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Research Article

EVALUATION OF SOLANUM NIGRUM LEAF EXTRACT AS A CORROSION INHIBITOR AND REDUCTANT FOR THE GREEN SYNTHESIS OF GOLD NANOPARTICLES

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ABSTRACT

Eco-friendly green methodologies have attracted the attention in every field of research owing to environmental pollution and the alarming development of global warming. In search for eco-friendly materials for the synthesis of metal nanoparticles and corrosion inhibition, the leaf-extract of Solanum nigrum have identified, as it displays remarkable antioxidant property. The leaf-extract was investigated by weight-loss method and potentio-dynamic polarization technique in carbon steel to study the corrosion inhibition. Surface and protective film analysis was carried out using scanning electron microscopy (SEM). The antioxidant property of the leaf-extract was exploited for synthesis of gold nanoparticles (GNPs). Excellent results were obtained when the leaf-extract was used to reduce AuCl4. The formation of GNPs was rapid and within a few hours AuCl4 was reduced into fine GNPs as evidenced by the appearance of deep ruby red colloidal dispersion. The UV–visible spectral analysis revealed the reduction of AuCl4 and showed a peak at ~545 nm originating from the surface plasmon resonance of GNPs. The GNPs were characterized using SEM with energy-dispersive X-ray spectroscopy, powder X-ray diffraction and zeta potential analysis. The average size, geometrical shape and the zeta potential were discussed.

Keywords: Leaf extract, Solanum nigrum, corrosion inhibition, gold nanoparticles, green synthesis.

INTRODUCTION

Corrosion is defined as the deterioration of a metal due to its interaction with the environment. Due to corrosion many useful properties of metals such as malleability, ductility and electrical conductivity are compromised. Nowadays, carbon steel has become an important part of our life due to its extensive applications in household appliances as well as in automotive, petroleum, construction, marine and chemical industries. The use of corrosion inhibitors is one of the most practical methods for protection against corrosion. Synthetic organic compounds are widely used as corrosion inhibitors for the prevention of corrosion of many metals and alloys in various aggressive environments. Because of their hazardous nature, researchers focus their attention on developing cheap, non-toxic, biodegradable and environment friendly natural products of plant origin as corrosion inhibitors [1–5].

Some of the remarkable properties of GNPs such as intense plasmon resonance, electrical, magnetic, chemical, anti-bacterial, anti-HIV, anti-angiogenic, anti-malarial and anti-arthritic properties as well as thermal conductivity, catalytic activity and biostability of GNPs have found tremendous interest over last few decades [6, 7]. Owing to the antioxidant property of the plant extracts, they are currently explored for the chemical reduction of metal salts and oxides. Consequently, some plant extracts have been shown to be useful in the preparation of metal nanoparticles. Currently, biosynthesis of GNPs has gained much attention and emerged as one of the active areas of research in the field of nanotechnology. Solanum nigrum, a medicinal plant found throughout Tamil Nadu, belongs to the family Solanaceae. The leaves of S. nigrum are widely used to cure mouth ulcer [8]. In the present work, we have evaluated its

leaf extract as a green corrosion inhibitor for carbon steel [9] and as a reductant for the synthesis of gold nanoparticles (GNPs) [10].

The aim of the present study was to investigate S. nigrum as corrosion inhibitor for carbon steel in ground water collected from Holy Cross College (Tiruchirappalli, Tamil Nadu, India) and as a reductant for the synthesis of GNPs. The leaf-extract was investigated by weight-loss method and potentio-dynamic polarization technique in carbon steel. Surface and protective film analysis was carried out using scanning electron microscopy (SEM). The GNPs were characterized using SEM with energy-dispersive X-ray spectroscopy, powder X-ray diffraction and zeta potential analysis. The average size, geometrical shape and the zeta potential were discussed.

Material and methods

Preparation of specimen

Carbon steel specimen (0.026% S, 0.06% P, 0.4% Mn and 0.1% C and rest Fe) of the dimensions 1.0 × 4.0 × 0.2 cm were polished to a mirror finish and degreased with acetone and used for the weight-loss method and surface examination studies.

Weight-loss study

Carbon steel specimens in triplicate were immersed in 100 ml of the ground water containing various concentrations of the inhibitor in the presence and absence of inhibitor for 1 day [11]. The weights of the specimens before and after immersion were determined using a ACCULAB Electronic top loading balance, with readability/sensitivity of 0.1 mg in 210 g range Then the inhibition efficiency was calculated using the formula

$$IE = 100\ [1 - (W2/W1)]\ \%$$

where W1 and W2 are corrosion rate in the absence and presence of inhibitor respectively.

