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Corrosion inhibition efficiency of human black hair extract on mild steel in 1 M H_2SO_4 media

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The use of human black hair (HBH) extract as corrosion inhibitors of mild steel in 1 M H_2SO_4 medium has been studied using weight loss, potentiodynamic polarization, electrochemical impedance spectroscopy. The results show the corrosion inhibition efficiency increased on increasing the concentration of the inhibitor. The extract is s a mixed type inhibitor with optimum concentration of 0.040% v/v in potentiodynamic polarization. The adsorption characteristics of the inhibitors have been determined from the results. Scanning electron microscopy (SEM) study confirm that the inhibition of corrosion of mild steel is through adsorption of the extract molecules on surface of metal.

Keywords: Corrosion inhibition, Mild steel, Human hair extract, Acid medium, Electrochemical, SEM, EDAX

Mild steel (MS), also known as plain carbon steel is widely used material in the fabrication of heating and cooling water system in many industries. Its price is relatively low and provides material properties that are acceptable for many applications[1,2]. Although, mild steel has remarkable economic and substantial applications, its deprived corrosion resistance in acids limits the usage. Acid solutions are essentially used on metal finishing industries, acidizing of oil well, cleaning of boilers and heat exchangers[3-6].

The use of inhibitor is one of the most practical methods for preparation against, especially in acidic media[7]. A number of heterocyclic compounds have been reported as corrosion inhibitors and the screening of synthetic heterocyclic compounds is still being continued. Several other inorganic inhibitors were used as inhibitor and they showed good anti-corrosive activity[7]. But most of them are highly toxic to both human beings and environment[8.]

Due to increased awareness about toxicity of the chemical inhibitors and strict environmental regulations, attention has been focused towards the development of non-toxic alternatives to synthetic inhibitors used so far[9]. There were several succeeding reports on non-toxic, eco-friendly green inhibitors[10-13].

The aim of this present work is to study the inhibitory action of human hair on the surface of mild steel using weight loss, potentiodynamic polarization, Electrochemical impedance spectroscopy and scanning electron microscope.

Human hair is considered a waste material in most parts of the world and its accumulation in waste streams causes many environmental problems. Preventing the waste of such a material requires both addressing the problems in the current usage and developing its utilization systems at locations where they are missing. Both in rural and urban areas this waste causes many problems since it takes several years to decompose. Due to slow degradation, it stays in the dumps and occupies more volume of space over time; leachate from these dumps increases the nitrogen concentration in the water bodies, causing problems of Eutrophication. Burning of human hair produces foul odour and toxic gases such as ammonia, Sulphur dioxide, phenols, nitriles, pyrroles and pyridines[14,15].

The best way to solve the problem is to develop systems which utilize the waste material as a resource. In addition to reducing waste, it contributes to the economy.

Constituents of hair

Human hair consists of proteins, lipids, water, trace elements and pigments.Compounds present in ethnic HBH sample were reported using GC-MS analysis. They are listed as follow[16-18]:

Alanine, Arginine, Aspartic acid, Cystine, Glutamic acid, Glycine, Histidine, Isoleucine, Leucine, Lysine,

Methaionine, Phenylalanine, Prolyne, Serine, Threonine, Tryptophan, Tyrosine, Valine.

Experimental Section

Preparation of the sample extract

Ethnic hair samples were collected and cut into small pieces (1cm length), washed with double distilled water and then ethanol and dried well. 10 mL of 1M NaOH is taken in a clean 1000 mL beaker and to that 10 g of hair sample is digested at 70-90°C. After digestion it is made upto 500 mL using distilled water and thus 2% stock solution of the inhibitor was prepared.

Preparation of specimens

Working electrodes of mild steel containing 0.09% P, 0.37% Si, 0.01% Al, 0.05% Mn, 0.19% C, 0.06% S and the remainder Fe, of dimensions 2.5 cm × 1 cm × 0.1 cm were used for the electrochemical polarizations and impedance measurements. The specimens were abraded into the uniform surface successively using the emery papers of 150, 180, 320, 400, 600 and 1000 grade. The surface were then degreased with acetone and washed with double distilled water before the experiment.

Electrolyte

The electrolyte of 1M H_2SO_4 solution was prepared by diluting Conc. H_2SO_4 using double distilled water which is used as a corrosive solution.

