

Determination of the Composition of the Waste Product Disposed From Calcium Carbide Use in South Eastern Nigeria Using X-ray Diffraction

Nobert Okechinyere Osonwa¹, Anayo Jonathan Nduka¹, Ferdinand Nnanna Aliegu², Charles Collins Ogbu²

1, Department of Ceramic and Glass Technology

2, Department of Metallurgical Engineering

AkanuIbiam Federal Polytechnic UnwanaAfikpoEbonyi State Nigeria.Email: nobertosonwa@gmail.com

Abstract:

A quantity of disposed waste of calcium carbide was collected from a dump site, near a mechanic garage in Afikpo, Ebonyi state of Nigeria. The sample was soaked and washed in ample distilled water for seven days, until no further effervescence from unreacted carbide was observed. The washed sample was dried in open air for seven days. The dried sample was grinded in a ball mill and sieved using mesh 100 sieve. The sieved sample was analyzed on an Empyrean diffractometer. The generated diffraction pattern was matched against the powder diffraction database. Analysis of the test data revealed that the major compounds present in the powder are calcium hydroxide (portlandite) $\text{Ca}(\text{OH})_2$, calcium oxide (calcite) CaCO_3 , and titanium oxide (rutile) TiO_2 . X-ray fluorescence analysis revealed the major element present in the sample to be calcium (69.20 weight concentration), as well as presence of trace amounts of some heavy metals such as silver (3.39 weight concentration), yttrium (3.30 weight concentration), niobium (2.85 weight concentration), and vanadium (1.41 weight concentration). The weight concentration of some of these heavy metals could have potential public health implications, as they could seep into ground water and contaminate it. It is recommended that regulatory agencies and chemical waste generators ensure proper and safe disposal of such wastes.

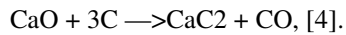
Keywords- Calcium carbide, x-ray diffraction, x-ray fluorescence, diffractometer, d-spacing, portlandite, calcite, rutile.

I. INTRODUCTION

Calcium carbide is a chemical compound with the formula CaC_2 . It is widely used in the production of acetylene and calcium cyanamide which is used as a fertilizer, [1]. Other uses include in steel making and as a raw material for the production of polyvinyl, [2]. The major use of calcium carbide in Nigeria is for the generation of acetylene gas for oxy-acetylene welding, especially by car body mechanics.

Technical grade calcium carbide often have a gray or off-white colour and consist of about 80 – 85 % of CaC_2 , with impurities such as calcium oxide (CaO), calcium phosphate (Ca_3P_2), calcium sulfide (CaS), etc, making up the rest, [3].

Thomas Leopold Willson accidentally discovered the process for the commercial production of calcium carbide in 1892. In this process, a mixture of lime and coke is heated to 2000°C in an electric arc furnace. At that temperature, the lime is reduced by the carbon to calcium carbide and carbon monoxide according to the equation



Lime for the reaction is usually made by calcining limestone and carbon can come from petroleum coke, metallurgical coke or anthracite coal. It is common knowledge that impurities in the furnace charge remain in the calcium carbide product, [5].

Natural lime supplies could contain significant concentrations of rare earth metals and heavy metals, [6]. Some of these impurities could remain in the discarded residue, after the use of calcium carbide by gas welders. Regulatory agencies and chemical waste generators must determine whether a discarded chemical is a hazardous waste, [7].

X-ray analysis provides a reliable method for the analysis and identification of unknown chemical substances. Two commonly used x-ray analytical methods are x-ray diffraction and x-ray fluorescence. X-ray diffraction provides a detailed information about the crystallographic structure, chemical composition and physical properties of materials.

Both x-ray diffraction and x-ray fluorescence techniques use an x-ray source and a detector. Both measure the response of the x-rays interacting with a substance and both provide measurements that help identify a substance, [8]. The distinguishing features between the two x-ray analytical methods is that x-ray fluorescence analyses for chemistry, (i.e elemental analysis), while x-ray diffraction determines the mineralogy/compounds present, [8].

In order to identify an unknown substance, the powder diffraction pattern is recorded with the help of a camera or a diffractometer and a list of d-values, (i.e the inter planer spacing), and the relative intensities is prepared. These data are then matched against a data base of known compounds, because each mineral has a unique set of d-spacings, [9]. Files for d-spacings for hundreds of thousands of inorganic compounds are available from the International Center for Diffraction Data as the Powder Diffraction File PDF, [10].

The reference patterns are usually represented by sticks and thus usually known as stick patterns. The position and intensity of the reference sticks should match the data, although a small amount of mismatch in peak position and intensity is acceptable experimental error, [11].