The corrosion rate (CR) was calculated by using the formula

CR = [(weight loss in mg)/(area of the specimen in dm2×immersion period in days)]

Potentio-dynamic polarization study

Potentio-dynamic polarization studies were carried out using CHI electrochemical impedance analyzer, model 660A. A three electrode cell assembly was used. The working electrode was a rectangular specimen of carbon steel with one face of the electrode (1 cm2 area) exposed and the rest was shielded with red lacquer. A saturated calomel electrode (SCE) was used as the reference electrode and a rectangular platinum foil was used as the counter electrode.

The working and platinum electrode were immersed in medium in the absence and presence of inhibitor. SCE was connected with the test solution through a salt bridge. Potential (E) versus log current (I) plots were then recorded. Corrosion potential (Ecorr) and Tafel slopes ba and bc were determined from E versus log I plots [12].

AC impedance measurements

A CHI electrochemical analyzer (model 660A) was used for AC impedance measurement. A time interval of 5–10 min was given for the system to attain its open-circuit potential. The real part (Z') and imaginary part (Z") of the cell impedance were measured in ohms at various frequencies (f). The values of the charge transfer resistance Rt and the double layer capacitance Cdl were calculated.

Cdl values were calculated using the following relationship [13]

$$C_{dl} = \frac{1}{2 \times 3.14 \times Rt \times f\ max}$$

Scanning electron microscopy

The carbon steel specimens immersed in presence and absence of inhibitor for 1 day were taken out, rinsed with double distilled water, dried and subjected to the surface examination. The surface morphology measurements of the carbon steel surface were carried out by SEM using vega3 tescan (TESCAN ORSAY HOLDING, Czech Republic).

Preparation of leaf extract

Fresh samples of S. nigrum were collected from the sub-urban areas of Tiruchirappalli. The leaves were washed thoroughly 2–3 times with running tap water and the leaves are air dried under shade. After complete shade drying, the leaves were grinded in the mixer, the powder was kept in small plastic bags with proper labeling. About 50 g of the powder was soaked in a 250 ml of methanol under cold percolation method. At regular intervals of time the extract was filtered and distillation was carried out to collect the crude extract. The extract was stored in an amper bottle and refrigerated [14].

Green synthesis of GNPs

4.5 ml of S. nigrum methanolic extract was mixed with 50 ml of 1mM chloroauric acid and 5 ml of 1% PVA in 250 ml conical flask. The reaction mixture was kept aside in a micro oven for 60 seconds. The color change from yellow to deep ruby-red indicated the formation of GNPs. The reaction mixture was centrifuged at 14,000 rpm for 15 min and the supernatant was discarded. The GNPs obtained as a pellet was dispersed in deionised water for further studies [15].

Characterization of GNPs

UV–visible spectroscopy

UV–visible spectrophotometer is the one of the important techniques for analysis of synthesized GNPs. After the synthesis, the pure GNPs were characterized by UV–visible absorption spectrophotometer (SPECORD 200 plus, Analytikjena, Germany). The color change in reaction mixture (metal ion solution + plant extract) was recorded through visual observation. Synthesized GNPs was confirmed by sampling the absorption maxima was scanned by UV–visible spectrophotometer at the wavelength of 400–800 nm.

SEM with EDAX spectroscopy

The mean particle size and surface morphology of green-GNPs were measured by field emission electron microscopy (FESEM) (Hitachi, SU6600 Scanning Electron Microscopy, Japan) coupled with energy-dispersive spectroscopy (Horiba, 8121-H, Japan). Dry powder of green-GNPs was loaded on the stub using double sided adhesive conductive carbon tapes and analyzed.

Zeta potential measurement

The zeta potential of green-GNPs was measured on a Zeta seizer (3000SH, Malvern Instruments, Ltd., UK). Green-GNPs were suspended in double distilled water and placed in an electrophoretic cell (3 ml) and the electrophoretic mobility was measured at room temperature. The zeta potential was calculated as described by Clogston and Patriusinga built-in software.

X-ray diffraction

The powder XRD studies were carried out on SEIFERT, JSO-DEBYEFLEX 2002, Germany X-ray diffractometer, operating at a voltage of 40 kV and a current of 20 mA. The scanning rate was maintained at 1.6 min^{-1} over a 2Θ range of 10–70 employing the reflection mode for scanning.

