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Investigating prospects of *Phyllanthus muellerianus* as eco-friendly/sustainable material for reducing concrete steel-reinforcement corrosion in industrial/microbial environment

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Abstract

Characterization and experimental modelling were employed for investigating prospects of *Phyllanthus muellerianus* as eco-friendly/sustainable material for reducing concrete steel-reinforcement corrosion in industrial/microbial environment. Atomic Absorption Spectroscopy/Fourier Transform Infra Red Spectroscopy of the inorganic and organic constituent of the leaf, as well as total-corrosion effects of the leaf-extract admixture in steel-reinforced concrete were requisitely analyzed and interpreted. Results showed that *Phyllanthus muellerianus* is constituted of non-toxic level of inorganic/heavy metals, useful phytochemical constituents and essential heteroatoms that makes it potent with excellent reduction effects on the total-corrosion model of steel-reinforcement in concrete immersed in the industrial/microbial simulating-environment studied.

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1. Introduction

Deterioration of environmental-quality by the industrial and the microbial environments affects human health, the built environment and the eco-system [1-3]. For instance the built environment, for which steel-reinforced concrete is the most widely used construction material due to its relatively low cost, includes buildings, bridges, car parks and garage, towers and foundations both of wind energy structures and of electric transmission lines as well as community sewage system [4-8]. Sulphur dioxide (SO_2) emission from industrial environment [2,9-10] and hydrogen sulphide (H_2S) production by microbial activities in sewage and underground (therefore microbial) environments [3,9,11-13] are of increasing aggressiveness on the corrosivity of construction materials [14]. SO_2 from industrial environment and H_2S from microbial environments react with other environmental agents that result in their conversion to sulphuric acid [2-3,10,12,15]. The sulphuric acid, so produced, reacts with hydrated products of concrete matrix to form gypsum and ettringite that are expansive within the concrete such that the resulting hoop stress cracks the concrete and renders the reinforcing steel in the concrete susceptible to unimpeded corrosion attacks [3]. This culminates in loss of structural integrity and affects durability of steel-reinforced concrete structures and infrastructures even as it also poises safety risks to life and loss of properties, especially, when the deteriorating structure is not subjected to timely and proper management, usually, through costly repairs and maintenance [4,16]. Definitely, the quality and sustainability of the environment would be improved through solution that appropriately manage and mitigate the safety risks to the built environment by the acidic product from industrial/microbial activities.

Many methods have been studied for mitigating the menace of the acidic product from industrial/microbial environments on corrosion-deterioration of steel-reinforcement and, in effect, the steel-reinforced concrete in the built environment. Notable among these include the surface protection methods such as paintings, coatings and waterproofing membrane applications and the steel-reinforced concrete resistance improvement methods through use of admixtures and/or corrosion inhibitors [4-5,10-13,17]. Neither of these two methods is without its own setbacks/disadvantages. The surface protection method requires highly skilled expertise for appropriate design, construction and workmanship for satisfactory performance [17] and for avoiding defective waterproofing, a violation of which is known to have caused lots of damages to building structures in many countries [18]. Although, the use of admixtures and/or inhibitors is relatively economical and labour saving, compounds of chromates and nitrites that are known as effective admixtures at mitigating steel-reinforced concrete corrosion suffer the setback that they affect environmental-quality by being toxic and hazardous to the environmental ecosystem. For example, compounds of nitrites were employed as reference inhibitors for comparing performance of other admixtures in acidified environments in [10,12] while it was shown in other reported works that compounds of chromate inhibited concrete steel-rebar corrosion in industrial/microbial simulating environment [19-20]. The carcinogenicity and toxicity to living organs is increasing restrictions against usage of chromates and nitrites compounds as inhibitors in many countries [21]. By this, research studies are being directed towards search for heteroatoms, aromatic ring, π-electron or long carbon chain containing substances, which are now being identified as potent at halting both anodic and cathodic reactions and by this effectively inhibit concrete steel-reinforcement corrosion [21-22]. These conditions are generally met by organic compounds that are constituted in natural plants, which combine non-toxicity with biodegradability, renewability and ready-availability, all of which constitute properties of sustainable material.

