#### **RELEVANCE OF SURFACE CHARACTERIZATION OF** *BAMBUSA VULGARIS* **(L.) LEAF EXTRACT DOPED IRON NANOPARTICLE**

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## **ABSTRACT**

*In the present study, green nano scaled zero-valent irons (nZVI) were synthesized from Bambusa vulgaris leaf extract at room temperature. The plant extract was evaluated for the presence of reducing agents (bioactive compounds) phytochemically using standard methods. nZVI was systematically characterized using Scanning Electron Microscopy (SEM), X-Ray Diffraction (XRD), Ultraviolet-Visible (UV-Vis), and Fourier Transform Infra-Red (FT-IR) techniques. The SEM and XRD results showed that after the reduction process for 4 hr at 25<sup>o</sup>C, the iron powder obtained was a single phase of lamellar or platy α-Fe crystals with the presence of medium nanoparticles (10-100 nm) having crystal size, L ranging from 40-46 nm (42.2 nm) and particle size, D ranging from 53-70 nm (56.1 nm) for both 0.3 M and 0.9 M B. vulgaris-nZVI. It has been demonstrated that B. vulgaris leaf extract was capable of producing iron nanoparticles that shows good stability in solution and under the UV-Visible wavelength, nanoparticles exhibited quite good surface plasmon resonance behavior in the range of 220-740 nm. The FT-1R spectra revealed that the vibrational bands at 3722.76 cm-1 and 3706.4 cm-1 represented the O-H stretching due to the presence of polyphenol as a functional group in addition to tannins, saponins, and others which may be responsible for the reduction of ferrous ion (Fe<sup>3+</sup>) to zero-valent iron (Fe<sup>o</sup>). This study reveals that B. vulgaris-nZVI possessing short-time synthesis protocol, anisotropism, and eco-benignity could be promising lowcost matrix for remediation (both soil and water), adsorption, catalysis, and other applications.*

**Keywords:** Green synthesis, phytochemical, morphology, extract, particle size, zero-valent iron

#### **INTRODUCTION**

Nanoscience popularly referred to as nanotechnology since the advent of Industrial Revolution in the  $18<sup>th</sup>$  century has made a tremendous impact on material makeup and accounting. The rapid change in dimension of materials from milli, kilo, deci, etc due to the advancement of science and technology to nanoscale has brought progressive turnout in quality, composition, handling, and costeffectiveness (Saif *et al*., 2016; Bhateria *et al*., 2021; Anju *et al*., 2021; Aguomba *et al*., 2022; Vijayaram *et al*., 2024).

The products of nanotechnology are many and varied emanating either from bottom-up (building up atoms and molecules into nanoscale) or top-down (breaking down of larger molecules) approaches and they include nanoparticles, nanorods, or nanotubes with different dimensions. All these nano-sized products have different specific roles. They may vary according to their size and shape, chemical nature, and crystalline, amorphous, solid-state of occurrence, etc (Karunakaran *et al*., 2018; Batool *et al*., 2021).

The limitless synthesis of nanomaterials has found relevance in diverse fields such as catalysis, electronics, drug delivery, monitoring, treatment of environmental pollution, etc (Bonaiti *et al*., 2017; Hu *et al*., 2020; Chen *et al*., 2020; Liu *et al*., 2020; Wang *et al*., 2020; Nkosinathi *et al*., 2020).

Nanomaterials have shown major importance in environmental pollution remediation, especially in the field of soil and water pollution since the remediation of soil, wastewater and groundwater, and the availability of potable water is a critical environmental problem faced by several countries (Majumder *et al*., 2019; Mondal *et al*., 2020; Oladotun *et al*., 2020; Aguomba *et al*., 2022).

There are different ways of synthesis of nanomaterials but green synthesis however is an alternative to the physical and chemical methods. The green synthesis route provides an Eco-friendly and better economical way to synthesize nanoparticles. It can be achieved using yeasts, molds, algae, plants, etc or their products (Pattanayak and Nayak, 2013; Saif *et al*., 2016; Nadaroglu *et al*., 2017; Ayomide *et al*., 2023). The molecules of these plant materials facilitate nanoparticle synthesis by reduction. However, the synthetic process of green metallic nanoparticles is initiated by adding plant extracts to aqueous solutions of metal ions. These plant extracts could be from leaves, roots, corms, stems, fruits, etc. The bioactive compounds present in these plants such as sugar, polyphenols, flavonoids, polymers, enzymes, etc act as reducing agents (stabilizing/capping agents) to produce the desired nanoparticles (Nadaroglu *et al*., 2017; Lakshminarayanan *et al*., 2021).

