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Synthesis And Characterization of Nanosilica from Palm Kernel Shell Ash

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ABSTRACT: This study explores the potential of transforming palm kernel shells, an abundant and inexpensive agricultural waste product into silica nanoparticles, which could be adopted for environmental applications like as an adsorbent, dye removal and catalyst support. Nanosilica could also find applications in other fields like industrial applications in the reinforcement of concrete, additive in paints and coatings, as nano fertilizers e.t.c. Palm kernel shells are the hard, woody endocarp of the oil palm fruit which is obtained as a byproduct of palm oil production. Nigeria, being a major palm oil producing country would naturally lead to the generation of a large amount of palm kernel shells as a byproduct, which could cause environmental pollution when not properly disposed. This study provides an environmentally friendly means for the valorization of these waste byproducts to obtain a substance of increased value and more diverse industrial and environmental applications. The silica nanoparticles were synthesized using the precipitation method. FTIR, SEM and XRD characterization techniques were used to characterize the synthesized nanoparticles to ensure its physical and chemical properties correlate. The XRD chart showed a large hump like peak at about 20-30^0, which confirmed amorphous silica when compared to existing works, the SEM data obtained showed peaks which confirmed the predominant presence of Si and O groups, and the peaks from the FTIR analysis at 790.07 cm-1, 1065.40 cm-1, 3410.56 cm-1, 1635.36 cm-1, 453.00 cm-1, 418.75 cm-1, 406.56 cm-1 respectively confirmed the presence of Si-O bond vibrations. All data on this study closely correlated with those from existing works. This study holds significance economically and research wise since palm kernel shells are low-cost, sustainable material for producing nano silica, while also offering an eco-friendly and economic solution for addressing agricultural byproduct pollution in the environment.

KEYWORD: Nanomaterials, silica nanoparticles, Palm kernel shell ash , Agricultural waste, Byproducts valorization

America, Asia, and Africa, especially in Nigeria are home to a large quantity of oil palm trees (Byerlee and Viswanathan, 2018). Elaeis guineensis commonly known as palm kernel consists of different parts (Mortimer et al., 2018). Palm kernel shell PKS is the rigid endocarp of palm fruit which borders the seed and is alternatively known as Oil Palm Shell (Adinkrah-Appiah et al., 2018). It is gotten as the remaining waste in the removal of the kernel in the nut once palm oil has been extracted from the mesocarp of oil palm pod (Anyaoha et al., 2018).

Palm Kernel Shell Ash (PKSA) is the residue left after the combustion of palm kernel shells (PKS), a byproduct of the palm oil industry. Due to its high silica content, porosity, and alkaline nature, PKSA has gained attention for its applications in environmental remediation, construction, catalysis, and agriculture. It's chemical composition varies depending on burning conditions, but it typically contains: Silica (SiO_2) :40–60%, Alumina (Al_2O_3): 10–25%, Calcium oxide (CaO) :5–15%, Potassium oxide (K_2O), Magnesium oxide (MgO), and Iron oxide (Fe_2O_3) in smaller amount

It's physical roperties includes; Fine powder after grinding which improves its reactivity, Porous structure which enhances its adsorption properties, High Surface area which is useful in catalysis and adsorption, Dark Gray to Black Color due to carbon content and metal oxides

Nanomaterials exhibit several interesting physical and chemical properties due to their high surface area and new quantum properties at nanoscale size. Such properties, like chemical stability, redox properties, high reactivity e.t.c. (Kalapathy et al., 2002). make them suitable candidates for a wide range of applications in different fields of science and technology.

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Conventionally, a variety of methods has been adopted in literature for synthesizing nanomaterials, including pyrolysis, vapor-phase reaction, vacuum arc discharge, chemical vapor deposition and laser evaporation, sol-gel and Solution precipitation (Al Mubarak et al., 2014). However, most of these methods involve energy intensive techniques and complicated methodologies . An alternative method, relying on the extraction of nanomaterials from natural biological sources such as microorganisms, plants and agriculture by-product and waste, has gained significant attention of researchers in recent years.

