Current Journal of Applied Science and Technology

24(2): 1-9, 2017; Article no.CJAST.23053
Previously known as British Journal of Applied Science & Technology
ISSN: 2231-0843, NLM ID: 101664541

X-Ray Diffraction Characterization of Sedimentary Rocks in Demsa Local Government Area of Adamawa State, Nigeria

B. L. Penuel[1], O. N. Maitera[2], M. E. Khan[3*] and Y. Ezekiel[4]

[1]Department of Chemistry, Adamawa State University, Mubi, Nigeria.
[2]Department of Chemistry, Modibbo Adama University of Technology, Yola, Nigeria.
[3]Department of Chemistry, University of Agriculture, Makurdi, Benue State, Nigeria.
[4]Department of Geography, Adamawa State University, Mubi, Nigeria.

Authors' contributions

This work was carried out in collaboration between all authors. Authors BLP and ONM designed the study, wrote the protocol and wrote the first draft of the manuscript. Authors MEK and YE performed the X-ray diffraction characterization, managed the analyses of the study and literature searches. All authors read and approved the final manuscript.

Article Information

DOI: 10.9734/CJAST/2017/23053
Editor(s):
(1) Jon S. Gold, Professor, Department of Chemistry, East Stroudsburg University, East Stroudsburg, PA, USA.
Reviewers:
(1) Ahmet Karakaş, Kocaeli University, Turkey.
(2) Angelo Paone, USA.
(3) José Martínez Reyes, Mexico.
(4) Thomas F. George, University of Missouri-St. Louis, USA.
Complete Peer review History: http://www.sciencedomain.org/review-history/21632

Original Research Article

Received 12th November 2015
Accepted 5th February 2016
Published 30th October 2017

ABSTRACT

This work involves the qualitative analysis and mineral (elemental) composition of rock samples in six sites (locations) in Adamawa State (Kenya) using several physical properties including colour, streak, hardness, specific gravity, luster, magnetism, taste and reactions to Hydrochloric acid (HCl). X-Ray Diffraction (XRD) spectroscopy was used in the identification of minerals in rock samples including:calcite, quartz, rutile, augite, srebrodolskite, sanidine and plagioclase. The samples were analyzed using an Emission-Transmission spinner stage with the theta-theta settings. Two theta starting positions were 0.0001 ending at 75,000, with a two steps 0.026 at 3.57 seconds per step. The tube current was 40mA and a generator voltage of 45V untilizing a copper anode. Qualitative analysis indicated the

---

**Corresponding author: E-mail: emamulu@yahoo.com;*

presence of carbonate ions in all the rock samples. Calcite was found to be predominant in four study locations. Therefore the possibility to harness calcite for indigenous economic development is possible.

Keywords: Sedimentary rock; minerals; x-ray diffraction; calcite; analysis.

1. INTRODUCTION

Nigeria is richly endowed with natural resources which spread across all the states of the Nation. Development and utilization of natural resources should therefore be largely promoted and supported by all states as a strategy for socio-economic development of their States since every State has specific comparable advantage(s) in available natural resource(s) [1]. According to Opeloye and Dio [2], Adamawa State is endowed with mineral resources on account of varied geologic rock types. Various basements and sedimentary rocks have their attendant mineral enrichment evenly distributed within the State. Currently known mineral resources can be categorized into three: - Industrial minerals, Metaliferous minerals, and Energy resource / strategic minerals.

Minerals originate from rocks which constitute the earth's crust, consisting essentially of assemblage of various minerals. They can be broadly classified, depending on their mode of origin, into three major divisions: The sedimentary, metamorphic and igneous rocks [3]. Unlike the igneous rocks, sedimentary rocks are made up of a very small selection of minerals, viz quartz, feldspars, clay and carbonates. Carbonate rocks (limestone) are the most important rocks in this group. Limestone including dolomite (magnesium-rich carbonate rocks), comprises 25% of all sedimentary deposits. The term limestone is normally used for rocks containing 50% or more of calcium carbonate. They are widely variable in appearances and in modes of formation [4]. These include calcite, dolomite, huntite, magnisite and many others [5], which are useful for industrial applications.

