Assessment of Kalambaina Limestone for Quicklime Production

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Abstract

Quicklime of high quality is an important mineral for many industries (such as building, agriculture, water treatment, sugar refining, tannery, paper, glass, etc) all over the world. In Nigeria most of the demand for this chemical product is met through importation of quicklime. The Nigerian Kalambaina limestone was subjected to physical, chemical, mineralogical and thermal characterization to determine its suitability for direct use in quicklime production. The physical, chemical, mineralogical and thermal characterization were carried out using Accelerated Surface Area Porosimeter Braunner-Emmet-Teller (ASAP-BET) and Scanning Electron Microscope (SEM), Inductively Coupled Plasma Optical Emission Spectrometer (ICP-OES) and X-Ray Diffractometer (XRD) respectively. The results of the chemical analysis showed that the limestone contains 99.04, 0.894, 0.0414, 0.0085 and 0.006 percents of calcium, magnesium, silicon, iron and aluminum respectively. The XRD result shows that Kalambaina limestone is composed of calcite mineral with small amount of silica. The result of SEM shows that Kalambaina limestone has fine grain of crystals with homogeneous texture. Thermal characterization using TGA shows that weight loss due to release of carbon dioxide from Kalambaina limestone sample is 44.6% and optimum calcination temperature for obtaining quicklime of highest yield in nitrogen atmosphere is 800^{o}C. Also, the physical characterization using ASAP BET method shows that Kalambaina limestone has a surface area of 2.7764 m^2/g. The results of physical, chemical, mineralogical and thermal characterization shows that Kalambaina limestone is highly suitable for the production of quicklime making beneficiation treatment little or unnecessary.

1.0 Introduction

Limestone is a naturally occurring mineral that consists principally of calcium carbonate. It generally contains some magnesium carbonate as a secondary component. Limestone is found in many forms and is classified in terms of its origin, chemical composition, structure, and geological formation (Oates, 1998). Thus, limestone may be pure calcite, a mixture of calcite and dolomite or pure dolomite (Benton, 1973).

Large limestone and dolomite occurrences have been reported by Bell (1963), Ola (1977), Gwosdz et al (1996), RMRDC (2010) and Akande (2015). They are widespread in the sedimentary basin of Nigeria. Nigeria Ministry of Mines and Steel Development reported that Nigeria has an estimated reserve of about 3 trillion tonnes of limestone which are scattered in different states of the country. Those states include Abia, Anambra, Benue, Cross River, Ebonyi, Enugu, Ogun, Sokoto, Gombe, Nassarawa, Edo, Borno, Yobe, Adamawa and Kebbi (RMRDC, 2010).

Kalambaina limestone quarry belongs to the Paleocene marine sediments deposited in the Sokoto Basin during the second phase of sediment deposition when the Tethys Sea moved southwards through the Sahara (Nyong, 1995). The exposed section measured ca.19.5m in thickness. The lower part of the section consists of limestone-shale inter-beds while the upper part is phosphatised shale (Adekeye and Akande, 2002).

Quicklime is a solid material that is produced from thermal decomposition of limestone from which carbon dioxide gas is evolved and upon hydration, forms white powder and releases large amount of heat to form hydrated lime. Hydrated lime is an important industrial mineral because of its physical, chemical and mineralogical properties, as well as its commercial importance and ease of production. Foraminifera (2012) reported that Nigeria has high demand for quicklime based on its wide range of uses. The industries that use quicklime include building, agriculture, water treatment, sugar refining, tannery, paper and glass. The bulk demand for quicklime in Nigeria is for water treatment, soft drink bottling, tannery, breweries and food processing (Foraminifera, 2012).

