

Characterization and structural investigation on diorite rocks of Botswana

Mr Gaesennngwe Gaesennngwe and Dr. G. Dahna
Department of Chemical, Materials and Metallurgical Engineering
Botswana International University of Science and Technology
Palapye, Private Bag 16, Botswana
info@biust.ac.bw

Mr Gaesennngwe Gaesennngwe
Chemical, Materials and Metallurgical Engineering Department
BIUST University
Palapye, Botswana
gaesennngwe.gaesennngwe@studentmail.biust.ac.bw

Abstract

Clear investigation on mineral rock characterization is essential for establishing solid bridges between the mineral resource location and area of application, i.e. rock provenance. Major scientific revolutions in the industry was made possible via technologies of characterization (XRD, XRF, SEM etc...) providing improved processing routes that effectively address arising challenges and problems faced by our local quarries and mining industries, therefore attempting also to explaining fundamental treatment techniques that were adopted anciently. Modern civil engineering activities, mining and construction entail clear statistical data on material composition, texture, petrographic and mechanical properties of the source material or mineral ore they are handling for clear documentation. Visual inspection was carried out on Diorite samples acquired from various locations. Microscopic examination of component were classified in order to rationalize unique and recognizable features (mineral type, structure and color patterns), and estimations for specific purpose the rock is suited for (i.e., as a sculpting material or fabrication tool). Characterization therefore, provided crucial information to the interpretation on quality of stone and relevant processing factors relating to energy input and effective processing technique. In this work, *In-situ* X – ray powder diffraction analysis was performed at rtp conditions on powdered samples of Diorite (a) and (b). Various components were identified which revealed that phase composition is significant in determining the strength of material and processing parameters.

Keywords

In- Situ X – Ray Diffraction (XRD), Characterization, Crystallography, Powdered X-ray diffraction (PXRD).

Introduction

Since the dawn of civilization powder diffraction (PXRD) has continually been employed as a standard analytical tool at both research faculty institutions and the industry (Spectrometer n.d.)(Wills and Finch 2016). Powder diffraction technology offer reliable estimations about material property information with X- ray diffraction scan measurements that are expeditious, information evaluation simultaneously displaying on a computer monitor for straightforward analysis as well as classification (Spectrometer n.d.). In-situ x-ray diffraction consider the crystal structure of polycrystalline materials identifying crystallinity of components, their relative quantity and phase magnitude relative to the whole matrix (Scanned Powdered Diorite Rock). Diorite rock is an intrusive material intermediate of gabbro and granite rocks in terms of composition (Austin 1963). It finds location mainly in mountainous landscapes where it occurs in substantial amounts as batholiths. With rock composition of about 5-50% dark minerals

(melanocratic) and a white appearance, the plutonic rock composite closely relates to andesite in appearance and textural similarities, consequently containing mostly hornblende (amphibole), plagioclase and/ or pyroxene (augite) and approximately 52% - 63% silica content (K et al. 2013)4]. In this study, Diorite obtained at various localities were measured and 100g material weighed using laboratory scale then pulverized, therefore converting it into a powdered phase via a laboratory ball mill operating with revolution speed, 250 rpm, for 20 minutes before the final sample products were subjected to x-ray diffraction scan to identify various mineral components with varying lithological units. Only two samples were prepared, with sample (a) obtained at Master Quarries in Serowe and sample (b) obtained at Francistown in Panda Quarries. The physical and chemical composition of any mineral deposit play a significant role in determination of processing techniques and providing vital data for prediction of behavior during processing (Bogoch, Avigad, and Weissbrod 2002). Consequently grain structure of diorite when observed under a microscope aids in establishing these very important estimation and thus enhanced expected outcome of results following size reduction (Simonov 1991). The grain size of crystalline minerals contribute largely towards processing and handling treatment since the amount of energy spent during breakage is proportional to the grain size of components of the rock ore. Generally the grain size of intrusive rock resemble that for scattered mineral grains which occasionally reach 5 – 6 mm in length but having an average length throughout the rock not exceeding 1.5 – 2.0 mm (Austin 1963). Diorite closely resemble andesite in mineralogy but revealing manly inhomogeneous distribution of fine – grained structure (small xenoliths) due to rapid cooling at solidification stages. Igneous rocks have texture of mafic type with micro-granular enclaves and geochemical characteristic similar to calcium rich silicates (Austin 1963).