Weight loss measurements

Gravimetric measurements were performed on mild steel coupons with the above mentioned dimensions in 1M H_2SO_4 solution with and without of inhibitors. Initial weights of the polished mild steel plates were noted. The sample was immersed using glass hooks in triplicate in 1M H_2SO_4 solution contained in a 100 mL beaker, both in the absence and presence of inhibitor for different intervals of time (Table 1). The specimens were taken out, washed with deionized water, dried and reweighed. The corrosion rate (CR) and inhibition efficiency (IE %) was calculated using the formula (1) and (2) respectively.

$$Corrosion\ rate = \frac{87.6 \times W}{D\ A\ T} \quad \ldots (1)$$

where W is weight loss in mg, D is density in mg, A is area of exposure in cm^2, and T is time in hours.

$$IE\ (\%) = \frac{Wo - Wt}{Wo} \quad \ldots (2)$$

W_0 is weight loss without inhibitor. W_t is weight loss with inhibitor. The effects of temperature on the corrosion inhibition performance for the various concentrations of the HBH extract were studied in the range of (303 to 323 K). The solution temperature was thermostatically controlled at desired temperatures.

Electrochemical measurements

Electrochemical studies were carried out by using an electrochemical work station, Bio-logic SP 200 instrument. A three electrode compartment cell was used for the electrochemical measurements. A saturated calomel electrode (SCE) and a platinum electrode were used as the reference and the mild steel as working electrode (WE), respectively. The area of the WE exposed to the solution was approximately 1 cm^2. The specimen was pre-treated similarly as done in the gravimetric measurements. Before each potentiodynamic polarization and electrochemical impedance spectroscopy studies, the electrode was allowed to corrode freely and its open circuit potential (OCP) was recorded as a function of time up to 20 min, which was sufficient to attain a stable state. The polarization measurements were carried out from a potential range of -200 mV to +200 mV with respect to open circuit potential in absence and presence of inhibitor at scan rate of 1 mV/sec. Electrode potentials were measured with respect to SCE. The polarization studies were done immediately after the EIS studies on the same electrode without any further surface treatment. The AC impedance was performed in the

Table 1 — CR and IE (%) of HBH extract in 1M Sulphuric acid at different concentrations and different immersion periods.

S.No.	Inhibitor conc. (% v/v)	1 h		3 h		5 h		7 h		24 h	
		CR (mpy)	IE (%)	CR (mpy)	IE (%)	CR (mpy)	IE (%)	CR (mpy)	IE (%)	CR (mpy)	IE (%)
1.	Blank	0.8518	**	0.6293	**	0.5940	**	0.668	**	0.618	**
2.	0.006	0.1612	81.08	0.0921	85.37	0.0783	86.82	0.069	89.66	0.098	84.16
3.	0.012	0.1151	86.49	0.0614	90.24	0.0599	89.92	0.066	90.15	0.059	90.53
4.	0.018	0.0921	89.19	0.0537	91.46	0.0506	91.47	0.039	94.09	0.052	91.61
5.	0.024	0.0691	91.89	0.0460	92.68	0.0414	93.02	0.036	94.58	0.045	92.70
6.	0.030	0.0460	94.59	0.0307	95.12	0.0368	93.80	0.033	95.07	0.034	94.57
7.	0.040	0.0460	94.59	0.0153	97.56	0.0368	93.80	0.030	95.57	0.031	95.03

frequency range from 20 kHz to 200 Hz with single amplitude of 10 mV. Fresh solution and fresh mild steel samples were used after each sweep.

Surface analysis

The surface morphology of mild steel specimen immersed in 1M H_2SO_4 in the absence and presence of hair sample extract at room temperature for 3 h was studied using a Scanning Electron Microscope (SEM).

Results and Discussion

Effect of inhibitor concentration and temperature

The corrosion rates and inhibition efficiencies of mild steel with different concentrations of human hair extract in 1M H_2SO_4 was determined for different immersion periods (1, 3, 5, 7 and 24 h) at room temperature are given in Table 1. The corrosion rate (CR) and percentage inhibition efficiency (IE %) were calculated using the equation 1 and 2. From the weight loss data, plot of Inhibition efficiency Vs Concentration and Corrosion rate Vs time were made and shown in Figs. 1 and 2. It was observed from the plots that the IE (%) increases with increase in hair sample extract. Maximum inhibition efficiency of 97.56% was obtained at 0.04% v/v hair sample extract concentration at room temperature for 3 h immersion.