Quicklime is produced from limestone that varies in properties from one location to another. Dogu (2000) reported that limestone can be obtained from a huge variety of sources and various limestones differ considerably in their chemical composition and physical structure. The chemical reactivity of different limestones shows a large variation due to their differences in crystalline structure and also because of the nature of impurities such as silicon, iron, magnesium, manganese, sodium and potassium present in them (Antonia et al, 2000). There are several references in literature concerning factors that may affect the quality of quicklime. Antonia et al (2000) reported these factors as characteristics of the limestone, calcination temperature, pressure acquired in kilns, rate of calcination, and fuel quality. The effect of calcination parameters on quicklime produced from various limestone deposits have been reported by the following authors (Khraisha and Dugwell (1989); Rao et al (1989); Khinast et al (1996); Dogu and Irfan (2001); Demir et al (2004); Muaazu et al (2011); Okonkwo and Adefila (2012) and Rashidi et al (2012)). Nevertheless, no studies have been reported, to the best of our knowledge, about the suitability of Kalambaina limestone for producing commercial quicklime. Hence, in this study, the characterization of Kalambaina limestone was carried out to determine its suitability for producing commercial quicklime.

2.0 Experimental

2.1 Materials and Procedure

Quicklime was produced from limestone obtained from Kalambaina quarry found in Northern part of Nigeria.

2.2 Analytical methods and techniques

The analytical procedures described below shows the techniques used in characterizing Kalambaina limestone.

2.2.1 Chemical Analysis

ICP-OES was used to determine the metals present in Kalambaina limestone in their elemental form. A mass of 50 g of Kalambaina limestone was dried at temperature of 105^{o}C. 0.5 g subsample of the dried limestone was subsequently digested in 10 ml aqua regia for a period of 1 hr and temperature of 140^{o}C. The digested limestone sample was further heated to temperature of 200^{o}C until the mixture produced a residue. The residue was subsequently dissolved in 2.5 M HNO_3 to form a solution. The solution was filtered and diluted to 50 ml solution. The diluted solution elemental composition was then analyzed using ICP-OES.

2.2.2 XRD Analysis

Kalambaina limestone mineral phases were determined using XRD. The diffractometer was put on and set to a voltage and current of 40kV and 30 mA respectively. The XRD fitted with was also operated at a wavelength of 1.5406. The XRD was scanned at a 2θ range from 20° to 60°. The diffractogram was also scanned at a rate of 3°/min.

2.2.3 Thermal Analysis

Differential thermal and thermogravimetric analyses (simultaneous DTA/TG) were carried out on Kalambaina limestone using Mettler Toledo, TGA/SDTA851 model. 50 mg of the limestone the limestone sample placed in an alumina crucible was weighed. The weighed limestone sample was heated from room temperature to 900°C at a heating rate of 10°C/min and nitrogen flow of 100 mL/min.

2.2.4 SEM Analysis

The texture, shape, and size of the limestone sample was determined using a SEM instrument model, HITACHI, S-3400N. The limestone powder was mounted onto the SEM stubs (layered with sticky carbon tape). 50 mg of the limestone the limestone sample was mounted on SEM stub and placed in a sputter coater (EMITECH, K550X) for five minutes. This is to provide coating with gold so that high reflectivity of the limestone sample during the scanning process. The samples were further heated to temperature of 40°C in an oven before SEM analysis.

2.2.5 Physical Properties Analysis (ASAP-BET-BJH)

Adsorption of nitrogen was performed on the limestone sample to evaluate the specific surface area, total pore volume, and pore size distribution, using a Quantachrome instrument (model Autosorb-1). The physical characterization consisted of three steps, including dehydration, degassing under low vacuum pressure, and nitrogen gas adsorption at 77 K. The isotherm obtained was analyzed using the BET and Barrett-Joyner-Halenda (BJH) methods.

