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Research Article

EVALUATION OF PHYSICAL, CHEMICAL PROPERTIES AND CHARACTERIZATION STUDIES OF ORGANIC INHIBITOR (*AZADIRACHTA INDICA GUM*) USING CONCRETE CORROSION

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ABSTRACT

The present research work mainly focused on the influence of natural admixtures using concrete with enhanced properties and to prevent the corrosion using natural inhibitors. Corrosion of reinforcement is one of the drawbacks in reinforced concrete structure to overcome corrosion problem an attempt has been made in the present investigation by use of corrosion inhibitors. The organic inhibitors extracted by water from *Azadirachta indica* (Gum). In this study. the effect of *Azadirachta indica* gum as set retarding admixture in cement pastes. The various concentrations of 0.2 mg/L to 1.6 mg/ L of the weight of cement and w/c ratio of 0.50. In the physical properties study, the inhibition efficiency was increased with increase in the concentration of inhibitors for mild steel aqueous medium the maximum concentration (1.6 mg/L). The effect of aqueous extract of (*Azadirachta Indica Gum*) on the corrosion inhibition of mild steel in various concentrations of 0.2 mg/L to 1.6 mg / L) solution was investigated by electrochemical study and weight-loss experiments at temperatures ranging from 303 K. The percentage of inhibition increased with the increase of the concentration of the inhibitor. At a concentration of 1.6 mg/ L, the percentage inhibition reached about 85.24% at 303 K. The SEM analysis showed that inhibitors adsorbed on mild steel surface to protect mild steel from corrosion. The aqueous extract was found to be an excellent potential corrosion inhibitor because of the high content of alkaloids. The results of inhibitors showed the natural gum has better corrosion inhibition efficiency. It is also observed that the efficiency of inhibitors depends on the exposure environment.

Keywords: *Azadirachta Indica Gum*, organic inhibitor, weight loss, SEM and Electrochemical studies.

INTRODUCTION

Concrete is a composite material made up of cement with different chemical admixtures[1]. The usage of admixtures is currently very low cost; It is expected to increase strength of materials. In industries concrete reinforced with rebar is considered the indispensible basic material for the building[2]. Cement based compound together with residue free composites belongs to the most highly developed materials used in construction. Since 20th century the uses of reinforced cement materials have become common practice due to its excellent properties compared to other construction materials like natural plant materials (Bamboo, barks and mammoth ribs etc.). The resistant cement materials comparatively cheap and easy maintains. Water is permeability of concrete is believed as the key property related to the serviceability and durability of concrete[3]. The corrosion of metals in many industries is a serious problem. In order to prevent corrosion inhibitors are generally used system like organic and inorganic or mixed inhibitors on the metal surface to form a complexity type of precipitate on its surface. The prevention measures used in the construction to increase service life of concrete structures. The advantages of green inhibitors over chemical inhibitors as they are readily available less toxic, biodegradable its eco-friendly and also easily renewable[4]. Based on the report of sustainability and the search for environmentally available materials, a new approach of corrosion inhibitor is extracted from natural plants. This widespread to the application of requires convey of inhibitors to the strengthening where it must reach sufficient on the surface of steel against corrosion. The aqueous extracts from different parts of natural plants have been found to be good corrosion inhibitors for many metals[5-9]. In the present study deals with *Azadirachta Indica Gum* natural admixture used with cement mortar to enhance the durability study of concrete. From the study were characterized by performance through phase behaviour observation, scanning electron microscopy (SEM), elemental analysis (EDX) Fourier transforms infrared (FT-IR) spectrum, X-ray diffraction (XRD). The aim of this research to study the potentiodynamic and weight-loss measurements on the corrosion inhibition of mild steel by aqueous extract of *Azadirachta indica gum*.

MATERIALS AND METHODS

Materials

The gum of *Azadirachta Indica Gum* was collected from Yercaud hills, Salem district; Tamil Nadu. An expert taxonomist has identified the plants at the Botanical Survey of India, Tamil Nadu, Agriculture University campus, Coimbatore, India. Voucher specimens (BSI /SRC/5/23/2016/TECH/166) are stored in the department of civil engineering laboratories and it is available for further reference.

Preparation of Plant Extract

The *Azadirachta Indica Gum* powered materials (150g) is extracted in 1.5 L (100 % v/v) water on a mechanical shaker (orbital shaker) for 8 hours. The extract is filtered using a

Buchner funnel and Whatman No.1 filter paper. The filter is concentrated under reduced pressure at 52 ± 5^0C to dryness for 48 hours by using freeze dryer.

