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Physicochemical Parameters and Heavy Metal Oxide Concentrations in Fluorspar Deposits from Liji Hills, Yamaltu-Deba LGA, Gombe State, Nigeria

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ABSTRACT

Fluorspar mining poses significant environmental and health risks, including toxicity, as it contaminates nearby water bodies, which can lead to fluorosis, characterized by brown teeth and skeletal deformities among residents. This study analyzed two fluorspar mineral samples—Greenish brown (A) and Bluish brown (B)—collected from Liji Hills, near Gombe, to determine their metal oxide composition, crystal structure, and physicochemical properties, and to compare these values with the WHO permissible limits. Physicochemical analysis revealed that the pH of sample A decreased with time (mean = 6.91), while sample B fluctuated (mean = 6.71). Electrical conductivity decreased with time for both samples, averaging 0.25 N S cm^{-1} for A and 0.29 N S cm^{-1} for B. Ash content was 3.7 % for A and 2.9 % for B, while moisture content was 4.16 % and 1.0 %, respectively. X-ray fluorescence (XRF) analysis of sample A indicated major oxides in descending order: CaO = 91.5 mg L^{-1} > SiO_2 = 3.36 mg L^{-1} > Al_2O_3 = 0.818 mg L^{-1} > P_2O_5 = 0.593 mg L^{-1} > TiO_2 = 0.463 mg L^{-1} > Fe_2O_3 = 0.418 mg L^{-1} > MnO = 0.0246 mg L^{-1} > K_2O = 0.0051 mg L^{-1}. Sample B showed CaO = 81.4 mg L^{-1} > Fe_2O_3 = 6.63 mg L^{-1} > SiO_2 = 5.65 mg L^{-1} > TiO_2 = 0.879 mg L^{-1} > P_2O_5 = 0.612 mg L^{-1} > MnO = 0.0204 mg L^{-1}. X-ray diffraction (XRD) revealed that sample A had an average crystallite size of 75.87 nm with a face-centered cubic structure and Bragg angles between 25.75° and 68.77°, while sample B had a size of 13.65 nm with similar symmetry and Bragg angles from 25.94° to 56.17°. Both samples exceeded WHO limits for CaO and Fe_2O_3, indicating contamination risk. Although Pb and As were not detected, the greenish-brown sample exhibited a higher metal oxide content and greater structural quality, suggesting that Liji Hills fluorspar may contribute to environmental pollution and adverse health outcomes in nearby communities.

INTRODUCTION

Fluorspar, also known as Fluorite (CaF2), is a popular industrial mineral used as a raw material in the metallurgical, ceramic, and chemical industries, as well as for optical applications. It serves as a source of fluorine raw material for the production of hydrogen fluoride or hydrofluoric acid, which is used as a feedstock for numerous organic and inorganic chemical compounds. Fluorspar was then recognized to be calcium fluoride [1]. The use of uranium hexafluoride in the separation of uranium isotopes, along with the development of organic fluorine compounds of industrial importance, made fluorine an industrial chemical of considerable use [1]. The mineral fluorspar, popularly known as Fluorite (CaF2), has been used for centuries as a flux in the washing of various metallurgical processes. The name fluorspar is derived from the Latin fluere, "to flow." The mineral subsequently proved to be a source of the Calcium and Fluorite elements, which were accordingly named fluorine.

Fluoride constitutes a naturally occurring chemical substance present in minor quantities in air, water, soil, plants, animals, and humans [2]. It is the most electronegative of all chemical elements and is therefore characterized by a bluish tinge when illuminated, a property known as fluorescence. Fluorspar is the rock containing the mineral fluorite (CaF_2), a purple product used in the glazing industry, making of fiberglass,

manufacturing of toothpaste, aluminium, steel, uranium fuel, refrigerants, and making of fiberglass, manufacturing of toothpaste, aluminium, steel, uranium, fuel, refrigerants, also used in uranium fuel, refrigerants, and insulating foams [2]. Most of the minerals especially fluorspar accumulated heavy metals such as lead, mercury, uranium, chromium, fluoride, and Arsenic are toxic and the constituent element found in the deposits mineral and areas affected by fluoride concentration between 2.5-3.9 mg/L experience mottling of teeth [2], which indicated that unique fluorspar is commonly associated with other minerals such as quartz, barite, calcite, galena, siderite, celestite, chalcopyrite and phosphates [3]. Thus, the values of oxides of metals and crystal structure in Fluorite mineral samples from the Liji mining area were compared with the WHO permissible limits to ascertain the level of contamination.

