Synthesis and characterization of Silicon Oxide Nanoparticles using Plantain Peel as a Precursor

Nsikak Bassey Essien, Chukwu Obaji Daniel, Raphael, Mmenyene Paul and Ukeme Ekop

Abstract: This study explores the synthesis and characterization of silicon oxide nanoparticles (SiONPs) using plantain peels as a sustainable precursor. SiONPs hold immense promise in various fields due to their exceptional attributes, and there is a growing need for eco-friendly synthesis methods. The research addresses this need by using agricultural waste materials, specifically plantain peels, as a renewable and cost-effective source for SiONP production. In the study, plantain peel samples were dried, ashed and converted to Si(OH)$_2$ after a series of reactions with HCl and NaOH respectively. The Si(OH)$_2$ obtained was calcined at 700 °C for two hours. The silicon oxide nanoparticles obtained were characterized with an ultraviolet-visible spectrophotometer, X-ray diffractometer and nitrogen adsorption study based on Brunaer-Emmett-Teller as well as other models. The results obtained showed that the XRD spectrum indicated a principal peak at 69, which was attributed to Si(111) or Si(40). The crystallite size of the silicon oxide nanoparticles obtained from the plantain peels was 0.23 nm while the evaluated particle size was 3.012 nm, confirming a mesoporous dimension. The absorption peaks obtained from the ultraviolet-visible analysis indicated a wavelength of maximum absorption at 342 nm and a corresponding bandgap of 3.6 eV. The materials are regarded as a highly porous semiconductors with unique potentials for environmental, optical, electrical and other applications. These results collectively highlight the eco-friendly synthesis and versatile applications of SiONPs, emphasizing their significance in advancing nanotechnology across diverse industries and scientific disciplines.
Keywords: Nanoparticles, precursor, plantain peels, production, properties

Nsikak Bassey Essien*
Department of Chemical Science, Akwa Ibom State Polytechnic, Ikot Osurua, P. M. 10…
Ikot Ekpene, Akwa Ibom State, Nigeria
Email:
nsikak.essien@akwaibompoly.edu.ng.
Orcid id:0000-0002-6664-3274

Chukwu Obaji Daniel
Department of Science Laboratory Technology, University of Nigeria, Nsukka, Enugu State, Nigeria
Email: daniel.chukwu.246878@unn.edu.ng

Raphael Mmenyene Paul
Department of Chemical Science, Akwa Ibom State Polytechnic, Ikot Osurua, P. M. 10…
Ikot Ekpene, Akwa Ibom State, Nigeria
Email:
Raphael.mmenyene@akwaibompoly.edu.ng

Ukeme Ekop
Department of Chemical Science, Akwa Ibom State Polytechnic, Ikot Osurua, P. M. 10…
Ikot Ekpene, Akwa Ibom State, Nigeria
Email:

1.0   Introduction

The applications of metal and non-metal oxide nanoparticles in various aspects that affect life have been widely reported in the literature because of their unique properties (Eddy and Garg, 2021; Garg *et al*., 2023). Nanoparticles have extraordinary properties that guarantee

their applications for various purposes such as environmental remediation, medical, firefighting, catalysis, and fabrication of improved materials, among others (Albertini *et al.*, 2024; Meijerink , 2024; Pajor-Świerzy *et al.*, 2023; Su *et al.*, 2023; Tapia *et al.,* 2024). Notable properties of nanoparticles are associated with their porosity, particle size, crystallite size, electrical conductivity, optical properties, thermal stability, mechanical stability, reusability, etc (Eddy *et al.*, 2024 a-b; Eddy *et al.*, 2023a-c). Given the listed features of metal and non-metal oxide nanoparticles, the commonest ones in the literature concerning synthesis are metal oxide nanoparticles such as CaO, ZnO, MgO, CuO, TiO_2 and MnO nanoparticles. Non-metal and metalloids can also form oxides nanoparticles, notably, graphene oxide and silicon oxide nanoparticles (Chavali and Nikolova, 2019; Naseem and Durrani, 2021; Negrescu *et al.*, 2022).

