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ASSESSMENT OF PURITY AND CRYSTALLINITY OF QUARTZ (SiO_2) MATERIALS USING X-RAY FLUORESCENCE AND FOURIER TRANSFORM INFRA – RED SPECTROPHOTOMETRIC METHODS

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ABSTRACT : The aim of the investigation was to carry out assessment of purity and crystallinity of quartz materials using X-ray fluorescence and fourier transforms infrared spectrophotometric methods. The objectives of the study were to modify standard analytical methods and to provide analytical/geochemical data which 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. The purity analysis of the samples was carried out using X-Ray Fluorescence Spectrophotometric methods, while the crystallinity was determined using Fourier Transform Infrared Spectrophotometric method. The XRF analysis of quartz samples revealed the presence of 68.12 ± 0.08 to 75.63 ± 1.05 % SiO_2, 7.34 ± 0.15 to 8.36 ± 9,20 % Al_2O_3, 1.16 ± 0.09 to 4.33 ± 0.15 % CaO and 0.24 ± 0.06 to 0.60 ± 0.34 % MgO. A sharp absorption band at 1010.18 cm^{-1} has been observed and seems to fit with symmetrical stretching of Si–O–Si bond. Symmetrical bands at 777.12 and 693.12 cm^{-1} have been observed and correspond to Si-O symmetrical bending vibration. These peaks confirmed the presence of quartz. In addition, we observed other bands at 588.91 and 475.14 cm^{-1} which coincided with Si–O–Si symmetrical and asymmetrical bending respectively. The presence of Si-O and O-Si-O vibrations in our sample again confirmed the presence of quartz. The values of crystallinity indices ranged from 0.25 to 0.50. It was also found that the structure of this quartz materials is crystalline in nature. X-Ray Fluorescence and Fourier Transform Infrared spectrophotometric methods were utilized to evaluate the utility and reliability as well as the merits of these analytical methods in the analysis of quartz and other minerals. The output of the study can serve as a practical manual for Geochemists, Laboratory Technologists/Scientists, Mineralogists and Engineers.

KEYWORDS: Quartz, Silica, Crystallinity indices, XRF, FTIR, Beneficiation, Nanoparticles, Solar

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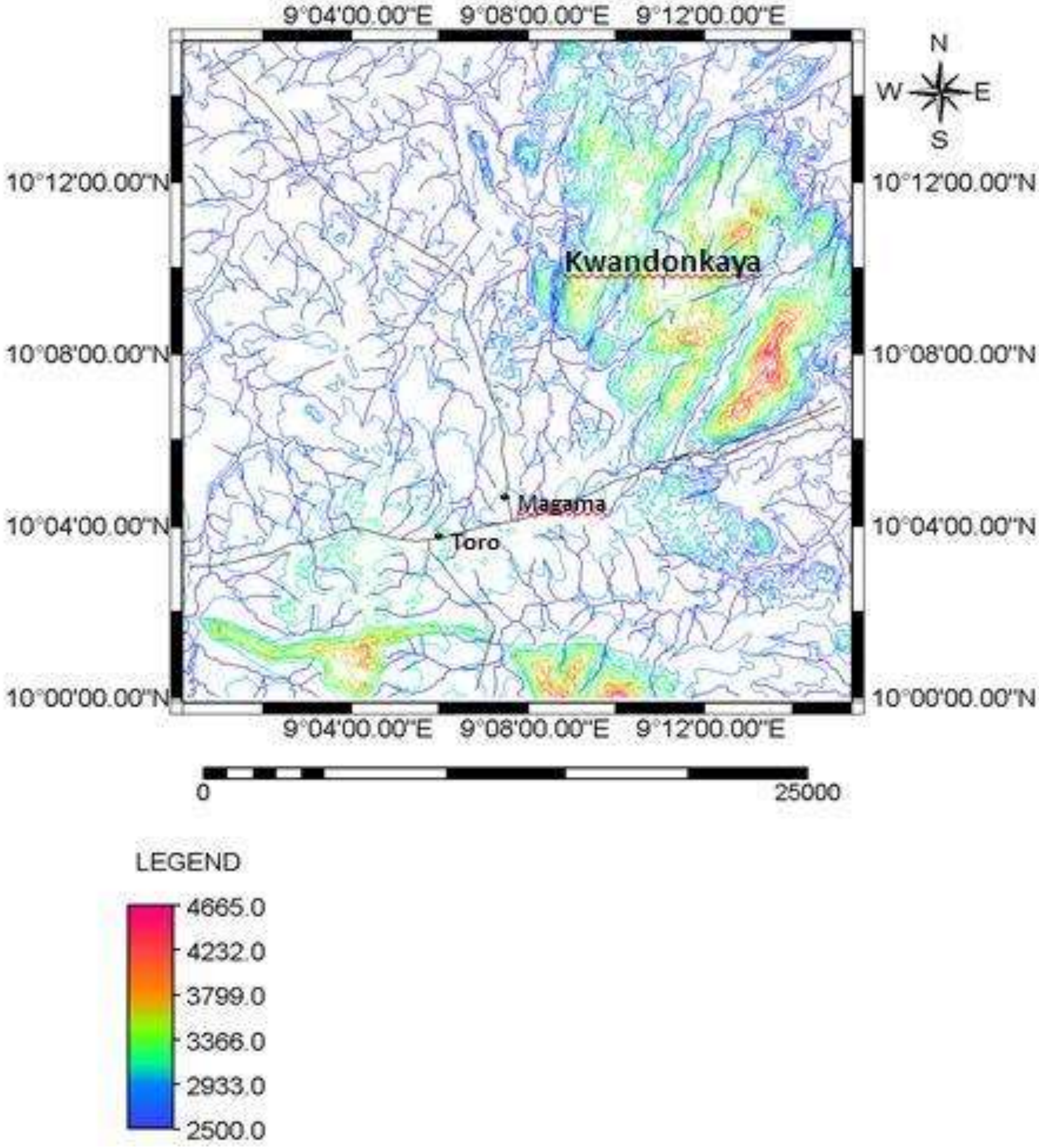
I. INTRODUCTION

