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SYNTHESIS AND CHARACTERIZATION OF QUARTZ (SiO_2) MATERIALS USING HYDROTHERMAL, X-RAY FLUORESCENCE AND FOURIER TRANSFORM INFRA-RED SPECTROPHOTOMETRIC METHODS

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ABSTRACT: The aim of the investigation was the synthesis and characterization of quartz materials using hydrothermal, X-ray fluorescence and Fourier transform infrared spectrophotometric methods. Upgrading of the quality of the quartz material was carried out by the synthesis of nanoparticles of SiO_2 from rock samples using hydrothermal process. The analytical/geochemical data obtained would be a guide to aid future beneficiation of the quartz materials from rocks, particularly for the economic exploration and exploitation of quartz and silicate minerals which had wide range of applications in various industries like solar cells and solar panels, glass, nanoparticles, ceramic and paint industries. The study involved collection of rock samples containing quartz minerals from Kwandonkaya, Magama Gumau and Toro within Toro District of Toro Local Government Area of Bauchi State, Nigeria. In the hydrothermal process, formation of sodium silicate solution was required. The synthesis steps were: (1) silica sand powder was mixed with 7 mol dm^{-3} sodium hydroxide solution and then stirred thoroughly at a temperature of 90 $^{\circ}C$ for 2 hours to obtain $Na_2{\cdot}xSiO_3$ used as a precursor, (ii) formation step to produce silicite involved titrating sodium silicate solution with hydrochloric acid (7 mol dm^{-3}) until reaching normal pH (~7) and producing silicite ($Si(OH)_4$) in a gel form, (iii) cleaning with deionized water to release NaCl from silica precipitate and (iv) drying the precipitate at a temperature of 80 $^{\circ}C$ for 24 hours. Characterization of the product was carried out using X-Ray Fluorescence Spectrophotometric method and the crystallinity as well as the minerals present was identified using Fourier Transform Infrared Spectrophotometric methods. The result of XRF analysis of synthesized quartz material revealed the presence of 83.40 ± 1.84 to 85.70 ± 1.62 % SiO_2, 1.94 ± 0.21 to 5.60 ± 0.56 % Al_2O_3, 0.68 ± 0.22 to 1.21 ± 0.13 % CaO and N.D. MgO. The IR band at 3964.00 cm^{-1} is due to the stretching vibration of H_2O molecules. Correspondingly, the IR band at 1659.53 cm^{-1} is assigned to – OH bending vibrations of H_2O molecules. The very strong and predominant absorbance peaks at 1079.31 cm^{-1} range usually assigned to the mode of the Si – O – Si asymmetric stretching vibrations. The IR band at 945.87 cm^{-1} can be assigned to silanol groups. In the case of alkali silicate glasses, this band is assigned to Si – O – stretching vibrations. The IR band at 802.04 cm^{-1} can be assigned to Si – O – Si symmetric stretching vibrations, whereas the IR band at 471.97 cm^{-1} is due to O – Si – O bending vibrations. The values of crystallinity indices ranged from 0.33 to 1.33. The synthesized nanoparticles of SiO_2 contained both crystalline and amorphous phases.

KEYWORDS: Quartz, Silica, Hydrothermal, XRF, FTIR, Beneficiation, Nanoparticles, Precursor

I. INTRODUCTION

The main use of high purity quartz is to manufacture silica glass which has a wide range of applications due to resistance to extreme fluctuation in temperature, its chemical durability in acidic environments and its ability of transmitting light from near ultraviolet to infra-red parts of the electromagnetic spectrum (Larsen *et al*., 2000 and Haus *et al*., 2012). In addition, silica glass has a wide range of applications in metallurgical, chemical and optical industries, as well as in communication technology for the production of optical wave-guides and as a raw material in the development of high – performance solar panels for energy production (Larsen *et al*., 2000).
Chemical processes such as extractions, purifications and syntheses of SiO_2 nanoparticles based on natural materials have been widely conducted. For instance, amorphous silica nanoparticles with purity of 95.00 % have been prepared by means of chemical methods (sol gel and hydrothermal) using organic materials and baggase ash (Affandi *et al.*, 2009) and rice husk ash (Kalapathy *et al.*, 2002; Nittaya and Nuntiya, 2008). Inorganic materials like silica sands were employed to produce SiO_2 nanoparticle via energy milling (Ahmad *et al.*, 2013). Diorite sand with Na_2CO_3 addition was sintered at a temperature of 1030 ^{o} C (Trabelsi *et al*., 2009). Waste colored glass was also used for obtaining SiO_2 nanoparticle by means of extraction process using alkali

compounds (KOH and NaOH) at sintering temperatures of 360 and 500 ^{o}C to form sodium silicate (Mori, 2003 and Munasir *et al.,* 2013).