It had been indicated in [23] that extract from the leaf of *Phyllanthus muellerianus* is non toxic to tested living organ in that study. Also, both *Phyllanthus amarus* and *Phyllanthus muellerianus* have been identified in studies [24-25] with good adsorption properties and high effectiveness performance on the inhibition of metallic corrosion in acidic media. However, in none of these were the inorganic and organic constituents characterized. Therefore, the objective of this paper was to employ characterization and experimental modelling for gaining insights into the prospects of *Phyllanthus muellerianus* as an eco-friendly/sustainable material for improving environmental-quality through reduction of concrete steel-reinforcement corrosion in industrial/microbial environment. The characterization modelling will include analyses for inorganic and organic constituents in *Phyllanthus muellerianus*. The experimental modelling will include analyses of test-results of corrosion experiment from steel-reinforced concrete samples that were admixed with *Phyllanthus muellerianus* and immersed in industrial/microbial simulating test-environment.

Nomenclature

Ctrl	Control sample without *Phyllanthus muellerianus* admixture
_Dup	Duplicate of steel-reinforced concrete sample with similar concentration of admixture with its duplicate
Tc	Total-corrosion

2. Experimental Materials and Methods

2.1. Experimental Material

Fresh leaves of *Phyllanthus muellerianus* (*P. muellerianus*) *Euphorbiaceae* that were collected from Okeho, Oyo State, Nigeria and identified at the Forestry Herbarium Ibadan (FHI), Nigeria, with the voucher FHI. No. 109496, were dried in a well aerated room maintained at 20 °C.

2.2. Experimental Methods

Inorganic (heavy metal) characterisation by the AAS (Atomic Absorption Spectrometer): dried and pulverised leaves portion of *P. muellerianus* was ashed by heating up to and maintaining, for 2hrs, at 500°C in a muffle furnace. This gave an ash content yield of 2.95%. Ashed material of 0.25g was soaked overnight in 5ml of 1:1 nitric-perchloride acid mixture and heated, thereafter, at 150°C in a reflux condenser, first for 1 hr and then the temperature was gradually raised to 235°C, from where continuous heating was maintained for another 2 hr until the occurrence of dense fumes. This was followed by removal from the heating block and cooling to 100°C before the addition of 1:1 HCl that was then followed again by heating to white fumes till obtaining colourless solution. This was poured into 100ml flask, washed five times with water, with each of the washing added to the flask in which volume was then made up with water to the 100ml mark. The filtrate from this resulting solution was then taken to the S Series, AA Spectrometer that uses Hollow Cathode Lamps of Thermo Electron Corporation®.

Characterization of organic constituent by Fourier Transform Infra Red Spectrometer: Extract from the dried *P. muellerianus* leaf was obtained through standard procedure that had been detailed in studies [24,26]. KBr pellet of the plant extract was then mounted on the Perkin-Elmer® FT-IR System, Spectrum BX®. The absorptions of organic constituents in the resulting FTIR spectra, from the Perkin-Elmer® FT-IR System, were interpreted as prescribed in [27].

Phytochemical characterization: By using the procedures described in [28-29], 2g of *P. muellerianus* leaf-extract was mixed and made up to 20ml of distilled water, for obtaining aqueous solution that was used for conducting the following phytochemical tests.