It is observed from the plot of CR Vs immersion time (Fig. 2) shows that at room temperature, CR of mild steel decreased on increasing hair sample extract concentration. This behavior could be attributed to the increase in adsorption of the amino acids present in the HBH extract at the metal solution interface on increasing its concentration.

Effect of temperature

The influence of temperature on the corrosion of mild steel in 1M H_2SO_4 without and with the presence of human hair extract was studied using weight loss method in the temperature range of 303 K - 333 K with 1 h immersion. Table 2 shows the values of corrosion rate and inhibition efficiency obtained from weight loss measurements at various temperatures mentioned below. The variation of CR of mild steel as a function of different concentrations of human hair extract is shown in Fig. 3. The data in Table 2 shows the CR and I.E (%) values with and without inhibitor at different temperatures for 1 h immersion time. It is seen that the corrosion rate increases with increase in temperature in both uninhibited and inhibited solutions. This can be attributed to the fact that the rate of corrosion reaction increases with increase in temperature. But the rate of corrosion is less in inhibited solutions compared with uninhibited solutions. Inhibition Efficiency decreases after 323 K this may be due to the fact that as the temperature

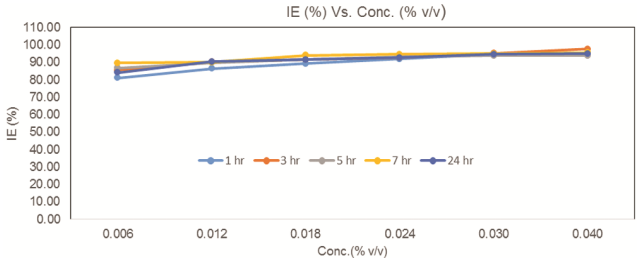

Fig. 1— Variation of IE (%) with concentration of HBH extract in 1M H_2SO_4 medium

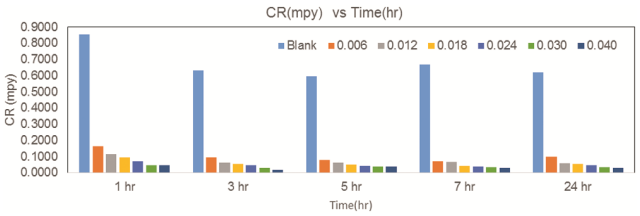

Fig. 2 — Effect of Corrosion rate with time in presence and absence of HBH extract in 1M H_2SO_4 medium

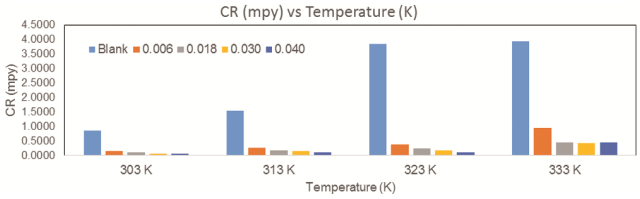

Fig. 3 — Variation of corrosion rate with temperature at different concentrations of HBH extract in 1M H_2SO_4 medium

Table 2 — CR and IE (%) of HBH extract in 1 M Sulphuric acid at different concentrations and different temperature

S.No.	Inhibitor Conc.(% v/v)	303K		313K		323K		333K	
		CR (mpy)	I.E. (%)	CR (mpy)	I.E. (%)	CR (mpy)	I.E. (%)	CR (mpy)	I.E. (%)
1.	Blank	0.8518	**	1.5425	**	3.8447	**	3.9368	**
2.	0.006	0.1612	81.08	0.2763	82.09	0.3914	85.088	0.9439	80.751
3.	0.018	0.1151	86.49	0.1842	88.06	0.2532	90.351	0.4604	90.610
4.	0.030	0.0691	91.89	0.1612	89.55	0.1842	92.982	0.4374	91.080
5.	0.040	0.0691	91.89	0.1151	92.54	0.1151	95.614	0.4604	90.610

increases the number of adsorbed molecules decreases, leading to a decrease in the inhibition efficiency. A decrease in inhibition efficiency with the increase in temperature in this case may also be due to weakening of physical adsorption[19].