Silica nanoparticles (SiO_2 NPs) are nanoscale particles of silicon dioxide (SiO_2), typically ranging from 5 to 200 nm in size. Due to their high surface area, tunable porosity, and chemical stability, they are widely used in environmental, biomedical, and industrial applications (Usman & Rufai, 2012).

Many agriculture by-products and wastes are very rich in silica, particularly rice husk, palm kernel shells and wheat straw (Salakhum et al., 2018; Velmurugan et al., 2015; Okoronkwo et al., 2016). Currently, there is a growing interest in the extraction of silica nanoparticles from agriculture by-product and waste due to their abundance and renewability.

The production of nanosilica from agricultural by-products and waste offers several advantages compared to conventional methods including, simplicity, lower costs, higher margin of safety and less environmental pollution of the synthesis process. In particular, the extraction of nanomaterials from agriculture waste could be used as a sustainable source both for solving problems of environmental degradation and economic substitute for industrial raw material. Nanoilica is a very important material. It has many important applications, such as thixotropic agents, thermal insulators, composite fillers and use as an adsorbent (Carmona et al., 2013).

The synthesis of nanosilica from palm kernel shells (PKS) is a highly significant area of study due to its environmental, economic, and technological implications. It addresses the critical environmental, economic, and societal challenge of pollution while also generating a value-added product. It is well known that there is an ever-growing global population and a relatively proportional increase in the amount of bio-waste produced, without proper disposal or recycling, it would pose a threat to the environment. By transforming agricultural waste into a valuable material for water treatment, the study promotes sustainability, economic development, and technological innovation as well as a cost-effective, eco-friendly, and globally relevant solution to environmental pollution from agricultural byproducts.

II. ROUTES FOR THE SYNTHESIS OF SILICA NANOPARTICLES

Several techniques have been developed to synthesise and produce silica nanoparticles with controlled shape, size, dimension, and structure from different waste materials such as rice husk ash, coal fly ash, paddy husk ash, and others. The techniques for extracting silica particles can be classified into two (Dlomo et al., 2020), which are top-down or physical approach and bottom-up or chemical approach as illustrated in Fig. 1. The top-down approach is defined by applying special size reduction techniques to reduce the dimension of the original size. Meanwhile, the popular method for producing silica nanoparticles from the atomic or molecular scale is the bottom-up approach (Reverchon et al., 2006).

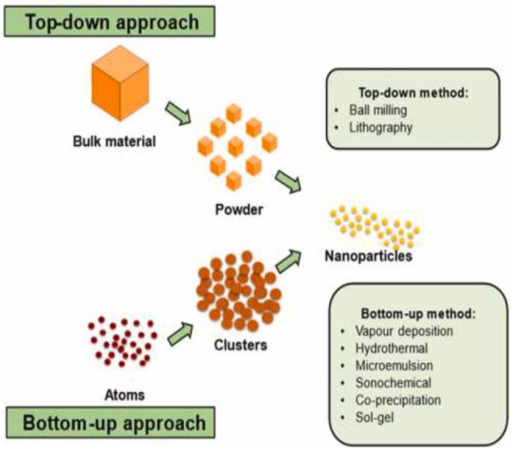

Figure 1: Top-down and bottom-up approaches (Rahman and Padavettan, 2012)

***2.1 Top-down approach*:** Top-down or physical approach is defined by reducing the dimension of the original size by using special size reduction techniques (Shankar et al., 2018). This approach is generally more straightforward and relies either on bulk material removal or division or on miniaturisation of the bulk fabrication processes to achieve the desired structure/surface properties (Setiawan et al., 2015). The milling technique is an example of a top-down approach that breaks down bulk materials to reach fine particles (Made-Joni et

al., 2020). Ball milling is a shear-force dominating technique in which the particle size is reduced by impact and attrition. It uses metallic balls (usually Zirconia or steel balls) as a grinding media and a rotating shell to produce centrifugal force (Rahman & Padavettan, 2012). This technique offers a number of benefits including cost-effectiveness, high product purity, low-temperature reaction, controllability and reproducibility (Priyadarshana et al., 2015).