XRD measurement and data processing

According to investigation, the quartz in diorite is much complicated by the presence of numerous small occurrence of granite structures confined inside the rock matrix along large grain boundaries (Nidhi et al. 2011). In-situ x-ray diffraction (XRD) technique was employed to qualitatively quantify phase presence in pulverized samples of diorite rocks. Powder patterns were obtained using a PAN-analytical X'Pert PRO x-ray diffractometer equipped with a sealed proportional detector device with a $\text{CuK}\alpha$ radiation, operating at a voltage of 45KV and current 40mA. A vertically adjustable multi-purpose sample stage was prepared and powdered rock specimen placed symmetrically on an aluminum x-ray sample holder which constantly was aligned to bisect the x-ray beam (Nidhi et al. 2011). Structural analysis of the material was carried out at normal temperature and pressure (20 – 30 °C) with the $\text{Cu K}\alpha$ radiation such that, each rock specimen were held under radiation for a period not exceeding 1200s prior a general scan for peak deflection zone identification that was done for 120s in order to guarantee accuracy and stability of the scan to represent the whole rock area under study. The main focus for this investigation was inclined also on observing the phases present in the material together with the fraction of compound particles occurring in both specimen, how they interact with each other inside the material matrix as well as how they differ with respect to the other sample material scanned. Consequently, in terms of gain size, phase orientation, phase distribution and phase concentration the comparison and different similarities where established, therefore assisting to justify the distinct material properties inherent in both rock samples (Toderas et al. 2017). In order to accurately measure the peak position within the powdered rock sample and their intensity, all the scan results were automatically analyzed through match3 software-program with the aid of an expert and data analysis was successfully achieved. X-ray penetration depth estimation value is a factor that differs from material to another, depending on the linear absorption coefficient of that particular material which is highly dependent on the measurement criterion during the experiment (symmetric, asymmetric measurement mode) as well as homogeneity of component alignment of the general rock structure (Nidhi et al. 2011). For Diorite rock samples, the calibration for penetration depth maybe accomplished as follows

Theory

The intensity of diffracted beam on the detector is generated by the following equation for a small volume of sample material under scan.

$$dl_D = abll_0 \exp\left(-\mu x \left(\frac{1}{\sin(\Omega)} + \frac{1}{\sin(2\theta - \Omega)}\right)\right) dx$$

Where: I_D Is the diffracted intensity, a is the volume fraction of sample specimen containing particulate material with the correct orientation for formation of diffraction patterns, b is the fraction of the incident beam that has been diffracted by one unit of volume sample, l is the illuminated specimen region, I_0 is the incident intensity or beam, μ is the linear absorption coefficient of the material, Ω is the angle of incidence while $(2\theta - \Omega)$ is the diffracted angle (Nidhi et al. 2011).

On Integration of equation (1) above, assuming ideal conditions are observed on the specimen together with its surface (Nidhi et al. 2011). Data from its surface to a certain depth would be analyzed and interpreted to be representative of the diffracted intensity only sustaining to a particular depth with the material, therefore diffracted intensity would be expressed as a function of depth x (Nidhi et al. 2011). X – Ray beam energy constantly entering the material is absorbed by the specimen on penetration and therefore being reduced to a lower energy factor to follow an exponential decay as the wave energy travel further into the material. Through the exponential equation below, the principle can be easily understood:

$$I = I_0 \exp(-\mu L)$$

Such that L represent the distance travelled by the x-ray wave into the material (Nidhi et al. 2011). Information receiving via the detector bears a portrayal of the whole sample thickness throughout the powdered sample material specimen. However, x-ray beam particles that have penetrated beyond the critical depth of the material will only be reporting as negligible intensity on the detector device while diffracted beam intensity reporting significant peak information of the scan will to a greater degree be due to material above this critical depth (Nidhi et al. 2011). Accordingly, this critical depth is thus referred to as the effective penetration depth. Since the graphical illustration of the procedure estimates an exponential decay which ever approach zero but never reaching zero, the negligible beam intensities below the critical depth report to the detector as background noise level, N , while detected significant signal, S_0 , are largely from the surface above the critical depth as can be depicted in the figure 1.0 below.