Results and discussion

The qualitative phytochemical screening of S. nigrum leaves [16] was done and the results are shown in Table 1.

Table 1 Qualitative phytochemical screening of S. nigrum L.

Name of the phytochemicals	Presence/absence
Alkaloid	+
Saponin	+
Tannin	+
Flavonoids	+
Protein	+

The physicochemical parameters of ground water are given in Table 2.

Table 2 Physico-chemical parameter of ground water

Parameters	Value
pH	7.4
Total hardness as $CaCO_3$	672 mg/l
Calcium	134 mg/l
Magnesium	81 mg/l
Nitrate	8 mg/l
Chloride	452 mg/l
Fluoride	0.4 mg/l
Sulphate	19 mg/l
Total dissolved solids	1645 mg/cm
Electrical conductivity	2350 mho/cm
Total alkalinity as $CaCO_3$	424 mg/l

Analysis of results of weight-loss study

The weight loss studies were done in ground water in the presence and absence of various concentration of the leaves extract ranging from 10 ppm to 700 ppm. Using the weight-loss data, the corrosion rate, inhibition efficiency and the optimum concentration of the extract have been calculated. From Table 3, it was found that with the addition of the leaves extract, the weight-loss of the carbon steel decreased, and the corrosion rate also decreased. The optimum concentration of S. nigrum was found to be 500 ppm with maximum inhibition efficiency of 86.36%.

Table 3 Corrosion rates of carbon steel in ground water by weight-loss method

S. No.	Concentration (ppm)	Corrosion rate (mdd)	Percentage inhibition
1	Blank	20.958	–
2	10	5.9880	71.42
3	50	5.2395	75
4	100	4.2857	79.55
5	200	3.5714	82.95
6	300	3.5714	82.95
7	400	3.7425	82.14
8	500	2.8571	86.36
9	600	4.3859	79.07
10	700	6.578	68.613

Table 4 Corrosion rates of carbon steel in ground water due to the effect of duration of immersion on the IE of leaf extract

S. No.	Time period (days)	Concentration (ppm)	Corrosion rate (mdd)	Percentage inhibition efficiency
1	1	500	2.8571	86.36
2	3	500	4.1916	80
3	5	500	5.3442	74.5
4	7	500	6.3083	69.9
5	8	500	7.9760	61.9

Table 5 Corrosion rates of carbon steel in ground water due to the effect of pH on the IE of leaf extract

S. No.	Concentration (ppm)	pH	Corrosion rate (mdd)	Percentage Inhibition efficiency
1	0	3	24.8538	–
2	500	3	20.8333	16.17
3	0	5	22.6608	–
4	500	5	18.2748	19.35
5	0	7	20.958	–
6	500	7	2.8571	86.36
7	0	9	10.000	–
8	500	9	3.570	64.3
9	0	11	14.6198	–
10	500	11	8.0409	45

Table 6 The corrosion parameters for carbon steel in ground water with 500 ppm of *S. nigrum* from polarization studies

S. No.	Environment	E_{corr} (mV Vs SCE)	b_a (mV)	b_c (mV)	I_{corr} (A)
1	Blank	-575.54	138.30	212.82	28.75
2	Inhibitor system	-517.78	122.13	258.02	24.77

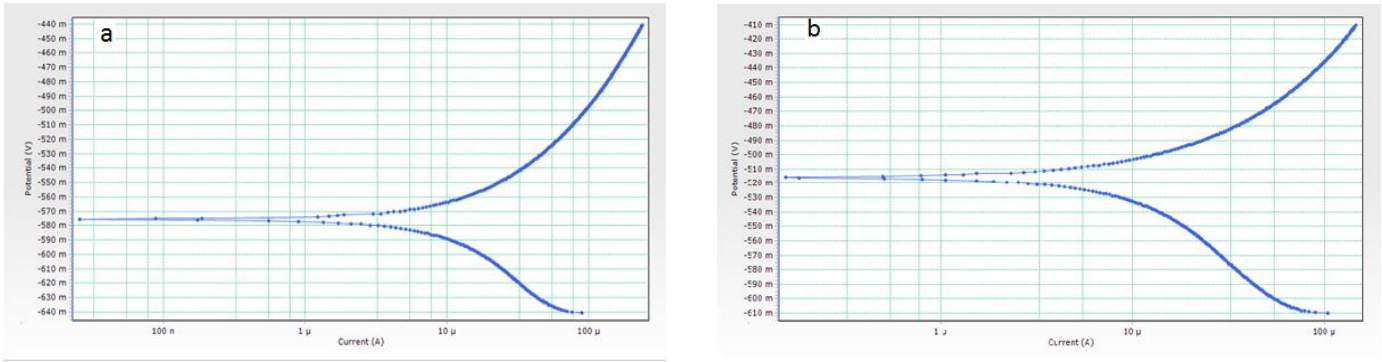

Fig. 1 Potentiodynamic polarization curves for carbon steel in ground water (a) and ground water + *S. nigrum* (b) Electrochemical impedance spectroscopy.