METHODOLOGY

Study Area

The Liji Fluorspar mining area is located at the outskirts of the Gombe metropolis, approximately 1km from the Gombe abattoir, but is correctly situated in Liji ward of Yamaltu Deba Local Government. The area is situated at a latitude of 10.32 ° North and a longitude of 11.38 ° East, with an altitude of 239.00 m (784.12 ft) above sea level [4], as illustrated in **Figs. 2, 3** and **4**). The topography of the study area has a moderately gentle to undulating or strong slope with a hilly structure composed of valuable minerals, such as granite and fluorspar; the vegetation is of the tropical Sudan Savanna type. Human interference through construction, quarrying of stones, mining, and farming activities has helped modify the vegetation. The exploration of minerals in the area contaminated the water, soil, and air quality by releasing harmful pollutants into the environment [4].

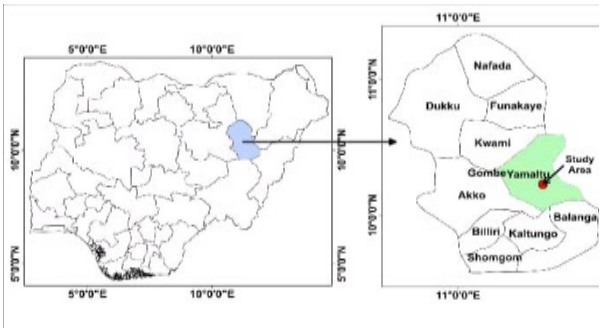

Fig. 1. Map of Nigeria Showing Yamaltu Deba Local Government [4].

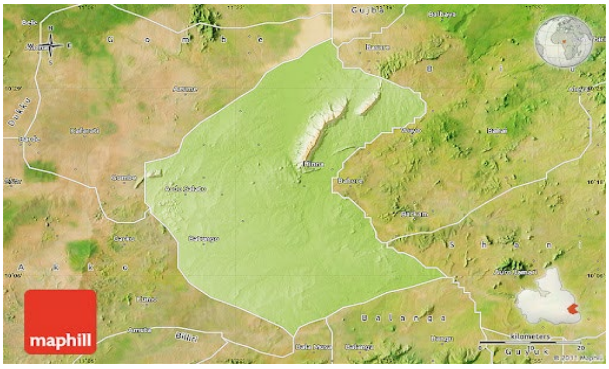

Fig. 2. Elevation map of the Yamaltu Deba Local Government area [4].

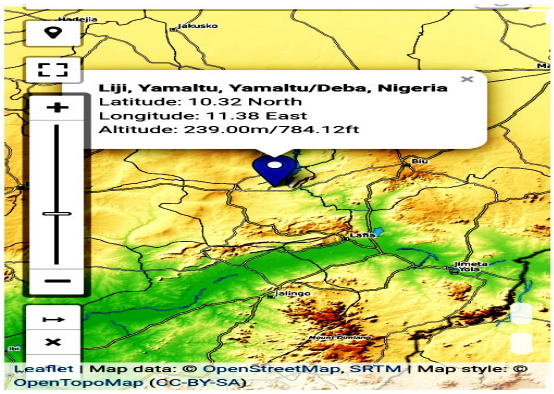

Fig. 3. Sampling area of the two fluorspar minerals.

Sample Collection

Two fluorspar minerals, Green Brown (A) and Bluish Brown (B), were collected at their respective mine locations, which are about 200 meters apart. The samples collected were placed in a clean sample bottle and transferred to the Geology Department at Gombe State University, which was identified by the geologists and adopted with slight modifications by [5].

Sample Preparations

Analysis of pH Samples

Fluorspar samples in the solid form was crushed into a powder form. About 2 g of the sample was be accurately weighed and placed into a 25 cm^3 beaker and 20 $cm^3$of distilled water was added. It was stirred with a glass rod and shaken moderately, and then allowed to become a homogeneous mixture. A pre-calibrated pH meter (Jenway 3150) was inserted into the slum, and pH values were recorded as adopted by [5]. The pH content of the fluorspar was determined to investigate the relationship between the concentration of heavy metals and pH values at the mining sites.