Today, high purity quartz has become a vital mineral, with its particular physicochemical properties being employed in a wide range of nanotechnologies such as glass fabrication, optics and microelectronics, semiconductors and telecommunications (Moore, 2005). Quartz contains silicon, which can be exploited in various fields, including medicine, sensor and bio-sensing, photonics, energy technologies and solar application (Hernandez *et al.*, 2015). Quartz is characterized as high purity, only when it contains less than 50.00 $\mu g\ g^{-1}$ of impurities including structurally bound trace elements (B, Li, Ge, Ti, Fe, Mn, Ca, K, Na and P) in the quartz lattice, but also micro minerals inclusions and entrapped liquids, Ultra-pure quartz is rare in nature and larger deposits thereof even more rare. The few ultra-pure found around the world include some kinds of quartz-rich pegmatite and hydrothermal quartz veins (Műller and Ihlen, 2007). Crystallinity is found to be helpful in determining the colour alteration in bedded cherts, as there is a relationship between quartz crystallinity index, grain size of quartz and conodont colour alteration index (Teji *et al.*, 2002). It is well known that natural occurrences like lightning striking quartz sand or sandstones can cause vitreous silica to develop (Saikia *et al.*, 2008 a). It is also recognized as an indicator of crystalline forms of other minerals associated with it. When crystallinity of quartz is at peak, the associated minerals are expected to be well crystalline form. The peak value of crystallinity denotes an ordered state of the minerals, while the least value denotes a disordered state. It is therefore paramount to find quartz's crystallinity rather than the associated minerals (Saikia *et al.*, 2008 a).

II. MATERIALS AND METHODS

Description of the Study Area : The area is about 60.00 km Southwest of Bauchi capital of Bauchi State, between metric grid coordinates between 500554.37 to 5275574.49 and 1105629.11 to 1133101.25 which corresponds to Latitudes 10 o 00’ N to 10 o 15’ N and Longitudes 9 o 00’ E to 9 o 15’ E.