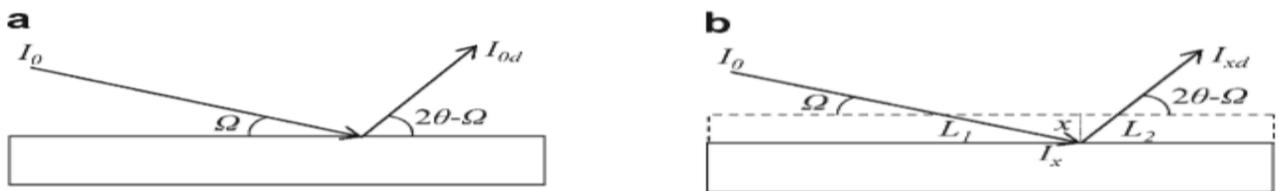


Figure 1.0 depiction of an x-ray incidenting and diffracting on the surface of a polarized sample material (a) rock prior entering material and (b) entering inside the material towards critical depth x (Nidhi et al. 2011)

Rearranging the information for neat analysis, it can be seen that:

$$\frac{S_0}{S_0 + N} = 1 - \exp\left(-\mu z \left(\frac{1}{\sin(\Omega)} + \frac{1}{\sin(2\theta - \Omega)}\right)\right)$$

Which can also be simplified to represent the linear absorption coefficient of the material, being taken as the fraction of the diffracted intensity only pertaining to the surface until the critical depth z , and thus the penetration depth for the powdered rock sample specimens (Nidhi et al. 2011).

$$z = \frac{\ln\left[\frac{S_0}{(S_0 + N)}\right]}{-\mu\left(\frac{1}{\sin(\Omega)} + \frac{1}{\sin(2\theta - \Omega)}\right)}$$

