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Potentials of *Commelina benghalensis L* and *Acanthospermum hispidum DC* Plant Extracts for use as Green Corrosion Inhibitors

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Authors' contributions

This work was carried out in collaboration among all authors. Author JNA designed the study, performed the experiments, wrote the protocol and wrote the first draft of the manuscript. Authors GBN and DTG managed/supervised the analyses of the study. Author JNA managed the literature searches. All authors read and approved the final manuscript.

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ABSTRACT

Phytochemical analysis was carried out on the leaves and stem extracts of *Commelina benghalensis L* and *Acanthospermum hispidum DC* plants to determine their corrosion inhibition potentials. The leaves and stem parts were processed by washing, air-drying under shade and then ground into powder. Stock solutions were then extracted from these materials using ethanol according to standard procedures. Quantitative and qualitative analysis were carried out on the extracts in accordance with standard procedures. These analyses showed the presence and composition of the extracts as alkaloids (7.2%), flavonoids (11.2%), saponins (4.1%), tannins (10.3%) and phenols (5.6%). The qualitative analysis also confirmed in addition, the presence of carbohydrates, glycosides and steroids. Anthraquinones were, however, found to be absent. These chemical substances are known to contain oxygen, nitrogen and sulphur atoms in their molecules which are regarded as adsorption centers in the process of corrosion inhibition. FTIR analysis was also used to identify the presence of functional groups in these extracts. The results of these analyses suggest that extracts of these plants could serve as materials for the production of corrosion inhibitors since they contain phyto-constituents that are known to have inhibitive properties against mild steel corrosion.

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1. INTRODUCTION

Phytochemical compounds are natural bioactive compounds which exist in plant tissues. These plant products are derived from plant leaves, stem bark, fruits, roots, seeds and flowers which are found to contain important active compounds that can be used for medicinal and other purposes. In recent times attention has been drawn to the use of materials of plant origin as green corrosion inhibitors due to environmental and safety issues associated with inorganic and synthetic inhibitors [1]. Studies have shown that extracts from plant tissues could be used as corrosion inhibitors for the protection of metallic structures against corrosion attack [2]. The use of inhibitors has been found to be one of the best options of protecting metals and their alloys from corrosion attack [3]. A corrosion inhibitor is a chemical substance that when added in a small quantity decreases the corrosion rate of a material especially a metal or an alloy [4]. The role of the inhibitor is to form a barrier (or protective film) on the surface of the material which prevents access of corrosive substances to the metal surface and consequently slows down the corrosion rate [5].

Due to increasing demand for a clean environment in the world today, researchers are now interested in developing alternative inhibitors that are non-toxic and ecofriendly, for corrosion protection and control. Plants extracts are naturally available and less expensive and non-toxic materials which can be used for corrosion inhibition. The extracts from plant leaves, stem bark, fruits and roots comprise of mixtures of organic compounds containing nitrogen (N), phosphorous (P), sulphur (S) and oxygen (O), which have been reported to function as effective corrosion inhibitors of metals and alloys in different aggressive environments (Quarashi et al., 2009).

Ebenso et al. [6] studied corrosion inhibitive properties and adsorption behavior of ethanol extract of piper guinensis as a green corrosion inhibitor for mild steel in H_2SO_4 solution. The study found that the plant extract retarded the acid induced corrosion of mild steel. Makanjoula et al. (2011) investigated corrosion of mild steel in hydrochloric (HCl) acid by tannins from *rhizophora raceumosa* and concluded that tannin treated steel at the concentration of 140 ppm gave 72% corrosion inhibition. Adzor et al. [7] also investigated the corrosion inhibitive potential of Hibiscus sabdariffa calyx extract for low carbon steel in 0.5M H_2SO_4 acid solution and found out that the inhibition efficiency was up to 95.01% at higher concentration. So, the potentials of plant extracts as green corrosion inhibitors in metal corrosion mitigation have been reported to be effective in many other studies as well [8-10] (Renita et al., 2015;).

In this study, phytochemical analysis of leaves and stem extracts of *Commelina benghalensis L* and *Acanthospermum hispidum DC* was carried out. *Commelina benghalensis L* (CBL) is commonly known as Benghal dayflower or tropical spider worth. It belongs to a family of *Commelinaceae* comprising of over 500 species with distinct characteristics. The plant is native only to tropical Asia and Africa and it can be found in Nigeria, Ethiopia, Kenya, Senegal and Cameroon. It is a widely distributed herbaceous weed that usually invades agricultural sites and disturbed areas. This rapid growing plant is one of the most troublesome weeds for 25 crops in 29 countries; hence it is found on the International Union for Conservation of Nature (IUCN) red list of species (Webster et al. 2005).