Electrical Conductivity Analysis of Fluorspar

Approximately 2g of the samples was shaken in 500 cm^3 of distilled water in an extraction bottle using a mechanical shaker for 1 hour. The suspension was filtered twice to remove turbidity, and two drops of 0.1% of Na_2PO_3 was added to the filtrate. To determine the electrical conductivity of fluospar and water, a probe of the conductivity meter was inserted, and values were recorded in NS cm-1, as adopted by [6].

Analysis of Moisture Content of Fluorspar

This was done according to the modification, as 2 g of the sample (fluorspar mineral) was weighed (W1) into a pre-weighed crucible (Wo) and placed in a hot drying oven at 1050 °C. The crucible was removed, cooled in a desiccator, and weighed. The process of drying, cooling, and weighing was repeated until a constant value was obtained. The weight loss due to moisture content was then calculated using the equation [6].

$$\% \text{ Moisture} = \frac{W1-W2}{W1-W0} \quad X\ 100$$

X-Ray Fluorescence (XRF) analysis'

Fluorspar mineral samples were carefully cleaned using distilled water to remove adhering soil and organic debris. The samples were dried in an oven at 105 °C for 2 hours to eliminate moisture.

The dried sample was then crushed using an agate mortar and pestle and then sieved through a 75 µm mesh to obtain a homogeneous fine powder suitable for X-Ray Fluorescence (XRF) analysis. Approximately 5 g of the powdered sample was mixed with a few drops of polyvinyl alcohol (PVA) solution as a binder. The mixture was compressed under a hydraulic press at 20 tons cm^{-2} to form a smooth, compact pellet of 32 mm diameter. The prepared pellets were stored in a desiccator to prevent atmospheric contamination and moisture absorption prior to analysis. Elemental composition of the sample was determined using a PANalytical Epsilon 1 Energy Dispersive X-Ray Fluorescence (EDXRF) spectrometer equipped with a rhodium (Rh) anode X-ray tube operated at 50 kV and 1 mA.

The system was calibrated with certified reference standards to ensure accuracy. Measurements were performed under vacuum to enhance detection sensitivity for light elements. Each sample was analyzed in triplicate, and the average value was recorded. The XRF spectra obtained were processed using Omnian software for semi-quantitative oxide determination. The results were expressed as percentage weight of corresponding metal oxides (e.g., CaO, SiO_2, Al_2O_3, Fe_2O_3). The accuracy of results was validated by comparison with certified reference material (NIST SRM 2711a). The concentration of detected oxides was further compared against the World Health Organization (WHO) permissible limits for trace metals in soils and minerals to evaluate potential health and environmental risks.

RESULTS AND DISCUSSIONS

pH Analysis of Fluorspar

The physicochemical parameters results of different characterization techniques done on this research, which showed the pH Analysis that was carried out using a pH meter of model (3150 Jenway). The result of the pH analysis for sample A and B Fluorspars mineral samples is illustrated in Fig. 4, where the pH was recorded at different times in an interval of one hour. Sample A at 1 hour (7.01), 2 hours (6.75), 3 hours (6.89), and 4 hours (6.99) revealed a Total pH mean of 6.91. The pH values shown are fluctuating with an increase in the time interval of dissociation. While the pH value of sample B was obtained at different time intervals, the 1-hour interval was used. (6.75), 2Hr (6.55), 3Hr (6.80), 4Hr (6.74) showed a total mean of 6.71. The pH values for sample B trended downwards at the second hour, while they fluctuated with increasing time of dissociation at the final tested hour. The result obtained was closer to the pH values range of 4.61 to 6.73 as reported by the findings of [5]. The result obtained is contrary to the findings of [7], which showed a pH value range of 0.33 to 12.59, where alkalinity and acidity increase with an increase in dissociation time.