Mineralogical Examination Results

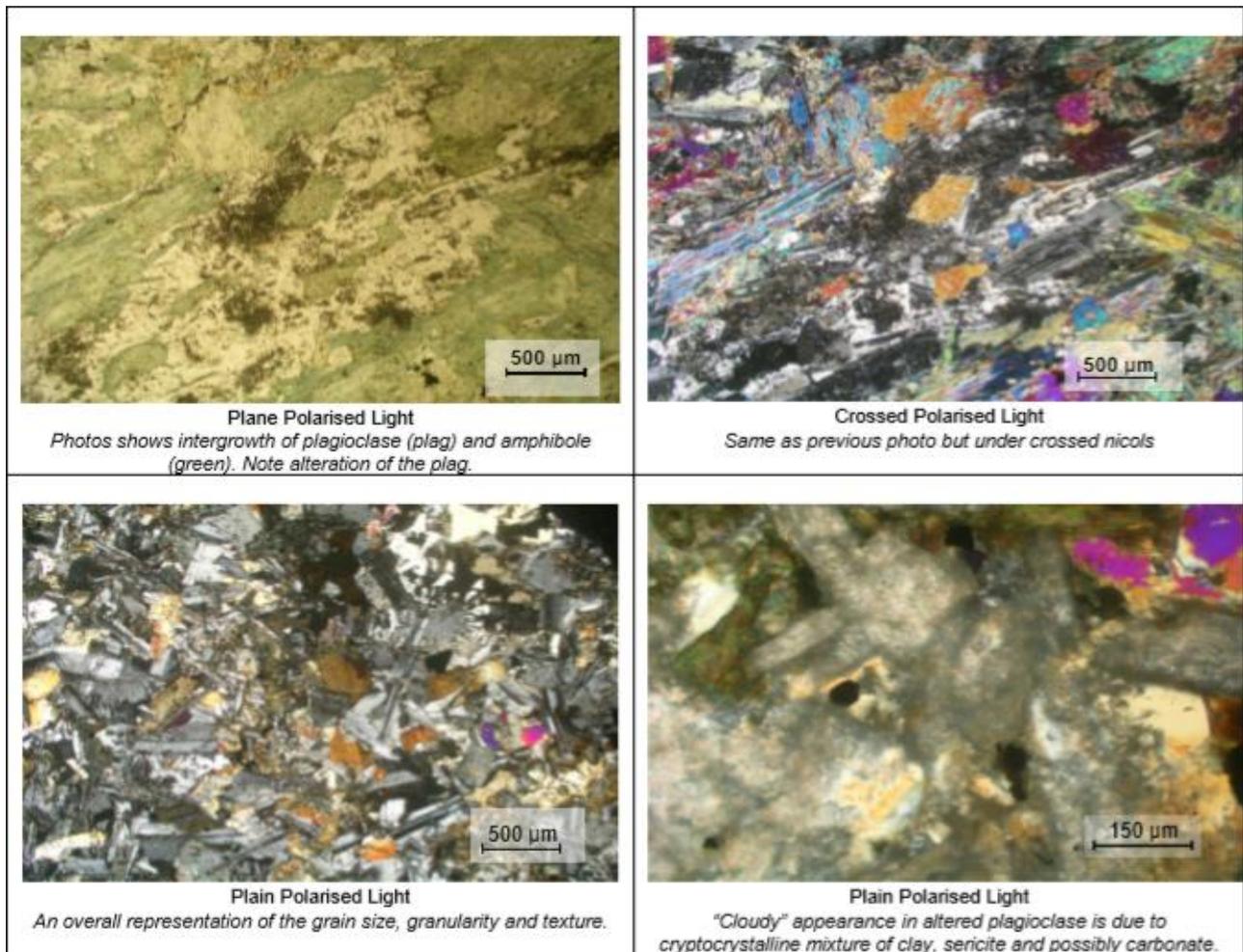


Figure 2.0 images taken under a light microscopy (plain and crossed polarized light) to reveal the surface of grain-structure for diorite complex, (a) ingrowth of plagioclase inside amphibole matrix (light green), (b) crossed pulverized light with various grain size, granularity and texture, (c) fragments of featured clay, sericite and carbonate inclusions.

The Grain size, granularity and texture for diorite rock sample as observed under light microscopy can easily address some of its physical properties that are inherently adopted due to structural patterns

(Simonov 1991). The sample is mostly fine – grained with an average crystalline diameter not exceeding < 1 mm, and therefore with an overall intergranular texture (Austin 1963). The relative size of the crystalline components are equigranular, consequently having an approximate size (Schmeide et al. 2014). Some micrographic intergrowth were also observed between particulate crystals manifesting as twin plates within the grain components (Kurpaska 2018). Observable grain size relates to the number of crystals seen in a given field view in the micrograph which were taken under the same magnification. The diorite specimen under investigation presented a ground mass that contained inter-growths of altered plagioclase, amphibole as well as clinopyroxene constituents, while occurring in minor trace amount were relatively biotite and quartz minerals (Nidhi et al. 2011). Plagioclase however occurs as a fresh mineral phase deposit, and some slightly altered traces of the complex into a clay phase composition, also clinopyroxene appeared to have been largely altered to amphibole phase structure. The “cloudy” impression depicted within the image is due to cryptocrystalline mixture of clay, sericite and possibly carbonates that are opaque in nature. Mica occurs ubiquitously such that it was identified as insignificant or as trace component of the material. The opaque minerals are made up of the finely dispersed minerals that are disseminated up to $\pm 16 \mu\text{m}$, and highly constitute of ilmenite which is sporadically distributed throughout the rock, as well as sparsely distributed trace amounts of sulphides (mainly pyrite and chalcopyrite minerals).

The crystalline phases detected in the panda quarry sample specimen via XRD are listed in table 1.0 below together with the diffractogram of the rock specimen in figure 3.0.

Table 1.0 presentation for prospected Panda quarry’s diorite mineralogy and their relative abundance %

Mineral	Crystal System	Approximate Formula	Approximate Abundance %
Hornblende	Monoclinic	$\text{NaAlSi}_3\text{O}_8 - \text{CaAl}_2\text{Si}_2\text{O}_8$	39.20
Amphibole	Monoclinic/ Orthorhombic	$(\text{Ca-Mg-Fe-Al-OH-Si-O})$	31.74
Quartz	Trigonal +hexagonal	SiO_2	12.26
Chlorite (chamosite)	Monoclinic – prismatic	$(\text{Mg.Fe})_6(\text{Si,Al})_4\text{O}_{10}(\text{OH})_8$	6.92
K-feldspar (Orthoclase)	Triclinic or Monoclinic	KAlSi_3O_8	5.70
Dolomite	Trigonal – Rhombohedral	$\text{CaMg}(\text{CO}_3)_2$	2.78
Mica	Monoclinic	$\text{KAl}_2(\text{Si}_3\text{Al})\text{O}_{10}(\text{OH,F})_2$	1.40
Total			100.00