CBL can be an annual or perennial herb; it grows as a perennial herb in tropical climates and as an annual herb in temperate areas. It produces seeds within 40-45 days of emergence and has multiple generations per year. The leaves are ovate or elliptical, 2.5-7.5 cm long, 1.5 -2.5 cm wide, with parallel venation (Plate 1). CBL is used for different purposes in different countries; it is used as a medicinal herb with diuretic and anti-inflammatory effects and as vegetables, and in livestock feeds in China, Pakistan, and Africa to combat infertility.

Acanthospermum hispidum DC (AHDC) is commonly known as bristly starbur, goathead, gypsy-thorn or hispid starbur. It is an annual herbaceous plant in the family of Asteracease which is native to Central and south America. It grows abundantly during the rainy season in many parts of the world including North Eastern Brazil, North and South America, Central America, Australia, Africa, India, Indonesia and Nigeria (Holm et al.,1997).

AHDC (Plate 2) is an invasive weedy plant species on agricultural lands and grows well on a wide variety of soils from sandy to clay up to 15-50 cm tall. It has a characteristic light, slightly sweet aroma. The stem is covered with fine hairs, the leaves are stalk less, covered with stiff hairs and borne in pairs along the stem, bitter in taste, simple and opposite, measuring on the average 2-12.5 cm long and 1-3 cm wide.

AHDC, has been traditionally used as medicine for treating asthma, bronchitis, fevers, as an expectorant and for intestinal disorders in North Eastern Brazil (Morais et al., 2005; Torres et al., 2005). It is also used as forage for feeding cattle during the dry season.

In this work quantitative and qualitative phytochemical analyses were conducted on the plants to identify and determine the actual phytochemical substances present and the quantities of each constituent in the leaves and stem extracts.

2. MATERIALS AND METHODS

2.1 Preparation and Extraction of Plant Materials

The fresh leaves and stem of CBL and AHDC were cleaned and air- dried separately under shade for four weeks. The dried leaves and stem materials were then separately ground into powder using a mortar/pestle to coarse particle size.

Cold extraction process was carried out using procedures described by Eddy and Ebenso [11]. Fifty grammes (50 g) of the prepared powdered plant materials were soaked in 500 mls of absolute ethanol for 48 hours with occasional agitation.

The solution obtained from this procedure was filtered with Whatman No. 42 grade of filter paper. The filtrate was then dried by evaporation and concentrated using water bath at 45^{o}c and collected in a tight sample bottle and stored in a refrigerator at 4^{o}c for future use as stock solution.

Phytochemical analysis: Both qualitative and quantitative screening of the four samples comprising of leaves and stem extracts of CBL and AHDC plants respectively.

Qualitative phytochemical screening tests were conducted on the materials to know their active constituent elements using the following procedures:

Test for Carbohydrates (Molisch Test): To a small portion of the extract in a test tube few drops of Molisch reagent were added and concentrated sulphuric acid also added down the side of the test tube to form a lower layer, a reddish colored ring at the interface indicates presence of carbohydrates [12].

Test for Anthraquinones (Bontragers Test): To 0.5g of the extract in a dry test tube ,5ml of 10% hydrochloric acid was added and boiled for 2-3mins.This hydrolyzed the glycosides to yield aglycones, which are soluble in hot water. This was filtered and the filtrate cooled and extracted with 5 ml of benzene. The benzene layer was pipetted off and shaken gently in a test tube with half of its volume of 10% ammonium hydroxide (NH_4OH). If the lower ammonia layer becomes rose pink to cherry red, the material contains anthraquinones derivative [12].

Plate 1. CBL

Plate 2. AHDC

Test for Glycosides (Fehling Test): To 0.5 g of the extract, 5 ml of dilute sulphuric acid was added and boiled on water bath for 10-15 mins, this was cooled and neutralized with 20% KOH and then divided into two portions. To the first portion, 5ml of a mixture of Fehling's solution A and B was added and boiled; a brick red precipitate shows the release of reducing sugar as a result of hydrolysis of glycoside [12].