Silicon nanoparticles have been identified as one of the most useful nanoparticles especially in biochemical applications, catalysis, energy conversion, environmental remediation, gas storage, etc (Bai *et* al., 2023; Owusu *et al.*, 2023). Several literature is in support of the application of SiO_2 nanoparticles as a widely useful material because of some outstanding properties such as crystalline: amorphous character, porosity, large surface area, tunable bandgap, etc (Adach *et al.*, 2021; Roustaei and Bagherzadeh, 2015; Garg *et al.*, 2022). However, the basic challenges facing the production and application of silicon oxide nanoparticles may be toxicity arising from synthetic routes relying on toxic chemicals (Eddy *et al.*, 2023d).

2.0   Materials and Methods
2.1   *Sample collection and synthesis*

Plantain peels were obtained as a waste product after the edible portions were removed. They were washed severally with distilled water and sun-dried to constant weight. The dried samples were grounded to a powder form and re-dried to a constant weight in an oven set at 105 °C. 100 g of the powder sample was mixed with 2 M HCl, stirred and allowed to stand for 24 hours. After 24 hours, the sample was sieved, dried and made to react with 20% NaOH to produce silicon hydroxide. The by-product (NaCl) was removed by washing with water. The dried hydroxide was finally calcined at 800 °C for two hours.

2.2   Characterization

Shimadzu UV visible spectrophotometer. FTIR instrument (Agilent infrared spectrophotometer, Carry 630 FTIR spectrometer) was also employed to obtain the infrared absorption spectrum of the SiO_2 -NP. XRD Model Nr. ARLXTRA-Xray-XRD was used for crystallography investigation while Brunaeur-Emmett-Teller machine (Nova4200e made in USA made in Japan) was used for particle size determination.

3.0   Results and Discussion
3.1 Absorption spectrum

The UV visible absorption spectrum of the silicon oxide nanoparticles (SiONPs) synthesized from plantain peel is shown in Fig. 1. The spectrum reveals maximum absorption within the ultraviolet region, which corresponds to a peak value of 341 nm. The observed absorption maxima for the SiONPs are in agreement with the ranges of values reported in literature values such as 259 nm (Saravanan and Dubey, 2020; Nimah *et al.*, 2023). Also, a x_{max} of 297 nm has been reported by Biradar *et al.* (2021) for SiONPs and 485 nm by Intartaglia *et al.* (2012).

The wavelength of maximum absorption is a significant parameter in identifying a compound and in the calculation of the bandgap of the nanomaterials. The calculation of the bandgap can be achieved through Planck's or Tauc's equations. For the synthesized SiONPs, the bandgap was evaluated using Planck's equation represented by equation 1 (Ogoko *et al.*, 2023)

$$E_{BG} = \frac{hc}{x_{max}} \qquad\qquad (1)$$

where h is the Planck constant and c is the speed of light. The insertion of the numerical constants and x_{max} into equation 4.1 led to the evaluation of the band gap of the nanoparticles as follows

$$E_{BG} = \frac{6.63 \times 10^{-34} \, J/Hz \times 3.0 \times 10^8 m/s}{341 \times 10^{-9} \, m} =$$
$$6.416 \times 10^{-19} \, J \qquad (2)$$
$$I \, J = 6.642 \times 10^{18} \, eV, therefore \, 5.832 \times 10^{-19} J = 3.87 \, eV$$
$$(3)$$

The observed x_{max} shows some agreements with the range of values in the literature such as 9.2 and 3.6 eV reported by Guler *et al.* (2020) and Hussin *et al.* (2016).