Nanolithography is a key technique for creating nanoscale structures that involves pressing a mould with three-dimension (3D) surface patterns against a superheated cast (Barabadi et al., 2020). To achieve high-resolution topography, lithography is generally coupled with deposition and etching. Masked lithography and maskless lithography are the two types of lithography. Masked lithography includes photolithography. Photolithography, the semi-conductor industry's reliable technique for high-volume production, has been used directly to produce discrete monodisperse colloidal particles. This technique offers several distinct benefits including exceptional shape consistency up to the submicrometer level and excellent output (Jose et al., 2019; Merkel et al., 2010) Although offering numerous advantages, top-down synthesis method needs further improvement due to the formation of flaws in the structure of the produced material. This is a significant limitation since the surface chemistry and other physical characteristics of the nanoparticles are strongly dependent on the material structure. In addition, they might have a lot of contaminants in the produced material. Despite these main drawbacks, this method is considered suited for large-scale manufacturing as it allows for deposition over a large substrate and requires no chemical purification (Modan and Plaiasu, 2020).

2.2 Bottom-up approach: The bottom-up approach includes a chemical aggregation of silica nanoparticles from atom to atom or molecule to molecule. This technique is considerably more prevalent for nanoparticle elaboration because it allows for precise particle size control. Bottom-up approaches are more versatile in which this method has resulted in a wide range of nanoparticle synthesis methods based on the self-assembly principle (Modan and Plaiasu, 2020). The significant advantage of this method is the high yield purity (Zobir et al., 2018).

CVD (chemical vapour deposition) is a bottom-up technique where vapour-phase substances are condensed to form a solid-phase material (Tahir et al., 2020). It is a well-known chemical technique utilised in a variety of industries for high-temperature protection, erosion protection, and a combination of the two (Madhuri et al., 2020). According to Huang et al. (Huang et al., 2018), CVD is the most common method to produce large-scale and high-purity fumed silica glass. This process involves injecting a precursor into H_2/O_2 flame to synthesise SiO_2. The silica glass is then formed by transporting SiO_2 to the substrate and depositing it on the substrate; hence, SiO_2 production inside a chamber is crucial to control this process. In an industry, halide compounds such as silicon tetrachloride ($SiCl_4$) are widely utilised as a precursor for CVD synthesis of silica glass. However, this technique requires high temperatures and its growth rate is rather slower. Furthermore, it utilises hazardous reagents such as $SiCl_4$, $Ni(CO)_4$ and B_2H_6. Some of these reagents are highly explosive and corrosive (Karande et al., 2021).

In addition to CVD, hydrothermal synthesis is another method to produce silica. In general, hydrothermal processes are carried out in a high-pressure enclosed compartment under subcritical or supercritical solvent conditions. The utilization of traditional hydrothermal method exhibits some drawbacks including poor yield, extensive process durations, and heterogeneous structure. This flaw may be remedied by utilizing a microwave as a heater. This method allows the control of the resulting nanoparticles morphology, able to produce material that is unstable near the melting point, and large crystals of high quality (Fernandez et al., 2018). However, it requires high temperature furnace and heating devices. Fernandes et al. synthesized MSN KIT-6 via hydrothermal synthesis whereby hydrochloric acid (HCl) was used to keep the medium acidic, the triblock copolymer P123 as an organic structural template, and tetraethylorthosilicate (TEOS) as a source of silica. Silmi et al. (2021) reported that hydrothermal is one of the most well-known and successful synthesis techniques for generating MSN KCC-1 with tuneable pore and particle size.Furthermore, microemulsions are self-aggregated colloidal system that gives a controllable system with potential use of nanoreactors. They can function as pools within which nanoparticle characteristics may be easily regulated (Cid, 2018). According to Narayan et al. (2018), this approach is presented as a new

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nanosilica synthesis technique. This method is able to control the size, shape, and crystal structure of the metallic nanoparticles; thus, generating a series of seeds with various shapes and sizes using the same reagents. Furthermore, controlling reaction duration, temperature, and reaction conditions will create various geometries that could be potentially used in many research fields (Cid, 2018). However, this method requires a large amount of surfactant and its stability is influenced by the pH of the microemulsion.

