Original Article

Green technology extraction and characterisation of silica nanoparticles from palm kernel shell ash via sol–gel

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1. Introduction

America, Asia, and Africa, especially in Nigeria are home to a large quantity of oil palm trees [1–3]. Elaeis guineensis commonly known as palm kernel consists of different parts [4]. Palm kernel PKS is the rigid endocard of palm fruit which borders the seed and is alternatively known as Oil Palm Shell [5,6]. 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 [7]. PKS was utilized by scientists as lightweight aggregates (LWA) to substitute traditional normal weight aggregates (NWA) in essential elements and motorway construction [8–11]. PKS has also been utilized in various applications extending from energy storage, biomass, bio-fertilizer, to supercapacitor electrode [12–15]. Silica nanoparticles are also used for biomedical applications, including drug delivery, due to the large surface area [16]. It constitutes environmental waste if not utilized [17].

Silica gel is a stiff three-dimensional linkage of colloidal silica. Silica gel is categorized based on the synthesis process as aqua gel, xerogel and aerogel. The aqua gel contains holes
The process of producing nano-silica from PKSA involves several steps, as illustrated in Fig. 1. The extraction begins with the dispersion of Palm Kernel Shell Ash in 3 M NaOH, followed by boiling with stirring for 2 hours. The filtrate is then filtered and allowed to cool. The filtrate is then titrated with 3M HCl (pH 7.5 - 8.5) and allowed to age for 24 hours. Crush gel is prepared by mixing the gel with water and centrifuging at 3,000 rpm for 5 minutes. The gel is then allowed to dry at 80°C for 24 hours. This process yields a Xerogel. The Xerogel is then refluxed with 3M HCL (4 hrs) and washed to dissolve in 3 M NaOH and titrated with 3 M H₂SO₄ (pH 7.5). The material is then washed with warm deionized water and dried at 80°C for 48 hours. This process yields Nano Silica.

The cumulative interest in the synthesis of SiO₂ nanoparticles is owed to the wide usage as a basic raw material in the growth of new classes of innovative materials for high-tech applications [35–37]. Silica nanoparticles have been produced using Chemical Vapour Condensation (CVC) [38], Reverse Micro Emulsion (RME) [39], precipitation method [40]. Sol–gel is methods used to synthesis silica, porcelain materials and glass owing to its capability to produce pure consistent products at trifling situations [41–43]. This study produced silica nanoparticles from agricultural bioresources of PKSA using green technology via the modified sol–gel method. It has a combined advantage of reducing disposal as well as pollution problems and making valued silica particles at a lesser cost. The prepared silica nanoparticles from palm kernel shell ash (PKSA) were characterized using XRD, SEM with EDX, FTIR, BET and TG techniques.

2. Materials and methods

2.1. Materials

The palm kernel shell PKS was obtained from southwestern Nigeria. PKS collection, washing and drying were performed in the open air. Combustion of PKS was done for 3 h at 750 °C with a heating speed of 10 °C/min and allow to cool in the muffle furnace.

2.2. Extraction of silica

Extraction of silica from palm kernel shell ash, (PKSA) was done using the sol–gel, template-free method as previously reported by Okoronkwo et al. [44]. 500 ml rations of 3 M NaOH was added to 50 g PKSA samples and heated for 2 h using a hot plate with continuous stirring to dissolve the silica present in the ash and producing a silicate solution. Ashless filter paper
was used to filter the solutions and 100 ml boiled distilled water was used to wash the residue. After cooling, the filtrate was titrated with 3 M HCl to pH in the range of 7.5–8.5 with continual stirring and nurtured for 24 h to allow gel development. The gel was softly broken after ageing and centrifuged at 4000 rpm for 4 min. The supernatant was discarded while the aqua gels were placed inside an oven to dry at 80 °C for 24 h to yield silica xerogels.

2.3. **Extraction of nano silica**

The silica xerogels were refluxed at 70 °C with 3 M HCl for 4 h and repeatedly washed with deionised water after which it was dissolved with 3 M NaOH by uninterrupted stirring with a magnetic stirrer for 10 h and the pH adjusted in the range of 7.5–8.5 by adding concentrated H₂SO₄. The silica precipitate was collected and repetitively washed using warm deionised water till the silica became wholly alkali-free and were dried for 48 h at 80 °C in a vacuum oven. A flow illustration of the extraction procedure is presented in Fig. 1.

2.4. **Material characterization**

XRD of palm kernel shell ash (PKSA) and extracted silica was scanned using GBC EMMA X-ray diffractometer having CuKα emission at 25 kV acceleration voltage and 400 μA current from 2θ 15° to 60° at a speed of 4.00°/min. Morphology and particle dimension of produced nano-silica were observed with an SEM (Zeiss Ultra Plus) and EDX at Secondary Electron Image (SEI) and high vacuum (HV) mode with 20 kV accelerating voltage. FTIR spectra were recorded using Tianjin GangDong FT-IR 650 in the range of 4000–350 cm⁻¹. Specific surface area (S_{BET}) was estimated using the Brunauer–Emmett–Teller (BET) method. Thermal gravimetric analysis (TG) was assessed using Perkin-Elmer Pyris 6 TGA analyzer. Sample weight of 10 mg was heated from 40 to 800 °C with a heating rate of 10 °C/min in nitrogen atmosphere.