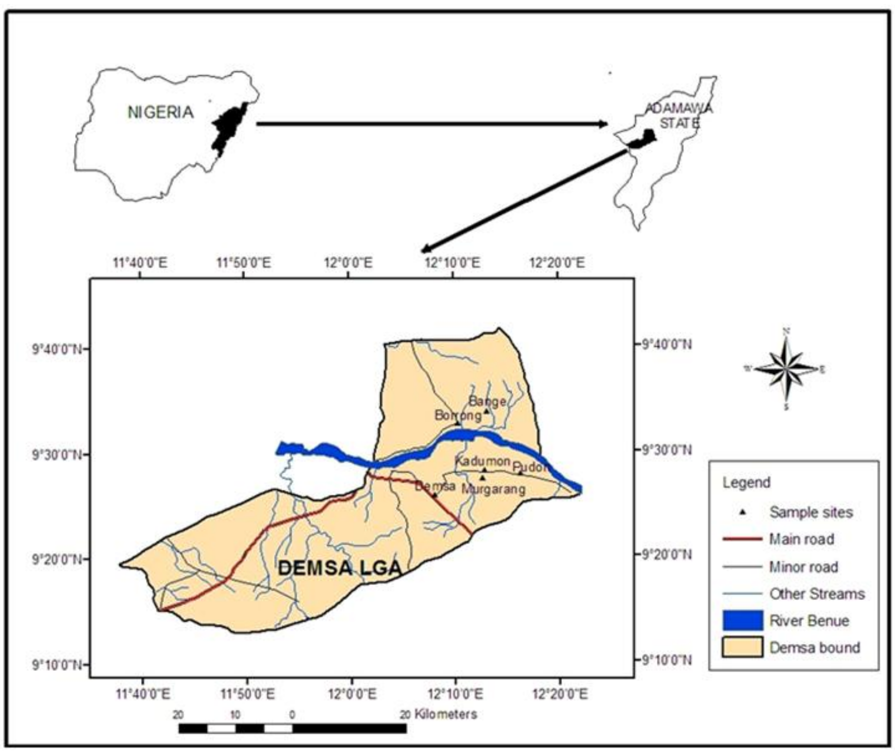

Fig. 1. The study location

Minerals are the foundations of industries ranging from constructions, manufacturing, agricultural technology to cosmetics [6].

Adamawa State is endowed with industrial minerals such as clay, gypsum, limestone, benthonite, trona, graphite, feldspar and quartz [2]. The need to identify these rocks to ascertain their mineral content will therefore include some qualitative analysis of the rock samples using some of their physical properties like colour, streak, hardness, specific gravity, luster, magnetism, taste and reactions with Hydrochloric acid (HCl). Instrumental methods using XRD which has been reported to be efficient and provides more reliable and accurate additional information about the chemical and physical properties of the suspected sample are used, [7].

Demsa Local Government Area is located between latitudes 9°12'N and 9°42'N of the Equator and between longitudes 11°38'E and 12°20'E of the Prime Meridian (Fig. 1). It is situated at the confluence of the Yola and Gongola arms of the Benue trough which was subjected to several depositional cycles that resulted in the formation of the Albian and Cenomanian sedimentary rocks of varied compositions and ages [8,9,2]. Consequently, the area is characterised by sedimentary rock varieties which include calcareous sandstones, feldspathic sandstones, limestones and shales with sandstone intercalations as well as shelly limestones.

2. MATERIALS AND METHODS

2.1 Sample Identification and Collection

The rock samples were identified by Dr. Yonnana Ezekiel of Geography Department, Adamawa State University, Mubi. Site identification of the rock was made by adding some few drops of dilute hydrochloric acid (HCl) on the rock which showed effervescence due to release of gas that turned lime water milky, inferring its carbon dioxide gas from carbonate rock. Other physical properties like colour, streak, luster, hardness, and specific gravity were carried out to confirm the presence of carbonate ions in the rock. The sampling methods by [10,11] were modified and adopted.

Sampling points were spaced two meters (2 m) apart for possible variations in their constituents. Small pieces of limestone were chiseled and hammered out. Samples were randomly collected from ten points (10) in each sampling site, mixed to give a representative sample. These were transferred into labeled polythene bags and preserved for analysis.

2.2 Sample Preparation

Samples were prepared according to [12]. The collected rocks were thoroughly dried in an oven at 31°C for 12 hours. The dried samples were crushed and ground to powder using pestle and mortar and reduced in sizes after crushing, by coning and quartering until desired size / quantity of sample for analysis was obtained. These were kept in air-tight polythene bags for analysis.