Table 1.0 presentation for prospected Panda quarry’s diorite mineralogy and their relative abundance %

Mineral	Crystal System	Approximate Formulae	Approximate Abundance %
Plagioclase	Triclinic	$(\text{Ca,Na})_{2-3}(\text{Mg,Fe,Al})_5(\text{Al,Si})_8\text{O}_{22}(\text{OH,F})_2$	49.9
Berlinite	Trigonal	$\text{Al}(\text{PO}_4)$	18.2
Bazzite	Hexagonal	$\text{Be}_3\text{Sc}_2\text{Si}_6\text{O}_{18}$	16.1
Quartz	Trigonal – hexagonal	SiO_2	8.4
Lazurite	Cubic	$(\text{Na,Ca})_8[(\text{S,Cl,SO}_4,\text{OH})_2](\text{Al}_6\text{Si}_6\text{O}_{24})$	4.8
Chlorite	Monoclinic-primitive	$(\text{Mg.Fe})_6(\text{Si,Al})_4\text{O}_{10}(\text{OH})_8$	1.4
Sericite (mica)	Monoclinic	$\text{KAl}_2(\text{AlSi}_3\text{O}_{10})(\text{OH})_2$	1.2
Total			100

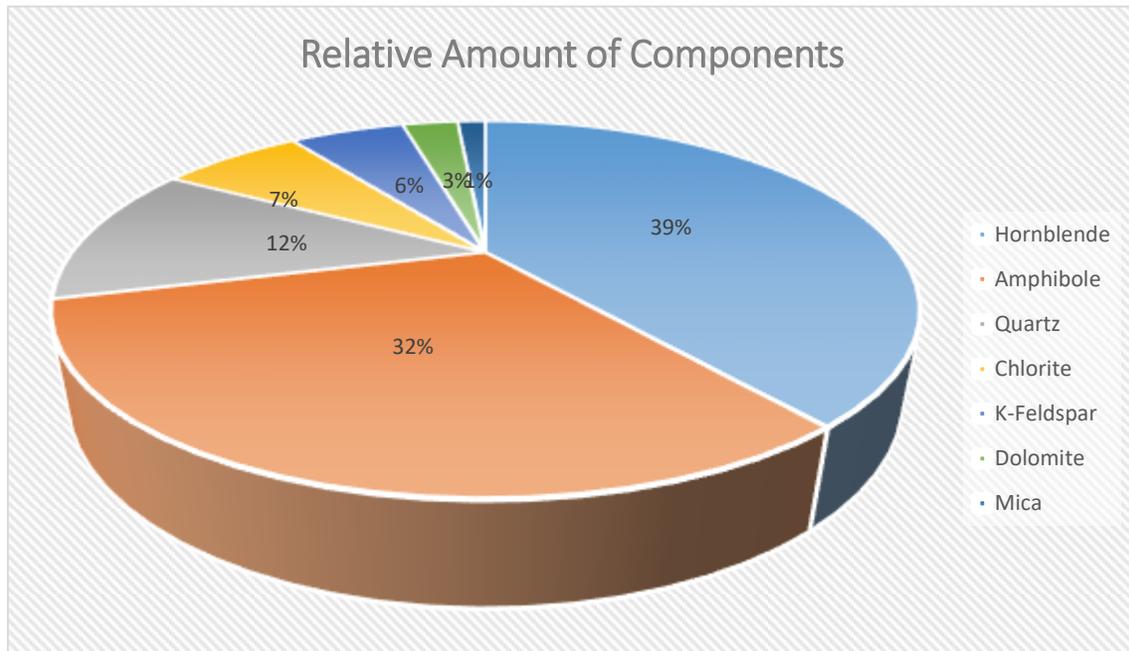


Figure 1 Relative amount in phase composition for rock samples acquired at Panda-North of Botswana