Test for cardiac glycosides (Keller-Kiliani Test): To 0.5 g of the extract was dissolved in 1 ml of glacial acetic acid containing traces of ferric chloride solution. This was transferred to a dry test tube and 1 ml of concentrated sulphuric acid was added down the side of the test tube to form a lower layer at the bottom. The appearance of purple –brown ring at the interphase indicates the presence of deoxysugars and a pale green color in the upper acetic acid layer indicates the presence of cardiac glycosides [12].

Test for Saponins (Frothing test): About 10 ml of distilled water was added to 1 g of the extract and shaken vigorously for 30 seconds. The tube was placed in a vertical position and observed for 30 mins. A honey comb froth that persists for 10-15 mins indicates the presence of saponins [12].

Test for Steroids and Triterpenes (Liebermann Burchard test): To 0.5g of the extract, equal volume of acetic acid anhydride was added and mixed gently, 1 ml of concentrated sulphuric acid was added down the side of the test tube to form a lower layer. Colour changes were observed immediately and over a period of one hour. Blue to blue-green color in the upper layer and a reddish, pink or purple color indicates the presence of triterpenes [12].

To 0.5g of the extract, 2-3 drops of concentrated sulphuric acid was added at the side of the test tube. Any immediate color changes at the interphase of the extract and sulphuric acid were noted over a period of one hour. Cherry red color usually indicates the presence of unsaturated steroids [12].

Test for Tannins (Ferric chloride test): To 0.5g of the extract, 3-5 drops of ferric chloride solution were added. A greenish-black precipitate indicates presence of tannins while hydrolysable tannins give a blue or brownish-blue precipitate (Evans, 1996).

Bromine Test: Few drops of bromine water were added to 0.5 g of plant extract in a test tube, a buff-colored precipitate indicates condensed tannins while hydrolysable tannins give no colour at all (Evans,1996).

Test for Flavonoids (Shinoda test): A portion of the extract was dissolved in 1-2 ml of 50% methanol with metallic magnesium chips and few drops of concentrated hydrochloric acid added. The appearance of red color indicates presence of flavonoids [12].

Test for Alkaloids (Dragendoff"s test): To 1g of the extract, few drops of Dragendoff reagent was added and a reddish-brown precipitate indicates presence of alkaloids.

Wagner's Test: To 1g of the extract, few drops of Wagner's reagent was added and a whitish precipitate indicates presence of alkaloids [12].

The results from the above tests are presented in Tables 1 and 2.

Quantitative Determination of phytochemicals: Quantitative phytochemical screening is used to determine the concentration or amount of the individual phyto-constituents present in the particular plant under study. This was carried out using the standard procedures as described by Harborne (1998) as follows:

Determination of Alkaloids: About 5 g of powdered plant sample was taken into 250 ml beaker; 200 ml of 10% acetic acid was added into the beaker, it was covered and allowed to stand for 4 hours. Content in beaker was filtered and the extract was concentrated on a water bath to a one quarter of the original volume. Concentrated ammonium hydroxide was added drop wise to the extract until the precipitation was complete. This solution was allowed to settle. The precipitate was collected, washed with dilute ammonium hydroxide and filtered.

The remaining residue which is alkaloid was completely dried and finally weighed

$$\% \text{ alkaloids} = \frac{W_a}{W_s} \times 100\ \% \quad (1)$$

where, W_s is the weight of extract used (g) and W_a is the weight of alkaloid residue obtained (g).

Determination of Flavonoids: 10 g of the plant sample was extracted repeatedly with 100 ml of 80 % aqueous methane at room temperature. The whole solution was filtered through

Whatman filter paper No. 42 (125 mm). The filtrate was later transferred into a crucible and evaporated to dryness over a water bath and weighed to a constant weight.

$$\% \text{ Flavonoids} = \frac{W_f}{W_s} \times 100\%, \quad (2)$$

where, W_s = weight of extract used (g), and W_f = weight of flavonoid residue obtained (g).

Determination of Tannins: The quantity of tannins is determined by using the spectrophotometric method.