2.3 Qualitative Analysis

Qualitative analysis of the rock samples were carried out based on the methods of [13,14] to confirm carbonate rock using the minerals` physical properties such as colour, Moh's hardness, luster, magnetism, streak, taste, specific gravity and reaction with hydrochloric acid.

2.4 X-Ray Diffraction Analysis (XRD)

X-ray diffraction was used for the identification of unknown crystalline materials (e.g. minerals, or inorganic compounds). Determination of unknown solid was critical in the characterization of crystalline materials, and in the determination of unit cell dimension, and measure of sample purity.

The samples were analyzed using X-ray diffraction spectrometer (Panalytical Empyrean Rayons). This worked by the combination of other components like the water chiller which cooled x-ray tube and maintained a uniform temperature. Compressed air also helped in opening and closing of the cabinet door.

The analysis was done as described by [15]. The materials to be analyzed were finely ground and sieved to pass through 63 microns sieve. The pulverized sample was then prepared using prepared blocks and compressed into a flat sample that was later mounted on the sample stage in the XRD cabinet.

The sample was analyzed using the Emission-Transmission spinner stage with the theta-theta settings. Two theta starting positions were 0.0001 ending at 75,000, with a two steps 0.026 at 3.57 seconds per step. Tube current was

40mA and the generator voltage of 45V with a copper anode was used.

The intensity of diffracted X-ray was continuously recorded as the sample and detector rotated through their respective angles. Peak intensities occurred when the mineral containing lattice plane with d-spacing appropriate to different x-rays at a value of θ. passed through the detector.

3. RESULTS AND DISCUSSION

The XRD results presented as peak position at 2θ and x-ray counts (intensity) in form of an x-ray plot (Figs. 2-7).

The Spectrum below shows Major mineral composition as Identified by XRD in Bange. The peaks indicate the mineral quantities.

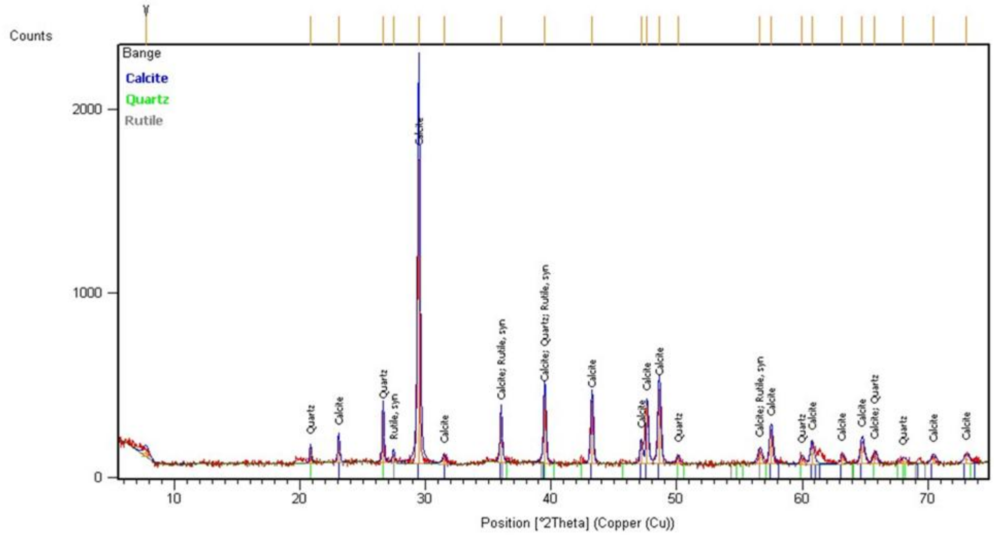

Fig. 2. Major mineral composition as identified by XRD in Bange

Below is the Spectrum showing Major mineral composition as Identified by XRD in Borrong. The peaks indicate the mineral quantities.