Table 7 The corrosion parameters for carbon steel in ground water with 500 ppm of *S. nigrum* from impedance measurements

S. No.	Environment	R_{ct} (ohm)	C_{dl} (F)
1	Blank	125	12.13×10^{-6}
2	Inhibitor system (500ppm)	301	2.67×10^{-6}

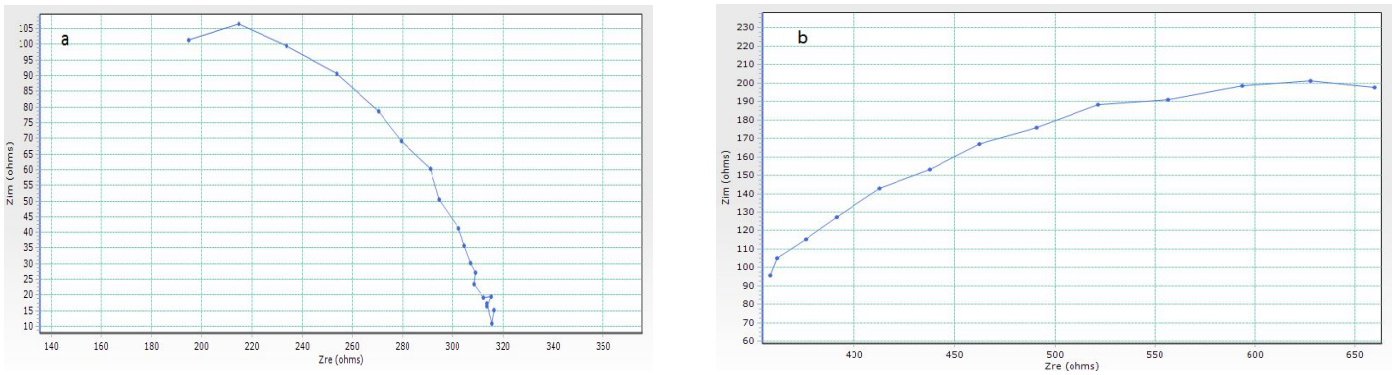

Fig. 2 AC impedance of carbon steel in (a) ground water (b) ground water + *S. nigrum*.