Preliminary Phytochemical Analysis

Preliminary screening for the presence of phytoconstituents is carried out by the reported protocol[10]

Cement

The cement used for this study is Chettinad Portland pozzolanic cement and it is confirmed to Indian Standard (IS) 1489 of Grade 43[11].

Sand

The sand is collected by a local area. The sand has been sieved in 1.18mm (passed) and retained in 600µ, taken the 500 grams of sand and the sieve plates are used in 1.18mm, 600µ, 300µ, 212µ, 106µ and 53µ. The sand is passed through the 1.18 sieve plate weighed and retained in 600µ. After that, the retained sand is weighed. The procedure is continued every half an hour the fineness modulus of sand.

Coarse Aggregate

The Aggregate is the major ingredients of concrete constituting 70-75 %. They provide a rigid skeleton structure for concrete and act as economical space fillers. The clean river sand is available locally complying with the requirements of grading zone – II specified by IS 383- 1970 of fine aggregate is used for the investigation. Similarly, fineness modulus of fine aggregate 2.73, locally available well-graded granite aggregate of maximum size 20 mm was used along with 12 mm sized aggregate and 6 mm chips as coarse aggregate.

Water

Bore well water collected from the Institution used to cost the concrete specimens. The water was acids and alkalis and more soluble chloride content of 150 mg/lit, sulfate 20 mg/ L, TDS 926 mg/ L, Total hardness 600 mg/lit which is high compared to permissible limit these results are testing from CIRT- water analysis Lab Salem, Tamil Nadu, India. As per IS 456-2000, the permissible limit for chloride is 500 mg/ lit of reinforced concrete.

Viscosity Modifiers

The *Azadirachta Indica Gum is* sparingly soluble in water but swells in contact with it giving a highly viscous solution. It is a polyuronide consisting of arabinose, galactose and glucuronic acid in the proportion of 10:7:2-mole rhamnose is present in traces. There are several reports about the successful use of hydrophilic plant polymers as a tablet, binders, emulsifiers and gelling agents. Suspending agents, stabilizers and thickener.

Specimen Preparation

The mild steel compositions of Fe: 99.75 %, Mn: 0.01 %, Cu: 0.01%, Si: 0.02%, P: 0.02% and C: 0.18% is used in this work. The specimens of dimension 1 cm length and 0.5 cm width are used. The specimens were polished using bench polisher (model. No :) and pickling solution was used to wash the mild steel till the metal is clear and washed with distilled water, acetone and kept in the oven for immediate usage. All the samples were characterized by FT-IR, SEM, EDX and XRD.

Weight Loss Method

The percentage of iron is more in mild steel, which improves the corrosion rate in industries. Here by adding the crude extract of *Azadirachta Indica Gum* the inhibition efficiency of mild steel increases with increase in concentration by reducing the rate of corrosion. The weight loss methods were carried out by weighing the specimens before and after immersion in water in the absence and presence of inhibitor. From the initial and final mass of the specimen, the weight loss was calculated. The inhibition efficiency (IE) and corrosion rate were determined.

$$I.E\ \% = \theta.100\ \%$$

Where θ (Surface coverage) = $\Delta W_u - \Delta W_i / \Delta W_u$

Where ΔW_u and ΔW_i Weight loss of the metal in the absence and presence of inhibitor**.**

Electrochemical Studies

The surface of the electrode is prepared by taking MS specimen of an area of 0.5 cm width and 0.5 cm thickness set with Teflon coated was added. The coated MS was polished bench polisher.

Electrode Cell Assembly

The electrochemical studies (CH Instrument) Model 608 E Series were used for polarization studies only. In this study were used to three different electrodes i) auxiliary electrode is platinum electrode ii) Standard electrode is saturated calomel electrode (SCE) and iii) MS specimen of an area 0.5 cm used as a working electrode.

Procedure and Calculation

In electrochemical study was carried out in the presence and absence of inhibitor, the values were recorded and the percentage of inhibition efficiency was calculated[12].

$$I.E\ \% = [1 - (i'_{corr} / i_{corr})] \times 100$$

Where i'_{corr} and I_{corr} are the corrosion current density of mild steel in the absence and presence of inhibitor.