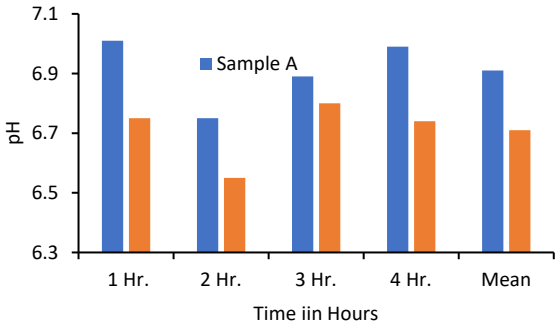

Fig. 4. pH values of sample A fluorspar mineral.

Conductivity Test of Samples

The electrical conductivity of the two fluorspar mineral samples, A (Greenish brown) and B (Bluish brown), was measured using a conductivity meter at hourly intervals. For sample A, conductivity values increased gradually over time—1 h (0.21 N S cm^{-1}), 2 h (0.25 N S cm^{-1}), 3 h (0.27 N S cm^{-1}), and 4 h (0.28 N S cm^{-1})—with an overall mean of 0.25 N S cm^{-1}. Similarly, sample B showed a steady increase in conductivity with time—1 h (0.27 N S cm^{-1}), 2 h (0.28 N S cm^{-1}), 3 h (0.29 N S cm^{-1}), and 4 h (0.30 N S cm^{-1})—and a mean value of 0.29 N S cm^{-1}.

These findings indicate that both mineral samples were found to exhibit a rising ionic mobility with the increase in contact time. This is likely due to dissolution of ionic species from the fluorspar matrix into the aqueous medium. The conductivity trend observed in this study shows notable similarities to that reported by Usman and Maitera [7], in which the latter investigated the proximate and quality assessment of coal deposits at Maiganga in Akko Local Government Area, Gombe State. In both studies, it was found that the conductivity increased progressively with time, demonstrating enhanced solubility and ion release during the dissolution process.

The similarity suggests that both mineral systems exhibit appreciable ionic constituents that contribute to measurable conductivity over time, with a notable difference lying in the magnitude and source of ionic species. In the coal samples analyzed by Usman and Maitera [7], it showed lower conductivity values associated primarily with organic matter and trace mineral content, while the fluorspar samples in this study recorded higher conductivity values (0.25–0.30 N S cm^{-1}), which is attributed to the predominance of calcium and fluoride ions from CaF_2 and associated oxides. This indicates that fluorspar minerals, being more ionic in nature than coal, can contribute a greater ionic strength to the solution, which enhances the electrical conductivity relative to the coal deposits as examined in [7].

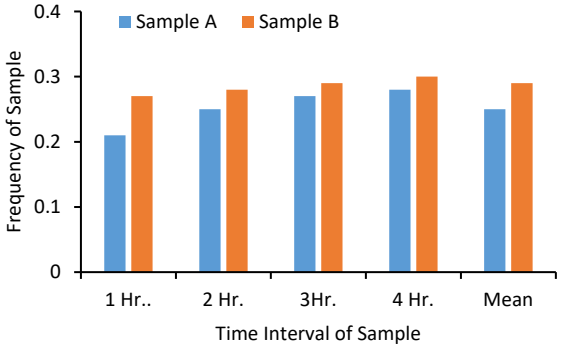

Fig. 5. Conductivity values of Samples A and B fluorspar minerals.

Moisture Content

The moisture Analysis was conducted on Fluorspar samples, as represented in Fig. 6. The results of weighed samples using a crucible and placed in an oven for one hour were calculated for both samples. The parameters used for this analysis were A = 4.15% and B = 1% of moisture content investigated. The result obtained was contrary to the reported moisture content of 9.5% as found by [7].

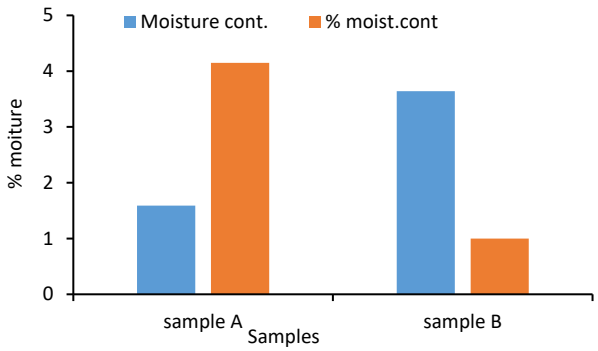

Fig. 6. Percentage values for the moisture content of the two fluorspar minerals.