II. EXPERIMENTAL METHODS

The raw materials used for this study were rock samples from Kwandonkaya, Magama Gumau and Toro (Toro District, Bauchi, Nigeria). The method used by Munasir, 2015 were modified and adopted. In the hydrothermal method, formation of solid sodium silicate was required. The synthesis steps were (i) Purification of SiO_2 from impurities which involves weighing of a known mass of the rock sample powder X g (based on the percentage SiO_2 analysis obtained) was mixed with 100. 00 cm^3 of 7.00 mol dm^{-3} sodium hydroxide solution in a 250 cm^3 beaker and heated on a hot - plate at a temperature of 90 ^{o}C for 2 h to form sodium silicate solution ($Na_2O.xSiO_2$). (ii) Titration of sodium silicate obtained with 7.00 mol dm^{-3} hydrochloric acid contained in a burette at a pH 7 to produced silicate gel (Si $(OH)_4$). (iii) Cleaning of the silica precipitate (gel) was carried out thoroughly using deionized water and continuous stirring to ensured sodium chloride was completely removed. (iv) Drying the precipitate was carried out in an oven at 80 ^{o}C for 24 h. The synthesized nanoparticles of SiO_2 were then characterized using XRF to evaluate the elemental composition and FTIR to study the functional groups and the crystallinity indices.

III. RESULTS AND DISCUSSION

Elemental Composition of Major Oxides in Nanoparticles of SiO_2

The percentage composition of major oxides in nanoparticles of SiO_2 using X – Ray Fluorescence method is presented in Table 1. The percentage range of silica (SiO_2) in the three locations within Toro district is 83.40 (Toro) to 85.70 % (Kwandonkaya)

Table 1: Percentage Composition of Major Oxides in Nanoparticles of SiO_2 using X -Ray Fluorescence Method

% Composition Oxides	KWK	MMG	TORO
SiO_2	85.70 ± 1.62	84.76 ± 1.12	83.40 ± 1.84
Al_2O_3	5.60 ± 0.56	1.94 ± 0.21	4.98 ± 0.74
Fe_2O_3	1.60 ± 0.47	1.40 ± 0.41	3.40 ± 0.34
MnO	0.03 ± 0.02	0.69 ± 0.12	0.24 ± 0.06
Cr_2O_3	0.003 ± 0.02	0.77 ± 0.32	0.14 ± 0.04
TiO_2	0.68 ± 0.19	0.83 ± 0.23	0.30 ± 0.26
P_2O_5	1.15 ± 0.11	1.84 ± 0.52	1.40 ± 0.34
Na_2O	N.D.	N.D.	N.D.
K_2O	0.19 ± 0.26	0.48 ± 0.02	0.27 ± 0.08
CaO	1.15 ± 0.13	1.21 ± 0.19	0.68 ± 0.22
MgO	N.D.	N.D.	N.D.

Values are mean ± standard deviation (n = 3). N.D. = Not Detectable. KWK = Kwandonkaya and MGM = Magama Gumua.

Table 1 presents the elemental composition of synthesized Nanoparticles of SiO_2 from Kwandonkaya, Magama Gumau and Toro using XRF. The samples were prepared by the Hydrothermal method, titrating $Na_2.xSiO_2$ solution with hydrochloric acid at neutral pH (~7). Silica gel were obtained with SiO_2 content of 85.70 ± 1.62, 84.76 ± 1.12 and 83.40 ± 1.84 respectively, the ANOVA results revealed no significant difference between the means ($p > 0.05$) of all analysed samples and was followed by the reduction of CaO, K_2O and Fe_2O_3, which were not completely removed. The results obtained is comparable to that of Munasir *et al.,* (2013, 2015).

Functional Groups and Crystallinity of Nanoparticles of SiO_2 : The functional groups from FTIR analysis result of Nanoparticles of SiO_2 from the three different sampling locations in Toro district are presented in Table 2.