Bamboo leaves belong to the subfamily of Bambusoideae within the grass family Poaceae and are botanically referred to

*Bambusa vulgaris*. It is well-known for its evergreen nature and rapid perennial growth with certain species capable of extending to 91 meters. Bamboo leaves are integral to its identity, not just as a physical characteristic but as a vital component of its growth and environmental role (Figure 1). In Nigeria, Yoruba tribe call it Oparun, Bine tribe refer to it as Iko while Ibo tribe call it Atosi. It is found in tropical and subtropical areas, especially in the monsoon and wet tropics (Yakubu *et al*., 2014).

The surface characterization of bamboo leaves doped nanoparticle is crucial in nanotechnology because of its enhanced hydrophobicity, antimicrobial properties, mechanical properties, drug delivery, biocompatibility, optical properties, catalytic activities, reduced toxicity, dispersion and stability, etc. By understanding the surface properties of bamboo leaves doped nanoparticles, researchers can tailor their characteristics for specific applications, unlocking their full potential in nanotechnology. The nanoparticles crystalline structures, functional groups and vibrational bands, surface morphology, particle size, basal spacing can be evaluated by using the techniques of X-Ray Diffraction (XRD), Fourier Infra-red Transform (FTIR), Scanning Electron Microscopy (SEM), particle ionizer, Transmission Electron Microscopy (TEM), etc (Pattanayak and Nayak, 2013; Shakeel *et al*., 2016; Lakshminarayanan *et al*., 2021; Asghar *et al*., 2023; Mohamed *et al*., 2023; Vijayaram *et al*., 2024).

The metallic nanoparticle of interest in this study is zero-valent iron. Zero-valent iron nanoparticles (nZVI) are the tiniest particle of iron metal with a large surface area and high reactivity (Ayomide *et al*., 2023). They are non-toxic. nZVI have excellent dimensional stability and also possess high thermal and electrical conductivity, high surface area, and are highly magnetic (Batool *et al*., 2021). nZVI can oxidize immediately when exposed to water or air and produces free Fe ions for reduction forming iron oxide/hydroxide

shell/core. This core-shell structure is the major reason for the adsorption and reduction of nZVI (Anju *et al*., 2021). It is worthy to note that different heavy metals possess different mechanism but ZVI nanoparticles mechanism occurs due to its difference in standard electrode potential of −0.44 V. However, reductive precipitation occurs in metals with much higher standard electrode potential than nZVI. In addition, sorption and partial chemical reduction occur in metals which have slightly more positive standard electrode potential than ZVI. More so, sorption or surface complexation occurs in metals with a more negative standard electrode potential than nZVI (Anju *et al*., 2021).

Generally, the removal or degradation or remediation mechanism by ZVI nanoparticles depends on various parameters such as type of pollutant (inorganic or organic), presence of oxygen, pH, temperature, etc (Kuang *et al*., 2013; Huang *et al*., 2014). There are numerous applications of nZVI (Ayomide *et al*., 2022; Aguomba *et al*., 2022).

nZVI particles synthesized from the plant extracts such as *Rosa damascene* (RD), *Thymus vulgaris* (TV), and *Urtica dioica* (UD) for the removal of Cr (VI) from aqueous solution showed high removal efficiency of 90% at a time of 10 mins (Fazlzadeh *et al*., 2017). The study also highlighted that the removal efficiency was dependent on the bioactive components of the plant extracts. Similar study by Mohamed *et al*. (2023) showed that adsorption of  $Cd^{2+}$  and  $Ni^{2+}$  on the iron oxide surface was found to be heterogeneous, and the mechanism of chemisorption is involved in the stage of determining the rate.

A novel green synthesized ZVI nanoparticles were used to activate persulphate and subsequently used for the oxidation of metronidazole (MTZ) and the results obtained showed a good effect on the activation of the PS and the PS/nZVI process had a high ability

in degradation of MTZ from aqueous solutions (Asghar *et al*., 2023).

Currently, ZVI nanoparticles have been found useful in biotechnological applications. Mohamed *et al*. (2023) and many other researchers have observed that the iron nanoparticles exhibit antimicrobial and anticancerous properties and their mechanism adversely affects cell viability, division, and metabolic activity (Nkosinathi *et al*., 2020; Mohamed *et al*., 2023; Vinod *et al*., 2023).

In the light of establishing more effective and efficient plant mediated zero valent iron nanoparticle with respect to stability, surface morphology, reactivity, functionality, etc, this study has embarked on the characterization of *Bambusa vulgaris* L leaf extract doped zero valent iron nanoparticle using Fourier Transform Infra-red (FTIR), Ultra-Violet (UV), X-Ray Diffraction (XRD), and Scanning Electron Microscopic (SEM) techniques.