Ash Content

The Ash content of two samples of Fluorspar mineral was shown in **Fig. 7**. The residue of the analyzed samples obtained as ash content revealed the moisture content, and the samples' ash content was determined using the required calculated parameters, the percentage of the ash content was calculated as sample A = 3.7% and sample B = 2.9% respectively. The percentage of Ash content determined was contrary to the calculated ash percentage range of 10%, as reported by [6].

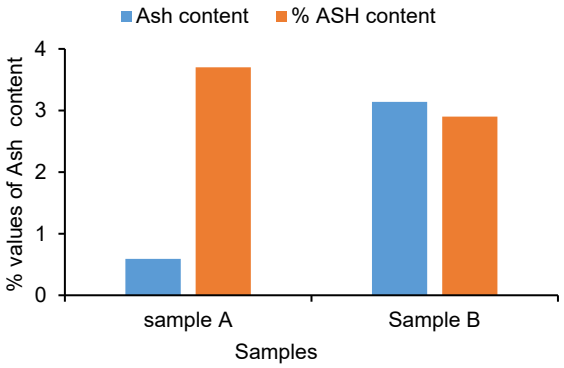

Fig. 7. Percentage of Ash content of the two fluorspar minerals.

X-Ray Fluorescence Analysis of Sample A

The XRF Analysis of sample (A) was determined on **Fig. 8** and the result obtained the percentage oxides of metals and the concentration of oxides are shown in decreasing order of; CaO = 91.5 % > SiO_2 = 3.36 % > Al_2O_3 % = 0.818 % > P_2O_5 = 0.593 % >, TiO = 0.463 % >, Fe_2O_3 = 0.418 % > MnO = 0.0246 % respectively. The percentage oxides revealed on the investigated samples showed that all the concentrations displayed were not beyond the WHO permissible limits of Ca and Mg, which are 50mg/kg, while Fe, Cr, Mn, Co, and Ni ranged between 0.1 and 0.003 mg/kg. The result obtained was contrary to the oxides of fluorspar, as stated by [8]: CaO = 2.8%, Al2O3 = 0.9%, SiO2 = 5.46%, Fe2O3 = 0.13%, and P2O5 = 0.012%.

X-Ray Fluorescence (XRF) Analysis of Sample B

The XRF result presented in **Fig. 9** shows the distribution of metal oxides present in the fluorspar mineral sample B. The oxides are arranged in decreasing order of abundance as follows: CaO = 81.4 %, Fe_2O_3 = 6.63 %, SiO_2 = 5.65 %, TiO_2 = 0.879 %, P_2O_5 = 0.612 %, and MnO = 0.0204 %. The data reveal that calcium oxide (CaO) is the dominant component, indicating that sample B is calcium-rich and typical of fluorspar minerals.

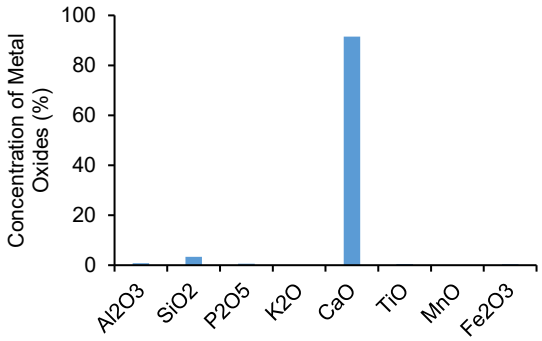

Fig. 8. X-Ray Fluorescence Analysis of Sample A.

When compared with sample A, sample B exhibited a slightly lower CaO concentration but higher levels of Fe_2O_3 and SiO_2, suggesting a marginal increase in iron and silica impurities. The CaO concentration (81.4%) exceeds the World Health Organization (WHO) permissible limit of 50 mg kg^{-1} for mineral content in environmental materials, implying a potential contamination risk if released into the surrounding soil or water systems.