Table 2: Functional Groups present in the Nanoparticles of SiO_2 using Fourier Transform Infrared Spectrophotometric Method

Band (cm^{-1})	Bond (Vibration mode)	Compound
3417.97 – 3964.00	H – O – H (Stretching vibration)	Water
2926.36	C – H (Stretching vibration)	Aliphatic
1011.47 - 1079.31	Si – O – Si (Symmetrical stretching)	Quartz
1384.35 – 1384.41	CO_3^{2-} (Asymmetrical stretching)	Calcite
1633.83 – 1659.53	H – OH (Bending vibration)	Water
945.87	Si – O (Symmetrical stretching)	Quartz
802.04	Si – O (Symmetrical bending)	Quartz
731.88 – 793.42	Si – O (Symmetrical stretching)	Quartz
607.79 – 666.76	Si – O – Si (Symmetrical bending)	Quartz
515.71 – 596.68	Si – O – Si (Symmetrical bending)	Quartz
426.60 – 471.97	Si – O – Si (Asymmetrical bending)	Quartz

Figure 1: FTIR Spectrum of Kwandonkaya Nanoparticles of SiO_2

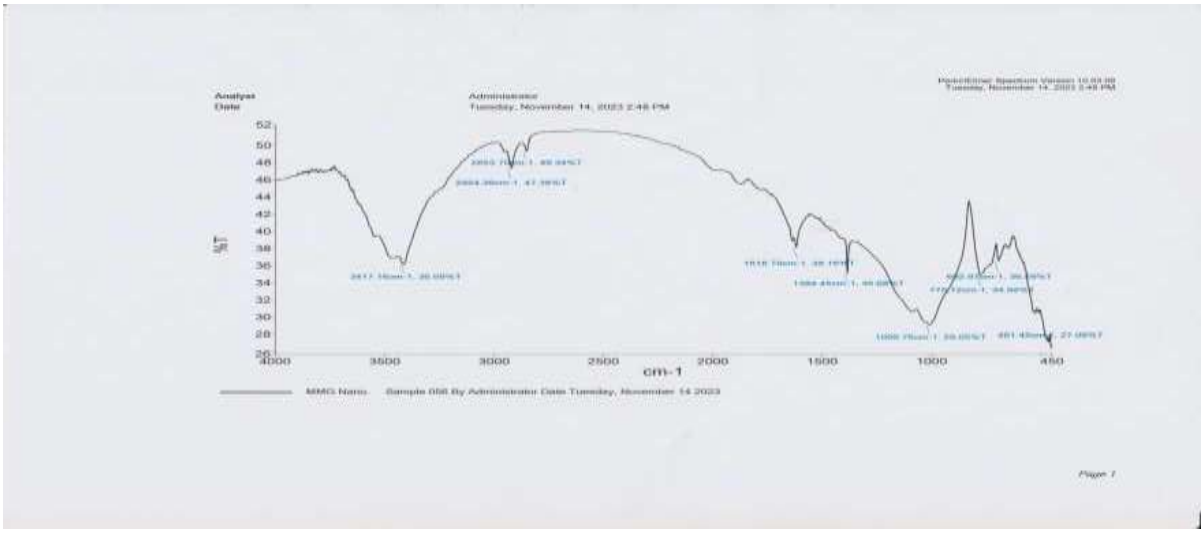

Figure 2: FTIR Spectrum of Magama Gumau Nanoparticles of SiO_2

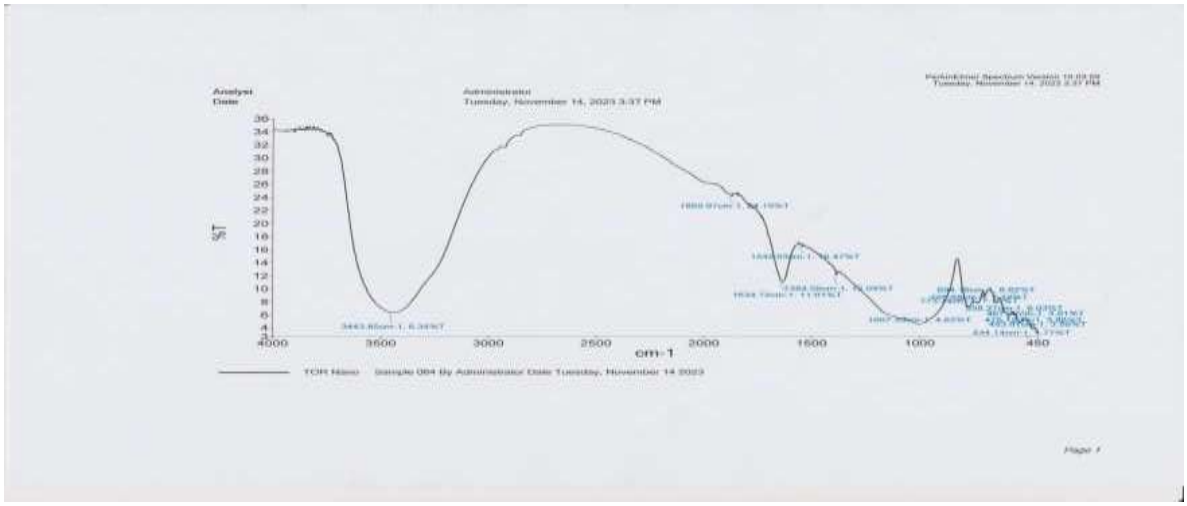

Figure 3: FTIR Spectrum of Toro Nanoparticles of SiO_2

The baseline method was used for the calculation of crystallinity index (CI) of synthesized Nanoparticles of SiO_2. It involves selection of absorption bands of analyte at 693.19 cm^{-1} and 777.12 cm^{-1}. The value of the incident (I_o) radiation was obtained by drawing a straight line tangential to the spectral absorption curve at the position of samples absorption band. The transmitted light (I_t) was obtained at the maximum absorption. The Beer's law was followed with apparent deviation from the law. Where the absorbance A_α at wavenumber α is given as:

$$A_\alpha = \log \frac{I_o}{I_t} \text{ - - - - } 1$$

The result of the calculated crystallinity indices of Nanoparticles of SiO_2 from three different sampling locations of Toro district is shown in Table 2.1.

Table 2.1: Calculated Crystallinity Indices of Nanoparticles of SiO_2

Sample	693.19 (cm^{-1})		777.12 (cm^{-1})		$A_{693.19}$	$A_{777.12}$	CI= $\frac{A_{693.19}}{A_{777.12}}$
	Io	I	Io	I			
KWK	34.00	37.00	32.00	34.00	-0.04	-0.03	1.33
MMG	18.00	23.00	14.00	19.00	-0.11	-0.13	0.84
TOR	20.00	22.00	26.00	30.00	-0.04	-0.06	0.66

KWK = Kwandonkaya, MMG = Magama Gumau and TOR = Toro