3.0 Results and Discussion

The metals present in Kalambaina limestone sample determined using ICP-OES method is shown in Table 1. The main elements found in it are Ca and Mg, with low levels of Mg and other impurities, such as Fe, Si, K, Na, Al and others. Those impurities either are found in the matrix of the crevices and other strata excavated along with the limestone (Chan et al, 1970). Table 1 shows

Kalambaina limestone has Ca content of 99.04%. The 99.04% calcium content of Kalambaina limestone is higher than 98.5% required for limestone used for commercial quicklime production (Oates, 1998). Kalambaina limestone is a higher purity limestone, since it is higher than 98.5% required for higher purity limestone (Cox et al, 1977). The CaO content, i.e. the Ca content (Table 1) less the LOI content (Table 2) for Kalambaina limestone is 54.44%. The 54.44% calcium oxide content of Kalambaina limestone falls within the range of 50% to 65% required for limestone for quicklime production (Plumpton, 1984). The CaO content is also very close to 54.1% determined for limestone to be used for pilot scale iron-making in India. The CaO content of the raw Kalambaina limestone is higher than 43.05% for Indian Salem Madras deposit and is only slightly lower than the flotation concentrate of Salem limestone with 52.9% CaO (Ryemshak et al, 2012). The Kalambaina CaO content is also far higher than 43.44% and 38.68% for low grade raw limestone under consideration for upgrading by Indian Tata Iron and Steel Ltd. and Rhotas Industries, respectively (Ryemshak, 2012).

Table 1: Elemental Chemical Composition of Kalambaina Limestone using ICP-OES

Chemical Compound	Ca	Mg	Si	Fe	Al	Other Oxides
Kalambaina Limestone (% ppm)	99.040	0.8938	0.0414	0.0085	0.006	0.0157
Commercial limestone (% ppm)	98.5	0.5	0.35	0.6	0.2	0.050

The Mg content of 0.474% in the raw Kalambaina limestone is slightly higher than 0.5% standard specified for commercial quicklime production (Oates, 1998). The Mg content which is a function of MgO present in Kalambaina limestone is lower than minimum of 2.48% standard specified for limestone for direct quicklime production (Plumpton, 1984). The MgO content of Kalambaina limestone is lower than 1.0% MgO content for a limestone for pilot scale iron-making in India (Ghandy, 1966). MgO forms the low melting point magnesium silicate with silica during iron-making. These results strongly suggest that the Kalambaina limestone with low Mg content is a high-grade limestone suitable for use in quicklime production as the Mg content which is present as $MgCO_3$ in limestone produces MgO at high calcination temperature and make the quicklime to sinter (Gunasekaran and Anbalagan, 2008). This sintering effect reduces the yield and reactivity of quicklime and this occur because MgO is produced from dolomite at a lower temperature than CaO and so crystallites tend to grow larger and less reactive (Harrison et al, 1998).

The iron content of 0.004% in Kalambaina limestone is lower than 0.6% standard specified for commercial quicklime production (Oates, 1998). The iron oxide content of 0.004% suggest that Kalambaina limestone will produce quicklime of high yield and reactivity as iron present as Fe_2O_3 reacts with produced quicklime at high temperature to form bellite which reduces the yield and reactivity of quicklime.

The 0.006% aluminum content in Kalambaina limestone is lower than 0.6% standard specified for commercial quicklime production (Oates, 1998). Since aluminum present as alumina is a highly refractory oxide, its low content in Kalambaina limestone strongly indicate Kalambaina limestone as a high-grade limestone. Alumina acts as a base or as an acid depending on the conditions imposed. For example, at high temperature, it may form alumina silicates, while in the presence

of a strong base like quicklime it may form undesired calcium aluminate that reduces the porosity of quicklime (Ryemshak et al, 2012).