Other oxides such as SiO_2, Al_2O_3, and P_2O_5 were found within safe limits (0.1–5 mg kg^{-1}) as stipulated by WHO (2010) [9]. However, when compared with the findings of Al Hameed et al. (2017) [8], who reported lower oxide levels in indigenous fluorspar (CaO = 2.8 %, Al_2O_3 = 0.9 %, SiO_2 = 5.46 %, Fe_2O_3 = 0.13 %, and P_2O_5 = 0.012 %), the present study shows significantly elevated CaO and Fe_2O_3 contents. This variation may be attributed to differences in geological formation, mineralization processes, and impurity incorporation within the Gombe deposit compared with other reported fluorspar sources. Overall, the XRF results confirm that sample B contains a high concentration of calcium oxide and trace amounts of other oxides, which together reflect the mineral's geochemical characteristics and potential environmental impact.

Comparative Oxides of Sample A and B

The comparative analysis of the two samples of Fluorspar mineral investigated is shown in Fig. 8, which indicates that sample A has higher oxide values of A = 91.5%, while sample B has oxide values of B = 81.4%. Also, oxide of SiO_2 in sample A mineral showed A=3.36% while sample B mineral showed oxide of B = 5.65 %. Other oxide of Al_2O_3 in sample A mineral A= 0.818% while sample B mineral showed B=0.595 %. The Fe_2O_3 in sample A mineral showed A = 0.418%, while sample B mineral showed B = 0%, or, eventually, no Fe_2O_3 was present in the mineral. The TiO in sample A mineral showed A 0.463 % while in sample B mineral B= 0 % or totally absent in the mineral composition of sample B mineral.

Sample A mineral showed P_2O_5 = 0.593 % while sample B mineral showed P_2O_5 = 0.612 % respectively. Samples which is greenish brown colour contain the highest percentage of metal oxides with a reasonable composition of Fe_2O_3 and TiO2 oxides. However, Sample B, which is a blue-brown mineral, contains a lower composition of metal oxides, with no or zero composition of Fe2O3 and TiO; hence, it is a low-quality mineral than Sample B. Two giant and persistent Fluorite veins occurred at the surface of the mining area, which showed the presence of two distinct fluorite samples, as reported by [1].

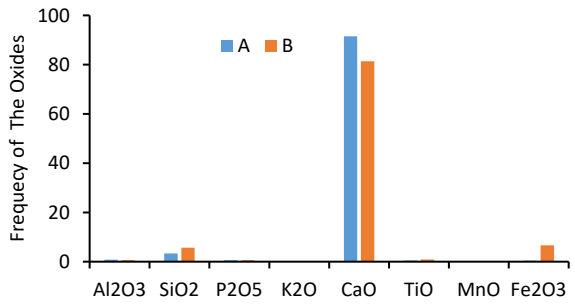

Fig. 9. X-Ray Diffraction (XRD) Analysis of the two fluorspar minerals.

XRD Analysis of Sample A

Fig. 10 shows the result obtained for the XRD analysis of sample A fluorspar mineral, with prominent peaks observed at 2θ = 25.57°, 28.37 °, 42.35 °, 47.04 °, and 50.02 °, which reveal the presence of CaO > SiO2 > Al2O3 > P2O5 > TiO2, respectively. The XRD of crystal structure angles obtained a range of 25.6 ° to 50.02 °, which was contrary to the angles of the crystal structure range of 20.8 ° to 78.8 ° of the Fluorspar mineral investigated by [8]. The peaks observed are in relation to the plane of symmetry angles (111), (101), and (210). It shows Face Centered Cubic [FCC] structure and the average crystalline size of 75.87 nm. The result obtained was also contrary to the crystal structure of nanoparticles in the planes of (111), (200), (220), and (311). It displays a Face-Centered Cubic (FCC) structure and an average crystalline size of 75.31nm, as reported by [10]. The diffraction peaks are indexed according to the Scherrer equation, as calculated. The result obtained corresponded to the literature reported by Usman et al. [11], which shows that the crystal size obtained was FCC (face-centered cubic). However, the two results contradicted each other with respect to the plane of symmetry (111), (110), (211), and (210), with a difference in average crystalline size of 52.64nm between them.

X-Ray Diffraction Analysis of Sample B

Fig. 11 shows the result obtained from the XRD analysis of the fluorspar mineral, where prominent peaks were observed at 2θ = 25.94 °, 28.73 °, 42.64 °, 47.44 °, and 56.17 °, which reveal the presence of CaO > Al2O3 > SiO2 > Fe2O3 > P2O5, respectively. The XRD analysis showed that the crystal structure angles obtained ranged between 25.9° and 56.17°, which was contrary to the angles of the crystal structure range of 20.8° to 78.8° of the Fluorspar mineral investigated by Zeeshan et al. [12]. The angles are with respect to the plane of Symmetry angle (111) and (101), it shows Face Centered Cubic [FCC] structure and the average crystalline size of 13.65 nm.