RESULTS AND DISCUSSION
Tests for Fresh Concrete

Fineness modulus of fine aggregate 2.73, similarly locally available well-graded granite aggregate of maximum size 20 mm was used along with 12 mm sized aggregate and 6 mm chips as coarse aggregate. Portable water was used to cost the concrete specimens. The water was free from oils, acids, and alkalis and had a water soluble chloride content of 140 mg/ L which is very much less than the permissible limit. As per IS 456-2000, the permissible limit for chloride is 500 mg/ litre for reinforced concrete. The gum dissolved in cold water giving light brown.

The values obtained from the various tests performed on the fresh concrete mixes as shown in table 1.

Table 1: Test values of fresh concrete mixer

S.No	Gum mg/ l	Slump mm	Compacting factor	Vee Bee Time 'Secs'	Final setting time minutes
1.	Blank	65	0.89	6	280
2.	0.2	68	0.90	5	290
3.	0.4	70	0.90	5	310
4.	0.6	72	0.92	5	315
5.	0.8	78	0.92	4	320
6.	1.0	82	0.94	4	328
7.	1.2	84	0.94	4	335
8.	1.4	86	0.96	3	340
9.	1.6	90	0.96	3	365

In fresh state, *Azadirachta Indica* gum powder plays an important role in the fluidity of concrete which is commonly expressed in such phenomenological measurements of workability compatibility. Setting and finish ability addition of neem gum powder has a significant influence on the rate of hydration reactions as well as on the effectiveness of chemical admixture. Based on the test result it was observed that the slump, compacting factor and final setting time of concrete increased whereas, on the other hand, the veebee time decreased with increase in the percentage of neem gum added. All the above finding, indicate that with an increase in the percentage of neem gum powder was added in cement concrete increase the workability of concrete. The values obtained from the various mechanical tests performed on the hardened concrete specimens are given below.