# **MATERIALS AND METHOD**

# **Reagents and Chemicals**

Analar grade of 0.3 and 0.9 M iron sulphate (FeSO4.7H2O) obtained was used as salt. Freshly prepared triple distilled water was used throughout the experiment.

## **Preparation of Extracts**

Bamboo leaf (Figure 1) were collected from the local region of Elele town in Rivers State Nigeria. They were washed and cleaned with triple distilled water and dried with water absorbent paper. Then it was cut into small pieces with an ethanol sterilized knife and crushed with mortar and pestle dispensed in 10 ml of sterile distilled water and soaked overnight at room temperature. The extract was then filtered using Whatman'sNo.1 filter paper. The filtrate was collected in a clean and dried conical flask by standard sterilized filtration method and was stored for further characterization and synthesis.

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**Figure 1.** *Bambusa vulgaris* leaf

# **Phytochemical Test on the Extract**

Preliminary phytochemical test was performed on the *Bambusa vulgaris* leaf extract to identify the presence of various phytochemicals such as polyphenol, steroids, tannins, flavonoids, and saponins, using standard methods such as those defined by Harbone and Baxter (1993), AOAC (1990) and Sofowora (1993).

# **Green Synthesis of Zero-Valent Iron Nanoparticles**

During the synthesis of iron nanoparticles both the precursor (salt) and the reducing agent (*B. vulgaris* leaf extract) were mixed in a clean sterilized flask in 1:9 ratio (Ayomide *et al*., 2022) and the pH of the mixture was taken. For the reduction of Fe ions, 20 ml of filtered *B. vulgaris* leaf extract was mixed to 180 ml of freshly prepared 0.3 M aqueous of FeSO<sup>4</sup> solution with constant stirring at  $25^{\circ}$ C for 4 hr. Within that period of time, change in colour occurred from light green to dark green indicating the presence of nanoparticle and the pH of the product was also taken. This process was repeated for 0.9 M FeSO<sub>4</sub>. The mixture was incubated at  $60^{\circ}$ C for 30 min and later oven-dried at  $160^{\circ}$ C for 9 hr to obtain a solid product (Plate 1).

# **CHARACTERIZATION**

# **X-Ray Diffraction Analysis**

The sample undergoes size-reduction processes to meet a  $\sim$ 10-μm particle size fraction. Once in powdered form, the sample was packed into a sample tray and analyzed by XRD. Samples prepared were scanned with a Scintag® XDS2000 X-Ray diffractometer using CuKα radiation at 40 kV and 30 mA. The majority of the scans were performed using a continuous scan mode from 2 to 34° 2θ with a 0.02 step size at 2 degrees per min. The collected data were then analyzed using Jade 9+® software. To conclude the process, the results were assembled into easy-to-read spreadsheets, and the XRD trace for each sample was put into the form of a .jpg image. The crystal size, L and the particle size, D was determined using equations 1 and 2 as:

$$
L = \frac{K\lambda}{\beta \text{Cos}\theta} \tag{1}
$$

Where K = Shape factor (0.5),  $\lambda = 1.54 \text{Å}, \beta =$ Broadening dimension (0.02) and  $\theta$  = Bragg's angle.

$$
D = \frac{4}{3} \times L \tag{2}
$$

# **Fourier Transform Infra-Red Analysis**

A 0.5 g of each of the sample was mixed with 0.5 g of Kbr after which 1 ml of Nujol (a solvent for preparation of sample by Buck

M530IR-Spectrophotometer) was introduced into the sample with aid of a syringe to form a paste before introducing it into the instrument sample mold and allowed to scan at a wavelength of  $600-4000$  cm<sup>-1</sup> to obtain its spectra wavelength.

# **Ultraviolet-Visible Spectra Analysis**

The reduction of pure Fe ions to zero-valent (Fe<sup>o</sup>) was monitored by measuring the UV-Vis spectrum by sampling of aliquots (0.3 ml) of Fe Nanoparticle solution diluting the sample in 3 ml distilled water. UV-Vis spectral analysis was done by using UV-Vis spectrophotometer Systronics 118 at the range of 200-600 nm.

# **Scanning Electron Microscopy**

SEM was performed to examine the physical structure change of samples using SEM model PhenomProX, by phenom World Einhoven, Netherland. The sample was placed on double adhesive which was on a sample stub coated with sputter coater by quorum technologies model Q150R with 5 nm of gold. Thereafter it was taken to the chamber of SEM-EDX machine where it was viewed via NaVCaM for focusing and little adjustment, it was then transferred to SEM mode focused and brightness contrasting was automatically adjusted, afterward the morphologies of different magnification obtained.