Collection of Rock Samples: Rock samples containing quartz minerals were collected using Hammer from Kwandonkaya, Magama Gumau and Toro**.**

Identification of Rock Samples using Mohs Hardness Scale: The Mohs hardness scale was used as a convenient way to identify the minerals. A mineral’s hardness is a measure of its relative resistance to scratching, measured by scratching the mineral against another substance of known hardness on the Mohs Hardness Scale. A smooth unscratched surface of the rock specimen was selected for testing. The rock specimen was held firmly and scratched with the point of an object of known hardness, in this case a sharp quartz (H = 7) crystal was used. The point of the crystal was firmly pressed against the unidentified rock specimen and a feel of definite “bite” into the surface of the rock specimen was not felt, when a finger was used to observe a line etched (scratched) into the surface of the rock specimen. The test was repeated using a sharp point and fresh surface (NIMG, 2020).

Pre-treatment of Rock Samples : The rock samples collected were crushed using Jaw Crusher, sampled with a Riffle box sampler and milled into fine powder (Size: 200 mesh) using Ball Ring Machine Model Number: BV 1111, stored in a polyethylene bag, sealed and labelled appropriately for further analyses (NIMG, 2020).

Determination of Elemental Compositions of Quartz (SiO_2) Materials using X-Ray Fluorescence Spectrophotometric Method

In X-Ray Fluorescence analysis, high energy x-rays are generated in an x-ray tube by high velocity electron bombardment of metallic anode. These primary x-rays are directed upon the sample to be analyzed. Every element within the sample is excited and emits its own characteristic X-ray spectrum. The intensity of any characteristic X-ray generated is a function of the identity of the element present in the sample. These phenomena provide the basis for a method of chemical analysis. Specific elements within a sample can be identified by the radiation wavelength emitted. The amount of the specific element present can be ascertained by measuring the intensity of its characteristic radiation.

$2d \sin\theta = \lambda$ ------- 1

In practice, the various wavelength components of secondary fluorescent radiation emitted by a sample are dispersed by means of a movable diffracting crystal of known inter-atomic spacing, d. The X-ray fluorescence method of analysis has some distinct advantages over conventional wet-chemical methods in many applications. The method is non-destructive towards the sample being analyzed. Its results are not significantly affected by the state of oxidation or chemical combination of the elements analyzed. The powdered sample was inserted into the X-ray fluorescence spectrophotometer. The X-ray PC was used to select the program for the sample and then identified the sample. The analysis was started, which lasted for about 2-3 min and the result was displayed on the PC screen. Attention was always drawn to the total percentage concentration. Normally, after running the samples, if the total percentage concentration is below 98.00 %, the sample is removed and the monitor runs to standardize the instrument, after which the sample is re-run again (NIMG, 2020).

Determination of Crystallinity of Quartz using Fourier Transform Infra-Red Spectrophotometric Method : Infrared spectroscopy provides a rapid, simple and convenient nondestructive means of characterizing and identifying minerals. It can readily indicate the presence of specific atomic groupings within the crystal structure. It is probably the best technique for detecting the presence of water in a mineral and for indicating the form in which the water is present. It is also a powerful means of detecting carbonate and of providing clues to the nature of the silicate anion in the mineral structure. Infrared studies are able to detect non-crystalline phases and some minerals are more easily identified from their IR spectra than from their X-ray diffraction patterns.The powdered sample was homogenized in a spectrophotometric grade KBr (1: 20) in an agate mortar and pressed with 3 mm pellets using a hand press (Saikia *et al.,* 2008 b).

III. RESULTS AND DISCUSSION

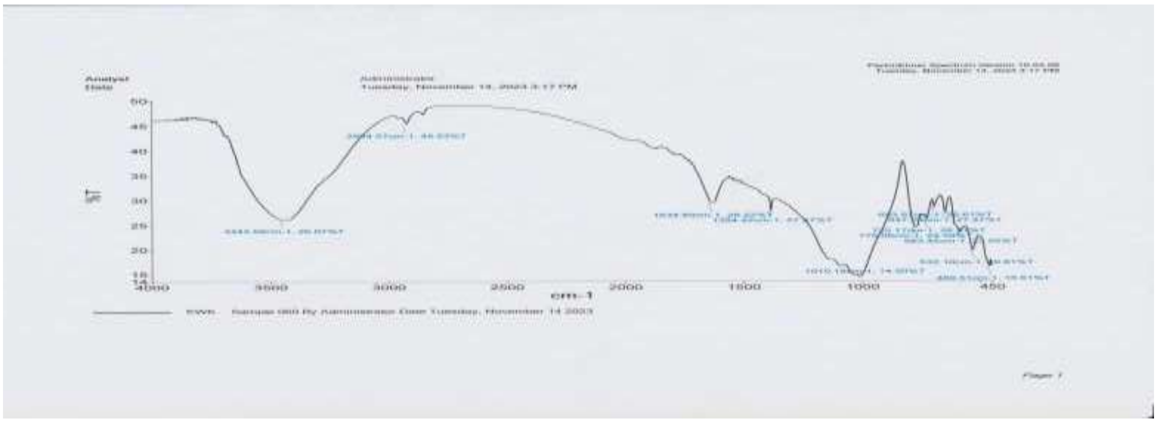
Elemental Composition of Major Oxides in Quartz Materials : The percentage composition of major oxides in quartz materials using X – Ray Fluorescence method are presented in table 1.