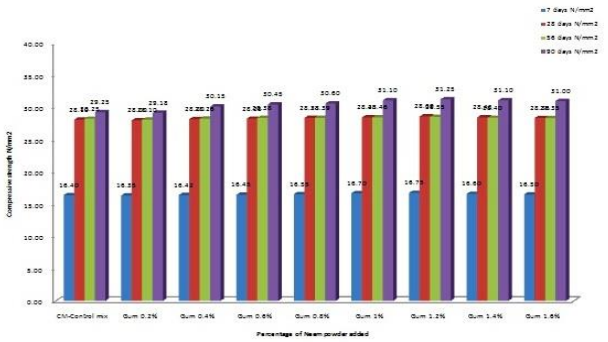

Figure 1: Compressive strength of concrete cubes

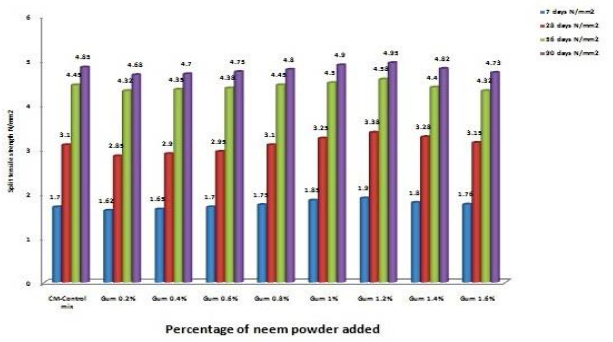

Figure 2: Split tensile strength of concrete cylinders

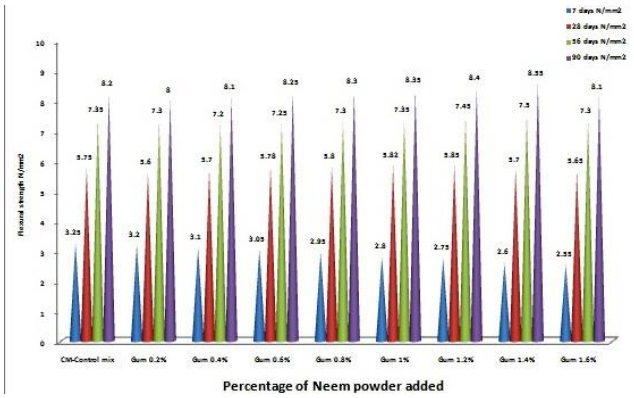

Figure 3: Flexural strength of concrete beams

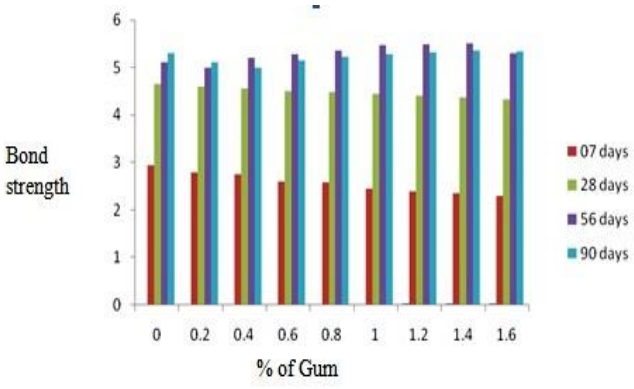

Figure 4: Bond Strength of concrete using different gum percentages

From the above figure.1, 2, 3 and 4 clearly indicate that Natural admixture using water extract of *Azadirachta indica* gum certain proportions contribute to the mechanical strength of concrete due to its pozzolanic reactivity. This phenomenon was observed in all the mechanical strength tests performed until the 28 days and 56th day of costing, there was a change in the trend. The slight increase in compressive flexural and bond strength particularly for 1.6 mg/ L added in concrete. This change in trend continued and ultimately the 90 days, the strength of all the specimens in all the specified tests clearly overtook the strength values attained by concrete. The split tensile strength of cylinder cost with 1.6 mg / L achieves the values in increases. Hence concrete developed greater strength at a later age exceeding that of concrete without gum. The Pozzolanic reaction natural gum only proceeds in the presence of aqueous extract or enough moisture should be available for a long time. Hence to achieve the specimens were continuously cured by complete immersion in water.

Water absorption test

Water absorption test carried out using cylindrical 60 mm diameter 120mm long. The specimen was immersed in water per 72 hours and retrieved from curing tank. This sample weighed in top loading balance immediately wiping the specimen with cotton. The percentage weight of water absorbed in the table.

Among this entire specimen, the control mortar absorbed very low water and which shows the percentage of 1.6 mg/ L highest 2.50 water absorption was found in NG admixed water. However, that can be stated that all the admitted mortar shows enhanced water absorption compared to control. In the case of gums, it is obvious that the gelation of the gum leads to increased absorption of water. In the present investigation, preliminary phytochemical screening has been done for the water extract of *Azadirachta indica* gum for the presence of phytochemical constituents namely Alkaloid**,** Saponin, and oils indicated in table.2.

Table 2: Preliminary Phytochemical Screening

S.No	Test	Water extract
1.	Alkaloid	+
2.	Saponin	+
3.	Tannins	-
4.	Flavanoid	-
5.	Terpenoids	-
6.	Coumarin	-
7.	Cycloglycoside	-
8.	Total phenol	-
9.	Quinine	-
10.	Steroids	-
11.	oils	+

+ Positive, - Negative

Tests for Durability

To determine the corrosion rate by diffusion of chloride cylindrical concrete specimens with pre-weighed rod centrally placed were used. The concrete specimen was immersed in 2.5mg/ L NaCl solution and positive rod was made anode (connected to positive terminal) with respect to an external stainless steel electrode (connected to negative terminal) serving as cathode on applying a constantly impressed voltage from D.C source, the variation of current was recorded with respect to time.

Table 3: Impressed voltage test

S.No	GUM mg/ L	Corrosion initiation time in hours
1.	Control	175
2.	0.2	169
3.	0.4	174
4.	0.6	180
5.	0.8	184
6.	1.0	189
7.	1.2	200
8.	1.4	205
9.	1.6	210

A sharp rise in current indicated the onset of corrosion and cracking of the concrete was usually visible thereafter. The time taken for initiation of the first crack was considered as a measure of their relative resistance against chloride permeability and reinforcement corrosion shown in table 3.

Weight loss Studies

The 16 mm diameter bars that were cut to specified lengths, embedded in concrete and utilized for the above three tests were initially immersed in (Gum powder + water equally quantity), cleaned and weighted. At the end of the tests, the specimens were broken and the corroded rebars were again immersed in the pickling solution, cleaned and weighed. The difference between the initial and final weights was taken as the weight loss of the specimen and it was converted into a reduction in thickness and expressed as a loss in thickness in mm per year as shown in figure.5.