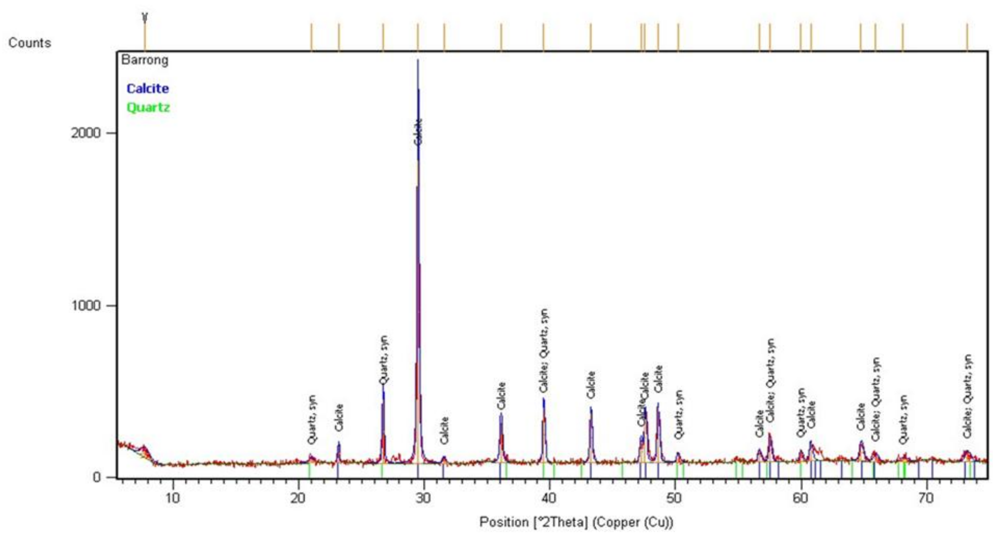

Fig. 3. Major mineral composition identified by XRD in Borrong

Spectrum below shows Major mineral composition as Identified by XRD in Kaumon. Peaks indicate the mineral quantities.

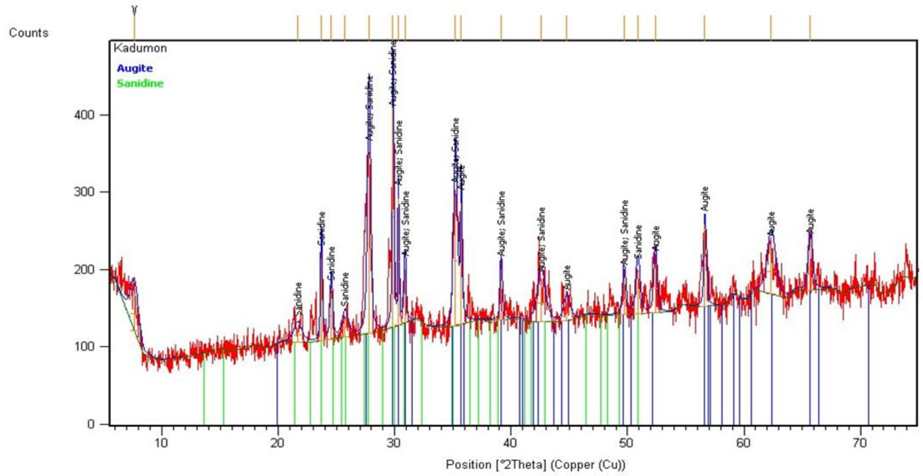

Fig. 4. Major mineral composition identified by XRD in Kadumon

The Spectrum below shows Major mineral composition as Identified by XRD in Murgarang. The peaks indicate the mineral quantities.

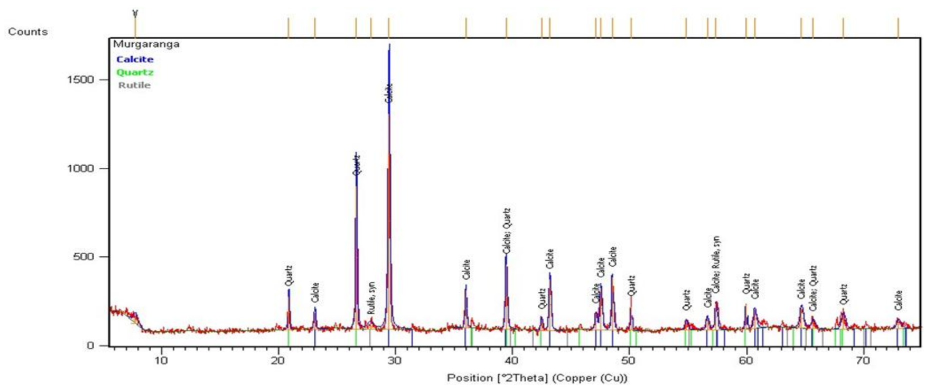

Fig. 5. Major mineral composition identified by XRD in Murgarang

Spectrum below shows Major mineral composition as Identified by XRD in Pudan. Peaks indicate the mineral quantities.