![Fig. 3 - SEM micrograph of nano-silica produced from palm kernel shell ash.](image)

3. **Results and discussions**

3.1. **XRD**

The silica yield was 54.35%. X-ray diffractogram of palm kernel shell Ash and extracted silica nanoparticles are presented in Fig. 2. The XRD pattern for the Ash shows the present quartz at theta = 20, 26, 40, 47 and 55° (SiO₂ PDF Card #331161) and Calcite at theta = 22, 44, 47 and 55° (CaCO₃ PDF Card #050586). The broad XRD array of extracted silica nanoparticles at theta = 22.5°, which is distinctive of amorphous solid, confirms the formation of amorphous silica; similar results were obtained by other researchers [20–34].
3.2. SEM

Fig. 3 shows the SEM micrograph of silica nanoparticles produced from palm kernel shell ash at ×200,000 magnification. The particles were observed to be spherical with reduced silica-silica agglomeration. The average particle sizes of the extracted silica particles sizes were found to be between 50 and 98 nm.

3.3. EDX

A strong intensity of Si and O as shown in the EDX spectra in Fig. 4, confirms silica (SiO₂) as the predominant element in the sample. This represents about 96.59% of the total element. This is negligible impurities commonly associated with green technology method.

3.4. FT-IR

FTIR spectral identified the key chemical compound existing in the nano-silica as revealed in Fig. 5. Band 463 cm⁻¹–475 cm⁻¹ is linked with vibration network of O—Si—O, while band 791 cm⁻¹–807 cm⁻¹ was allotted to symmetric stretching vibration network of Si—O—Si [34]. Band 1071 cm⁻¹–1090 cm⁻¹ was due to Si—O—Si irregular stretching vibration [14] and broadband at 1633 cm⁻¹–1645 cm⁻¹, is due
to O–H bond bending vibration from Si–OH silanol groups, while 3338 cm\(^{-1}\) to 34,750 cm\(^{-1}\) is due to O–H bond stretching vibration from Si–OH silanol groups and are due to adsorbed H\(_2\)O molecules on the silica surface [18,19].

3.5. BET

The surface area (\(S_{\text{BET}}\)) of prepared nano-silica produced from palm kernel shell ash was assessed after calcination at 750 °C for 2 h. Nitrogen adsorption/desorption isotherms of produced nano-silica are shown in Fig. 6. The isotherms exhibit the typical Type IV isotherms, typical of mesoporous materials [47,48]. Pore size distribution is shown in Fig. 7, reveals narrow pore size distributions with average pore diameters ranging from 2.2 nm to 6.3 nm.

The nano-silica \(S_{\text{BET}}\) surface area was found to be 438 m\(^2\) g\(^{-1}\). This specific surface area is greater than 327 m\(^2\) g\(^{-1}\) recorded for commercially available silica, and the values 120 to 288 m\(^2\) g\(^{-1}\) reported for the silica nanoparticles extracted from RHA [45]. It also seems comparable to the 413 m\(^2\) g\(^{-1}\) from rice straw and 422 m\(^2\) g\(^{-1}\) from mineral pumice rock powder [46]. However, this method produced a better yield.

3.6. TG

The results of thermal gravimetric analysis (TG) are shown in Fig. 8. Two-step weight losses were observed. The loss in weight up to 130 °C (step 1) is ascribed to dehydration caused by the loss of physically adsorbed H\(_2\)O. However, chemically bound water from the sol–gel production method was ascribed to the loss in weight from 130 to 570 °C (step 2) [49]. Above 600 °C, no further weight loss was observed indicating thermal stability of extracted nano-silica.

4. Conclusion

Silica nanoparticles were extracted from palm kernel shell ash (PKSA) using a modified sol–gel procedure. The results show that amorphous silica nanoparticles with 54.35% silica yield with negligible mineral contaminants can be produced from palm kernel shell ash using a modified sol–gel technique. XRD investigation revealed the presence of silica in the ash and amorphous nature of extracted silica. SEM micrograph shows silica nanoparticles in the range of 50–98 nm. The surface area of extracted silica nanoparticles was 438 m\(^2\) g\(^{-1}\) and SiO\(_2\) presence in the samples was confirmed by EDX result. Key chemical group existing in the samples were indicated by FT-IR data. This will contribute to knowledge and help reduce environmental pollution caused by the disposal of PKS and valuable silica nanoparticles for advanced materials for high-tech applications can be produced.

Conflicts of interest

The authors declare no conflicts of interest.

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