Sonochemical synthesis was one of the first techniques developed to produce engineered nanomaterials (Kamala et al., 2021). Ultrasound irradiation is a simple way to make a range of nanomaterials from inorganic precursors, mostly by primary, secondary or combined sonochemistry effects. When the secondary sonochemical synthesis uses the post-produced chemical in the liquid phase, the primary sonochemical synthesis refers to the extreme transitory circumstances such as high pressure and temperature from the collapse of the cavitation bubbles, Thus, this high-intensity ultrasound offers a unique approach to nanoparticle synthesis that avoids the need for bulk high temperatures, high pressures or extended reaction periods. However, this method produces low materials yield (Modan and Plaiasu, 2020).

Chemical precipitation is a prevalent bottom-up method in synthesising silica nanoparticles due to its energy efficiency and excellent processing time. Another effective soft chemical route to mix oxide materials is the co-precipitation technique also known as the solid-state analogue or a side-branch of the sol–gel process (Babooram, 2008). Co-precipitation reactions occur when nucleation, growth, coarsening, and/or agglomeration processes happen at the same time. This technique is simple, allows easy size and deposition control, requires minimal temperature, and is energy efficient (Rane et al., 2018). It also has the benefit of producing homogenous nanomaterials with small size and size dispersion directly into the solution through various chemical reactions (Liu et al., 2019). However, Rane et al,(2018) stated that this approach is not applicable to uncharged species and the product obtained may include precipitated trace impurities. Furthermore, it suffers from batch-to-batch reproducibility issues.

Lastly, because of its ability to produce a pure and homogenous product under mild circumstances, the sol-gel or precipitation method has been frequently utilized to produce silica nanoparticles (Dolmo et al., 2020). It is a simple, economical, and efficient method in producing high quality coating (Modan and Plaiasu, 2020). However, this synthesis technique necessitates the use of expensive raw materials and a high-temperature furnace or heating apparatus. In the presence of acid or base, this process includes the hydrolysis and condensation of metal alkoxides such as TEOS ($Si(OEt)_4$) or inorganic salts such as Na_2SiO_3. The dimensions and morphologies of the resultant materials are influenced by temperature, water content, pH, surfactant or copolymer properties, and the concentrations and sources of the silica employed in the process (Diagboya and Dikio, 2018).

III. METHODOLOGY

The precipitation or sol-gel method, which is a widely used technique for the laboratory synthesis of nanosilica from palm kernel shells is utilized in this study because of its low cost, availability of materials and simplicity compared to other routes. This process involves several steps; transforming the palm kernel shells into palm kernel shell ash by calcination, acid or alkali leeching, drying, crushing and the characterisation of the particles.

3.1 Preparation of the palm kernel shell ash

On obtaining the palm kernel shells commercially , they were washed with distilled water to remove impurities and air dried at room temperature, after which they were ground to reduce their sizes. Calcination at 600^0C for 5 hours was carried out to obtain palm kernel shell ash, which is cooled at room temperature in the presence of oxygen.

3.2 Preparation of sodium silicate solution

Palm kernel shell silica was extracted using an alkali extraction process, adopting the precipitation method. The experimental process was carried out by weighing and transferring 30 g of palm kernel shell ash on the electronic weighing balance into a glass beaker containing 500 mL of 3M NaOH solution. The solid PKA was mixed with the sodium hydroxide solution and boiled for 4 h with vigorous magnetic stirring during the whole reaction process. The temperature of the whole system was maintained at 80^0C. The final mixture was allowed to cool down to room temperature. The mixture was filtered to separate the filtrate which

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contains sodium silicate, while the insoluble residue which was discharged. The whole process was described in figure 2.

(a) Ground palm Kernel shells

(b) Palm Kernel shell ash

(c)Heating with NaOH.