Also, a x_{max} of 235 nm has been reported for silicon oxide nanoparticles synthesized by the sol-gel method by Hussin, *et al.* (2016). The evaluated bandgap suggests that the synthesized SiONPs is a semiconductor.
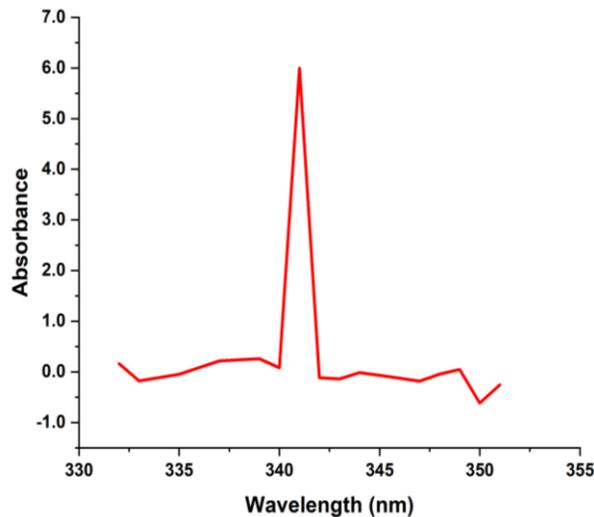
Fig. 1: UV visible absorption spectrum of SiONPs derived from plantain peels

3.2 XRD analysis

The XRD spectrum of the synthesized SiONPs is shown in Fig. 2. The principal peak in the spectrum is at diffraction angle of 68.08 °. Other peaks were found at diffraction angles corresponding to 29.96, 39.66, 44.5, 50.45, 50.46, 57.38, 68.16 and 69.18 °. Chi *et al.* (2017) described a principal XRD peak in SIONPs and attributed to peak to silicon water.

The exclusion rules expect the peak at 69 ° to be associated with only the (400) Muller indices. According to Hodhod *et al.* (2019), a broad XRD peak between 20 and 30 ° is attributed to amorphous SiONPs while Abdul Ghani *et al.* (2017) and Rahimzadeh *et al.* (2022) observed an amorphous peak between 15 and 35 ° diffraction angle. In this spectrum, amorphous character is minimal indicating a high degree of crystallinity. On the other hand, Daulay *et al.* (2022) observed absorption peaks at 28.38, 47.26, 56.08, and 69.08°, which are good matches with the observed spectrum. The peaks reflected those expected for crystalline SiONPs. Therefore, a crystalline nature seems to dominate the spectrum. The observation of less noise also confirmed the crystallinity of the synthesized SiONPs (Odoemelam *et al.*, 2023). . Scherer's equation (equation 2) can be used to calculate the crystalline size (D_{cryst}) of the synthesized CaONPs by substituting the values of FWHM (at various angles of diffraction) into the following equation (Canchanya-Huaman *et al* 2021; Kelle *et al.*, 2024)

$$D_{cryst} = \frac{kx}{(FWHM)cos x} \qquad (4)$$

where x is the wavelength of the Cu-K line excitation ($x = 1.5406$) and k is the Scherer's constant, which is numerically equal to 0.9.
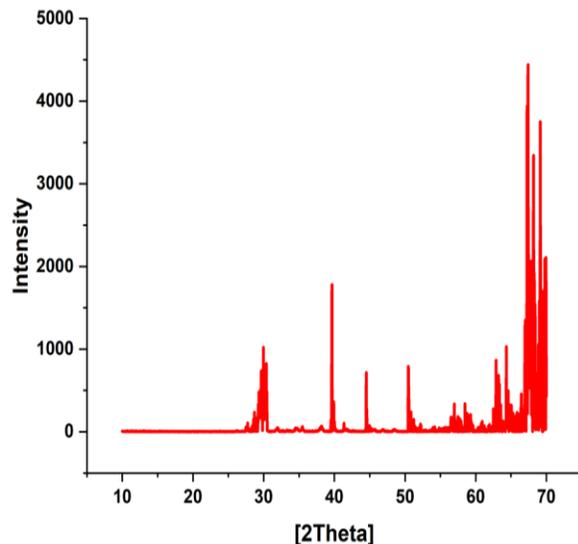
Fig. 2: XRD Analysis spectrum of SiONPs derived from plantain peels

The D_{cryst} for synthesized SiONPs was evaluated through FWHM (2.7414 nm) evaluated from Gaussian peak function using the Origin statistical package.

