

Fruit Extract as a Green Inhibitor for Copper Corrosion in Nitric Acid Solution

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ABSTRACT: The adsorption and inhibition effects of fruit extracts of *Citrus aurantium* (CAU), *Moringa oleifera* (MOL) and *Capsicum annuum* (CAN) on corrosion of copper were studied in nitric acid medium. Gravimetric, potentiodynamic polarization and electrochemical impedance spectroscopic techniques were used to evaluate the inhibition efficiency. Electrochemical analysis indicated that these fruit extracts acted as predominantly cathodic mixed inhibitors. The adsorption of fruit extracts on the copper surface, leading to inhibition of corrosion process, followed Langmuir adsorption isotherm.

KEYWORDS: Copper, Inhibitor, EIS, SEM, AFM

I. INTRODUCTION

Acid solutions are generally used in several industries for a variety of purposes, such as acid pickling, cleaning, descaling etc of a number of metallic objects. Because of the aggressive nature of acid solution, inhibitors are commonly used to tame their corrosive attack. Most of the research done in this field is concerned with steel – acid systems. There are scant references available in literature pertaining to use of inhibitors to counter the acid attack on those objects in the fabrication of which copper is extensively used [1-3]. Most of the traditional organic and inorganic inhibitors are synthetic compounds, which are costly and often lead to serious environmental problems [4-6]. To combat the problem of environmental hazards, the current researchers have focused their attention on using natural compounds, particularly plant extracts, as corrosion inhibitors.

The use of plant extracts in corrosion inhibition studies has gained momentum because these are not only cheap, easily available and eco-friendly but are also extracted with procedures at very low cost. The high yield of these natural products as well as their corrosion inhibition abilities largely depends on the particular part of plant and location in which the plants are found [7]. The extract of different parts of plant like - leaves, seeds, flower and fruits can be used as inhibitor to reduce the corrosion rate in acidic media. The extract of *Tagetes erecta* (Marigold flower) has been successfully used by Mourya et al, to inhibit the corrosion of mild steel in sulphuric acid medium to a very large extent at low concentration [8]. The inhibitive performance of *Gingko biloba* (Pankha plant) fruit extract on corrosion of steel in 3.5% NaCl solution saturated with CO₂ has been investigated by Ambrish Singh and coworkers [9]. They reported the maximum inhibition efficiency to the tune of above 97% for relatively higher concentration of 1000 ppm. Corrosion inhibitive effects of naturally occurring *Osmanthus fragran* (Tea olive) [10] have been studied in different concentrations of HCl for steel. The maximum corrosion inhibition efficiency was reported to be 94 and 96% for leaves extract at 298 and 318K, respectively.

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The extract of *Ricinus Communison* as are inhibitor for copper corrosion in HNO_3 solution was studied using weight loss and potentiodynamic polarization curves [2]. The maximum corrosion inhibition efficiency was reported to be 99.4% in 300 ppm at 298K. The influence of *Thymus vulgarise* extract on copper corrosion in HNO_3 solution was studied using weight loss and potentiodynamic polarization studies by Fouda and co-workers [11]. They concluded that the plant extracts could serve as effective corrosion inhibitor. The inhibition performance of plant extract is usually recognized to the presence of complex organic species, including tannins, alkaloids and nitrogen bases, carbohydrates and proteins as well as hydrolysis products in their composition.

Foregoing literature reveals that very little work has been done on copper corrosion protection using fruit extracts in nitric acid. In present study, we have chosen three fruits, *Citrus aurantium* (12), *Moringa oleifera* (12), *Capsicum annum* (13), commonly known as ‘Orange’, ‘Drumstick’, and ‘Capsicum’ respectively for preparing their extract. The aim of this paper is to evaluate and explain the corrosion inhibition of copper in nitric acid by these extracts. The inhibition study has been conducted by gravimetric, potentiodynamic polarization and electrochemical impedance spectroscopy measurements.

II. EXPERIMENTAL

2.1. Coupon preparation

Copper coupons of size $2 \times 2 \times 0.1 \text{ cm}$ and $3 \times 1 \times 0.1 \text{ cm}$ cut from the same metal sheets were used for gravimetric and electrochemical measurements, respectively. The specimens were mechanically abraded with 600, 1000 and 1500, 2000 grade of emery paper, washed successively with double distilled water and acetone. The samples were dried at room temperature and stored in a desiccator.