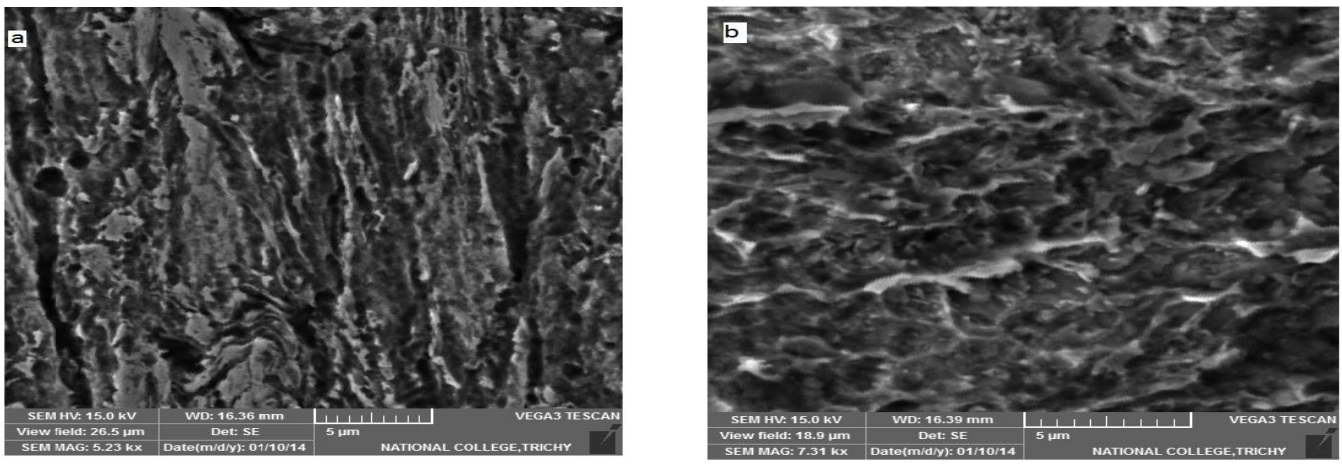

Fig. 3 SEM micrographs of carbon steel surface (magnification =1000×): Carbon steel immersed in ground water (a); Carbon steel immersed in ground water containing 500 ppm of *S. nigrum* extract (b)

Effect of duration of immersion on the IE of leaf extract

The effect of duration of immersion was studied for some period of days (Table 4). The results also show that the corrosion rate increased with increase in time. It was observed that as the duration of immersion increases, the inhibition efficiency decreases. This is due to the fact that as the duration of immersion increases, the protective film formed on the metal surface is not able to withstand the attack of Cl-. The film is broken and hence the IE decreases. Similar observation was made with Fe2+-curcumin system [17], Fe2+-henna leaves system [18]. The decrease in IE with time may be attributed to an increase in the iron ions concentration.

Effect of pH on the IE of leaf extract

It is seen from (Table 5) that at pH 7, the S. nigrum extract has 86.36 % IE. When pH is lowered to 5 by addition of dilute hydrochloric acid, the IE decreased to 19.35%. This is due to the fact that when the acid is added the protective film is broken by the aggressive H+ ion present in the acid. When the pH is increased to 9 by addition of diluted sodium hydroxide solution, the IE increased to 64.3% (when compared to an acidic medium). This is due to the fact that the phenolic-OH groups would have been ionized to phenolate anion, –O–Na+. This helped anchoring of phenolic O- on the anodic sites of the metal surface effectively and hence IE increased at higher pH values. Similar observation has been observed in the case of corrosion inhibition by curcumin extract and by henna extract: as the value of pH is increased the corrosion inhibition efficiency also increased [18, 19]. However this 64.3% IE in basic medium (pH 9) is lower than the IE of 86.36 % in neutral medium.

Potentio-dynamic polarization study

Polarisation study has been used to detect the formation of protective film on the metal surface. When a protective film is formed on the metal surface, the corrosion current Icorr decreases when carbon steel is immersed in ground water the corrosion potential is –575.54 mV versus SCE. The corrosion potential shift to –517.78 mV versus SCE by adding 500 ppm of S. nigrum extract. The cathodic slope is found to change from 212.82 to 258.02 mV/decade and the anodic slope from 138.309 to 122.136 mV/decade [20]. This shows that the inhibitor function as anodic inhibitor controlling both anodic and cathodic process, but predominantly anodic process [21].

Electrochemical impedance spectroscopy

AC impedance spectra have been used to detect the formation of a film on the metal surface. If a protective film is formed, the charge transfer resistance increases and the double layer capacitance value decreases [22, 23]. The AC impedance spectra of carbon steel immersed in various solutions are shown in Fig. 2 (Nyquist plot). The AC impedance parameters, namely, charge transfer resistance (Rct) and double layer capacitance (Cdl) are given in Table 7. When carbon steel is immersed in ground water, Rct the value is 125 ohm and the Cdl value is 12.13x10-6 F. When 500 ppm of S.nigrun is added, the Rct value increases from 125 ohm to 301 ohm and Cdl decreases from 12.13x10-6 to 2.67x10-6. This suggests that a protective film is formed on the surface of the metal is a diffusion controlled process [24].

Scanning electron microscopy

Scanning electron microscopy (SEM) provides a pictorial representation of the surface. To understand the nature of the surface film in the absence and presence of inhibitors and the extent of corrosion of carbon steel, the SEM micrographs of the surface are examined [25, 26]. The SEM micrograph (1,000×) of carbon steel specimen immersed in the ground water for 1 day is shown in Fig. 3a, 3b respectively. SEM micrographs of carbon steel surface (magnification 1,000×) (a) carbon steel immersed in ground water, (b) carbon steel immersed in ground water containing 500 ppm of S. nigrum extract.

The SEM micrograph of carbon steel surface immersed in ground water is shown in Fig. 3a. This shows the roughness of the metal surface which indicates the corrosion of carbon steel in ground water. Figure 3b indicates that in the presence of 500 ppm leaves extract in ground water, the surface coverage increases which in turn results in the formation of insoluble complex on the metal surface. In the presence of leaves extract, the surface is covered by a thin layer of inhibitors which effectively control the dissolution of carbon steel [27].