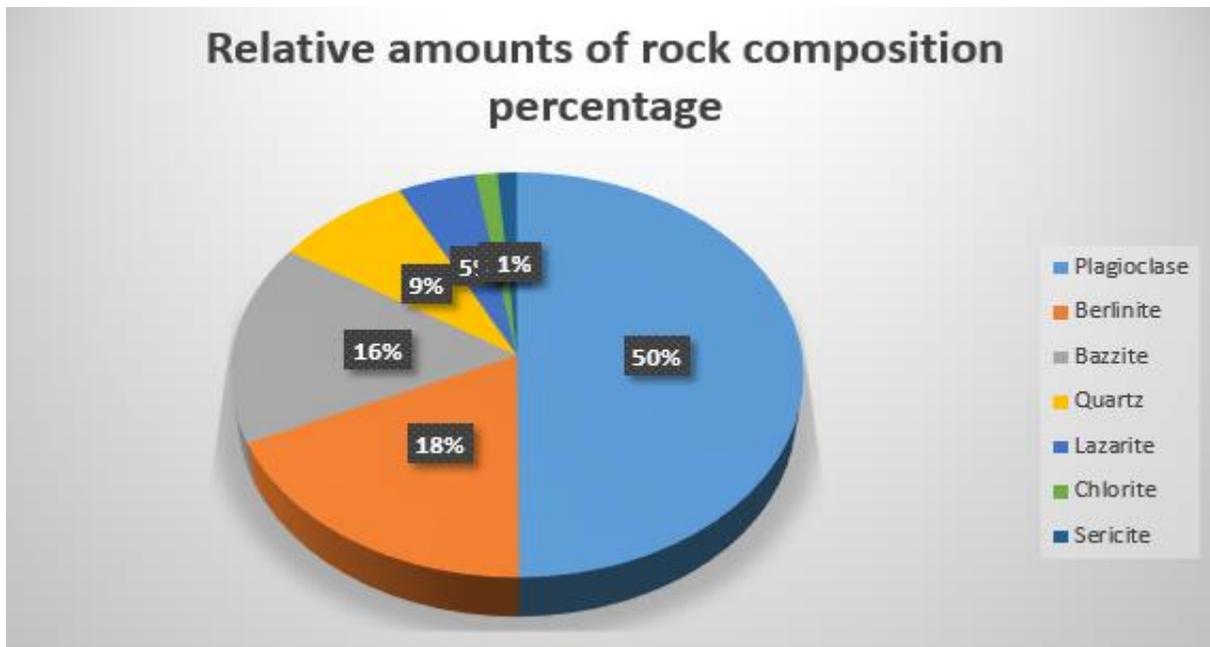


Figure 2 Relative amounts of diorite phase composition acquired in central of Botswana (Serowe)