0.5 g of plant sample is weighed into a 50 ml plastic bottle 50 ml of distilled water is added and stirred. This was left to stand for 30 minutes at room temperature being shaken every 5 minutes. At the end of 30 minutes, it was centrifuged and the extract obtained. 2.5 ml of the extract was dispersed into 50ml volumetric flask. Similarly, 2.5 ml of standard tannic acid solution was dispersed into a separate 50 ml flask. A 1.0 ml Folin-Dennis reagent was measured into each flask followed by 2.5 ml of saturated Na_2CO_3 solution. The mixture was diluted to mark in the flask (50 ml) and incubated for 90 minutes at room temperature. The absorbance was measured at 720 nm in a Genway model 6000 electronic spectrophotometer. Readings were within the reagent blank at zero. The tannin content was obtained as follows:

$$Tannin\ (g) = \frac{A \times DF \times GF}{W_s} \quad (3)$$

where, A = absorbance of test sample at 720 nm, DF = dilution factor, DF = $\frac{total\ volume}{aliquot\ volume}$; GF = gradient factor = slope of a standard tannic acid curve and W_s = weight of extract used (g).

$$\% \text{ Tannins} = \frac{tannin\ (g)}{Ws} \times 100\%; \quad (4)$$

Determination of Saponins: About 10 g of plant extract was repeatedly extracted with 100 ml of 80% aqueous methanol at room temperature. The whole solution was then filtered through filter paper and the filtrate later on transferred into a water bath and the solution was evaporated to dryness. The sample was weighed until a constant weight obtained [13].

$$\% \text{ Saponins} = \frac{Wr}{Ws} \times 100\%; \quad (5)$$

Where, Ws = weight of extract used (g), and Wr = weight of saponin residue obtained (g).

Determination of Phenols: The spectrophotometric method was used in the determination of total phenols. The fat free sample was boiled with 50ml of ether for the extraction of the phenolic component for 15 minutes, 5 ml of the extract was pipetted into a 50 ml flask, then 10 ml of distilled water was added. 2 ml of ammonium hydroxide solution and 5 ml of concentrated amylalcohol were also added. The samples were made up to mark and left to react for 30 min. for colour development and measured at 510 nm.

$$\text{Phenol (g)} = \frac{AxDFxGF}{Ws}, \quad (6)$$

where, A= absorbance at 510 nm, DF = dilution factor = Total volume /aliquot volume, GF = gradient factor = slope of a standard tannic acid curve, W_s = weight of extract used (g)

Phenol (g)

$$\% \text{ phenol} = \frac{phenol(g)}{Ws} \times 100\ \% \quad (7)$$

The results of the quantitative phytochemical analysis tests using equations (1) to (7) are presented in Table (3).

3. RESULTS AND DISCUSSION

The results of this study are presented in Tables 1-3.

The results show the presence (+) of carbohydrates, glycosides, cardiac glycosides, saponins, steroids, tannins, flavonoids and alkaloids; anthraquinones were, however, absent (-) in the extracts. This indicates the potential of these plants to be used as green corrosion inhibitors. These substances are known to contain oxygen (O), nitrogen (N) and sulphur (S) atoms in their molecules which are regarded as centers of adsorption of the extracts on the metal surface thereby aiding the formation of a protective film on the metal surface which acts as a barrier separating the metal from the corrosive environment as reported by previous researchers [5,14].

Table 1. Phytochemical constituents of *Acanthospermum Hispidum* DC leaves and stem extract using qualitative analysis

S/N	Constituents	Test carried out	Leave sample (AHL)	Stem sample (AHS)
1	Carbohydrates	Molisch test	+	+
2	Anthraquinones	Bontragers test	–	–
3	Glycosides	Fehling test	+	+
4	Cardiac glycosides	Keller-Killiani test	+	+
5	Saponins	Frothing test	+	+
6	Steroids and Triterpenes	Liebeman Burchard test	+	+
7	Tannins	Ferric chloride test	+	+
8	Flavonoids	Shinoda test	+	+
9	Alkaloids	Dragendorff test	+	+

Key: + present, - Absent

Table 2. Phytochemical constituents of *Commelina Benghalensis L* leaves and stem extract using qualitative screening

S/N	Constituents	Test carried out	Leave sample (CBL)	Stem sample (CBS)
1	C Carbohydrates	Molisch test	+	+
2	Anthraquinones	Bontragers test	–	–
3	Glycosides	Fehling test	+	+
4	Cardiac glycosides	Keller-Killiani test	+	+
5	Saponins	Frothing test	+	+
6	Steroids and Triterpenes	Liebeman Burchard test	+	+
7	Tannins	Ferric chloride test	+	+
8	Flavonoids	Shinoda test	+	+
9	Alkaloids	Dragendorff test	+	+