Table 1: Percentage Composition of Major Oxides in Quartz Materials using X – Ray Fluorescence Method

% Composition Oxides	KWK	MMG	TORO
SiO_2	75.63 ± 1.05	74.65 ± 1.14	68.12 ± 0.80
Al_2O_3	8.36 ± 0.20	7.34 ± 0.15	7.87 ± 0.69
Fe_2O_3	2.79 ± 0.87	5.08 ± 0.23	8.04 ± 0.54
MnO	0.01 ± 0.00	0.03 ± 0.01	0.22 ± 0.18
Cr_2O_3	N.D.	0.02 ± 0.00	0.02 ± 0.00
TiO_2	0.11 ± 0.02	0.75 ± 0.14	1.29 ± 0.29
P_2O_5	1.59 ± 0.04	1.68 ± 0.13	1.50 ± 0.11
Na_2O	1.52 ± 0.20	1.31 ± 0.08	1.71 ± 0.09
K_2O	7.82 ± 0.05	6.05 ± 0.08	6.04 ± 0.64
CaO	1.16 ± 0.09	2.11 ± 0.02	4.33 ± 0.15
MgO	N.D.	0.24 ± 0.06	0.60 ± 0.34

Values are mean ± standard deviation (n = 3). ND = Not Detected. KWK = Kwandonkaya and MGM = Magama Gumau

The results of XRF analysis of quartz materials from three different sampling locations in Toro district is shown in Table 1. It reveals that the predominant oxides were SiO_2, Al_2O_3, Fe_2O_3 and K_2O with the percentage ranged of 68.12 ± 0.80 – 75.63 ± 1.05, 7.34 ± 0.15 – 8.36 ± 0.20, 2.79 ± 0.87 – 8.04 ± 0.54 and 6.04 ± 0.64 – 7.82 ± 0.05 % respectively. Obiefuna *et al.* (2018) obtained similar results with SiO_2 content (70.58 %), Al_2O_3 (14.30 %), K_2O (5.44 %), Fe_2O_3 (2.73 %) and CaO (2.62 %). ANOVA results revealed no statistically significant difference between the two groups since ($p > 0.05$).



Functional Groups and Crystallinity of Quartz Materials : The functional groups and the calculated crystallinity indices found in the quartz materials from the three sampling locations within Toro district are presented in Table 2 and Table 2.1.

Table 2: Functional Groups present in Quartz Materials using Fourier Transform Infrared Spectrophotometric Method

Band (cm^{-1})	Bond (Vibration Mode)	Compound
3417. 16 – 3443.85	H – O – H (Stretching vibration)	Water
2869.97 – 2924.69	C – H (Stretching vibration)	Aliphatic
1542.03 – 1637.04	H – OH (Bending vibration)	Water
1364.46 – 1384.51	CO_3^{2-} (Asymmetrical stretching)	Calcite
1008.02 – 1010.18	Si – O – Si (Symmetrical stretching)	Quartz
725.17 – 777.12	Si – O (Symmetrical stretching)	Quartz
647.06 – 693.19	Si – O – Si (Symmetrical bending)	Quartz
530.50 – 588.91	Si – O – Si (Symmetrical bending)	Quartz
453.92 – 475.14	Si – O – Si (Asymmetrical bending)	Quartz