Adsorption isotherms

The values of surface coverage θ for different concentrations of the hair extract at different temperatures have been used to explain the best isotherm to determine the adsorption process. Various adsorption isotherms were tested and among them Temkin adsorption fits best for the experimental data values [20].

$$Log\ \theta/C = \log K - g\,\theta \quad \ldots (3)$$

where C is the concentration of the inhibitor

K adsorptive equilibrium constant

θ is the surface coverage

G is the adsorbate parameter

The plot of log C against θ yields lines as shown in Fig. 4. It is evident that the R^2 value is almost equal to 1 which indicating the adsorption of the inhibitor on mild steel surface follows Temkin adsorption isotherm.

Activation parameters

The dependence of corrosion rate on temperature can be expressed by Arrhenius equation

$$Icorr = A\exp(\frac{-Ea}{RT}) \quad \ldots (4)$$

where I_{corr} is the reaction rate, A is a constant, E_a is the activation energy of the reaction, T absolute temperature and R is the universal gas constant. The Arrhenius plot is given in Fig. 5. The related $E_a$values were given in Table 3. The activation energy were found to higher for inhibited acid solutions than uninhibitied acid solutions. These results indicate the adsorption is by physical adsorption mechanism[21].

Figure 6 showed the plot of log (CR/T) verses 1/T for the corrosion of mild steel in the absence and presence of inhibitors in 1M H_2SO_4. In order to calculate the activation parameters like ΔH_{ads} and ΔS_{ads} for the corrosion process, transition state equation (5) was used

$$CR = \frac{RT}{Nh}\exp\frac{\Delta Sads}{R}\,exp\frac{-\Delta Hads}{RT} \quad \ldots (5)$$

where h is Planck's constant, N is Avogadro's number, R is the universal gas constant, T is the absolute temperature, ΔS is the entropy of activation, and ΔH is the enthalpy of activation. Plot of log (CR/T) versus $1/T$ gave straight lines with slope $(-\Delta H_{ads}/2.303R)$ and intercept [log (R/Nh) + $(\Delta S_{ads}/2.303R)$], from which ΔH and ΔS were calculated and listed in Table 3. ΔH_{ads} and ΔS_{ads} were found to be negative in all the concentrations of the hair extract showing that the reactions are exothermic[22].

Free energy of adsorption (ΔG_{ads})

The values of ΔG_{ads} are given in Table 4. The negative values of ΔG_{ads} suggest that the strong

Table 3 — Activation parameters of 1 M H_2SO_4 on mild steel surface in both uninhibited and inhibited solutions.

S.No.	Conc % (v/v)	E_a (KJ mol^{-1})	ΔH_{ads} (KJ mol^{-1})	ΔS_{ads} (J mol^{-1})
1.	Blank	45.78082	-4.3158	-230.91
2.	0.006	46.98709	-4.4383	-188.53
3.	0.018	37.22204	-3.4618	-247.47
4.	0.030	47.15941	-4.4536	-252.06
5.	0.040	47.33174	-4.4709	-253.67

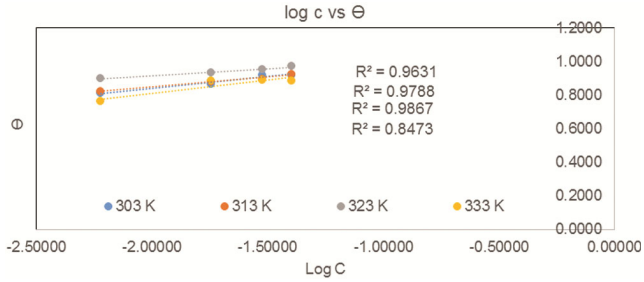

Fig. 4 — Tempkin plot

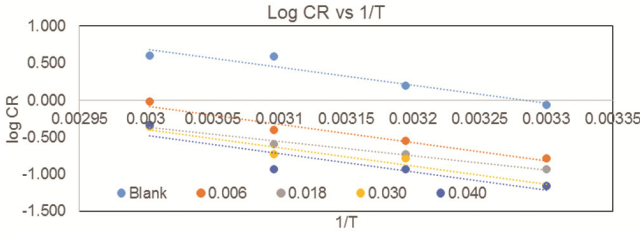

Fig. 5 — Arrhenius plot

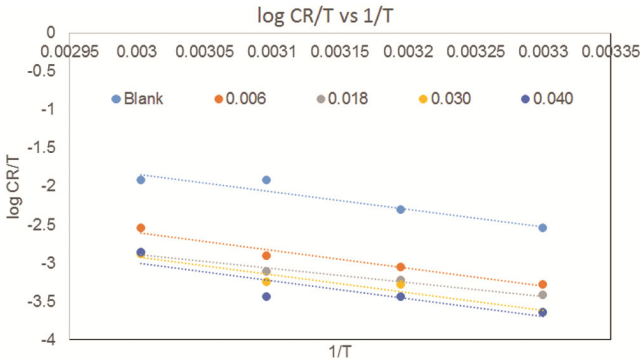

Fig. 6 — Transition state plot

Table 4 — Values of free energy changes for the corrosion on mild steel in 1 M H_2SO_4 in the presence of different concentrations of inhibitor at different temperatures.