2.2. Electrolytic solution

The corrosive solution, 3M HNO_3 , was prepared by dilution of analytical grade HNO_3 of predetermined normality with double distilled water. The concentration range of the extract used was $0.05\text{--}0.3 \text{ g L}^{-1}$ and the volume of electrolyte used was 150 ml for each experiment.

2.3. Inhibitor

Citrus aurantium, *Moringa oleifera* and *Capsicum annum* fruits were separately dried and ground to powder form 10 g of the powder was soaked in 500 ml double distilled water and refluxed for 4 h. It was filtered and the resulting filtrate was concentrated to 100 ml by evaporation. The obtained extract was used for the measurements of inhibitive properties. The amount of extract in the filtrate was determined by subtracting the weight of the residue obtained from the total weight of the dried fruit taken.

2.4. Gravimetric measurement

The abraded Cu specimens of size $2 \times 2 \times 0.05 \text{ cm}$ were weighed on a balance with 1 mg sensitivity and immersed for 4 h in 3M HNO_3 in absence and the presence of 0.05, 0.1, 0.2 and 0.3 g L^{-1} in the temperature range 308-323K. To compensate the volume change due to addition of inhibitor, volume correction was done while preparing 3M HNO_3 acid. The corrosion rate (C_R) in $\text{mg cm}^{-2} \text{ h}^{-1}$ was calculated from the following equation

$$C_R = \frac{\Delta W}{S \times t} \quad (1)$$

where, ΔW is the average weight loss, S the total area of the specimen and t is the immersion time. From the obtained corrosion rate, percentage inhibition efficiency ($\eta \%$) was calculated by using the same equation as described earlier [8].

2.5. Electrochemical measurements

Electrochemical tests were carried out in a conventional three electrode cell assembly. This assembly consisted of a flat bottom Pyrex glass flask with three openings one each for working reference and counter electrodes. A rectangular working electrode of copper metal with an exposed surface area of 1 cm^2 was attached to a self-designed holder and the rest of the surface was covered with lacquer. Silver-silver chloride electrode (3M KCl) and platinum wire were used as

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a reference and counter electrodes, respectively. The working electrode was first immersed in test solution for establishing a steady state open circuit potential (OCP) and after attaining this potential the cathodic and anodic polarization studies were conducted using electrochemical analyzer (CHI604C).

Linear polarization resistance (LPR) measurements were potentiodynamically performed in a potential range +10 to -10 mV with respect to OCP at a scan rate of 0.01 mV/s. Polarization resistance (R_p) values were obtained from slope of the resulting linear current density versus potential plot. From the measured R_p values, η % was calculated using the relation as mentioned prior [8]. In potentiodynamic polarization study the curves were obtained from -250 to +250 mV with respect to OCP at a scan rate of 0.5mV/s. AC impedance measurements were carried out using signals of 5mV amplitude in the frequency range from 100 kHz to 0.01 Hz. The experimental impedance data were analysed with Zsimpwin software. The charge transfer resistance (R_{ct}), was obtained from the diameter of the semicircle of the Nyquist plot.

III. RESULTS AND DISCUSSION

3.1. Gravimetric measurements

3.1.1 Effect of inhibitor concentration

The effect of addition of CAU, MOL and CAN fruit extracts at different concentrations (0.05-0.3 gL⁻¹) on copper corrosion in acid solution (3M HNO₃) was evaluated by gravimetric measurement at 308 K. The values of inhibition efficiency and corrosion rate obtained from gravimetric measurement are listed in Table 1. The corrosion rate decreases and inhibition efficiency increases with increase in inhibitor concentration up to 0.3 gL⁻¹. Beyond this concentration the increase inhibition efficiency was too small to consider worth for further study. The maximum inhibition efficiency observed at 0.3 g L⁻¹ was to be 85% for CAN, 71% for MOL and 62% for CAU. From Table 1 it is clearly seen that the order of inhibition efficiency of Fruit extract is as follows: CAU < MOL < CAN.

Table1. Corrosion parameters for copper in 3M HNO₃ in absence and presence of different concentrations of CAU, MOL and CAN gravimetric measurement at 308 K for 4 h.