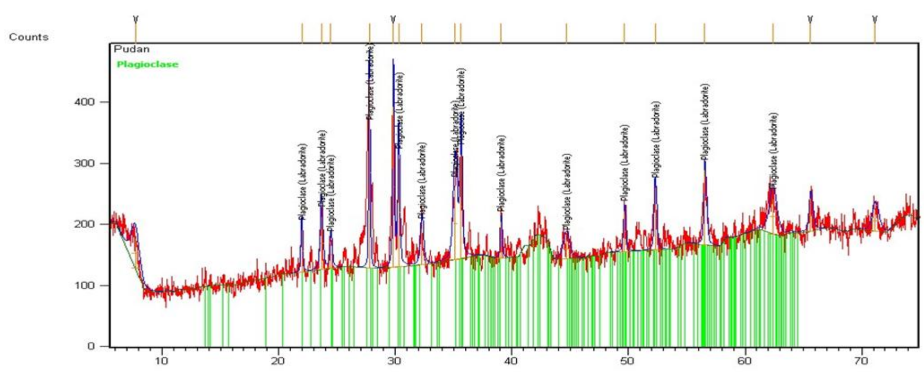

Fig. 6. Major mineral composition identified by XRD in Pudan

Spectrum shows (Fig. 7) Major mineral composition as Identified by XRD in Demsa. The peaks indicating the mineral quantities.

Table 1. Qualitative analysis of the rock samples portraying their various colours, Streak, hardness, luster, taste, magnetism, specific gravity and reaction with HCl

Location	Colour	Streak	Hardness	Luster	Taste	Magnetism	Specific gravity	Reaction to HCl
Borrong	Gray	White	3.5	Non-metallic	Tasteless	Non-magnetic	2.72	Effervescence vigorously
Demsa	Gray	White	3.5	Non-metallic	Tasteless	Non-magnetic	2.64	Effervescence vigorously
Murgarang	Brown	Cream	3.5	Non-metallic	Tasteless	Non-magnetic	2.74	Effervescence vigorously
Bange	Gray	White	3.5	Non-metallic	tasteless	Non-magnetic	2.70	Effervescence vigorously
Pudan	Black	Dark brown	3.5	Metallic	tasteless	Magnetic	3.00	Weak effervescence
Kaduman	Black	Dark brown	3.5	Metallic	Tasteless	Magnetic	2.95	Weak effervescence

Table 2. Minerals Identified (Names of compounds and formulae) in Sample by X-Ray Diffraction

Sample site	Mineral identified	Compound name	Chemical formula	Crystal system
Bange	Quartz	Silicon oxide	SiO_2	Hexagonal
	Calcite	Calcium carbonate	$CaCO_3$	Rhombohedral
	Rutile	Titanium oxide	TiO_2	Tetragonal
Borrong	Calcite	Calcium carbonate	$CaCO_3$	Rhombohedral
	Quartz, syn	Silicon oxide	SiO_2	Hexagonal
Demsa	Calcite	Calcium carbonate	$CaCO_3$	Rhembohedral
	Quartz	Silicon oxide	SiO_2	Hexagonal
	Srebrodolskite	Calcium iron oxide	$CaFeO_4$	Orthorhombic
Kadumon	Augite	Calcium Iron Magnesium Silicate	$Ca(Mg, Fe)\ Si_2O_6$	Monoclinic
	Sanidine	Potassium Sodium calcium Aluminium silicate	$K_{0.47}\ Na_{0.43}\ Ca_{0.10}\ Al_{1.1}\ Si_{2.9}O_8$	Monoclinic
Murgarang	Calcite	Calcium carbonate	$CaCO_3$	Rhombohedral
	Quartz	Silicon oxide	SiO_2	Hexagonal
	Rutile	Titanium oxide	TiO_2	Tetragonal
Pudan	Plagioclase (Labrodarite)	Calcium sodium aluminium silicate	$Ca_{0.65}\ Na_{0.35}\ (Al_{1.65}\ Si_{2.35}\ O_8)$	Anorthic