S.No.	Inhibitor conc. (% v/v)	-ΔG_{ads} (KJ/mole)			
		303 K	313 K	323 K	333 K
1.	0.006	26.652	27.706	30.351	28.456
2.	0.018	24.894	26.085	28.674	27.816
3.	0.030	25.047	25.146	28.209	26.562
4.	0.040	24.322	25.359	28.749	25.605

interaction of the inhibitor molecules onto the mild steel surface. The values of ΔGads is in the order of -20 kJ/mole indicating physical adsorption process. The negative sign also indicates that the adsorption of the inhibitor constituents on the metal surface is a spontaneous process[23].

Potentiodynamic polarization measurements

Polarisation study has been used to study the formation of protective film on the metal surface. The changes observed in the polarization curves are usually used as criteria to classify inhibitors as cathodic, anodic or mixed type. The potentiodynamic polarization curves of mild steel immersed in acidic medium in the absence and presence of inhibitors are shown in Fig. 7. The results in Fig. 7 suggested that the inhibitor affects both anodic and cathodic corrosion processes; hence, it reveals properties of a mixed type inhibitor. The addition of inhibitor changes the values of E_{corr} to the cathodic direction, so the human hair extract ca be considered as a slightly cathodic inhibitor. The electrochemical parameters, Corrosion current density (I_{corr}), anodic ($ß_a$) and cathodic ($ß_c$) Tafel constants and Polarization resistance (R_p) are shown in Table 4. The polarization resistance (R_p) from Tafel extrapolation method was calculated using the Stern-Geary Equation (6). The inhibition efficiency was calculated from the formula (7) and (8)

$$Icorr = \frac{\beta a \beta c}{2.303\,(\beta a + \beta c)} \frac{1}{Rp} \quad \ldots (6)$$

$$IE(\%) = \frac{Icorr(Blank) - Icorr(inh)}{Icorr(Blank)} \times 100 \quad \ldots (7)$$

$$IE(\%) = \frac{Rp\,(inh) - Rp(Blank)}{Rp(inh)} \times 100 \quad \ldots (8)$$

It is clear from the data in the Table 4 clearly shows that the I_{corr} values gradually decreases with increase in the concentration if the inhibitor, with respect to the blank. It is observed that the current density (I_{corr}) values decreased and the polarization resistance (R_p) values increased in the presence of inhibitor with various concentrations as expected.

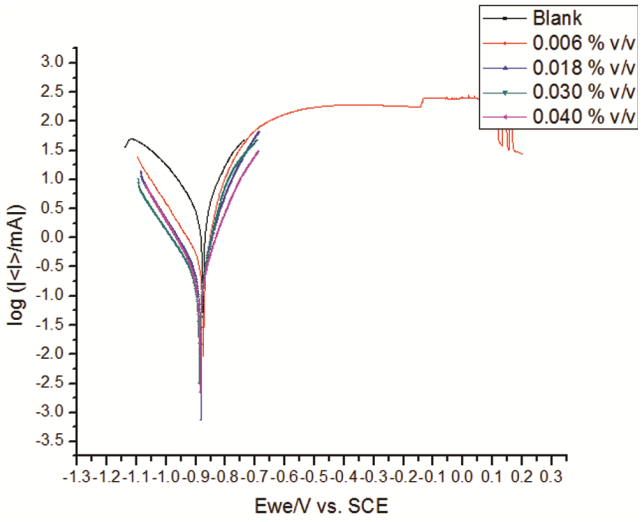

Fig. 7 — Potentiodynamic polarization curves for mild steel in 1M H_2SO_4 containing different concentrations of the inhibitor