Inhibitor	Concentration (g L ⁻¹)	Weight loss (mg cm ⁻² h ⁻¹)	C _R (mg cm ⁻² h ⁻¹)	η (%)
Blank	---	60	1.666	---
	0.05	34	0.944	43
	0.1	31	0.861	48
	0.2	26	0.722	57
	0.3	23	0.638	62
	0.4	24	0.666	60
CAU	0.5	25	0.694	58
	0.05	29	0.805	52
	0.1	25	0.694	58
	0.2	23	0.638	62
	0.3	17	0.472	71
	0.4	18	0.500	70
MOL	0.5	19	0.527	68
	0.05	19	0.527	68
	0.1	15	0.416	75
	0.2	11	0.305	81
	0.3	9	0.250	85
	0.4	10	0.277	83
CAN	0.5	12	0.333	80

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3.1.2. Effect of temperature

The effect of temperature was studied at various temperatures (308 - 323 K) in absence and presence of highest concentration (0.3 g L⁻¹) of all the three inhibitors in 3M HNO₃ solution. The variations in C_R and η% values with variation in temperature are listed in Table 2. It is observed that C_R increase with rise in temperature from 0.638 to 2.666, 0.472 to 2.111 and 0.250 to 1.555 mg cm⁻² h⁻¹ and η% decrease from 62-41, 71-54 and 85-66% for CAU, MOL, CAN, respectively.

Table 2. Various corrosion parameters for copper in 3M HNO₃ in absence and presence of optimum concentration (0.3 g L⁻¹) of CAU, MOL and CAN at different temperature

Inhibitor	Temperature (K)	C _R (mg cm ⁻² h ⁻¹)	Θ	η (%)
Blank	308	1.666	---	---
	313	2.388	---	---
	318	3.361	---	---
	323	4.583	---	---
CAU	308	0.638	0.62	62
	313	1.083	0.55	55
	318	1.750	0.48	48
	323	2.666	0.41	41
MOL	308	0.472	0.71	71
	313	0.777	0.67	67
	318	1.305	0.61	61
	323	2.111	0.54	54
CAN	308	0.250	0.85	85
	313	0.416	0.82	82
	318	0.750	0.78	78
	323	1.555	0.66	66

Decrease in the inhibition efficiency with increase in temperature is suggestive of physical adsorption mechanism. It has been shown that the logarithm of corrosion rate is a linear function with 1/T. The apparent activation energy (E_a) for corrosion process of copper in nitric acid solution is calculated by the using Arrhenius equation [14]. The values of E_a were calculated by using a plot between log C_R and 1/T with a slope of - E_a/2.303 R (Fig.1(a)). The value of E_a (Table 3) is higher in the presence of extract than for the uninhibited solution which suggests the formation of an energy barrier for mass and charge transfer in inhibited system.

Table 3. Activation parameters E_a, ΔH_a^o and ΔS_a^o, for the copper dissolution in 3M HNO₃ in the absence and presence of CAU, MOL and CAN at optimum concentration.

Inhibitor (gL ⁻¹)	Linear regression coefficient (R ²)	E _a (kJ mol ⁻¹)	ΔH _a ^o (kJ mol ⁻¹)	ΔS _a ^o (Jmol ⁻¹ K ⁻¹)
Blank	0.999	56.13	53.45	-67.69
CAU	0.999	78.84	76.62	18.67
MOL	0.999	82.86	80.72	29.22
CAN	0.998	103.96	101.74	61.29

CAN has highest E_a than other remaining two extracts. The values of the enthalpy of activation (ΔH_a^o) and entropy of activation (ΔS_a^o) were calculated using the equation of transition state [15].