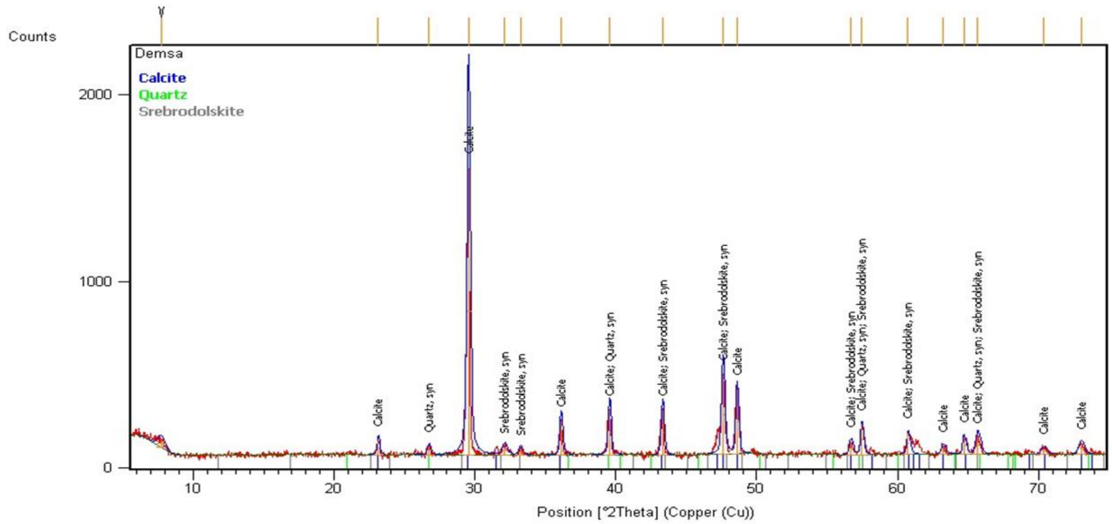

Fig. 7. Major mineral composition identified by XRD in Demsa

Fig. 1, Situates Adamawa State on Nigeria map and locate the sample points in Demsa LGA of the state. Its longitudes and Latitudes are also given.

From Table 1, the colours and streaks of the rock samples obtained in the study area, all agreed to the colours obtained in sedimentary rocks and limestone. Borrong, Demsa, Murgarang and Bange had typical limestone colours as described by [14], usually white, gray or colourless. Also, [16] stated that non-ferromagnesian silicate can vary in colour but rarely dark which agreed with this research. The colour of sedimentary rocks can be influenced by iron(II) oxide and iron(III) oxide which impart gray or greenish colour and reddish to brownish respectively [17]. The presence of organic materials could also give a rock black or gray colour [18]. This asserts to the colours obtained in samples in Pudan and Kadumon respectively.

The specific gravities of these rock samples met the values for common rock-forming mineral of 2.5 to 3.5. [19]. Specific gravity varies in materials depending on their composition and structure. The ferromagnesian silicates have specific gravity ranging from 2.7 to 4.3 whereas the non-ferromagnesian silicates vary from 2.6 to 2.9 [16]. Though the ranges of ferromagnesian and non-ferromagnesian silicates specific gravities overlapped, they can be ascertained after other parameters are considered. Results obtained in Borrong, Bange, Murgarang and Demsa felt within the specific gravities of non-ferromagnesian silicates (2.6 to 2.9) while Pudan and Kadumon showed slightly higher specific gravities (3.00 to 2.95) respectively and some showed ferromagnesian silicate specific gravity tendencies (2.7-4.3).

Pudan and Kadumon samples showed magnetic properties and have metallic lusters while the other sample areas indicated non-magnetic and non-metallic luster. The magnetic tendencies of Pudan and Kadumon indicated that the samples had high iron content [13].

Table 2 showed results of the mineral composition of the rock deposits. The results showed calcite ($CaCO_3$) in four locations with the exception of Pudan and Kadumon. The sample from Demsa apart from being rich in calcite, had quartz (SiO_2) and srebrodolskite. Bange and Murgarang had calcite and rutile (TiO_2). Borrong showed calcite and quartz. Pudan and Kadumon indicated no calcite. Pudan had plagioclase, while Kadumon portrayed more Sanidine and Augite. The peaks on the diffraction spectra Figs. 2-7 signified the abundance of each of these minerals in their different locations.

4. CONCLUSION AND RECOMMENDATION

From the research findings/analysis, there was a high qualitative presence of carbonate ions in all the rock samples. The XRD confirmed the presence of calcite, quartz, rutile, augite, srebrodolskite, sanidine and plagioclase in the rock samples in most of the locations.