Synthesis of gold nanoparticles

The qualitative phytochemical screening of S. nigrum leaves [16] was done and the results shows the presence of proteins, alkaloids, saponins, tannins, and flavonoids, which could be

explored for the preparation of different metal nanoparticles [28]. GNPs of different sizes have been obtained using plant extracts with various concentrations of added salt [29]. We envisioned that S. nigrum is rich in flavonoids with anti-oxidant property, would serve as a reducing agent in the preparation of GNPs from chloroauric acid. In addition to this, tannins also present in S. nigrum, tannins has poly phenols that would bind the metal nanoparticle surface. In a pilot experiment, when the solution of S. nigrum and chloroauric acid were mixed together a clear color change from pale-yellow to ruby-red was observed within a minute, indicating S. nigrum-mediated transformation of chloroauric acid into GNPs. As GNPs formed need to be stabilized (prevented from aggregation), 1% PVA was added as stabilizing agent, and irradiated in a microoven for 60 seconds. The reaction proceeded smoothly, and formation of deep ruby-red color confirmed the completion of the reaction as shown in Fig. 4.

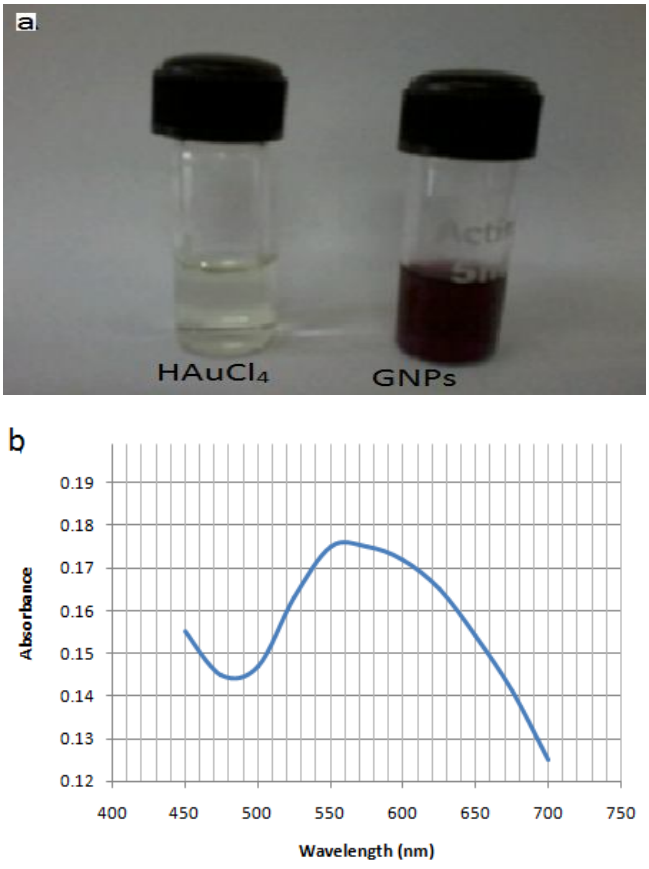

Fig. 4 Naked eye observation of formation of GNPs from $HAuCl_4$ and *S. nigrum* leaf extract (a). UV–visible spectra of GNPs (b)

UV–visible spectral analysis

The leaves of S. nigrum contain several compounds containing polyphenols and heteroatoms such as solasonine, solamargine, riboflavin, diosgenine, nicotinic acid and vitamin C [30]. The phenolic hydroxyl containing compounds can be used as an effective reducing agent for Au(III) which has a high oxidation–reduction potential to Au(0) [31].

The UV–visible absorption spectrum of the isolated GNPs showed a distinct surface plasmon resonance (SPR) band at 545 nm as shown in Fig. 5b confirming the formation of gold particles in nano dimension [32]. The position of SPR band observed at 545 nm reflects there is a red shift takes place due to agglomeration of nanoparticles. The position of SPR band observed at the initial stage of the reaction remind almost unaffected even after several hours implying the capping of nanoparticle surface by S. nigrum molecules. Such a capping of GNPs provides long term stability by preventing further aggregation of the nanoparticles.