# **RESULTS AND DISCUSSION**

# **Phytochemical Test**

*B. vulgaris* leaf extract was used to produce zero-valent iron nanoparticles in this study. The extract revealed the presence of polyphenol, tannins, saponins, and anthrone as presented in Table 1. The  $Fe<sup>3+</sup>$  ions were reduced into zero-valent (Fe<sup>o</sup>) nanoparticles when plant extract was mixed withFeSO<sub>4</sub> solution in 1:9 ratio. Reduction was followed by a change in colour from Light Green to Dark Green and change in pH of the solution was observed from 6.2 to 3.5 as shown in Tables 2 and 3. It is well known that iron sulphate exhibit bright yellowish colour in distilled water. On mixing the plant extract with the aqueous FeSO<sub>4</sub> solution it changed the colour of the solution immediately and reducing the pH, which may be an indication of formation iron nanoparticles.



**Table 1:** Bioactive compounds in *B. vulgaris* leaf extract

+ indicates presence while – indicates absence

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|   | <b>Colour change</b>             |                         |                        |                            |           |
|---|----------------------------------|-------------------------|------------------------|----------------------------|-----------|
| S/N   | <b>Solution</b>                  | <b>Before reduction</b> | <b>After reduction</b> | <b>Colour</b><br>intensity | Time (hr) |
|   | B. <i>vulgaris</i> -nZVI extract | Light yellow            | Dark green             | $+++$                      | 14        |
| $\bigcirc$  | $0.3 \& 0.9 M FeSO4$             | Light green             | Dark green             | $+++$                      | 14        |
| Colour intensity: $+$ = Light colour, $++$ = Dark colour, $++$ = Very dark colour |                                  |                         |                        |                            |           |



**Table 3:** Change in pH during iron nanoparticle synthesis



Result:  $+$  = Positive,  $-$  = Negative.



**(a) (b)**

**Plate 1:(a)** *B. vulgaris* extract before synthesis **(b)** *B. vulgaris*-nZVI

# **SEM Result**

The micrograph in Figures 2 and 3 revealed a uniform and non-uniform particle sizes and void space. The particles were discovered to be irregular in form, with sizes varying from 54 to 70 nm, with an average particle size of 56.1 nm. However, XRD results reveal the crystals formed to be anisotropic and lamellar or platy in shape since the shape factor (K) was 0.5. The findings were found to be similar to those of Kowshik *et al*. (2002); Sondia (2004); Kumar *et al*. (2015); Sravanthi *et al*. (2018); and Nhung *et al*., (2018).



**Figure 2:** SEM of 0.3 M Fe nanoparticle





# **XRD results of** *B. vulgaris***-nZVI**

The X-Ray diffractogram in Figures 4 and 5 revealed that the peak at  $2\Theta$  equals to  $45.2^{\circ}$  indicates the presence of zero-valent iron (Fe<sup>o</sup>) in *B. vulgaris*-nZVI. The results of the XRD study of *B. vulgaris* nZVI at  $2\Theta = 36.2^{\circ}$  peak revealed the presence of Fe<sub>2</sub>O<sub>3</sub> or Fe<sub>3</sub>O<sub>4</sub> in *B. vulgaris*-nZVI. However, other peaks may indicate iron oxyhydroxides and organic matter on the surface of *B. vulgaris*-nZVI. The result obtained was found to be very similar to those of Sun *et al*., 2006; Singh *et al*. (2011); Khasim *et al*. (2011); Saif *et al*., 2016; Saranya *et al*. (2017); Qayoom *et al*., 2020; Ayomide *et al*. (2022); Eldeeb *et al*., 2023 and Mohamed *et al*., 2023. The XRD result further revealed the presence of medium nanoparticles (10-100 nm) having crystal size, L ranging from 40- 46 nm (42.2 nm) and particle size, D ranging from 53-70 nm (56.1 nm) for both 0.3 M and 0.9 M *B. vulgaris*-nZVI (Nhung *et al*., 2018). This has shown that *B. vulgaris*-nZVI could be promising matrix for remediation (both soil and water), adsorption, catalysis, and other applications.



