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Synthesis and Characterization of Quartz (SiO_2) Materials Using Hydrothermal, X-Ray Fluorescence and X-Ray Diffraction Spectrophotometric Methods

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Abstract

The aim of the present study was the synthesis and characterization of quartz materials using hydrothermal, X-ray fluorescence and X-ray diffraction 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 °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 °C for 24 hours. Characterization of the product was carried out using X-Ray Fluorescence and X-Ray Diffraction 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 synthesized quartz materials XRD results showed structural properties such as crystalline size, planes and diffraction angles as well as the associated minerals present such as albite, anorthite, biotite, muscovite and orthoclase. The synthesized nanoparticles of SiO_2 contained multi-minerals phases.

Keywords: Quartz, Silica, Hydrothermal, XRF, XRD, Beneficiation, Nanoparticles, Precursor

1. 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) [5, 3]. 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) [5].

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) [1] and rice husk ash (Kalapathy *et al.,* 2002; Nittaya and Nuntiya, 2009) [4, 10]. Inorganic materials like silica sands were employed to produce SiO_2 nanoparticle via energy milling (Ahmad *et al.,* 2013) [2]. Diorite sand with Na_2CO_3 addition was sintered at a temperature

of 1030 ° C (Trabelsi *et al*., 2009) [11]. 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 °C to form sodium silicate (Mori, 2003 and Munasir *et al.,* 2013) [6, 7].

2. Experimental Methods

The raw materials used for this study were rock samples from Kwandonkaya, Magama Gumau and Toro (Toro District, Bauchii, Nigeria). The method used by Munasir, 2015 [8] 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 °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 °C for 24 h. The synthesized nanoparticles of SiO_2 were then characterized using XRF to evaluate the elemental composition and XRD to study the structural properties.

3. Results and Discussion

3.1 X-Ray Fluorescence Characterization of 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**	**TOR**
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, MGM = Magama Gumua and TOR = Toro.

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) [7, 8].

3.2 X-Ray Diffraction Characterization of Nanoparticles of SiO_2

The Structural and Mineralogical properties of the Nanoparticles of SiO_2 synthesized using hydrothermal method are presented in Table 2, 2.1, 3, 3.1, 4 and 4.1 respectively.

The range of crystalline sizes of Kwandonkaya nanoparticles of SiO_2 are 30.00 to 123.00 nm and the mineralogical phases present are quartz, anorthite, albite and orthoclase as shown in Table 2 and 2.1.

Table 2: Structural Properties of Kwandonkaya Nanoparticles of SiO_2

2θ°	(d) Å	h	k	l	FWHM (deg)	Crystallite size (nm)	Mineral	Intensity (%)
13.89	6.36	0	0	1	0.15	54.10	Albite	21.45
20.90	4.24	1	0	0	0.18	47.70	Quartz	16.29
22.06	4.02	1	2	-3	0.08	108.00	Anorthite	19.08
23.56	3.77	1	3	-1	0.09	91.30	Anorthite	17.48
25.52	3.48	2	2	-1	0.18	47.20	Orthoclase	25.49
26.67	3.33	1	0	1	0.13	62.10	Quartz	100.00
27.48	3.24	2	2	0	0.14	60.70	Orthoclase	75.17
27.90	3.19	0	4	0	0.12	69.70	Anorthite	89.62
30.11	2.96	1	3	-4	0.28	31.00	Anorthite	67.36
36.56	2.45	1	1	0	0.09	95.10	Quartz	74.37
45.76	1.98	2	0	1	0.07	123.00	Quartz	13.65
50.05	1.82	1	1	2	0.20	30.00	Quartz	36.31
54.91	1.67	1	7	1	0.11	85.60	Orthoclase	4.40
58.67	1.63	4	4	0	0.14	67.70	Albite	9.20
59.96	1.54	2	1	1	0.15	63.50	Quartz	9.46
63.78	1.45	1	1	3	0.17	57.70	Quartz	5.96
64.09	1.45	3	-7	0	0.14	71.20	Albite	8.86
68.17	1.37	3	0	1	0.18	54.70	Quartz	54.94

2θ° = Diffraction Angle, (d) Å = Interplanar distance, h k l = Miller indices and FWHM = Full Width at Half Maximun

Table 2.1: Qualitative and Quantitative Analysis Results of Kwandonkaya Nanoparticles of SiO_2

Phase Name	Chemical Formula	Percentage (%)
Quartz	SiO_2	45.30
Anorthite	$CaAl_2Si_2O_8$	31.50
Albite	$NaAlSi_3O_2$	19.10
Orthoclase	$KAlSi_3O_8$	4.00

The range of crystalline sizes of Magama Gumau nanoparticles of SiO_2 are 36.80 to 92.90 nm and the mineralogical phases present are quartz, anorthite, orthoclase and muscovite as shown in Table 4 and 4.1.