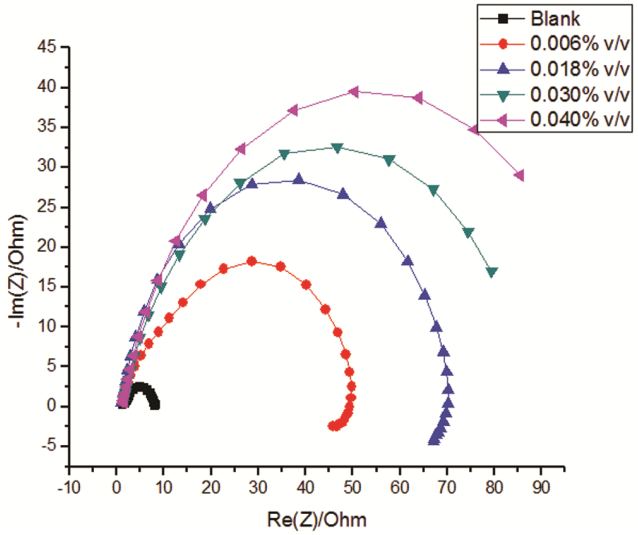

Fig. 8 — Nyquist plot of mild steel in 1 M H_2SO_4 in the absence and presence of various concentration of inhibitor

Because of the inverse relationship between I_{corr} and R_p, with increasing concentration of the inhibitor, it can be assumed that the adsorption of the inhibitor molecules on metal surface creates a physical barrier for the mass and charge transfer, providing a high degree of protection to the metals surface[24].

Electrochemical impedance measurements.

The corrosion behavior of mild steel on acidic solution in the absence and presence of inhibitors was investigated by the electrochemical impedance spectroscopy method at room temperature. Figure 8 shows the results of EIS experiments in Nyquist

representation. It is clearly evident from Nyquist plots that they are significantly changed with the addition of inhibitors. As a result, the following electrical parameters were determined and it is shown in Table 5.

The Nyquist plots are analysed with EC-Lab software. This program provides good information about circuit. In order to acquire more quantitative information about the adsorption mechanism, electrical analysis of the experimental data was performed. An equivalent electrical circuit that fitted the best impedance data was introduced in Fig. 9

From the impedance data, we notice an increase in charge transfer and decrease in double layer capacitance with increasing inhibitor concentration indicate that the hair sample extract inhibits the corrosion rate of mild steel by an adsorption mechanism. The decrease in the C_{dl} value suggests that the inhibitor molecules function by adsorption at the metal/solution interface as they displace the water molecules and other ions originally adsorbed on the surface leading to the formation of a protective adsorption layer on the electrode surface which increases the thickness of the electrical double layer[25]. The thickness of this protective layer is related to C_{dl} in accordance with Helmholtz model given by the equation:

$$\boldsymbol{Cdl} = \frac{\varepsilon\varepsilon \boldsymbol{oA}}{\boldsymbol{d}} \quad \ldots (9)$$

where Σ is the dielectric constant of the medium and Σ_o is the permittivity of free space ($8.854*10^{-14}$ F/cm) and A is the effective surface area of the electrode. From the equation, it is clear that as the thickness of the protective layer that is, the film formed by inhibitor molecules, increases the C_{dl} should decrease. Here the C_{dl} value was found to be highest for uninhibited solution and as the inhibitor concentration increases the C_{dl} value decreases.

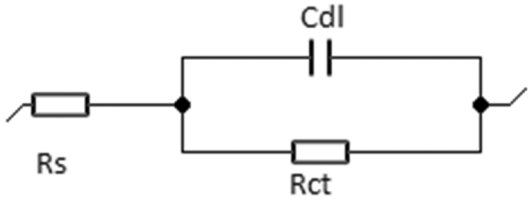

Fig. 9 — Equivalent electrical circuit

The charge transfer resistance R_{ct} values are calculated from the difference in impedance at low and high frequencies. The R_{ct} value is a measure of electron transfer across the mild steel surface and it is inversely proportional to the corrosion rate (Table 6). The inhibitor efficiency of inhibitors for the corrosion of mild steel in 1M medium is calculated using R_{ct} values as follows:

$$\boldsymbol{I.E}(\%) = \boldsymbol{Rct(i)} - \boldsymbol{Rct(o)} * \frac{\boldsymbol{100}}{\boldsymbol{Rct(i)}} \quad \ldots (10)$$

where $R_{ct(o)}$ and $R_{ct(i)}$ are the charge transfer resistance values in absence and presence of inhibitor respectively. The R_{ct} value was found to be highest for the uninhibited solution. The inhibitor efficiencies obtained from R_{ct} are in good agreement with those obtained from potentiodynamic polarization and weight loss measurements[26].