All these are minerals with different economic importance but the abundance of (calcite) in all locations is an indication that there are deposits of limestone in Demsa Local Government Area, of Adamawa State and thus further geological survey should be done to access its economic feasibility and viability for industrial purposes. For if these minerals are properly enhanced and harnessed, they could scaffold and diversify the Demsa locals and in general the Nigerian government economy from a single to a multiple revenue economy. There by uplifting the indigents and the locals /the common man, and ameliorating their life styles.

With the predominance of calcite signifying its abundance based on the spread, we therefore recommend the need for the Nigerian government to harness it for industrial purposes. This, if well harnessed could scaffold the Nigerian economy to greater highs and could even be the main stay of the economy. The Nigerian government is singly dependent on oil (petroleum) but if they can develop mineral deposits (mining like S. Africa), the economy will be diversified. Locals (indigenes) will be employed and this will improve their living standards.

ACKNOWLEDGEMENT

Our appreciations go to Dr. Yonnana Ezekiel of Geography Department, Adamawa State University, Mubi for his valuable assistance.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

REFERENCES

1. Onwuala AP. Optimizing the exploitation of natural resources endowment in the states for accelerated socio-economic development: Strategies and opportunities for investment (Paper Presented at the Technology Management Forum for Directors of Science and Technology in Nigeria). 2010;2-25.
2. Opeloye SA, Dio CJ. Geology and mineral resources of Adamawa State. In Adebayo, A. A. and Tukur, A. L; Adamawa State in Map. Paraclete Publishers, Yola; 1999.
3. Aidekoya JA. Negative environmental impact of mineral exploitation in Nigeria. International Journal of Physical Sciences. 1995;613 – 619.
4. Cox KG, Price NB, Harte B. The practical study of crystals, minerals and rocks. McGraw Hill International Editions. 1974; 104-124:201-207.
5. Adesakin AO, Olunlade BA. X-ray diffraction characterization of Huntile Found in Southern Nigeria.Chemical Society of Nigeria (CSN) Confrence Proceedings. ANA. 2011;171-172.
6. Hobert King. Mineral and chemical composition of pure limestone-Missouri Department of Natural Resources; 2005-2015. Geology.com.
7. Chung FH. Quantitative interpretationof x-ray diffraction patten of mixtures / matrix flushing method for quantitative multicomponent analysis. Journal of applied Crystallography. 1974;7:519.
8. Bawden MG. Physiography, geology, geomorphology and hydrology. In Tuley, P. (ed). Land Resources of North East Nigeria. The Environment: Land Resource Study No.9. Surrey, England. Land Resources Division/ Overseas Development. 1972;1.
9. Kogbe CA. Major transgressive episodes in the Benue Valley of Nigeria. Earth Evolutionary Science. 1981;2:139-143.
10. Alexander P, Maina HM, Barminas JT, and Zira SP. Quality of solid minerals in rocks of michika local government area of Adamawa State, Nigeria. Journal of Sciences and Multidisciplinary Research. 2011;3.23.
11. Magili ST, Maina HM, Maitera ON. Comparative assessment of limestone resources of Guyuk and Ashaka Areas for Industrial Utilization. (online) Journal of Physical Sciences and Innovation. (©2010 cenresin publications). 2010;2.
12. Okunola OJ, Ozam A, Ndukwe GI, Adunusi SG. Assessment of Cd and Zn in roadside surface soils and vegetation along some roads of Kaduna Metropolis, Nigeria in: Research Journal of Environment Sciences. 2008;2(4):266-274.
13. Bates RL, Jackson JA (eds.). Glossary of geology. American Geological Institute, Alexander, V.A. 1987;788.

14. Carlson DH, Plummer CC, McGeary D. Physical geology forth revealed, 7th edition. McGraw-Hill International Edition. 2008;360-376,241-249.
15. Dutrow LB, Clark MC. Geochemical instrumentation and analysis. X-ray Powder Diffraction (XRD); 2009.
16. Monroe SJ, Wicander R. Physical geology. Exploring the earth (3rd edn), Wadworth USA. 1998;35-60.
17. Lervin HL. The earth through time (3rd edn) Saunders College Publishing. 1987;57–60.
18. Tarburg E, Lutgen FK. Earth, an introduction to physical geology 96th edn), Prentice Hall. 1999;145v-146.
19. Monroe SJ, Wicander R. Physical geology. Exploring the earth (4th edn). Wadworth USA. 2001;106 -200.

Peer-review history:
The peer review history for this paper can be accessed here:
http://sciencedomain.org/review-history/21632