3.1 Thermal analysis

The TG and DTA curves of Kalambaina limestone obtained in nitrogen atmosphere is shown in Figure 1. The trend of the TG curves obtained for Kalambaina limestone sample shown in Figure 1 represents standard TG curves of calcite (Wagner, 2009). It shows that thermal decomposition with the formation of volatile reaction product (CO_2) occurred. Further examination of the TG curves (Figure 1), shows absence of multi-step decomposition which confirms calcite as the predominant mineral phase present in Kalambaina limestone (Wagner, 2009). This result is also in agreement with XRD, ICP and SEM analysis of the limestone samples. The DTA curve of Kalambaina limestone, shown in Figure 1, presents one endothermic peak at temperature above 750 °C. This confirms calcite as the predominant mineral phase present in the limestone. The typical TG curves of Figure 1 show that Kalambaina limestone presented a weight loss of 2.5% below temperature of 300°C. The weight loss detected in the temperature range of 100–300°C can be attributed to the chemically bound water (Gunasekaran and Anbalagan, 2008). This was followed by a weight loss attributed to the decomposition of carbonates. The thermal curves representing the carbonate mineral are characterized by endothermic peaks caused by the evolution of carbon dioxide. DTA curve of calcite shows one endothermic peak (Harrison et al, 1998).

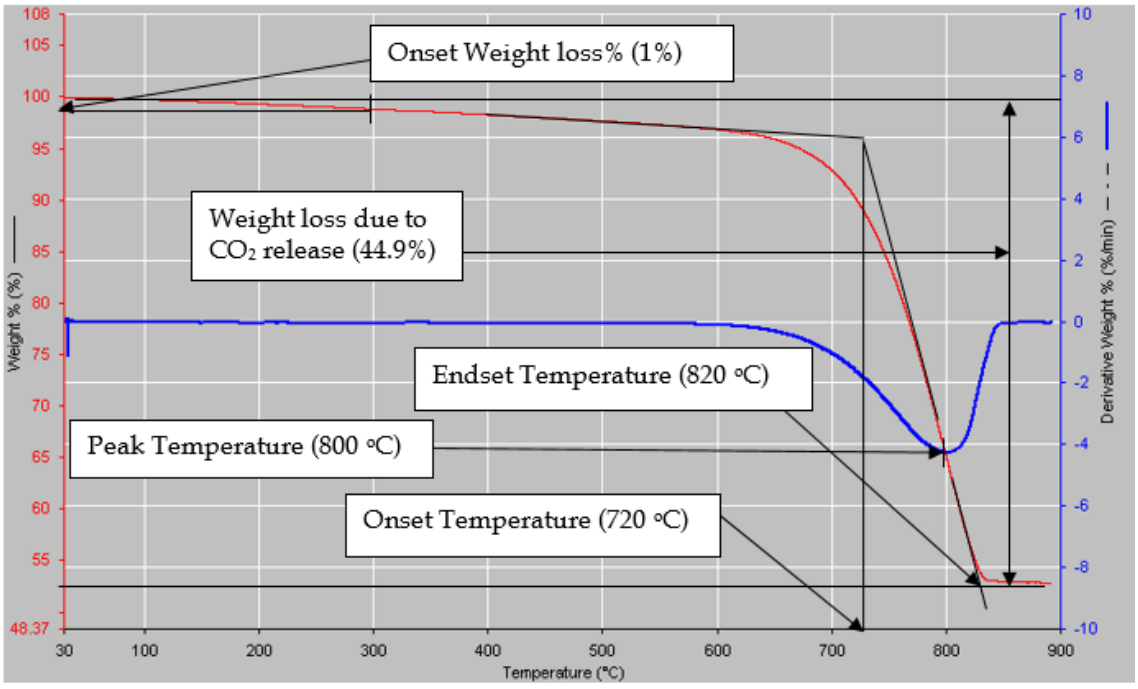

Figure 1: Thermogravimetry and Differential Thermal Analysis of Kalambaina limestone at heating rate of 20°C/minutes from 30°C to 900°C.

The calcination of Kalambaina limestone as shown in Figure 1 and Table 2 begins at 720°C, reaches a peak at 800°C and ends at 820°C. The presence of one peak temperature and absence of peak at lower temperature represents the decomposition of calcite and absence or non-significant

yield of dolomite (Anthony et al (2001); Gunasekaran and Anbalagan, (2008); Chan et al (1970); Harrison et al (1998) and Kilic and Mesut (2006)). The result of the TGA shows that optimum temperature for obtaining quicklime of highest yield in nitrogen atmosphere is 800^{o}Cas indicated in the peak temperature obtained in TGA plot of Figure 1.