Key: + Present, - Absent

Table 3. Quantitative Phytochemical screening of *Acanthospermum hispidum* and *Commelina benghalensis* leaves and stems

S/N	Constituent (%)	AHL	AHS	CBL	CBS
1	Alkaloids	7.2	2.1	6.7	1.8
2	Flavonoids	11.2	8.1	11.5	7.3
3	Saponins	4.1	1.1	3.2	1.0
4	Tannins	10.3	2.1	8.5	1.9
5	Phenols	5.6	1.7	4.2	1.5

Key: AHL-Acanthospermum hispidum leaves; AHS - Acanthospermum hispidum stem
CBL-Commelina benghalensis leaves; CBS - Commelina benghalensis stem

The quantitative phytochemical analysis (Table 3) shows that the leaves and stem extracts of AHDC contain alkaloids, flavonoids, tannins, saponins and phenols in different quantities. The presence of these phytochemical constituents, though in small amounts are indicative of the potentials of the extracts for use as corrosion inhibitors for mild steel corrosion in acidic medium.

Benali et al. [15], carried out a study on green corrosion inhibitor: inhibitive action of tannin extract of *Chamaerops humulis L* plant for the corrosion of mild steel in 0.5M H_2SO_4. The preliminary phytochemical screening result revealed the presence of tannins in the leaves (0.351%) and fruits (0.098%). Qualitative phytochemical analysis also revealed the presence of tannins, flavonoids and terpenoids in both leaves and fruits while saponins were found in leaves and steroids in fruits of *Chamaerops humulis* extract [16]. The presence of tannins in the leaves (0.351%) and fruits (0.098%) and other phytochemical substances in this plant

gave a good inhibition efficiency of 78.55% at maximum concentration of 100 mg/l. The current study has revealed that AH, AHS, CBL, and CBS extracts contain phytochemical substances with tannins in the range of 10.3%, 2.1%, 8.5% and 1.9% respectively, which is higher compared to 0.351% tannins found in *Chamaerops humulis* extract. This implies that plant materials in this current study with the same phytochemicals can be considered as potential inhibitors for mild steel corrosion.

The data in Table 3 has been presented in Fig. 1 to show at a glance the phytochemical constituents in AHL, AHS, CBL and CBS extracts. The peaks represent flavonoids and tannins which are in higher amounts, while saponins, alkaloids and phenols are present in smaller amounts in the various plant extracts.

3.1 Fourier Transform Infrared Spectroscopic (FTIR) Analysis

Fourier transform Infrared Spectroscopic analysis was also used to identify the chemical bonding and functional groups present in each of the extracts. The spectra produced for each extract were recorded and used in determining the functional groups present in the extract using peaks of typical infrared absorption frequencies in the plot of transmittance against wavenumber (cm^{-1}). The result is presented in Figs. 2 to 5. The functional groups identified using the IR Spectrum Table by Frequency Range are also presented in Tables 4 and 5.

The FTIR analysis has identified the presence of some functional groups in these extracts, notably, the O-H stretch at 3314.8 and 3324.8 cm^{-1}, C-H stretch at 2832.8, 2929.7 and 2944.5 cm^{-1}, C-O stretch at 1017.8 and 1110.7 cm^{-1}. There was a C≡C stretch at 2035.1, 2038.9 and 2099.9 cm^{-1}, C-H bend also observed at 1449.9 cm^{-1} and S=O stretch at 1408.9 cm^{-1}. The presence of these functional groups in these extracts would enhance their adsorption om the metal surface since they act as adsorption on the metal surface since they act as adsorption centers thereby increasing the inhibition potential of the extracts.