Based on the results, the average crystalline size of the nanoparticle is 3.65 nm which is within the range of values reported for SiONPs such as 20 nm (Abdul Ghani *et al.* 2017), 79 nm (Azib *et al.*, 2021), 24.58 nm (Daulay *et al.*, 2022).

3.3   Surface properties analysis

The determination of the surface area, pore volume and pore diameter of the synthesized nanoparticles was carried out using Brunauner Emmett Teller (BET) methods. Figs. 3 shows the BET isotherm as a representative plot among the fitted models (isotherms) adopted for the evaluation process.
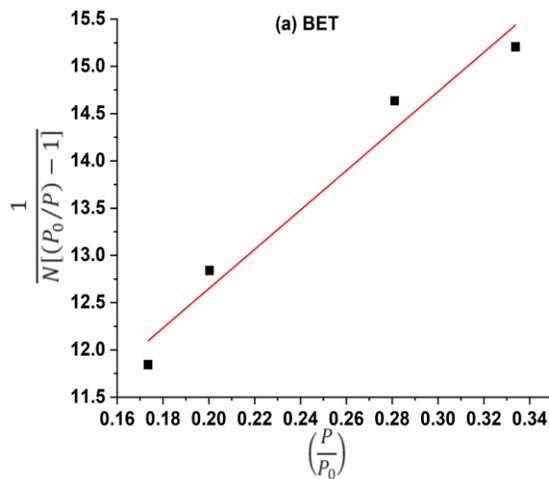
Fig. 3: Multi-Bet plot for SiONPs synthesized from plantain peel

The nitrogen adsorption study indicated that based on the Brunauer-Emmett-Teller (BET), Langmuir, Dullimore Heal (D-H) and Dubinin-Radushkevich (D-R) models, the surface area, pore volume and pore diameter are recorded in Table 1. The BET model predicted the surface area of the synthesized SiONP to be 5428 m^2/g while the Langmuir surface area was observed as 591.01 m^2/g. Higher values were obtained from the density functional theory (DFT) and Dubinin-Raduskevich (D-R) methods which indicated surface area values of 661.02 and 700.20 m^2/g respectively. Therefore, the surface area of the SiONPs lies between 528 and 700 m^2/g. Also, the evaluated particle size from the D-R and DFT models were 8.0971 and 3.012 nm as shown in Table 2. Therefore, the SiONPs is a mesoporous material because the particle size is within the range, of 2 to 50 nm (Eddy *et al.*, 2022). .

Based on particle size, the porosity of nanoparticles can generally be classified into microporous (particle size less than 2 nm), mesoporous (particle size between 2 and 50 nm) and microporous (particle size between 50 and 100 nm) (Eddy *et al.*, 2023b). However, the average pore volume was 0.0371 cc/g while the pore diameter was 5.5545 nm. Some reported values concerning the particle size of SiONPs are presented in Table 2. From the presented data, the synthesized SiONPs have uniquely mesoporous particle size. Some reported values of particles for SiONPs are 15 nm (Meng *et al.*, 2020) and 27.77 nm (Daulay *et al.*, 2022).

Table 1: Pore properties of the synthesized CaO nanoparticles

Model	Surface area (m^2/g)	Pore volume (cc/g)	Pore diameter (nm)
Multi-BET	528.00		
Langmuir	591.01		
D-R	700.20	0.0359	8.0971
DFT	681.02	0.0399	3.012

Table 2: Literature Values of Source of Synthesis, Characterization and Surface Properties of SiONPs