Table 2: Thermogravimetric Analysis Parameters of Kalambaina Limestone

Limestone Type	Onset Temperature (^{o}C)	Peak Temperature (^{o}C)	Endset Temperature (^{o}C)	Onset Weight (%)	Weight Loss due to CO_2 release (wt%)
Kalambaina	720	800	820	99	44.6

Table 2 and Figure 1 show that weight loss due to release of carbon dioxide from Kalambaina limestone samples is 44.6%. Furthermore, it is observed that the percentage of carbon dioxide of Kalambaina limestone is higher than the theoretical one (44%). This fact might be ascribed to the presence of a high content of dolomite in Kalambaina limestone (Antonia et al, 2000). Gunasekaran and Anbalagan (2008) also reported that the weight loss of calcite is attributed to the decomposition of carbonates. For pure calcite, the expected mass decrease in one step (at 900 ^{o}C) is 44.6% (Gunasekaran and Anbalagan, 2008). A value of LOI higher than 44%, are indications for the presence of metallic impurities in the limestone sample which is higher in Kalambaina (44.6%) limestone sample (Gunasekaran and Anbalagan, 2008). Additionally, the case of presence of low quartz (silica) present in Kalambaina limestone, as observed in XRD result (Figure 3) and ICP result (Table 1) leads to no fluctuation in Kalambaina DTA curve, at temperature of 360 - 680 ^{o}C (Gunasekaran and Anbalagan, 2008).

3.2 SEM analysis

The scanning electron microscopy (SEM) image of Kalambaina limestone is presented in Figure 2. The SEM image also show there may be presence of Oolites cemented by different types of calcites as observed in Kalambaina and Kalambaina limestones. The SEM analysis confirmed the granulometric characteristic of the calcite as microcalcite and syntaxial calcite in the core of the oolites. The SEM images also show that the crystal shapes of Kalambaina limestone are distributed homogeneously throughout the mass and the crystal size are fine grain, compact and homogeneous texture (Akande, 2015).

SEM image of Figure 2 shows that Kalambaina limestone with fine grain and homogeneous texture of crystals will produce quicklime with bigger surface area and high reactivity due to a fine-grained rock is relatively quick-burning as carbon dioxide diffuses rapidly through the large number of internal pores between small limestone crystals (Harrison, 1993). Kalambaina limestone with fine grain and homogeneous texture of crystals when decomposed, the limestone front will reach the core of the chippings faster and a quicklime product containing burnt carbonate as well as high reactive quicklime will be produced (Harrison et al, 1998).

3.3 XRD analysis

The XRD spectrum of Kalambaina limestone is shown in Figure 3. The horizontal axis caption, 2θ in the figures is doubled Bragg angle in XRD analysis while the vertical axis shows the peak intensity. Analyzing the diffractograms (Figure 3) and peak matching with JCPDS (Joint

Committee for Powder Diffraction Standards) data, the crystal phase predominant on the samples was calcite. As can be seen from Figure 3, the main peak of the XRD spectrum appears for calcite at 2θ = 29.5ºand minor peaks appear at 2θ = 26.6º, 29.5º, 31.4º, 36.0º, 39.4º, 43.2º, 47.2º, 47.5º and 48.5º, respectively. The mineral phase of limestone identified in Kalambaina limestone is similar to that obtained by the following authors (Navarro et al, 2009; Soares et al, 2008)