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**Figure 4:** XRD pattern of 0.3 M Fe nanoparticle



**Figure 5:** XRD pattern of 0.9 M Fe nanoparticle

#### **FT-IR Analysis**

FTIR spectroscopy was adopted to identify the presence of functional groups responsible for the synthesis of nZVI from an aqueous extract of *B. vulgaris* leaf. The absorbance bands of nZVI was in the range of wave region between  $600$  and  $4000$  cm<sup>-1</sup>as presented in Figures 6 and 7. The FT-IR spectra displayed four (4) distinct characteristics peaks at 1513.45, 1738.43, 2400.9 and 3722.76 cm-1 for 0.3 M *B. vulgaris*-nZVI while 1155.29, 1532.14, 2383.65 and 3706.4 cm-1 for 0.9 M *B. vulgaris*-nZVI. The vibrational band at 3722.76 cm<sup>-1</sup> and 3706.4 cm<sup>-1</sup> represented the O-H stretching due to the presence of polyphenol or alcohol as functional group in addition to tannins, saponins, and others which may be responsible for the reduction of ferrous ion  $(Fe<sup>3+</sup>)$  to zero-valent iron (Fe<sup>o</sup>). The peaks at 2383.65 and 2400.9cm<sup>-1</sup> confirmed the C-H stretching due to the alkane functional group. The band at  $1513.45$ ,  $1155.29$ ,  $1532.14$  and  $1738.43$  cm<sup>-1</sup> represent bending

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vibration of secondary N-H, C-O stretching, C=C stretching due alkenes, and C=O stretching due carbonyl group respectively. However, the FT-IR results showed varied vibrational bands from 696-844 cm<sup>-1</sup>, 1033-1856 cm<sup>-1</sup>, 2040-2975 cm<sup>-1</sup> and 3014-3836 cm<sup>-1</sup> representing C=C, C-O, C-N, N-H, C=O, C-H, and OH functional groups which played vital role in the synthesis of nZVI. The findings in this study were very close to those of Monalisa *et al*. (2013) and Adeyemi *et al*. (2020). The following works such as biologically synthesized iron nanoparticles (FeNPs) from *Phoenix dactylifera*, green synthesis of iron nanoparticles and their environmental applications and implications, green synthesis of iron nanoparticles using aqueous extract of *Musa ornata* flower sheath against pathogenic bacteria, etc revealed similar FT-IR pattern (Reichenbächer and Popp, 2012; Saif *et al*., 2016; Saranya *et al*., 2017; Batool *et al.* 2021; Eldeeb *et al*., 2023). The existence of phytochemicals such as polyphenol, tannins, saponins, and others as supported by the occurrence of related frequency bands or peaks in the FT-IR spectra in the *B. vulgaris* leaf extract were responsible for reducing effect on the nanoparticles (Sorbiun *et al*., 2018).



**Figure 6:** FT-IR spectrum of 0.3 M Fe nanoparticle



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**Figure 7:** FT-IR spectrum of 0.9 M Fe nanoparticle

#### **UV-Vis Result**

It was observed that the absorption peaks at 220-740 nm regions in Figures 8 and 9 were due to the excitation of surface plasmon vibrations in the nZVI solution, which were identical to the characteristics UV-visible spectrum of nZVI (Batool *et al*., 2021).



**Figure 8:** UV spectrum of 0.3 M Fe nanoparticle



**Figure 9:** UV spectrum of 0.9 M Fe nanoparticle

## **CONCLUSION**

Zero valent iron nanoparticles were produced successfully from ferrous precursor by means of low temperature reduction. Characterizations of prepared iron powder were investigated by modern analysis methods. The results showed that after the reduction process for 4 hr at  $25^{\circ}$ C, the iron powder obtained was single phase of lamellar or platy α-Fe crystals with a particle diameter concentrated on 41.4 nm for (about 100 %) and possesses an average size of 56.1 nm.It has been demonstrated that *B. vulgaris* leaf extract was capable of producing iron nanoparticles that show good stability in solution and under the UV-Visible wavelength, nanoparticles shown quiet good surface plasmon resonance behavior in the range of 220-740 nm. Ferrous sulphate with reducing agent i.e. *B. vulgaris* leaf extract has shown a remarkable color change with reduced change in pH of solution. The Fourier Transform Infra-Red spectra revealed that the vibrational bands at  $3722.76$  cm<sup>-1</sup> and  $3706.4$  cm<sup>-1</sup> represented the O-H stretching due to the presence of polyphenol as functional group in addition to tannins, saponins, and others which may be responsible for the reduction of ferrous ion  $(Fe^{3+})$  to zero-valent iron (Fe<sup>o</sup>). This study reveals that *B. vulgaris*- nZVI possessing short-time synthesis protocol, anisotropism, and eco-benignity could be promising low-cost matrix for remediation (both soil and water), adsorption, catalysis, and other applications. This study recommends further synthesis using variations of amounts of different salts in synergy and characterization using Transmission Electron Microscopy (TEM).

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## **Conflict of Interest**

The authors declare non-existent of any academic or financial conflict.

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