Figure 2: FTIR Spectrum of Magama Gumau Quartz Materials

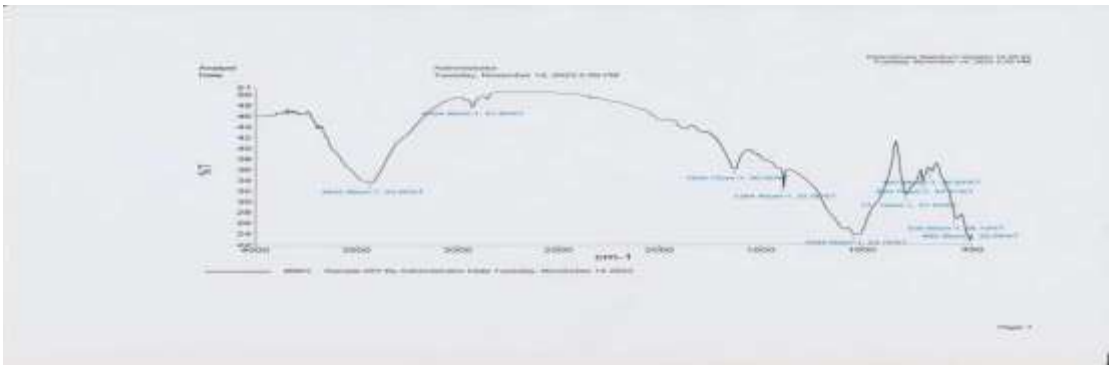


Figure 3: FTIR Spectrum of Kwandonkaya Quartz Materials

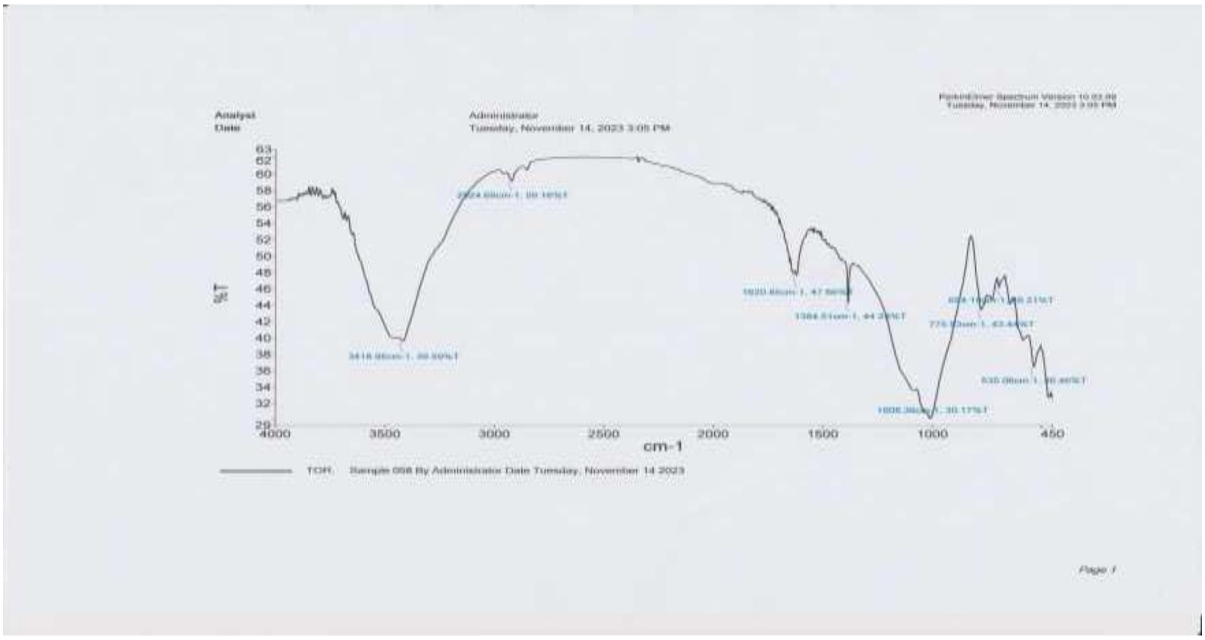


Figure 4: FTIR Spectrum of Toro Quartz Materials

The baseline method was used for the calculation of crystallinity index (CI) of quartz in the samples. 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 still followed with apparent deviation from the law. Where the absorbance A_α at wavenumber α is given as:

$A_\alpha = \log \frac{I_o}{I_t}$ - - - - 2

Table 2.1: Calculated Crystallinity Indices of Quartz Materials

Sample	693.19 (cm^{-1})		777.12 (cm^{-1})		$A_{693.19}$	$A_{777.12}$	$CI = \frac{A_{693.19}}{A_{777.12}}$
	I_o	I_t	I_o	I_t			
KWK	26.00	31.00	24.00	34.00	-0.08	-0.15	0.50
MMG	34.00	36.50	32.00	38.00	-0.03	-0.07	0.43
TORO	44.50	47.00	43.00	51.50	-0.02	-0.08	0.25

KWK = Kwandonkaya and MMG = Magama Gumau

From the FTIR spectrums we can identify the main components of our samples from the three sampling locations within Toro district. Table 2 summarizes the functional groups found in the quartz materials. In the range of the high wavenumbers, we see a high intensity absorption band at 3443.85 cm^{-1} which was due to stretching vibrations of hydroxyl groups (OH), a less intense band has been observed at 1637.04 cm^{-1}, which was due to the twisting of H-O-H. Also, absorption band have been observed at wavenumbers of 1384.51 cm^{-1}, which were due to CO_3^{2-}. A sharp absorption band at 1010.18 cm^{-1} has been observed and seems to fit with symmetrical stretching of Si–O–Si bond. Symmetrical bands at 777.12 and 693.12 cm^{-1} have been observed and correspond to Si-O symmetrical bending vibration. These peaks confirmed the presence of quartz. In addition, we observed other bands at 588.91 and 475.14 cm^{-1} which coincided with Si–O–Si symmetrical and asymmetrical bending respectively. The presence of Si-O and O-Si-O vibrations in our sample again confirmed the presence of quartz. Similar work by Aderibigbe and Ojuri (2017) shows quartz was found and its crystalline form was confirmed by the presence of Si - O symmetrical stretching vibrations at 1119.61 - 967 cm^{-1}, Si – O asymmetrical stretching vibrations at 1180 - 1120 cm^{-1} and Si - O symmetrical bending vibrations at 693 cm^{-1}. The silica was in the bands at 800 cm^{-1}, 822.79 cm^{-1} and 786.23 cm^{-1}, which showed that the silica was present as quartz. The crystallinity index (CI) of quartz in our samples was calculated by measuring the ratio between the absorbance of the bands 693.19 cm^{-1} and 777.12 cm^{-1} ($A_{693.19}/A_{777.12}$), where the absorbance A_α at wavenumber α is given as:

$A_\alpha = \log \frac{I_o}{I_t}$ - - - -3

The level of crystallinity index (CI) of quartz materials from three different sampling locations in Toro district shown in Table 2.1, indicates that the samples were crystalline in nature and within the range of 0.25 to 0.50. The result obtained is similar to that of Meftah and Mahbouh (2019).

IV. CONCLUSION AND RECOMMENDATIONS

Conclusion : X – Ray Fluorescence and Fourier Transform Infra – Red Spectrophotometric methods were utilized to evaluate the utility and reliability as well as the merits of these analytical methods in the analysis of quartz and other minerals. Purity, crystallinity, Si-O functional bonding were used to characterize the quartz materials. The results can be of benefit to the mining, ceramic, glass, nanoparticles, material science and metallurgical industries. The output can serve as a practical manual for Geochemists, Laboratory Technologists, Mineralogists, Scientists and Engineers in general.

Recommendations

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

- There should be result validation using other methods different from XRF and FTIR in the analysis of quartz and other solid minerals.
- Instrumental techniques such as XRD, SEM, X-ray Photo electron Spectroscopy (XPS) and Attenuated Total Reflectance – FTIR (ATR – FTIR) and Elemental Detector X-ray (EDX) should be used alongside XRF and FTIR to reduce matrix interference.
- A method for the separation of mineral components of rock sample is necessary for beneficiation of Quartz.
- Less crystalline rock should be utilized for a good source of (SiO_2) for industrial applications.
- Proper health monitoring of the residents and employees of the mining area for exposure to heavy metals and disease like silicolisis should be carry out.

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