Table 3: Structural Properties of Magama Gumau Nanoparticles of SiO_2

2θ°	(d) Å	h	k	l	FWHM (deg)	Crystallite size (nm)	Mineral	Intensity (%)
9.02	9.79	0	0	2	0.22	36.80	Muscovite	31.85
22.33	3.97	1	2	-3	0.11	74.30	Anorthite	4.94
25.48	3.49	2	2	-1	0.09	92.90	Orthoclase	21.62
26.92	3.30	1	0	1	0.15	55.30	Quartz	100.00
28.53	3.12	0	4	0	0.20	43.50	Anorthite	6.29
36.76	2.44	1	1	0	0.13	69.90	Quartz	6.29
40.53	2.22	1	1	1	0.11	82.10	Quartz	2.65
42.73	2.11	2	0	0	0.17	49.80	Quartz	13.93
51.51	1.77	4	3	-5	0.15	63.40	Anorthite	2.23
60.20	1.53	5	3	-1	0.13	69.20	Orthoclase	10.09
68.54	1.36	5	5	-3	0.26	39.20	Orthoclase	11.45

2θ° = Diffraction Angle, (d) Å = Interplanar distance, h k l = Miller indices and FWHM = Full Width at Half Maximun

Table 3.1: Qualitative and Quantitative Analysis Results of Magama Gumau Nanoparticles of SiO_2

Phase Name	Chemical Formula	Percentage (%)
Quartz	SiO_2	52.00
Anorthite	$CaAl_2Si_2O_8$	25.00
Orthoclase	$KAlSi_3O_8$	8.00
Muscovite	$KAl_3Si_3O_{10}(OH)_2$	14.00

The range of crystalline sizes of Toro nanoparticles of SiO_2 are 19.80 to 78.10 nm and the mineralogical phases present are quartz, albite, biotite and anorthite as shown in Table 4 and 4.1.

Table 4: Structural Properties of Toro Nanoparticles of SiO_2

2θ°	(d) Å	h	k	l	FWHM (deg)	Crystallite size (nm)	Mineral	Intensity (%)
8.97	9.84	0	0	1	0.28	29.40	Biotite	28.14
21.08	4.21	1	0	0	0.12	70.50	Quartz	24.93
23.29	3.81	1	1	1	0.13	65.20	Albite	8.75
24.07	3.69	1	-3	0	0.34	25.20	Albite	35.01
26.69	3.33	1	0	1	0.25	33.70	Quartz	100.00
27.55	3.23	2	0	-2	0.31	27.70	Albite	34.71
30.43	2.93	1	-3	1	0.11	78.10	Albite	9.61
35.57	2.52	3	1	-2	0.30	28.70	Albite	11.41
36.62	2.45	1	1	0	0.20	44.50	Quartz	3.92
37.56	2.39	3	-1	0	0.17	51.70	Albite	4.06
49.97	1.82	1	1	2	0.28	52.50	Quartz	22.30
58.75	1.57	2	-4	-4	0.17	57.50	Albite	6.26
59.95	1.54	3	3	-1	0.21	45.20	Biotite	6.87
68.28	1.37	0	2	3	0.51	19.80	Quartz	17.73

2θ° = Diffraction Angle, (d) Å = Interplanar distance, h k l = Miller indices and FWHM = Full Width at Half Maximum

Table 4.1: Qualitative and Quantitative Analysis Results of Toro Nanoparticles of SiO_2

Phase Name	Chemical Formula	`Percentage (%)
Quartz	SiO_2	55.00
Albite	$NaAlSi_3O_2$	23.60
Biotite	$(H.K)_2(Mg.Fe)_2Al_2Si_3O_{12}$	19.00
Anorthite	$CaAl_2Si_2O_8$	2.00

The XRD data analyses of the Nanoparticles of SiO_2 from the three locations within Toro district synthesized consists of quartz, albite, anorthite, biotite, muscovite and orthoclase with their corresponding planes and angles as indicated by the miller indices. In addition to the XRD data analysis of the mineralogical compositions of the Nanoparticles of SiO_2, the estimated crystallite size was calculated by the well-known Scherrer formula.

$$D = \frac{0.96\lambda}{\beta cos\theta} \quad (1)$$

Where, D, λ, β, θ and 0.96 are the Particle size, wavelength in nanometre, the Full width at Half Maximum, the Peak position and the Scherrer constant respectively.