Nanoparticles	Method	Synthetic condition	Characteristics	References
SiO-NPS	Sol gel method using $Si(OC_2H_5)_4$	650 °C, 4 hours, x = 58 nm,	FESEM, EDS	Chang *et al*. (2014),
SiO-NPS	Sol gel method using rice husk	550 °C, 4 hours, x = 3 nm, SA = 340 m^2/g	BET, XRD, TEM, SEM	Lee *et al* (2020)
SiO-NPS	Sol gel method using $Si(OC_2H_5)_4$	700 °C, 2 hours, x = 79.68 nm to 87.35 nm	FE-SEM, particle size analyser	Azlina *et al*. (2016)
SiO-NPS	Sol gel method using $Si(OC_2H_5)_4$	500 °C, 1 hour, x = 193 nm, $x_{max} = 259\ nm$	UV-V, FTIR, DLS,	Saravanan and Dubey (2020)
SiO₂-NPS	Modified Sol gel method using HCl, NaOH and ashed plant wastes	650 °C, 2 hours, X = 193 , d = 53 nm, $x_{max} = 259\ nm$	FTIR, XRD,	Nimah *et al*. (2023)
SiO₂-NPS	Sequential method employing sonication to complete sol gel synthesis using $Si(OC_2H_5)_{4,}$ NH_4OH as precursors	Calcination at 600 °C, 2 hours, x = 20 nm, d = 27 nm	UV-Vis, XRD, FTIR, TEM	Rao *et al*. (2005)
SiO₂-NPS	lyotropic liquid crystals formation method	x= 4 nm, SA = 587 m^2/g, PHZC = 3.1,	EDX, SEM, BET, PHZC	Arce et al. (2013)
SiO₂-NPS	Precipitation method using rice husk	650 °C , Purity = 95%, x = <100 nm	FESEM, XRF	Zarei *et al*. (2021)
SiO₂-NPS	Green synthesis using Rhus coriaria L. extract and sodium metasilicate	d =18 nm, x = 60 nm	XRD, TGA/DSC, FTIR, UV Vis, DLS	Rahimzadeh *et al*. (2022)
SiO₂-NPS	Facile approach	x = 67 nm	XRD, TEM, SEM, FTIR, TGA/DSC	Ismail *et al*. (2021)

SiO₂-NPS	Ultrasound-assisted sol-gel method using TEOS, NH₄OH and water	$d = 28$ nm, $x = 48$ nm, $E_{BG} = 4.8$ eV	SEM, TEM, XRD	Edriss and Adinehnia (2011)

4.0 Conclusion

The present study was conducted to synthesize silica Nano-particles from plantain peels. From the chemical composition of plantain peel wastes, there is evidence that indicates that the peel is rich in silicon. The organic content was removed by ashing and the silicon was removed as silicon chloride by reacting the ash with HCl and was subsequently calcined after conversion to silicon hydroxide. The prepared silicon oxide Nano-particles show properties similar to silicon oxide nanoparticles reported elsewhere such as XRD spectrum showing patterns that aligned with JCPD card for silicon oxide nanoparticles such as Si(111) and Si(400) peak around 69, crystallite size of 3.65 nm, surface area ranging between 528 and 700 m^2/g, pore diameter of 3.012 nm, pore volume of 0.0399 m3/g and surface area to volume ratio ranging from 1 323.31 nm-1 to 1754.39 nm^{-1} which makes them suitable candidates for various applications such as adsorbent, drug carrier, etc. The low bandgap (3.6 eV) observed for the nanoparticles also indicates that the mesoporous silicon oxide nanoparticles synthesized from plantain peels have good optical properties that can guarantee their application as photocatalysts and other related applications. The material's significant adsorption capacity is also observed by the high surface area. From the information obtained from the analysis and comparison with literature parameters, it can be concluded that plantain peels is a good precursor for the synthesis of silicon oxide nanoparticles with unique surface properties. However, the need for further characterization and trial application tests is recommended since further information concerning the nanoparticles can be obtained from such tests, especially their applications.

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Compliance with Ethical Standards
Declarations

The authors declare that they have no conflict of interest.

Data availability

All data used in this study will be readily available to the public.

Consent for publication
Not Applicable

Availability of data and materials

The publisher has the right to make the data public.

Competing interests

The authors declared no conflict of interest.

Authors' Contributions

COD:Conceptualization, Methodology and graphical plots. NBE, UE and COD: Writing, corrections: NBE, COD and UE.