(d)Filtering of solution

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(e)Clear sodium silicate solution

FIGURE 2. Preparation of sodium silicate solution from palm kernel shell ash

3.3. Precipitation of Nanosilica:

After obtaining the sodium silicate solution,The pH of the filtrate (sodium silicate solution) was reduced using controlled addition (drop wise) of concentrated hydrochloric acid (37%), while being stirred magnetically, to extract the nanosilica precipitate. when the pH of the solution approaches 7-8, silica gel is observed to begin to precipitate in the solution. After repeated washing of the filtered precipitate, the solids were collected and dried at room temperature for 72 h. The solids were crushed and ground to powder using a mechanical grinder for obtaining the nanosilica grains. The whole process of silica dissolution and precipitation is described by the following reactions:

$SiO_2 + 2NaOH \rightarrow Na_2SiO_3 + H_2O$ - - -
(eq.1)

$Na_2SiO_3 + 2HCl \rightarrow SiO_2 + 2NaCl + H_2O$ - - -
(eq.2)

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(a) silica gel ppt left to age

(b) Washing and filtering

(c) Dried and crushed nanosilica

FIGURE 3. Synthesis of Nanosilica from sodium Silicate solution

IV. RESULTS

4.1 Nanoparticles Characterization of

4.1.1 X-ray diffraction (XRD): X-ray Diffraction (XRD) is a powerful technique used to study the structure of materials. It helps identify phases, crystallinity, and structural properties by analyzing how X-rays interact with the atomic planes of a material. The XRD pattern of the synthesized silica nanoparticles are shown in figure 4.The diffraction pattern shows a broad hump like pattern typically around 20-30^0 which closely corresponds to those reported by Nnaemeka et al.,(2024). This information confirms the and amorphous nature of the nanoparticles synthesized and their use as an adsorbent. Very similar diffraction patterns and hump like peaks were observed in the study by Imosili et al., (2019).

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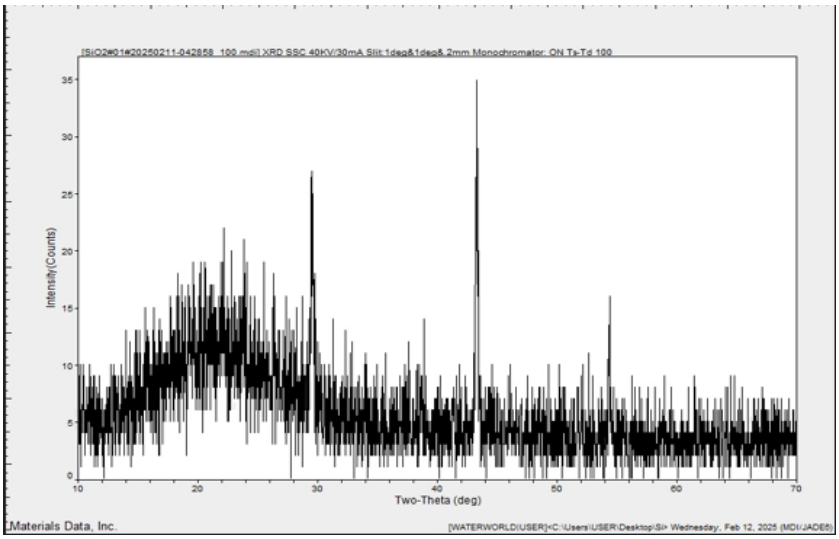

Figure 4.0. XRD of synthesized nanosilica

4.1.2. Scanning Electron Microscope (SEM-EDX): Scanning Electron Microscopy **(SEM)** is a powerful imaging technique used to examine the surface morphology and microstructure of materials at high magnifications. (SEM-EDX) analysis was employed to observe the physical morphology of sample surfaces and to analyze the element compositions. The synthesized nanoparticles were observed at 5000x magnification to be highly porous and to have and have an aggregated structure, typical of amorphous silica which similarly corresponds to reports from Nnaemeka et al., (2024). The particles seem to be clustered rather than isolated and have irregular shapes, which is common for nanosilica synthesized using the precipitation method. The surface texture suggests high roughness, which can enhance adsorption capacity. This is similar to reports made by Pattanayak et al., (2021).