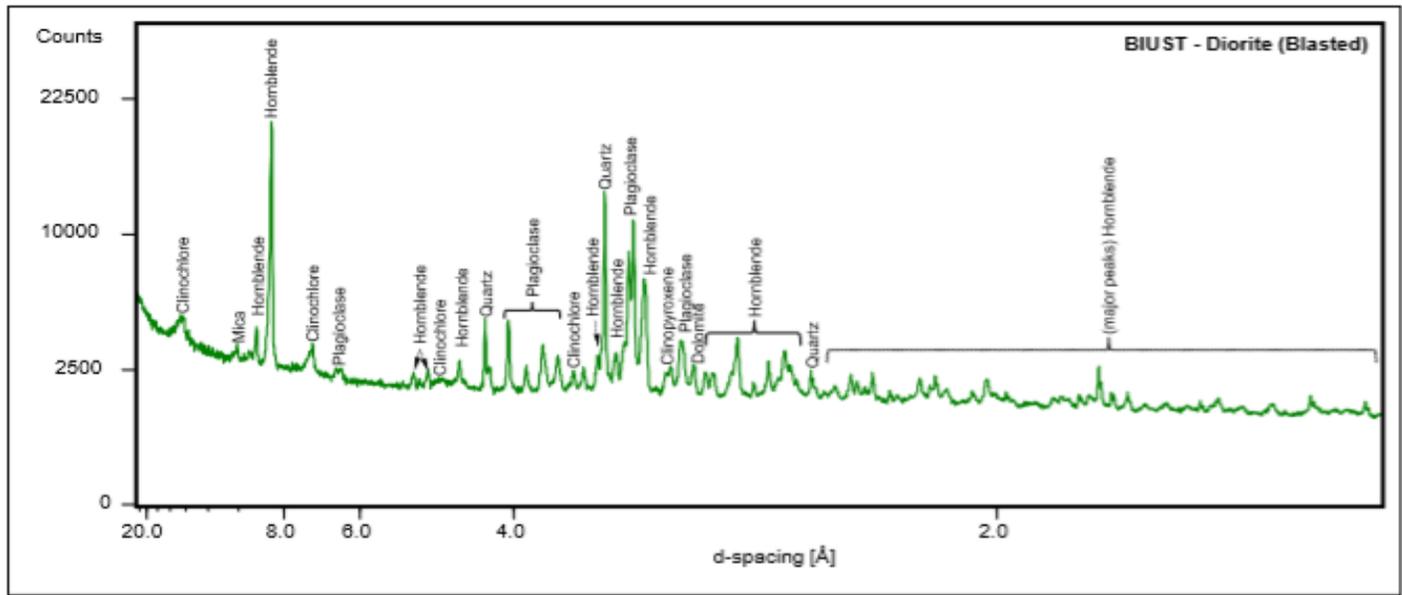


Figure 3 X-Ray diffractogram of the diorite complex obtained in Panda-north Botswana (Crystalline phases identified established presented the Panda Quarry Diorite sample to comprise Hornblende, amphibole, quartz, K-feldspar and some minor traces of chlorite and mica.)

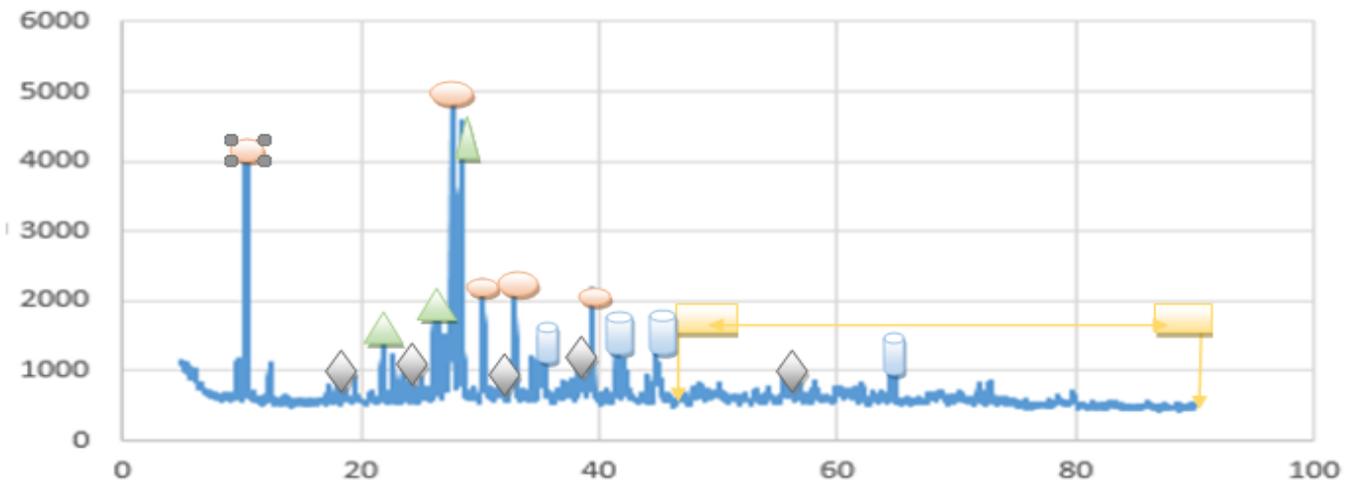


Figure 4 X-Ray diffractogram of the diorite rock sample obtained in Serowe Central Botswana (Crystalline phases identified at master quarries are mainly plagioclase, Berlinite, Bazzite, Quartz, Lazarite and other trace elements.)