Surface morphology

Surface analysis by FTIR

FTIR instrument is used to study the type of bonding for inhibitors adsorbed on the metal surface. FTIR spectra were used to analyze the inhibitor film formed on mild steel surface. FTIR spectrum of crude Black hair sample extract and FTIR spectrum of mild steel in 1M H_2SO_4 containing inhibitor. It was found that NH_2 or OH stretch at 3348.42 cm-1 was shifted to 3197.98 cm^{-1}, the C=C stretch at 1635.64 cm^{-1} was shifted to 1651.07 cm^{-1} indicating that these groups gets adsorbed on the metal surface.

Table 6 — Impedance parameters for mild steel in 1M H_2SO_4 in the absence and presence of various concentrations of inhibitor

S. No.	Conc. (%v/v)	R_{ct} Ohm	$C_{dl}F$	IE%	
				C_{dl}	R_{ct}
1.	Blank	4.346	0.359 * 10^-3	**	**
2.	0.006	39.94	43.83 * 10^-6	87.79109	89.12
3.	0.018	63.249	29.63 * 10^-6	91.74652	93.13
4.	0.03	62.691	22.38 * 10^-6	93.76602	93.07
5.	0.04	73.012	33.22 * 10^-6	90.74652	94.05

Table 5 — Kinetic parameters of mild steel in 1M H_2SO_4 containing different concentrations of the inhibitor

S.No.	Inhibitor conc. (% v/v)	$-E_{corr}$(mV)	I_{corr}(μA)	$ß_a$ (mV/dec)	$ß_c$(mV/dec)	R_p (Ω cm^2)	IE (%)	
							I_{corr}	R_p
1.	Blank	874.836	3727.789	112.5	189.3	82.19	**	**
2.	0.006	876.148	392.722	49	125.3	389.47	89.47	78.90
3.	0.018	883.449	228.162	57.7	120.6	742.74	93.88	88.93
4.	0.03	887.531	188.546	53.2	131.2	871.71	94.94	90.57
5.	0.04	886.348	143.809	65.6	104.8	1218.19	96.14	93.25

Surface analysis by Scanning electron microscope (SEM) – Energy Dispersive X- Ray Spectroscopy

Fig. 10 (a) and (b) presents image of the corrosion morphology of mild steel immersed in 1M HNO_3 and 1M HNO_3 with 0.040 % v/v extract after 2 h of immersion. The sulphuric acid aggressively corroded the sample in Fig. 10(a) causing a rougher surface, whereas the sample shown in Fig. 10(b) with smoother surface is noticed. The mild steel with smooth surface was attributed to the fact that the hair sample extract to form an adsorbed film on the surface of the mild steel, which is absent in mild steel[27].

Table 7 shows the percentage atomic content of Fe, C, O of the polished, uninhibited and inhibited mild steel surface determined by EDX. The percentage atomic content of Fe for plain polished mild steel, mild steel dipped in 1M H_2SO_4 medium and mild steel dipped in acid medium containing optimum concentration (0.40%) of the inhibitor is 6.61 , 33.28 and 46.96 % respectively. The decrease in Fe values confirms the formation of inhibitory film formed on the mild steel surface. The percentage atomic content of oxygen is increases for mild steel immersed in 1M H_2SO_4 corrosive acid medium, this confirms the attack of oxide ion on the mild steel surface. But on addition of inhibitor to the acid medium prevents the attack of oxide ion on the mild steel surface by the formation of passive layer, which is seen from the low O percentage. The Electron Dispersive X- ray spectra of Polished, uninhibited and inhibited mild steel specimens were shown in Fig. 11 (a), (b) and (c) shows the EDX[28-29].