EDX

The EDX (Energy-Dispersive X-ray) analysis of the synthesized nanoparticle showed peaks indicating the presence of C, Na, Mg, Al, Ca, Mn and Fe in negligible amounts which are most likely from the palm kernel shell precursor and the synthesis method as well as Si (45.2%) and O (48.1%) which makes up 93.3% of the compound and confirms silica (SiO_2)
as the predominant compound.

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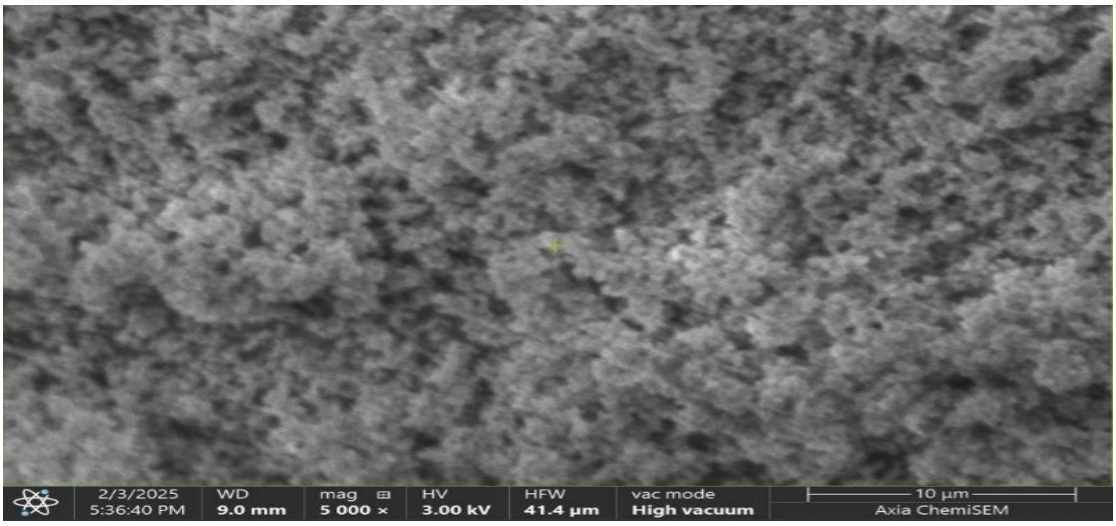
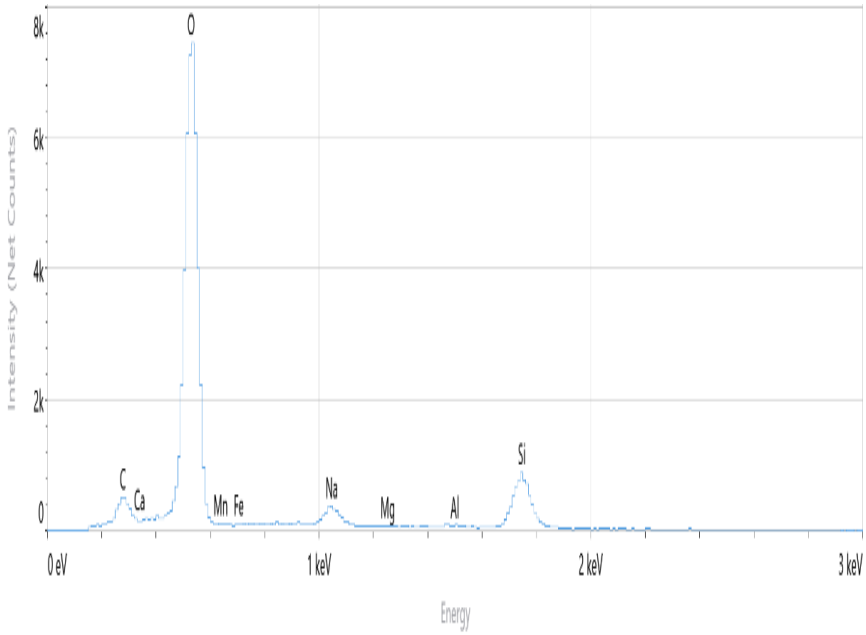