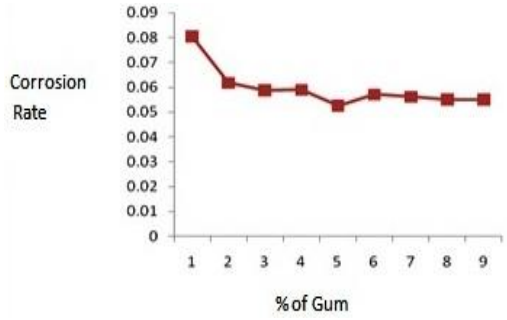

Figure 5: Corrosion rates from weight loss method

Potentiodynamic Polarization Studies

The Potentiodynamic polarization curves for mild steel in various concentrations of aqueous extract of *Azadirachta indica* gum without and with inhibitor are shown in Figure.6. The anodic and cathodic Tafel slopes and inhibition efficiency (IE) for the corrosion of mild steel in an aqueous medium at 303 K in the absence and presence of different concentrations are given in Table 4. The Polarization studies revealed that the corrosion current density (I_{corr}) markedly decreased with the addition of *Azadirachta indica* gum aqueous extract and the corrosion potential shifts to less negative values upon the addition of *plant* extract. Tafel slopes are slightly changed indicating that this behavior reflects the ability of the extract to inhibit the corrosion of mild steel in aqueous solution and the adsorption of *Azadirachta indica extract* on both anodic and cathodic. It was observed that the inhibition efficiency was found to be increased with an increase in the concentration of *Azadirachta indica* extract from 100 ppm to 1600 ppm. The maximum inhibition efficiency of 85.24 % was observed at 1600 ppm. The corrosion behavior of mild steel in aqueous extract of *Azadirachta indica* gum observed by weight loss method is in good agreement with the polarization studies.

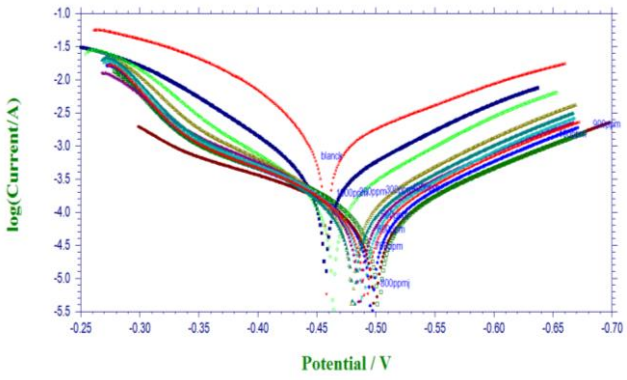

Figure 6: Potentiodynamic Polarization curves for mild steel in Aqueous extract of *Azadirachta indica* gum at 303 K

Table 4: Polarization parameters of Aqueous extract of *Azadirachta indica* gum on mild steel corrosion at 303 K

Conc. of *Azadirachta indica* (ppm)	E_{corr} (mV)	Tafel slopes (mV/dec)		I_{corr} ($\mu A/cm^{-2}$)	Inhibition efficiency (%)
		$-b_a$	$-b_c$		
Blank	-0.449	5.48	3.56	1348	-
200	-0.450	5.73	3.96	83.88	74.10
400	-0.462	5.80	4.07	89.43	75.19
600	-0.473	6.05	4.27	88.09	76.30
800	-0.481	6.06	4.28	81.99	78.01
1000	-0.483	6.09	4.29	65.02	79.25
1200	-0.485	6.13	4.31	62.40	80.31
1400	-0.488	6.15	4.35	48.11	81.33
1600	-0.494	6.18	4.38	47.92	82.25

FT-IR Spectral Analysis of aqueous extract of *Azadirachta indica* gum

The FT-IR spectrum of mild in aqueous extract of *Azadirachta indica* gum is shown in Figure 7. The assignment of speak appeared in the various region of FT-IR spectrum for the functional groups present in the ethanol extract of *Azadirachta indica* as follows. The FT-IR spectrum of *Azadirachta indica* extract showed the characteristic absorption at 1409 cm^{-1} (due to C -N bond), 1595 cm^{-1} (due to stretching vibration of C-O or C=O), 2945 cm^{-1} (due to stretching vibration C-H Asymmetric), 2411 cm^{-1} (due to stretching vibration C-H symmetric).