- Tannins: Boiling 0.4ml of the solution in 5ml water, filtering and adding a few drops of 0.1% $FeCl_3$ for observation of brownish-green or blue-black colouration.
- Phlobatannins: Boiling the solution in 1% HCl for observation of deposit that is red in colour.
- Saponins: Boiling 0.4ml of the solution in equal-part distilled water in a water bath, filtering, shaking the filtrate vigorously for stable persistent froth, and mixing this with 3 drops olive oil and shaking again vigorously for observation of emulsion formation.
- Flavonoids: Heating 0.4 ml of the solution with 2ml ethyl acetate over steam bath for 3 min, steeping the mixture and shaking 0.4 ml of the filtrate with 0.1ml dilute ammonia solution for observation of yellow colouration.
- Steroids: Adding 0.4ml of acetic anhydride to 0.1g of *P. muellerianus* leaf-extract with 0.4ml H_2SO_4 for observation of colour change from violet to blue or green.

- Terpenoids (Salkowski test): Mixing 1ml of solution with 0.4ml chloroform and carefully adding 0.6 ml concentrated H_2SO_4 such that a layer could be formed for observation of reddish-brown colouration.
- Glycosides (Keller-Killani test): Treating 0.5ml of solution with 0.2ml of glacial acetic acid which contains a drop of $FeCl_3$ solution and that had also been underlaid with 1ml concentrated H_2SO_4 for observation of brown ring and/or violet ring below the brown ring or green ring through the thin layer.
- Alkaloids: Adding 10ml of water to the solution from which 1ml was then taken for the addition of 2 drops of Wagner's reagent (Iodo-potassium iodide that was obtained through dissolution of 0.2g iodine and 0.6g KI in 1ml water) for observation of brown or reddish-brown precipitate.

Total corrosion test and modeling: For this, macrocell current were measured from 100mm × 100mm × 200mm steel-reinforced concrete samples, to which four variations of *P. muellerianus* leaf-extract was admixed in duplicates, during casting of the fresh concrete, and which were immersed in 0.5M H_2SO_4. The four variations of *P. muellerianus* include 0g/L (the control sample), 1.67 g/L, 3.33g/L and 6.67g/L of the concrete mixing water. Concrete formulation for these was as described in [18]. The use of 0.5M H_2SO_4 as the medium of steel-reinforced concrete immersion followed practice in reported works [4,9,11,30] for simulating microbial/industrial environment. The measurements of macrocell current, versus $Cu/CuSO_4$ reference electrode by the zero resistance ammeter [31] were carried out in five days interval for forty days and thereafter in seven days intervals for six weeks, thus totaling n = 15-point measurement in eighty-two days experimental period. The total corrosion, Tc (C), model was then obtained from the macrocell current i (A) as per ASTM G109-99a [32] through the formula:

$$Tc_j = Tc_{k-1} + \left[\left(t_k - t_{k-1}\right)\times\left(i_k + i_{k-1}\right)/2\right];\quad k = 2,\ 3,\ ...,\ n \tag{1}$$

Where: t is the time (s) elapsed between the previous, (k–1)th, and the present (kth) measurement of macrocell currents i_{k-1}, at time t_{k-1}, and i_k at time t_k .

3. Results and Discussion

3.1. AAS characterization of the leaf of P. muellerianus

Results of inorganic (heavy metal) characterization of *P. muellerianus* by the AAS technique are presented in Table 1. Subjecting the results to the Ecological Soil Screening Levels (Eco-SSLs) regulations of the United State Environmental Protection Agency (USEPA) showed that the constituents of heavy metals in *P. muellerianus* are below the Eco-SSLs, that is the concentrations of contaminants in soil that are protective of ecological receptors that commonly come into contact with and/or consume biota that live in or on soil, for plants [33]. Specifically, Eco-SSLs for plants for Ni = 38μg/g [34] compared to 29.3981μg/g, Cd = 32μg/g [35] compared to 2.4854 μg/g, Pb = 120μg/g [36] compared to 56.1165μg/g obtained in the study. The *P. muellerianus* studied does not contain chromium. In contrast to these results, the concentration of iron in the *P. muellerianus* was very high, Fe = 9904.85μg/g. This bare implication the *P. muellerianus* plant was able to absorb iron micronutrient that is needed for its growth, for the formation of chlorophyll and for the functioning of enzymes of its respiratory (photosynthesis) mechanism [37]. By these, the plant can thus be said to exhibit no toxicity, which exhibited agreements with the results of cytotoxicity from [23].