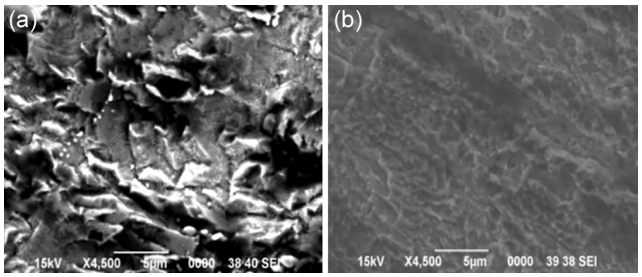

Fig. 10 — (a) and 11(b) corrosion surface morphology

Table 7 — Percentage atomic content of elements obtained from EDX spectra

S.No.		Fe %	O %	C %
1	Polished mild steel	63.61	17.18	18.55
2	Mild steel without inhibitor	33.28	19.00	46.40
3	Mild steel with inhibitor	46.96	14.00	37.63

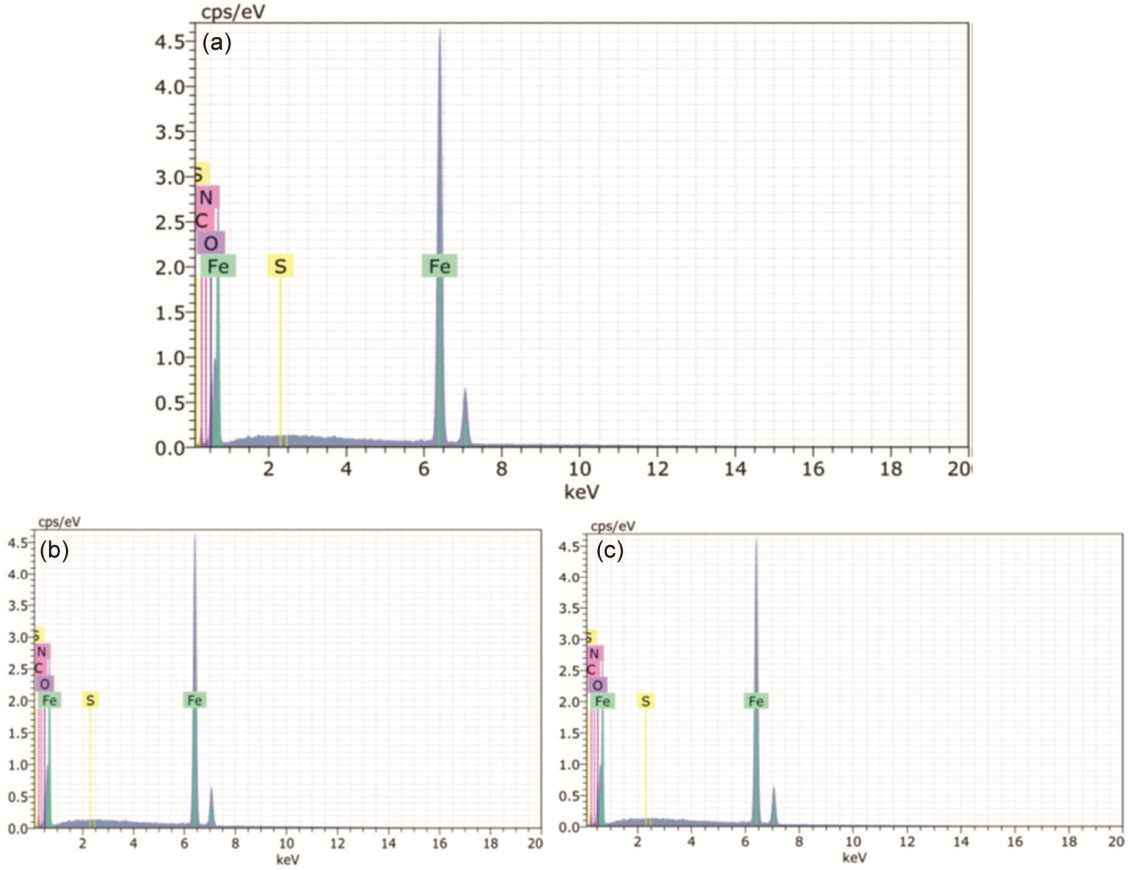

Fig. 11 — (a) Fresh mild steel, (b) Mild steel without inhibitor , & (c) Mild steel with inhibitor

Conclusion

The alkali extract of Human hair sample acts as a very good corrosion inhibitor for mild steel in 1 M H_2SO_4 medium. The inhibitor efficiency increases with increase in concentration and reaches an optimum time of 7 h in room temperature and optimum temperature of 323 K for 1 h immersion time. The free energy change ΔG carries a negative value around -20 KJ/mole indicates that the adsorption process is spontaneous and follows physical adsorption respectively. From the polarization studies it is evident that all the plant extract acts as mixed-type corrosion inhibitors. The increase in R_{ct} value and decrease in C_{dl} values confirm the formation of a protective layer over the mild steel surface, which was supported by SEM images.

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