The FT-IR evaluations of the spectra and the spectral data of synthesized Nanoparticles of SiO_2 are presented in Table 2. The IR band at 3964.00 cm^{-1} is due to the stretching vibration of H_2O molecules. Correspondingly, the IR band at 1659.53 cm^{-1} is assigned to – OH bending vibrations of H_2O molecules. The very strong and predominant absorbance peaks at 1079.31 cm^{-1} range usually assigned to the mode of the Si – O – Si asymmetric stretching vibrations. The IR band at 945.87 cm^{-1} can be assigned to silanol groups. In the case of alkali silicate glasses, this band is assigned to Si – O – stretching vibrations. The IR band at 802.04 cm^{-1} can be assigned to Si – O – Si symmetric stretching vibrations, whereas the IR band at 471.97 cm^{-1} is due to O – Si – O bending vibrations. Previous study revealed that Si – O group lies in a wave number range of 465 to 475 cm^{-1}, Si–OH group in 800 to 870 cm^{-1}, siloxane Si – O – Si group in 1115 to 1050 cm^{-1}, water molecule O – H group in 1625 cm^{-1} and O – H group in 3000 to 4000 cm^{-1}. The ranged of the calculated crystallinity indices of Nanoparticles of SiO_2 were 0.66 to 1.33 within the three different sampling locations of Toro district shown in Table 2.1. The results indicated that the synthesized quartz is crystalline. The FTIR evaluation of the synthesized Nanoparticles of SiO_2 is comparable to that obtained by Munasir *et al.* (2013, 2015).

IV. CONCLUSION AND RECOMMENDATIONS

Conclusion : The nanoparticles of SiO_2 synthesized from natural quartz materials using hydrothermal method, showed improve purity of SiO_2 and crystallinity indices as well as the presence of the silanol (Si–O), siloxane (Si–O–Si) and hydroxyl groups on the surface of the tested samples. This also, revealed that the nanoparticles of SiO_2 contained both crystalline and amorphous phases.

Recommendations

Based on the results obtained in this study, the following recommendations were made:

- There should be result validation using other synthesis methods such as sol – gel method for the synthesis of nanoparticles of SiO_2.
- The quartz should be calcine before the application of the hydrothermal method.
- Other alkaline based compounds such as sodium trioxocarbonate (IV) and potassium trioxocarbonate (IV) can be used for the synthesis of the nanoparticles of SiO_2.
- Instrumental techniques such as Scanning Electron Microscope (SEM) X-ray Photo electron Spectroscopy (XPS) and Attenuated Total Reflectance – FTIR (ATR – FTIR) and Energy Dispersive X – ray (EDX) should be used alongside XRF and XRD to reduce matrix interference.
- Good Measurement Practice (GMP) and Good Laboratory Practice (GLP) should be observed to ensure quality assurance of the result and the product.

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