Table 1. AAS Test-results of inorganic constituents from *P. muellerianus*

Inorganic Constituent	Nickel (Ni)	Cadmium (Cd)	Lead (Pb)	Chromium (Cr)	Iron (Fe)
Concentration (μg/g)	29.3981	2.4854	56.1165	–	9904.85

3.2. FT-IR characterization of the leaf-extract of P. muellerianus

FT-IR spectra characterising *P. muellerianus* leaf-extract is shown in Figure 1. From this, the free O–H stretching vibration of alcohol/phenol is prominent at 3728.00cm^{-1} with fingerprints of O–H in-plane bending at 1330.66cm^{-1} and O–H out-of-plane bend at 725.00cm^{-1}. Alkanes of CH_3, CH_2 and CH stretching are suggested by the absorption at 2855.77cm^{-1} with fingerprints indicating CH_2 and CH_3 deformation bending vibration at 1406.98cm^{-1} as well as the potency of CH_2 rocking overlap with the O–H out-of-plane bending at 725.00cm^{-1}. The 2350.71cm^{-1} absorption suggests S–H thiols while the 1156.70cm^{-1} absorption suggests C=S thiocarbonyl stretching vibrations. Acyl halide is implied by the C=O stretching absorption, suggesting presence of chloride or bromide, of carboxylic acid derivative at 1803.37cm^{-1} with fingerprint of C–O–H bending at 1406.98cm^{-1} while the 1688.00cm^{-1} absorption suggests C=O of amides with N–H fingerprint at the 1559.29cm^{-1} absorption. By this, the absorption at 1491.78cm^{-1} exhibited fingerprint potency of an amide bending vibration with extended conjugation that is known to usually lower absorption frequency. The inferences of carboxylic acid derivatives find confirmation from the 2 bands at 1002.78cm^{-1} and 911.64cm^{-1} indicating O–C stretching of the carboxylic acid/derivative group.