The result obtained was also contrary to the crystal structure of nanoparticles in the planes of (111), (200), (220), and (311). It displays a Face-Centered Cubic (FCC) structure and an average crystalline size of 75.31nm, as reported by [10], calculated using the Scherrer equation. The result obtained corresponded to the literature reported by Usman et al. [11], which shows that FCC (face-centered cubic) has planes of angle symmetry (111), (110), (211), and (220). Thus, a clear difference in the average crystalline size of 52.64 nm occurred, showing dissimilarities.

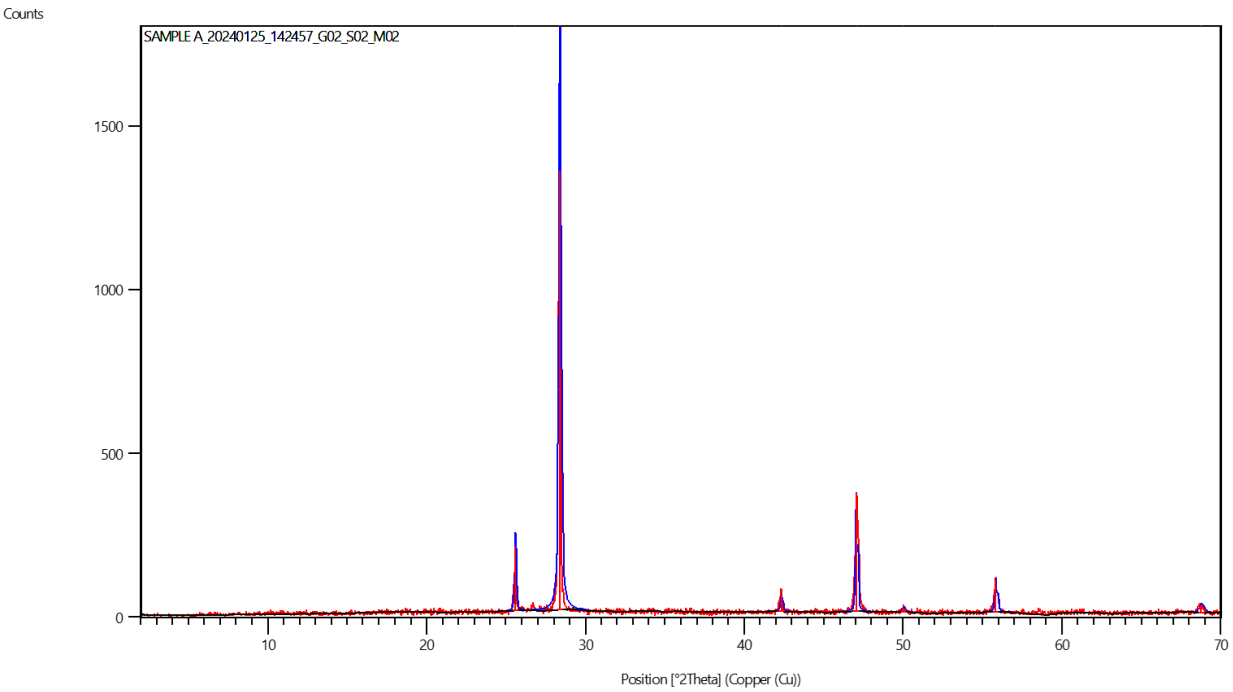

Fig. 10. XRD analysis of sample A, a fluorspar mineral.