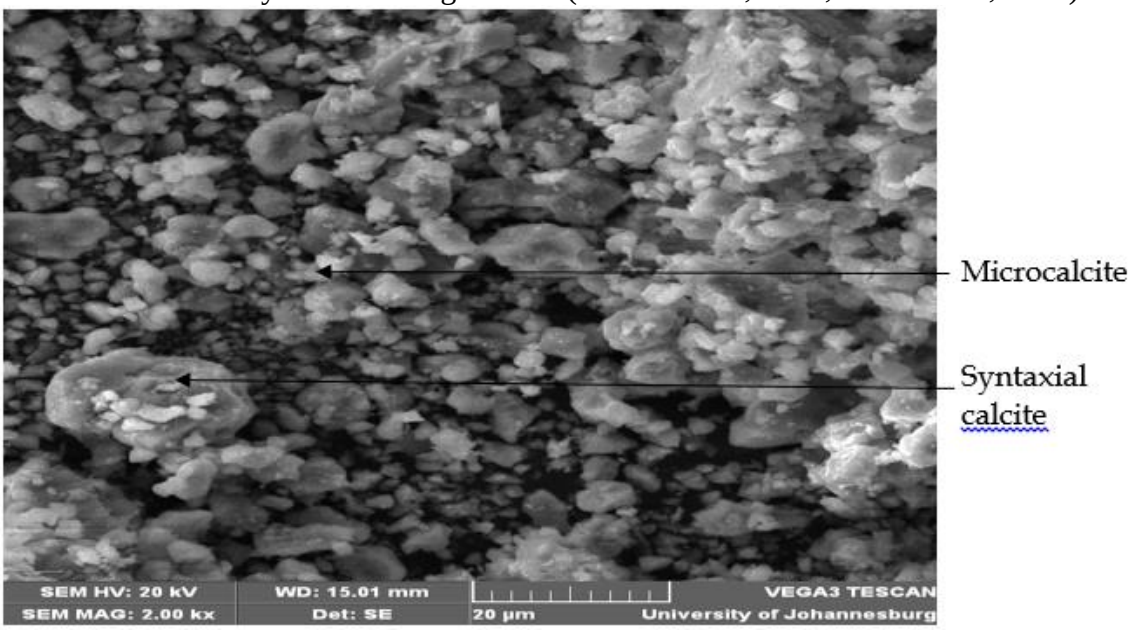

Figure 2: Scanning Electron Microscopic (SEM) Image of Kalambaina Limestone

Table 3 present the crystallinity parameters of limestone samples obtained from XRD analysis. By XRD examination using d-values spacing of $^{o}2\theta$values obtained above, the main constituent of the limestone samples is calcite. The XRD result shown in Figure 3 shows that Kalambaina limestone sample has small yield of quartz. This is corroborated by the silica level determined by ICP-OES (Table 1).

Table 3 shows that Kalambaina limestone has variable grain size of 428.19 *nm* respectively. This shows that Kalambaina has a high coherent scattering domain with perfect arrangement of unit cells at d-spacing (2θ) of 29.5º (Chen et al, 2007). Further examination of the diffractograms of Kalambaina shows that it has a calcitic strain of 0.18%. This shows that Kalambaina limestone is crystalline. The results of crystallite strain show that Kalambaina will produce quicklime of highreactivity and tendency to decrepitate during calcination is low (Harrison et al, 1998). Decrepitation decreases the porosity of the bed, retarding the flow of heat and reducing efficiency of the calciner (Harrison et al, 1998). Also, Kalambaina fine-grained rock is relatively quick-burning as carbon dioxide diffuses rapidly through the large number of pores between small lime crystals (Harrison et al, 1998).

Table 3: Crystallinity Parameters of Limestone Samples Analyzed Using XRD

LimestoneType	Crystallite Size		Strain (%)	
	Calcite	Quartz	Calcite	Quartz
Kalambaina	428.9	-	0.16	-