(a)SEM-EDX image of the nanoparticles



(b)Graph showing the peaks of various elements F

FIGURE 4.1: Scanning Electron Microscope SEM and EDX of Silica nanoparticles

Table 1: Element composition of synthesized nanoparticle

Element	Line	At. %	Wt. %	Net Counts	At. % Error	Wt. % Error
C	K	2.9	1.7	2 476	0.1	0.1
O	K	60.4	48.1	39 315	0.4	0.3
Na	K	3.6	4.1	1 941	0.1	0.2
Mg	K	0.1	0.1	46	0.1	0.1
Al	K	0.6	0.8	202	0.1	0.2

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Element	Line	At. %	Wt. %	Net Counts	At. % Error	Wt. % Error
Si	K	32.4	45.2	6 772	0.5	0.7
Ca	L	0.0	0.0	0	---	---
Mn	L	0.0	0.0	0	---	---
Fe	L	0.0	0.0	0	---	---

4.1.3. Fourier-Transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared Spectroscopy (FTIR) is a technique used to identify functional groups in a material by measuring how it absorbs infrared (IR) radiation at different wavelengths. It provides insights into the chemical composition and structure of the synthesized nanoparticle.

FTIR was used to analyze the surface functional groups on the silica nanoparticle synthesized to ascertain the types and number of functional groups present. The FTIR spectra of the nanosilica showed intense IR peaks at 790.07 cm^{-1}, which corresponds to the symmetric stretching vibrations of Si-O-Si, which is a characteristic of silica. It also shows peaks at 1065.40 cm^{-1}, indicating the asymmetric stretching vibration of Si-O-Si, confirming the presence of siloxane groups. There are strong peaks at 3410.56 cm^{-1} which corresponds to O-H stretching from adsorbed water and -Si-OH groups which are similar to reports by Nakamoto et al., (2009) and 1635.36 cm^{-1} corresponding to H-O-H bending, further confirming the presence of adsorbed water

Additionally, less intense peaks are observed at 453.00 cm^{-1}, which could be associated with H-O-H bending vibrations, further confirming the presence of adsorbed water. Lastly, there are peaks at 418.75 cm^{-1} and 406.56 cm^{-1}, possibly related to Si-O bending vibrations.