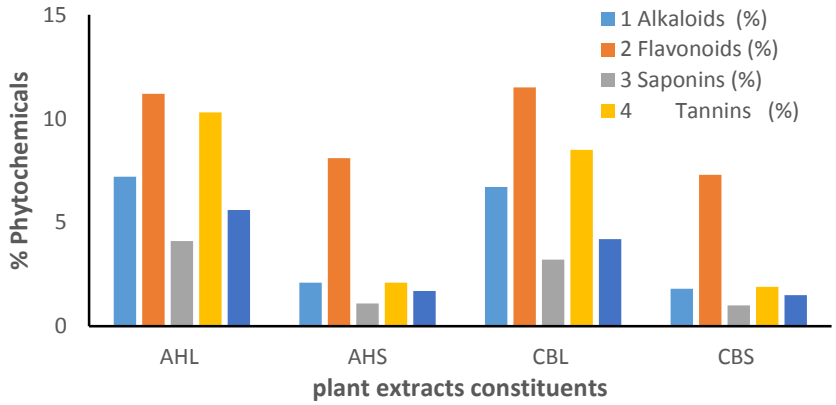

Fig. 1. Percentage composition of phytochemical constituents in AHDC and CBL extracts

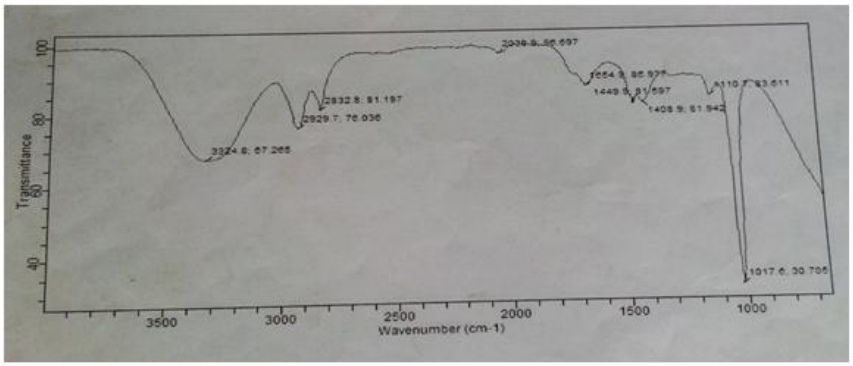

Fig. 2. Typical Infrared absorption frequencies for AHL

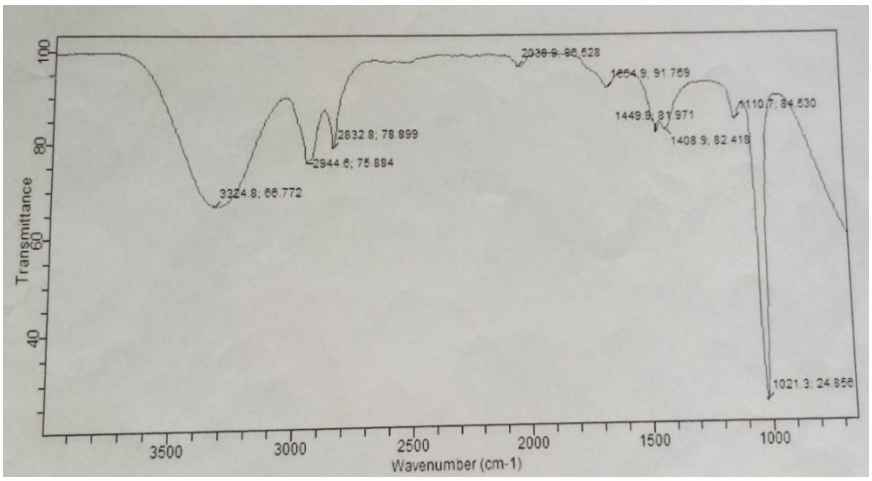

Fig. 3. Typical Infrared absorption frequencies for AHS

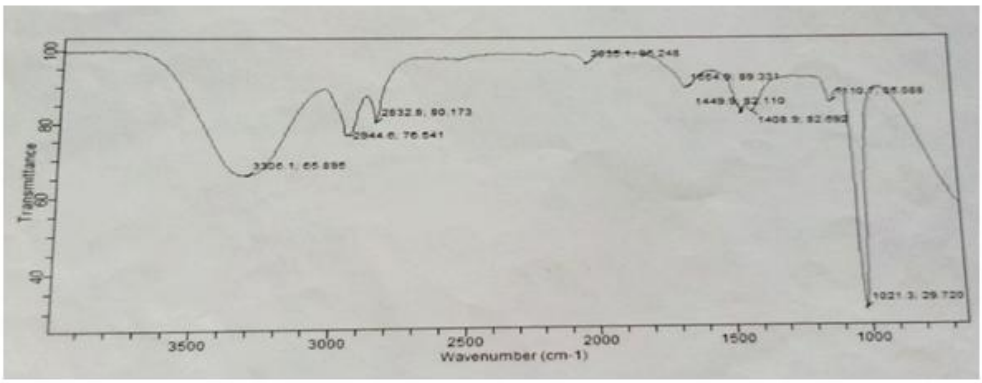

Fig. 4. Typical Infrared absorption frequencies for CBL

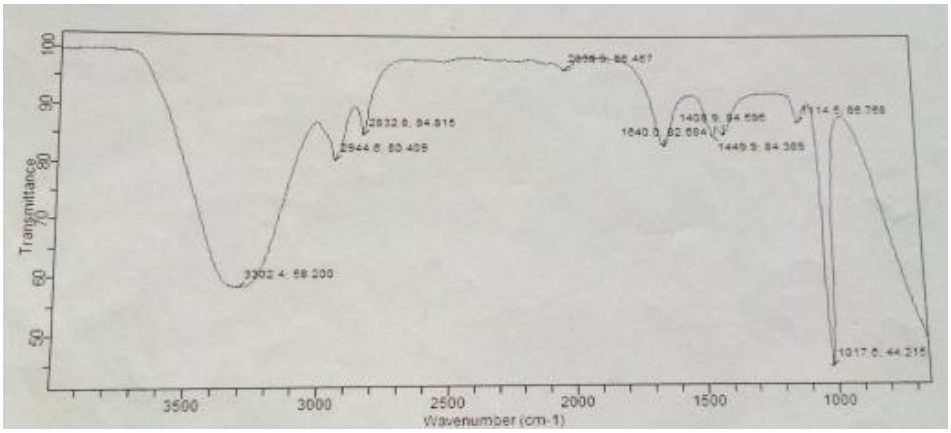

Fig. 5. Typical Infrared absorption frequencies for CBS

Table 4. Prominent peaks obtained from FTIR spectroscopy analysis for Leave and stem extracts of CBL and CBS

S/N	CBL		CBS	
	Frequency (cm^{-1})	Functional group	Frequency (cm^{-1})	Functional group
1	3306.1	O-H stretch	3302.4	O-H stretch
2	2944.6	C-H stretch	2944.6	C-H stretch
3	2832.8	C-H stretch	2832.8	C-H stretch
4	2035.1	C≡C triple bond	2099.9	C≡C triple bond
5	1654.9	C=C stretch	1640.0	C=C stretch
6	1449.9	C-H bend	1449.9	C-H bend
7	1408.9	S=O stretch	1408.9	S=O stretch
8	1110.7	C-O stretch	1114.5	C-O stretch
9	1021.3	C-O stretch	1017.6	C-O stretch

Table 5. Prominent peaks obtained from FTIR spectroscopy analysis for Leave and stem extracts of AHL and AHS

S/N	AHL		AHS	
	Frequency (cm^{-1})	Functional group	Frequency (cm^{-1})	Functional group
1	3314.8	O-H stretch	3324.8	O-H stretch
2	2929.7	C-H stretch	2944.5	C-H stretch
3	2832.8	C-H stretch	2832.8	C-H stretch
4	2038.9	C≡C triple bond	2038.9	C≡C triple bond
5	1854.9	C-O stretch	1654.9	C=C stretch
6	1449.8	C-H bend	1449.8	C-H bend
7	1408.9	S=O stretch	1408.9	S=O stretch
8	1110.7	C-O stretch	1110.7	C-O stretch
9	1017.8	C-O stretch	1021.3	C-O stretch

4. CONCLUSION

From the results of qualitative and quantitative analysis, it was concluded that these plant materials contain a wide range of natural compounds with different bioactive components. In the current study, the result has shown that the leaves and stem extracts from *Acanthospermum hispidum DC* and *Commelina benghalensis L* contain alkaloids, flavonoids, tannins, saponins and phenols. These chemicals contain atoms of oxygen(O), nitrogen (N) and sulphur (S) in their molecules which usually act as centers of adsorption on the metallic surface thereby producing a protective film that acts as a barrier separating the metal from the corrosive environment thereby protecting it from corrosion attack [17]. The analysis of these extracts using FTIR spectroscopy also identified the presence of some functional groups that could enhance their potential for use as inhibitive materials.

In conclusion, it be confidently said that these herbaceous plants, AHDC and CBL, have the potential to be used as green corrosion inhibitors as per their phytochemical analysis and IR spectra analysis [18].

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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