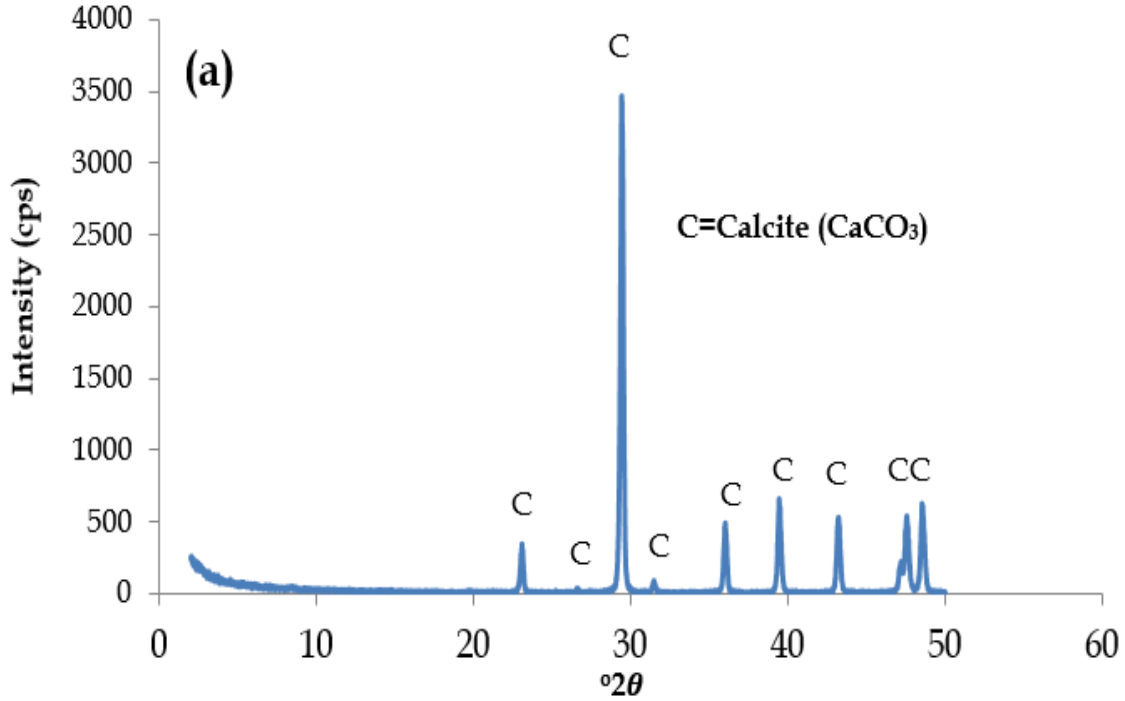

Figure 3: XRD pattern of Kalambaina Limestone

3.4 Surface area analysis using BET

The BET surface area, BJH adsorption cumulative surface area of pores, BJH desorption cumulative surface area of pores and pore volume are 2.7764m^2/g, 3.095m^2/g and 3.424m^2/g and 0.011328 cm^3/g respectively. The BET surface area of 2.7764 m^2/g in the raw Kalambaina limestone is higher than minimum of 1 m^2/g standard specified for limestone (Stanmore and Gilot, 2005). This high surface area greater than minimum shows that Kalambaina will produce more reactive quicklime as the calcination process is under the kinetics reaction control and therefore, the reaction rate is proportional to the BET surface area (Borgwardt, 1985). In addition, the high surface area of the sample will significantly improve the heat distribution within them and thus, accelerates better decomposition process (Mustakimah et al, 2012).

Table 4: Surface Area, BJH Adsorption cumulative surface area of pores, BJH Desorption cumulative surface Area of pores and Pore Volume of Kalambaina limestone

BET Surface Area (m^2/g)	BJH Adsorption Cumulative Surface Area of Pores (m^2/g)	BJH Desorption Cumulative Surface Area of Pores (m^2/g)	Pore Volume (cm^3/g)
2.7764	3.095	3.424	0.011328

4.0 Conclusion

This work has demonstrated the characterization of Kalambaina limestone using analytical techniques in order to determine its suitability for production of commercial quicklime. The following conclusions were obtained:

1. Kalambaina limestone has a calcitic mineral with fine grain homogeneous texture of crystals.
2. Kalambaina limestone is suitable for production of quicklime from limestone.
3. The optimum temperature for producing quicklime of highest yield in nitrogen atmosphere is 800^{o}C.
4. Kalambaina limestone has LOI due to release of carbon dioxide of44.6%.

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