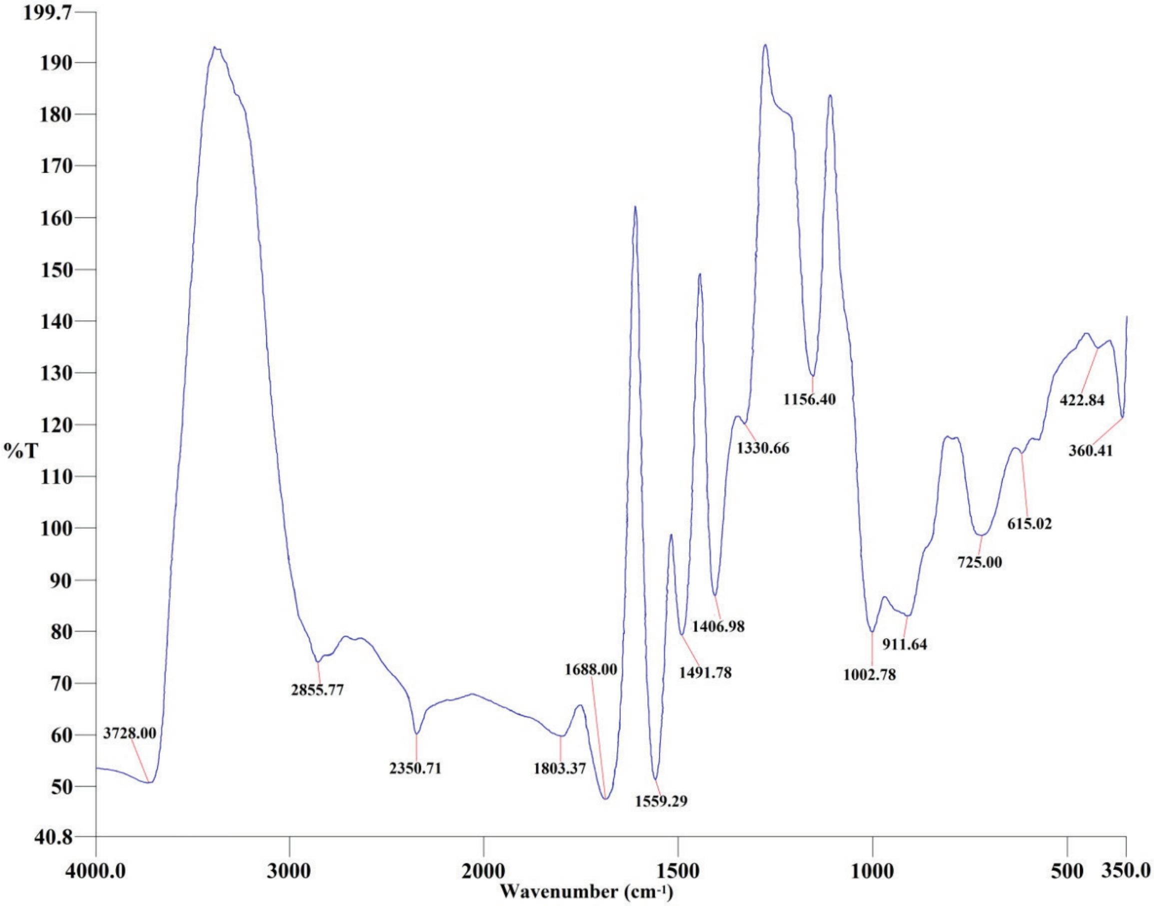

Figure 1. FT-IR spectra of the leaf-extract of *P. muellerianus*

It could be deduced from these interpretations of the FTIR spectra of *P. muellerianus* leaf-extract, that the natural plant is constituted of organic S–, N– and O– containing heteroatoms with lone pairs and π-electrons. It is worth noting also that plants that had been known to constitute these types of organic constituent had been identified in literature with good corrosion protection of metals in aggressive media.

3.3. Phytochemical analyses of the leaf-extract of *P. muellerianus*

Results of phytochemical analyses of the leaf-extract of *P. muellerianus* are presented in Table 2. It could be observed from the table that the leaf-extract of *P. muellerianus* was found to contain six out of the eight phytochemical constituents tested. Tannins, phlobatanins, saponins, flavooids, terpenoids and alkanoids are contained in *P. muellerianus* leaf-extract while it does not contain glycosides and steroids. While it is worth noting that plants containing these types of phyto-constituents have been identified with potency of protecting metals from corrosion in studies, the absence of glycosides in the *P. muellerianus* also positively enhances its eco-friendliness because glycosides had been reported to exhibit potency of toxicity in [37].

Table 2. Phytochemical constituent of *P. muellerianus*[*]

Tannins	Phlobatannins	Saponins	Glycosides	Flavonoids	Stenoids	Terpenoids	Alkaloids
+	+	+	–	+	–	+	+

* Present ≡ +; Absent ≡ –

3.4. Effect of *P.muellerianus* leaf-extract effect on the total corrosion of concrete steel-reinforcement

Total-corrosion effects of the different concentrations of *P. muellerianus* leaf-extract admixtures in duplicates of steel-reinforced concrete samples immersed in the H_2SO_4 medium are presented in Figure 2.