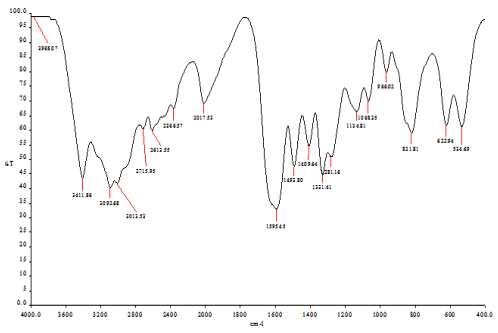

Figure 7: FT-IR spectrum of *Azadirachta indica* gum

Scanning Electron Microscopic Studies (SEM with EDX)

Surface analysis was carried out by SEM technique in order to evaluate the surface condition of the mild steel in contact with aqueous extract. The Surface image morphology of mild steel specimens in the presence and absence of different concentrations of *Azadirachta indica* gum aqueous extract immersed for 24 h at 30^0C. In the presence of *Azadirachta indica* gum, aqueous extract micrograph shows less evidence of pitting when compared with the concrete and smooth surface as shown in Figure 8. This result is due to the adsorption of *Azadirachta indica* gum extract around the pits. This passive film blocks the active site present on the mild steel surface thereby retarded the corrosion process. The SEM images clearly differentiate the formation of the protective layer on the surface of the mild steel.

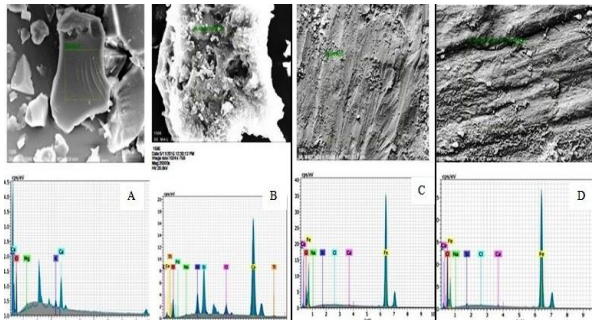

Figure 8: SEM images of mild steel in a water extract of *Azadirachta indica* gum in the absence and presence of the inhibitors A) Plain Rod B) Plain concrete C) *Azadirachta indica* gum D) *Azadirachta indica* gum + Rod E) Normal mix concrete.

The EDX Spectra patterns of natural gum extract using concrete the presence of elements, concrete (oxygen = 56.12 %), Plain rod (Fe = 75.56 %), Natural gum + concrete (oxygen = 53.29 and calcium = 35.96 %) and normally mixed with concrete (Fe= 54.42 % and oxygen = 44.00 %) respectively. It confirms the diffraction signals are from natural gum mixed with concrete. Other diffraction peaks corresponding to iron phases can also be observed, indicating that Iron droplets are not oxidized absolutely during the process the significant percentage of iron, calcium, and oxygen in the concrete are major constituents shown in figure.6.

X-ray diffraction (XRD) study

The XRD spectra of the films annealed at 400°C for 4 hours in ambient air. All films show pure natural gum with the monoclinic crystal structure. The major peaks formed were sharp due to crystalline nature showed if Figure.9. The oxide was further analyzed with XRD broad peaks at 2h = 27.3, 28.5, and 35.3 was observed. It suggests that the obtained oxide may contain a mixture of phases. The lack of clear peaks, low intention, and broadening of peaks indicate the amorphous nature and the small size of the crystalline grain

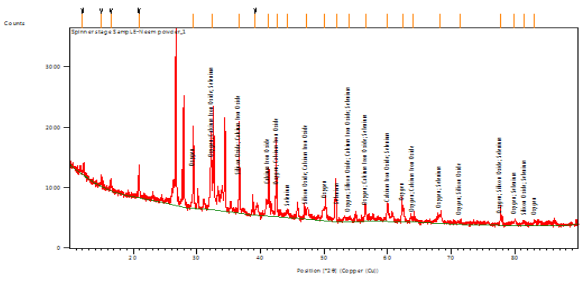

Figure 9: XRD spectra of the *Azadirachta indica* gum

CONCLUSION

The aqueous extract of *Azadirachta indica* gum was found to be a highly efficient inhibitor for mild steel in aqueous solution the maximum 85.24 % for 1.6 mg / L at 303 K. The rate of corrosion of the mild steel in aqueous medium decreased as the concentration of the gum extract is increased. The aqueous natural gum extract is an excellent inhibitor, eco-friendly, and very cheap corrosion inhibitor for mild steel in aqueous solution.SEM study confirms that the inhibition of corrosion of mild steel is through adsorption of *Azadirachta indica* gum aqueous extract were strongly adsorbed on mild steel surface. FT-IR analysis concluded that nitrogen and oxygen atoms present in the *Azadirachta indica* gum aqueous extract.

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