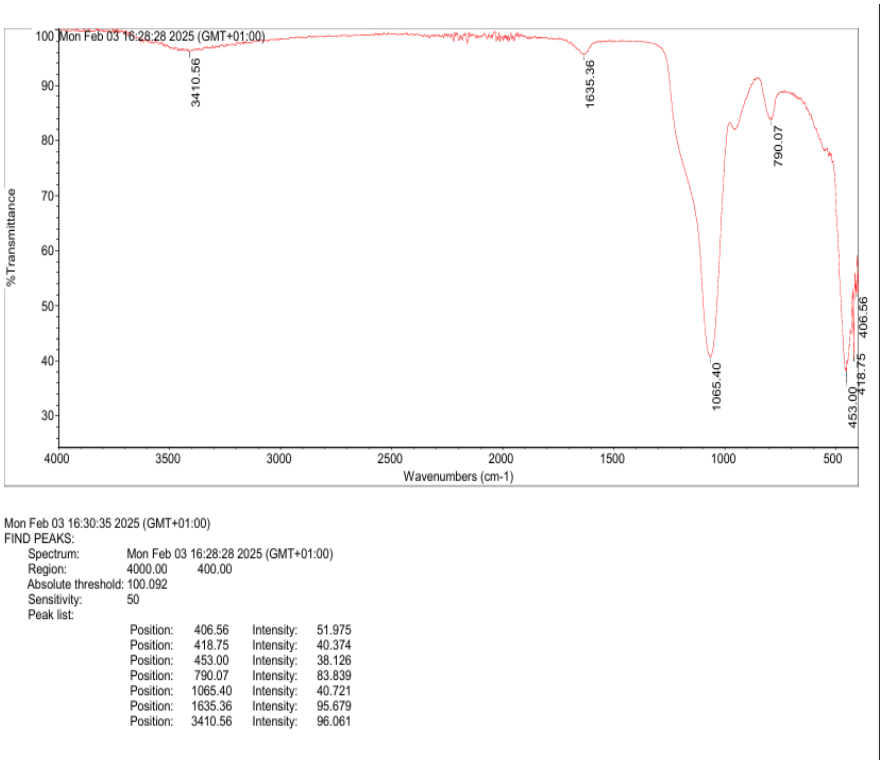

FIGURE 4.2. FTIR spectra of synthesized silica nanoparticles

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V. CONCLUSION

In this study, nanosilica was successfully synthesized from palm kernel shells, demonstrating the potential of agricultural waste as a sustainable and cost-effective silica source. The synthesis process effectively extracted silica through controlled chemical treatment and thermal processing, yielding nanoparticles with desirable properties. FTIR, XRD and SEM Characterization techniques confirmed the formation of nanosilica with high purity, amorphous structure, and nanoscale particle size.

The findings highlight that palm kernel shells, an abundant agro-waste in many tropical regions, can serve as an environmentally friendly raw material for nanosilica production. This not only adds value to waste materials but also supports the development of low-cost nanomaterials for potential applications in adsorption, catalysis, drug delivery, and composite materials. Further optimization of synthesis parameters could enhance the yield and functionality of the nanosilica, promoting its commercial viability

5.1.CONTRIBUTION TO KNOWLEDGE

This research has made several significant contributions to the field of environmental chemistry. It demonstrated that palm kernel shell ash (PKSA), an abundant agricultural waste in many tropical regions, can be effectively utilized as a viable and low-cost precursor for the synthesis of nanosilica. This approach promotes waste valorization and supports circular economy principles by converting agro-waste into a high-value nanomaterial.

The study demonstrates that palm kernel shells, an underutilized agricultural waste, can serve as a viable raw material for synthesizing nanosilica. It contributes to sustainable waste management practices and circular economy strategies in agricultural regions.

The research also shows that the precipitation method can be effectively adapted for nanosilica synthesis from palm kernel shells, providing a relatively simple, cost-effective, and scalable alternative to more complex synthesis methods.

The work provides detailed physical and chemical characterization of nanosilica derived from palm kernel shells. The FTIR and SEM results support the presence of Si–O bonds and elemental composition, adding to the body of knowledge on silica nanoparticle properties obtained from natural sources.

By confirming the successful synthesis of nanosilica with desired structural properties, the study opens up opportunities for using this material in water purification, drug delivery systems, nanocomposites, and other advanced materials applications

This research adds regional relevance by focusing on palm kernel shells, a common waste product in many tropical countries, especially Nigeria. It highlights a practical solution that can be replicated in similar agro-based economies

5.2 RECOMMENDATIONS

- Further research should explore the optimization of key synthesis parameters—such as pH, reaction time, temperature, and precursor concentration—to improve the yield, uniformity, and particle size control of the synthesized nanosilica.

- Comparative studies using other agricultural waste sources such as rice husks, corn cobs, or sugarcane bagasse could help determine the most efficient and cost-effective materials for nanosilica production.

- Future work should focus on functionalizing the synthesized nanosilica for specific applications such as heavy metal adsorption, drug delivery, or as a reinforcing filler in nanocomposites, to fully realize its practical potential.

- Studies should also investigate the scale-up potential of the synthesis process, including economic analysis, to assess the feasibility of commercializing nanosilica production from palm kernel shells.

- It is important to assess the environmental safety and potential toxicological impacts of the synthesized nanosilica, especially if intended for use in biomedical or environmental applications.

By addressing these recommendations, future studies can further enhance the efficiency, economic

viability, and environmental sustainability of palm kernel shell-derived nanosilica.

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