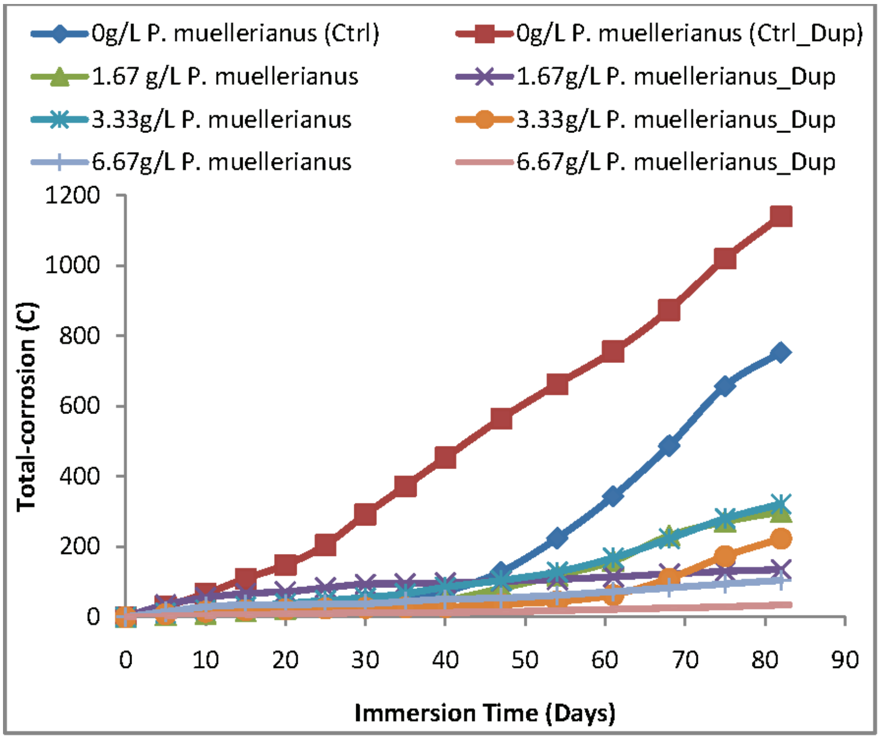

Figure 2. Plots of total-corrosion effects of *P. muellerianus* leaf-extract on concrete steel-reinforcement in industrial/microbial simulating medium

The figure showed that the total-corrosion was increasing in the control (Ctrl) samples with increasing number of immersion days while it could also be observed that the total-corrosion generally decrease with increasing concentration of *P. muellerianus* admixture. The most reduction in total-corrosion in the study could be noted from the duplicates of 6.67 g/L *P. muellerianus* admixed steel-reinforced concretes. In numerical terms, total-corrosion of 1140.48C by the control duplicate or of 753.54C by the control sample was mitigated to 104.69C or 34.28C respectively by the 6.67 g/L *P. muellerianus* admixed steel-reinforced concrete and its duplicate sample, in the tested corrosive environment. This support the indication that the essential non-toxic inorganic and organic constituents of *P. muellerianus* find usefulness in the corrosion protection of the steel-rebar embedment in the steel-reinforced concrete samples immersed in the acidic test-medium employed. This form of effective mitigation of reinforcing steel corrosion will definitely improve durability of steel-reinforced concrete designed for the industrial/microbial environment. The results of total-corrosion analyses and the non-toxicity of *P. muellerianus* therefore bear confirmations of the highly positive prospects and suitability of this natural plant as an eco-friendly and sustainable material for reducing corrosion of steel-reinforcement in concrete immersed in industrial/microbial environment. Based on this, further studies are recommended for investigating suitability of this eco-friendly, renewable and sustainable plant material for corrosion protection of reinforcing steel and other structural metals in other corrosive environments.

4. Conclusions

Study of inorganic (heavy metal) micro-constituents, using AAS, and the organic constituents by the FTIR and phytochemical analyses, exhibited agreements on the non-toxicity and environmentally-friendly potencies of *P. muellerianus* with the prospects of the suitability of the extract from the plant for metallic corrosion protection. The prospects find confirmation from this study based on the consideration that extract from the leaf of *P. muellerianus* mitigated total-corrosion of concrete steel-reinforcement when used as admixture in steel-reinforced concrete samples immersed in 0.5M H_2SO_4 medium, for simulating industrial/microbial environment. Further study is therefore recommended for investigating suitability of this eco-friendly, renewable and sustainable plant material for corrosion protection of steel-reinforcement and other structural metals in other types of corrosive environments.

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