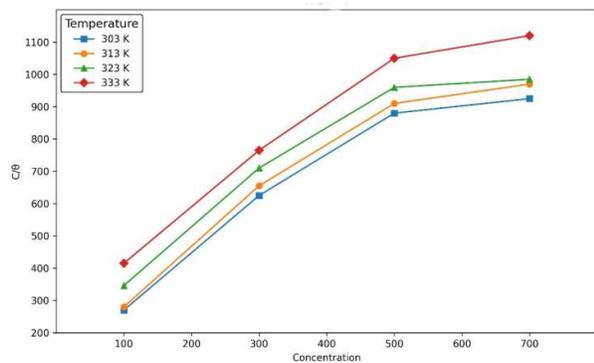


Figure-9 Langmuir adsorption isotherm of extracts of *Withaniasomnifera* in Acidic Medium

$$K = (1 / 55.5) \times \exp(-\Delta G / RT)$$

Here, R is the universal gas constant (8.314 J/mol/K), T is the absolute temperature in Kelvin, and 55.5 represents the molar concentration of water in the solution (mol/L).

Rearranging the equation allows calculation of the free energy of adsorption as:

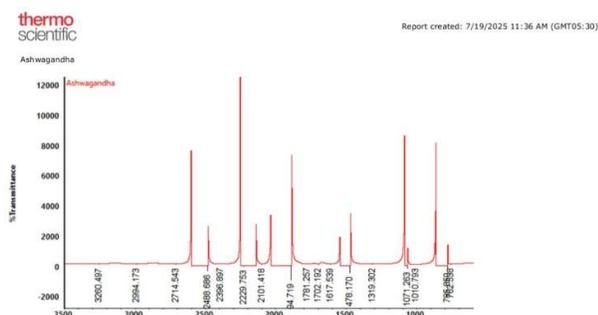
$$\Delta G = -RT \times \ln(55.5 \times K)$$

The calculated ΔG values for the adsorption of the *Withaniasomnifera* root extract on the copper surface are presented in Table 3. The negative values of ΔG indicate that the adsorption process is spontaneous and that a stable adsorbed film forms on the copper surface. Furthermore, as temperature increases, the absolute value of ΔG decreases, suggesting a reduction in adsorption strength and the stability of the inhibitor film. This trend also indicates that the adsorption process is endothermic. Since the calculated ΔG values are less than the typical threshold for chemisorption, the adsorption mechanism is best described as physisorption. This conclusion is further supported by the observed decrease in inhibition efficiency with rising temperature.

Table 3. Values of ΔG at different temperatures for *Withaniasomnifera* extract

| Medium | Temperature (K) | R ² | ΔG (kJ/mol) |
|-----------------|-----------------|----------------|---------------------|
| HCL | 303 | 0.78 | -13.45 |
| | 313 | 0.88 | -13.79 |
| | 323 | 0.85 | -13.87 |
| | 333 | 0.88 | -13.96 |
| NH ₃ | 303 | 0.99 | -15.04 |
| | 313 | 0.97 | -16.38 |
| | 323 | 0.97 | -16.66 |
| | 333 | 0.96 | -16.72 |

Spectral Data



FTIR Spectrum Interpretation of Ashwagandha Extract- X-axis: Wavenumber (cm⁻¹) – shows bond vibration frequencies. Y-axis: % Transmittance (or possibly intensity) shows how much light was absorbed.

Key Observed Peaks & Functional Group Assignments: Wavenumber (cm⁻¹) and functional group with possible compound in Ashwagandharelevance to corrosion inhibition

- 1) ~3260–3294 shows O–H / N–H stretch may have alcohols, phenols, withanolides Can form H-bonds or adsorb on metal surfaces
- 2) ~2924, ~2850 shows C–H stretch (aliphatic CH₂/CH₃) may have Steroidal structures which can help to create hydrophobic protective layer
- 3) ~2101–2200 shows Possibly ≡C–C (alkyne), or N=C=O (rare) may have minor groups (if any) Limited role; may indicate trace nitrile-like groups
- 4) ~1741–1760 shows C=O stretching (carbonyl group) Ketones, esters (from withanolides) Coordinates with metal to form protective film
- 5) ~1600–1650 shows C=C (aromatic), or amide C=O aromatic systems in flavonoids/alkaloids π-electrons interact with metal surface
- 6) ~1400–1500 shows CH₂, CH₃ bending may have aliphatic chains hydrophobic interaction on surface
- 7) ~1000–1300 shows C–O stretching, C–N may have alcohols, esters, amino acids polar groups that can bind metal via O or N atoms
- 8) ~600–900 shows aromatic C–H bending may have aromatic rings in alkaloids/flavonoids Confirms aromatic content useful in adsorption

Ashwagandha as a Corrosion Inhibitor- Ashwagandha contains withanolides, alkaloids, and flavonoids, which include: Hydroxyl (–OH), Carbonyl (C=O), Aromatic rings (C=C), Alkyl chains (C–H) and Possible amines (N–H). These functional groups Adsorb on the copper surface in acid medium, block active corrosion sites, Form a protective barrier against H⁺ or Cl⁻ ion attack. Evidence of Corrosion Inhibition- This FTIR spectrum confirms the presence of donor atoms like: Oxygen (from –OH, C=O), Nitrogen (from amines/alkaloids), π-electrons (from aromatic rings) These act as active sites for binding onto metal surfaces (like copper), forming a chemisorbed or physisorbed protective film.

CONCLUSION

FTIR spectrum of Ashwagandha extract shows characteristic peaks of –OH, C=O, C–O, aromatic rings, and CH stretches. These functional groups are strongly linked to corrosion inhibition, as they Bind to copper atoms in acidic media, Prevent the interaction of corrosive ions with the metal stabilize the surface by forming a complex or film. This supports that Ashwagandha is an effective eco-friendly corrosion inhibitor.

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