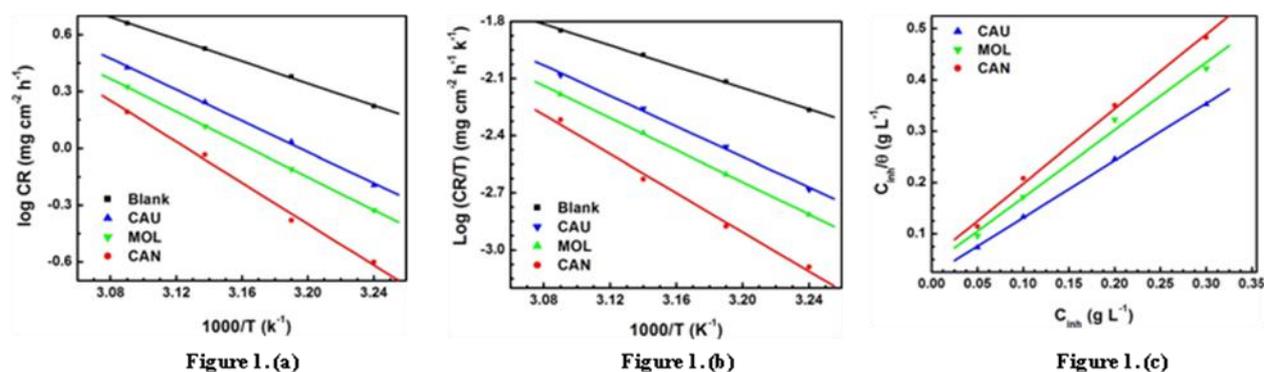


Figure 1. (a) Arrhenius plots (b) Transition - state plot (c) Langmuir adsorption isotherm plot for copper corrosion in absence and presence of optimum concentrations (0.3 g L^{-1}) of CAU, MOL and CAN in 3M HNO_3 .

A plot of $\log CR/T$ against $1/T$ gave a straight line Fig.2 (b) with a slope of $-\Delta H_a^o/2.303R$ and the values of ΔH_a^o and ΔS_a^o were calculated with an intercept of $\log(R/Nh) \Delta S_a^o/2.303$. These data are listed in Table 3. The negative value of ΔS_a^o is obtained in blank while the positive value is obtained in case of all these extract. The negative ΔS_a^o value indicates the activated complex is more ordered in the blank solution. The positive values of ΔS_a^o suggest that an increase in disordering takes place in going from reactants to the activated complex on metal - solution interface [10] which is driving force for the adsorption.

3.2. Adsorption isotherm

Organic molecules are usually effective inhibitor components found in plant extracts, the corrosion inhibition of these inhibitors is always explained based on molecular adsorption [16]. In addition, the adsorption isotherms describe the interaction of the inhibitor molecules with the active sites on the metal surface. The elucidation of the adsorption isotherm involves mainly the fitting of the data with various isotherms such as the Frumkin, Temkin, Freundlich and Langmuir isotherms. In present work Langmuir adsorption isotherms gave the best result.

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh} \tag{2}$$

where, K_{ads} is the equilibrium constant of the process of adsorption, C_{inh} the inhibitor concentration, and θ is the degree of surface coverage. The plot of C_{inh}/θ against C_{inh} shown in Fig.1(c) yield a straight line with regression coefficient ($R^2 > 0.999$) and suggests that the adsorption of CAU, MOL and CAN from 3M HNO_3 solution on copper surface obeys Langmuir isotherm.

The standard free energy of adsorption (ΔG_{ads}^o) is related to the adsorption constant (K_{ads}) with the following equation:

$$\Delta G_{ads}^o = -RT \ln(C_{H_2O} K_{ads}) \tag{3}$$

where $C_{H_2O} = 1000 \text{ g L}^{-1}$ in the solution. In this equation 1000 g L^{-1} is used for concentration of water, rather than 55.5 mole per liter because unit of K_{ads} has been reported here in terms of g^{-1}L [8]. The calculated value of ΔG_{ads}^o and K_{ads} of investigated inhibitors (Table 6) are in the following order as $\text{CAU} < \text{MOL} < \text{CAN}$.

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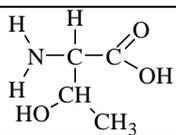
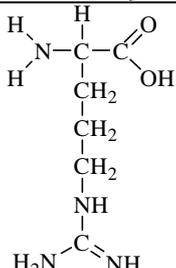
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Table 4. Thermodynamics parameters for adsorption of CAU, MOL, and CAN inhibitors on copper in 3M HNO₃ solution at 308 K.