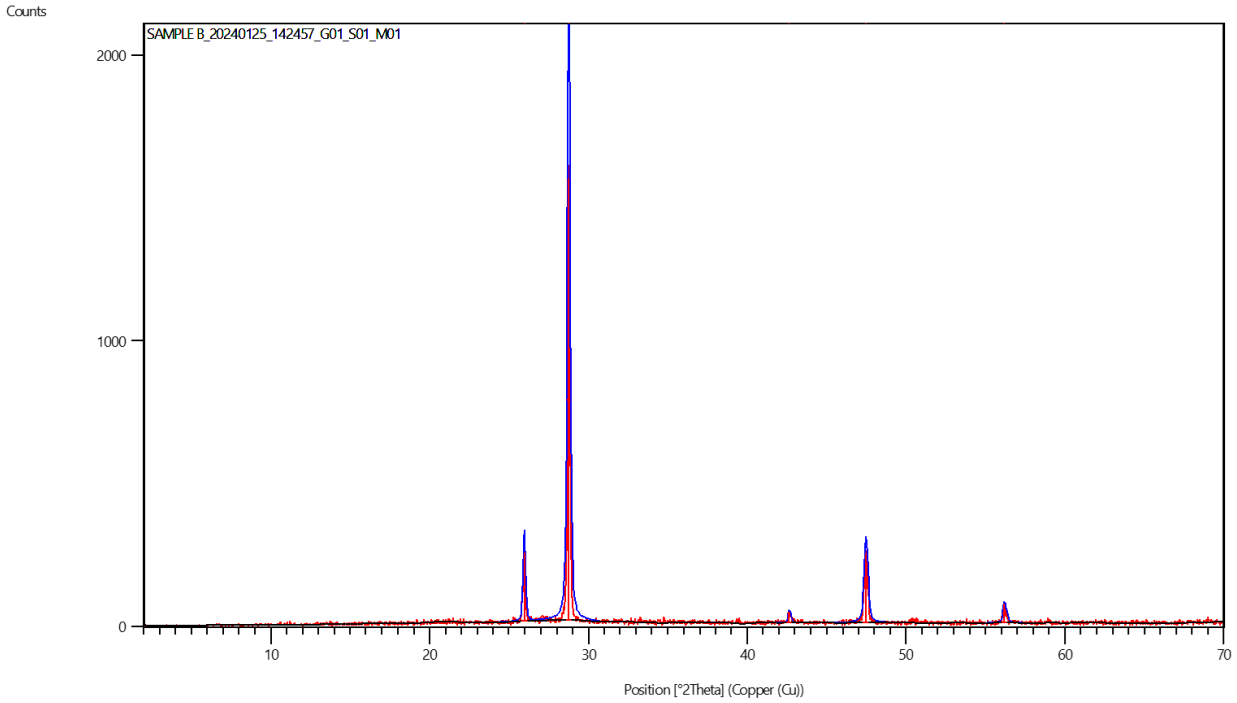

Fig. 11. X-Ray Diffraction of Sample B.

CONCLUSION

The heavy metal oxides and crystal structure of fluorspar minerals investigated at Liji Hills indicated that only Sample A, a greenish-brown fluorspar mineral, has a higher concentration of Ca above the WHO permissible limit of 50mg/kg, which may be carcinogenic and can affect health risks to the populace and the ecosystem at large. On the other hand, Sample B blush brown Fluorspar mineral contains trace metals of Ca and Mg, and some heavy metals such as Ti, Pb Fe Zn are either not detected or range between 0.001 mg/kg to 0.5 mg/kg at the apex within the WHO permissible limits, thus their concentration has no significant contamination threats to humans and the ecosystem. Physicochemical analysis showed a mean pH value of sample A to be 6.91, while sample B had a mean pH value of 6.71, indicating that sample B is more acidic than sample A. Conductivity showed sample A= 0.24 $NScm^{1,}$ while sample B= 0.29 $NScm^{1}$. The moisture content of sample A was 4.15%, whereas that of sample B was 1.0%. Ash analysis of the sample showed A = 3.7%, while sample B = 2.9%. The crystal structure of the sample and B fluorspar mineral showed a crystalline size of 13.8 nm with a plane of symmetry angle of Face Cubic Center (FCC) ranging between 25.9 ° and 75.56 °. Sample A showed Face Centered Cubic [FCC] structure and the average crystalline size of 75.87nm, with a crystalline size of 56.17nm. Therefore, sample A, a greenish brown Fluorspar mineral, is a more qualitative mineral than sample B, a bluish brown Fluorspar mineral, which can withstand metallurgy and other important extraction processes.

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CONFLICTS OF INTEREST

The Authors of this article does not show any misunderstanding issues nor any attempted willingness to declare any conflict of interest upon this research work carried out.

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