The XRD pattern of Kwandonkaya nanoparticles of SiO_2 as shown in Table 2 revealed that the diffraction peaks are related to the planes (1 0 0), (1 0 1), (1 1 0), (201), (112), (211), (113) and (301). According to the POWD-12++, (1997) standard pattern, these XRD peaks correspond to quartz with hexagonal crystalline structure and belonging to space group P3121 (152). We observed that the (1 0 1) peak has the highest intensity indicating preferred orientation. The d_{hkl} interplanar spacing has been calculated from the X – ray diffraction profile using the Bragg law:

$$2d_{hkl}Sin\theta = n\lambda \quad (2)$$

Where θ is the diffraction angle, λ is the wavelength of X – rays and n is the order of diffraction. We note that the calculated value of d_{hkl}- spacing (Table 2) matched very well with those of the standard international centre for diffraction data (ICDD). This table also shows that the spacing distances d_{hkl} of 4.24, 3.33 and 2.45 (Å) which is almost the same obtained by Meftah and Mahboub, (2019) and Aderibigbe and Ojuri, (2017) that is 4.43, 3.33 and 2.45 (Å) and hence this affirm the presence of the α – quartz phase in Kwandonkaya nanoparticles of SiO_2 which is the most stable phase of quartz at room temperature (Beddiaf *et al*, 2015). Table 3 showed that the value of β was equal to 0.13, that is full width at half maximum (FWHM) of the most intense diffraction peak, usually measured in radian with its corresponding position 2θ = 26.67° meaning the Bragg angle which is very useful when determining the crystalline size D of the quartz material using the Scherrer's formula.

It is clearly shown from Table 3 that the plane (1 0 1) with d_{hkl} – spacing of 3.30 in angstrom which corresponds to the highest peak having a value of 100.00 % followed by the plane (2 0 0) with intensity 13.93 % indicating proper orientation. According to POWD – 12 ++, the planes of Magama Gumau quartz also corresponds to hexagonal crystal system with space group P3121 (152), but at diffraction angle (2θ = 9.02) having intensity of 31.85 % meaning plane (0 0 2) it's purely muscovite with Anthorthic as crystal system; space group P – 1 (2). Other diffraction angles such as 22.33, 25.48, 28.53, 51.51, 60.20 and 68.54 correspond to planes (1 2 -3), (2 2 -1), (0 4 0), (4 3 -1), (5 3 -1) and (5 5 -3) respectively. It is a combination of quartz, Anorthite, Muscovite and Orthoclase minerals.

Diffraction angle of 26.69 Å corresponding to plane (1 0 1) has highest peak of 100 % as in Magama Gumau

nanoparticles of SiO_2 indicating proper orientation, followed by plane (1 0 0) having diffraction ($2\theta = 21.08^o$). All planes of this sample are purely quartz having a hexagonal crystal system with space group P3121 (152). Furthermore, Toro Quartz has a (Å) = b (Å) = 4.90 and c (Å) = 5.40. It is noted that the calculated value of d_{hkl} - spacing (Table 4) matched very well with those of international centre for diffraction data (ICCD), also from this, the spacing distances d_{hkl} of 4.21, 3.33 and 2.45 (Å) which are in conformity with that obtained by Meftah and Mahboub, (2019) and Aderibigbe and Ojuri, (2017) affirm the presence of the α – quartz phase in Toro nanoparticles of SiO_2.

4. Conclusion and Recommendations

4.1 Conclusion

The nanoparticles of SiO_2 synthesized from natural quartz materials using hydrothermal method, showed improve purity of SiO_2 and contained minerals such as quartz, albite, anorthite, biotite, muscovite and orthoclase.

4.2 Recommendations

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

1. There should be result validation using other synthesis methods such as sol – gel method for the synthesis of nanoparticles of SiO_2.
2. The quartz should be calcine before the application of the hydrothermal method.
3. Other alkaline based compounds such as sodium trioxocarbonate (V) and potassium trioxocarbonate (V) can be used for the synthesis of the nanoparticles of SiO_2.
4. 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.
5. 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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