Inhibitor (gL ⁻¹)	K _{ads} (Lg ⁻¹)	Linear regression coefficient (R ²)	-ΔG _{ads} (kJ mol ⁻¹)
CAU	5.43	0.999	10.95
MOL	8.16	0.998	11.28
CAN	18.88	0.999	12.04

The main constituents present in the fruit extract of CAU, MOL and CAN are Threonine, Arginine, and Capsaicin and Ascorbic acid, respectively, and these structures are shown in Table 5. CAN has heteroatom (O, N), fused ring and OH moiety in this structure, which facilitate its adsorption on copper surface. In case of MOL there are only four N atoms, in imine moiety and alkyl chain. The relatively less performance of CAU is owing to the presence of COOH (electron withdrawing) group, which reduces the electron density on nitrogen atom. Therefore, the adsorption process occurs either by the electrostatic interaction in charged molecule and constituents of inhibitors or by the interaction of unshared electron pairs of inhibitor molecules.

Table 5. Molecular structure and common name of main constituents present in fruit extracts

Fruit Extract	Constituents		
	IUPAC Name	Structure	Common Name
<i>Citrus aurantium</i> (CAU)	2 - Amino -3- hydroxyl - butyric acid		Threonine
<i>MoringaOleifera</i> (MOL)	2 - Amino -5- guanidine - pentanoic acid		Arginine

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Table 6. Electrochemical parameters obtained by Tafel polarization for copper in 3M HNO₃ solution in without and with of CAU, MOL and CAN at 308 K.

Inhibitor (gL ⁻¹)	E_{corr} (mV)	i_{corr} (μAcm^{-2})	β_a (mV dec ⁻¹)	β_c (mV dec ⁻¹)	η (%)	R_p ($\Omega\text{ cm}^2$)	η (%)
Blank	-31	1305	96	307	--	31	--
CAU	-54	470	115	207	64	84	63
MOL	-62	380	103	221	71	107	71
CAN	-89	159	97	249	88	263	88

3.3.2. Electrochemical impedance spectroscopic (EIS) measurement

The corrosion behaviour of copper in 3M HNO₃ solution in the presence of CAU, MOL and CAN was investigated by using electrochemical impedance technique at 308 K. Nyquist plots of copper in the absence and presence of all three inhibitors are presented in Fig.2 (b). It is clear from the figure that the impedance spectrum is not a perfect semicircle due to the frequency dispersion and surface inhomogeneity. The Nyquist plot displays a capacitive loop at high frequencies and a straight line - an inductive loop at low frequencies. A high frequency capacitive loop is explained by charge transfer during the corrosion process and a low frequency inductive loop is due to the diffusion- controlled mechanism [16].

To investigate technical details of impedance spectra, we used an equivalent circuit presented in Fig.2(c), has been used and calculated various parameters, viz., solution resistance (R_s), constant phase element (CPE) in parallel to the charge transfer resistance (R_{ct}), and R_{ct} is in series with Warburg impedance (W). To compensate for inhomogeneity in the system, the capacitance was implemented as a CPE that is defined by two values, ' Y_0 ' and ' n '. CPE is explained as follows [18].

$$Z_{CPE} = Y_0^{-1} (i\omega)^{-n} \tag{5}$$

Where ' Y_0 ' is a proportionality factor, ' ω ' is the angular frequency and ' n ' has the meaning of phase shift. The calculated parameters are listed in Table 7. It is observed that R_{ct} increased on addition of these extracts to the blank.

Table 7. Impedance parameter obtained fitting the Nyquist plots with the equivalent circuit in absence and presence of optimum concentration of CAU, MOL and CAN at 308 K.

Inhibitor	R_s ($\Omega\text{ cm}^2$)	R_{ct} ($\Omega\text{ cm}^2$)	CPE		Chi Square	W ($\Omega^{-1}\text{cm}^2\text{s}^{1/2}$)	η (%)
			Y_0 ($\mu\text{ }\Omega^{-1}\text{s}^n\text{ cm}^2$)	n			
Blank	4.3	23.05	160.2	0.85	0.0016	0.0045	--
CAU	3.5	60.1	96.6	0.84	0.0031	0.2772	62
MOL	4.8	82.5	97.1	0.87	0.0054	0.1468	72
CAN	5.2	181.7	110.4	0.89	0.0081	0.0981	87

VI. CONCLUSION

CAU, MOL and CAN fruit extracts are found to be good corrosion inhibitors for copper in 3M HNO₃ solution. The inhibition efficiency increases with increasing the inhibitor concentration and decreases with increasing temperature. The adsorption of fruit extract on copper surface followed the Langmuir adsorption isotherm. Polarization study reveals that these extracts behave as mixed inhibitors which are predominantly cathodic.

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