SEM–EDAX analysis

Energy dispersive X-ray (EDX) spectrum analysis of GNPs demonstrated the presence of GNPs in the sample (Fig. 5a). From a comparison of the different peaks in the spectrum, it could be concluded that the individual GNPs showed a signal of nanoparticles, and that the individual nanoparticles could be identified as consisting of gold.

The FE-SEM micrograph of GNPs sample shows the presences of spherical particles with a mean size of about 20 nm as shown in Fig. 5b. The fact that all the nanoparticles are well separated, and no clusters or aggregated mass is seen, clearly indicates the stabilization of individual nanoparticles by S. nigrum-capping. Even in the region where the nanoparticles are in loosely arranged cluster, individual nanoparticles could be easily recognized, indicating that the clusters are formed due to weak binding forces.

Zeta potential studies

Zeta potential (ξ) is an essential parameter to study the state of the nanoparticle surface and predict the long-term stability of the nanoparticles. The electrostatic potential at the electrical double layer surrounding a nanoparticle in solution is referred to as the zeta potential. Since the size of leaf-extract-coated-GNPs was greater than 10 nm, 0.1 g/ml suspension of GNPs was used for the measurement of zeta potential. The GNPs have the highest count with a ξ value of

−15 mV, indicating a moderately negative charge on the surface of the nanoparticles [33]. The moderate value of ξ might also reflect the adsorption of S. nigrum molecules on the surface of GNPs, and the electrostatically moderately stable form of GNPs coated with the molecules present in S. nigrum leaf extract.

(Bragg peaks) were produced at (1 1 1), (2 0 0) and (2 2 0) planes and were assigned to the diffraction lines of the face-centred-cubic (fcc) gold [34].