Petrographic Examination of the Sample (s)

Petrographic examination of the panda rock specimen estimated that the rock is fine-grained structure that contain slightly-to-partially altered structures of quartz imbedded within a diorite matrix material (Nidhi et al. 2011). The rock comprise mainly plagioclase as the major constituent of the rock and amphibole and/ or actinolite complex, with also small quantities of quartz, **SiO₂**, Potassium feldspar with inclusions of other trace elements and constituent compounds like chlorite and mica which were simultaneously identified via the x-ray analysis device. The amphibole distinguished within the specimen matrix dispenses itself to be initiated from clinopyroxene alterations, but the plagioclase resulted mainly due to nucleation through the process of argillisation. Trace elements and other members identified within the rock were mainly accounted as the clay fraction, sericite, ilmenite and sulphites deposited also as part of the petrographic investigation. Therefore, due to this large degree of alteration on phase composition, it is suggested that the panda rock material type will be best suited for road construction material rather than as an aggregate for concrete constituent. The diorite rock sample obtained from Serowe region display a random distribution of phase orientation within the matrix with rather a unique classification of minerals composition fraction of hornblende (45 – 55%), Berlinite (18 – 20%), Bazzite (10 – 15%), Quartz (5-12%) and other minor traces of chlorites mixed with mica mineral as depicted in the diffractogram above, information that was theoretically justified. The melanocratic minerals crystallized simultaneously together as the dark appearance of the rock texture suggests and this matrix is surrounded but the light colored mica trace constituents which only deposited along grain boundaries together with the randomly dispersed minor alteration products like sericite, chlorite and calcite compounds (K et al. 2013). The overall texture of the magmatic master quarry rock sample show a fine-grained texture which resulted due to rapid crystallization of the host mineral during formation together with a mixture of few equigranular grain particles of aligned hornblende laths with an ophitic texture. Consequently, due to this random orientation of components it is thus difficult to classify mafic igneous rocks with consistent mineralogy and texture since there is high variation with a short range of ordered particles.

Conclusion

From the data acquired by the diffractogram, it was visible that majority of crystalline phases were easy to identify with panda quarry rock sample as plagioclase (39.20%), amphibole (31.74%), Quartz (12.26%) and minor inclusions of mica mixed with chlorides compounds. For the analyzed scan results clear data obtained with less noise levels, N, made classification straightforward, presenting the material to possess a high fraction of crystallinity with larger particle components which consequently led to recommendation of the material as mainly suitable for road construction activities and infrastructural work due to the inherent character of large phase composition. The data analysis for master quarry diorite sample as mainly Hornblende (49.9%), Berlinite (18.2%), Bezzite (16.1%) together with traces of other complex, displayed less crystallinity of components while showing greater amounts of fine – grained distribution of phases thus suggesting a larger contribution to be occupied by clay and minor trace constituents of lazurite, mica and silicates. From the diffractogram analysis it can be suggested that the master quartz stone is much suited to use in infrastructural work like building and also civil industry. Due to great domination in phosphates (Al (PO₄)) and lesser crystallinity of components, master quarry rock would require less amount of breakage energy in comparison to the Francistown's panda quarry rock product with higher crystallinity and a well-defined component field. The signal-to-noise ratio is important during analysis and a material with a high noise level remain difficult to classify, from these specimen it is clear that for Serowe rocks the signal was two times greater than the noise level, 2:1 while for panda quarries the ratio was approximately 3:1, these difference was due to the material's diffraction characteristics and linear absorption coefficient of each specimen.

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Mr Gee Gaesennweg; A teaching assistant and MSc Student at the University of BIUST under the department of chemical, materials and metallurgical engineering. For the past 6 years I was enrolled with Leeds University where I pursued an undergraduate program, still as a material scientist. Growing up, I always dreamed of working as a metallurgist especially in the mining industry because the economy of our growing country was continually investing more into mining. Also, I believe our country to be blessed since it affords many opportunities of science and technological improvement especially due to high reliance on production of methods and techniques that are old (for example: dependence on coal for energy production rather than employment of solar voltaic systems for energy production something that can not only reduce carbon emission into the environment but also create jobs and fill our large unused fields.) Outside learning I enjoy music and meeting new persons!!! Thanks.