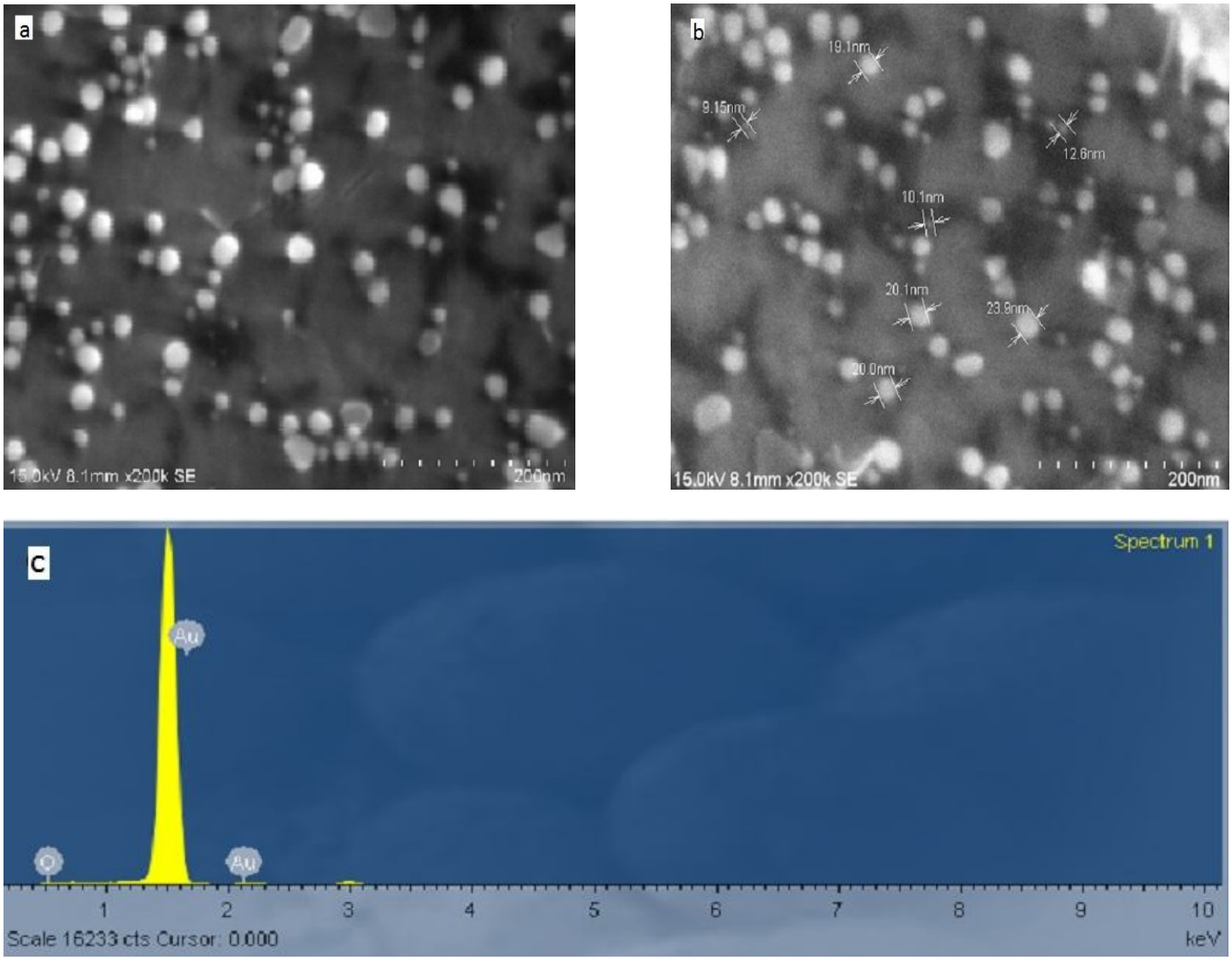

Fig. 5 FE-SEM picture of *S. nigrum* GNPs (**a, b**). EDAX spectrum of *S. nigrum* GNPs (**c**)

X-ray diffraction analysis

The powder XRD pattern (2Θ= 37.95, 44 and 64.44◦) of the sample is in agreement with the International Centre for Diffraction Data 10725392 of bulk gold, which further proves the formation of crystalline Au (Fig. 6). The crystalline peaks were identified as gold nanoparticles according to International Centre for Diffraction Data. The XRD analysis revealed that the intense peaks of Fig. 6 reflected radiation

Mechanism of corrosion inhibition

The photochemical analysis of the constituents of the leaf extract of S. nigrum shows the presence of many hetero atoms, double bond containing molecules and aromatic rings which are potential adsorption centers for adsorption on to the metal surface. Organic compounds containing π-electrons, hetero atoms and multiple bonds have been reported to function as effective inhibitors for the corrosion

of many metals in various media [35-37]. Since the S. nigrum extract contains many organic compounds, it is very difficult to mention a particular compound for the inhibition activity.

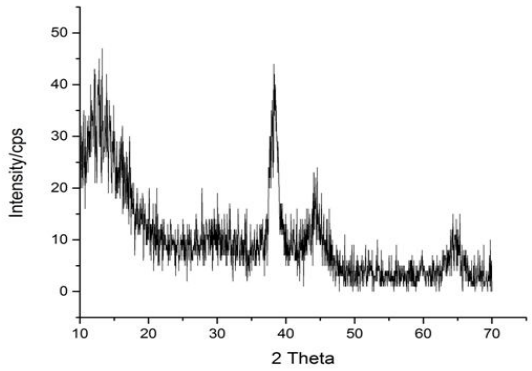

Fig. 6 The powder X-ray diffraction pattern of R-GNPS

When carbon steel is immersed in a neutral aqueous environment, the anodic reaction is

$Fe \rightarrow Fe2+ + 2e-$

The cathodic reaction is

$O2 + 2H2O + 4e- \rightarrow 4OH-$

Hence the proposed mechanism is the neutral molecules of leaf extract can be adsorbed on the surface of the carbon steel through chemisorptions mechanism, involving the displacement of water molecules from the carbon steel surface and the sharing electrons between the hetero atoms and iron. Since the S. nigrum extract [38] contains many organic compounds, it is very difficult to mention a particular compound for the inhibition activity. The inhibitive activity of the extract is attributed to the combined action of all the compounds present in the extract [39].

CONCLUSION

- Weight loss study reveals the 500 ppm of S. nigrum shows 86.36% IE.
- Polarization study reveals that this inhibitor function as both anodic and cathodic controlled but predominantly anodic process.
- AC impedance spectra reveal that a protective film is formed on the metal surface.
- The SEM image shows the surface coverage increases which in turn results in the formation of insoluble complex on the metal surface.
- The antioxidant (reducing) property of the leaf extract has been successfully explored for the first time in the green synthesis of gold nanoparticles.
- The SPR band at 545 nm confirms the formation of GNPs.
- The FESEM analysis shows the average size of the GNPs of about 20 nm.
- The Zeta potential shows -15 mV indicating the adsorption of S. nigrum molecules on the surface of GNPs.
- The XRD data shows the nanoparticles as face-centered cubic (fcc) gold.
- Exploring the pharmaceutical properties of the newly synthesized GNPs is part our current research.

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