In practice, for any unknown sample, the appearance of three most intense characteristic lines from the standard powder diffraction file (PDF) line pattern is sufficiently convincing evidence of the existence of the crystallographic phase in either a homogeneous substance or even in a multi component mixture. Reference [11] reported that the diffraction pattern of a mixture is the sum of the diffraction patterns of each individual phase. In some cases, a distinction between two or more possible phases can be done by comparing the other characteristic lines, [9].

II. DESCRIPTION of STUDY AREA

Sample preparation, including grinding, sieving, etc, were carried out at the ceramic workshop of Akanulbiam Federal Polytechnic UnwanaAfikpoEbonyi state of Nigeria. X-ray analyses of the powder sample were carried out at the Nigeria Geological Research Laboratory Kaduna Nigeria.

III. MATERIALS and METHODOLOGY

The calcium carbide waste used for this research was obtained from a dump site near a mechanic garage at AfikpoEbonyi state of Nigeria. The calcium carbide waste was soaked in ample distilled water in a clear transparent bucket. The sample was vigorously stirred with a wooden stirrer and bubbles of gas from unreacted carbide were continuously released as the sample was stirred.

Excess water was decanted as well as some organic impurities which floated at the top of the bucket. The sample was allowed to soak for seven days, with the excess water decanted daily and refilled with fresh distilled water until no more bubbles were released from the sample.

The excess water was finally decanted and the wet sample was spread out on a drying mat and allowed to dry in open air for seven days. The dried sample was then ground in a ball mill for six hours and later sieved using a mesh 100 sieve. The sieved sample was then scanned on an Empyrean diffractometer.

The diffraction pattern generated was automatically matched against the PDF database.

IV. RESULTS and DISCUSSION

Figure 1 shows the generated diffraction pattern superimposed on the matching stick patterns from the powder diffraction file database. Table 1 shows the list of diffraction peaks observed from the scan of the powder sample. Table 2 shows the results of the x-ray fluorescence analysis with the percentage composition of the elements in the powder sample.

Table 1. List of observed diffraction peaks.

Pos.[°2Th.]	Height [cts]	FWHM	Left[°2Th.]	d-spacing [Å]	Rel. Int. [%]
4.0965	715.31	0.3070	21.57008	21.85	
8.1479	67.59	0.4093	10.85149	2.06	
18.0707	1346.39	0.2047	4.90905	41.13	
23.0415	177.44	0.2047	3.86004	5.42	
27.4979	834.82	0.1023	3.24375	25.50	
28.6711	548.55	0.2047	3.11364	16.76	
29.4140	2192.89	0.2047	3.03667	66.99	
34.1172	3273.44	0.3070	2.62805	100.00	
35.9323	311.52	0.1791	2.49935	9.52	
39.3610	394.59	0.2303	2.28918	12.05	
43.0849	350.59	0.1023	2.09956	10.71	
47.1196	1379.14	0.2558	1.92876	42.13	
47.5469	767.28	0.2047	1.91242	23.44	
48.4677	507.34	0.2303	1.87822	15.50	
50.7971	1120.01	0.2558	1.79742	34.22	
54.3524	518.27	0.1791	1.68796	15.83	
56.4677	99.42	0.3070	1.62964	3.04	

57.3782	188.79	0.3582	1.60593	5.77
59.3891	104.50	0.3070	1.55627	3.19
60.6569	127.59	0.4093	1.52674	3.90
62.6371	394.45	0.3070	1.48315	12.05
64.5774	373.00	0.2558	1.44320	11.39
65.7389	99.64	0.3070	1.42049	3.04
71.7831	249.52	0.2047	1.31502	7.62

TABLE 2: RESULTS OF THE X-RAY FLUORESCENCE ANALYSIS

Element Symbol	Weight Concentration.	Oxide Symbol	Stoichiometric weight Concentration
Ca	69.20	CaO	69.14
Ag	3.39	Ag ₂ O	2.60
Y	3.30	Y ₂ O ₃	2.99
C	2.97	CO	2.12
Nb	2.85	Nb ₂ O ₅	2.91
Si	2.48	SiO ₂	3.79
Ba	2.14	BaO	1.71
Fe	2.03	Fe ₂ O ₃	2.07
S	1.82	SO ₂	2.60
Al	1.72	Al ₂ O ₃	2.32
K	1.64	K ₂ O	1.41
V	1.41	V ₂ O ₅	1.80
Cl	1.32	Cl	0.94
Mn	1.02	MnO	0.94
Ti	0.89	TiO ₂	1.06
Mg	0.48	MgO	0.57
P	0.40	P ₂ O ₅	0.65
Na	0.25	Na ₂ O	0.24
F	0.21	F	0.15

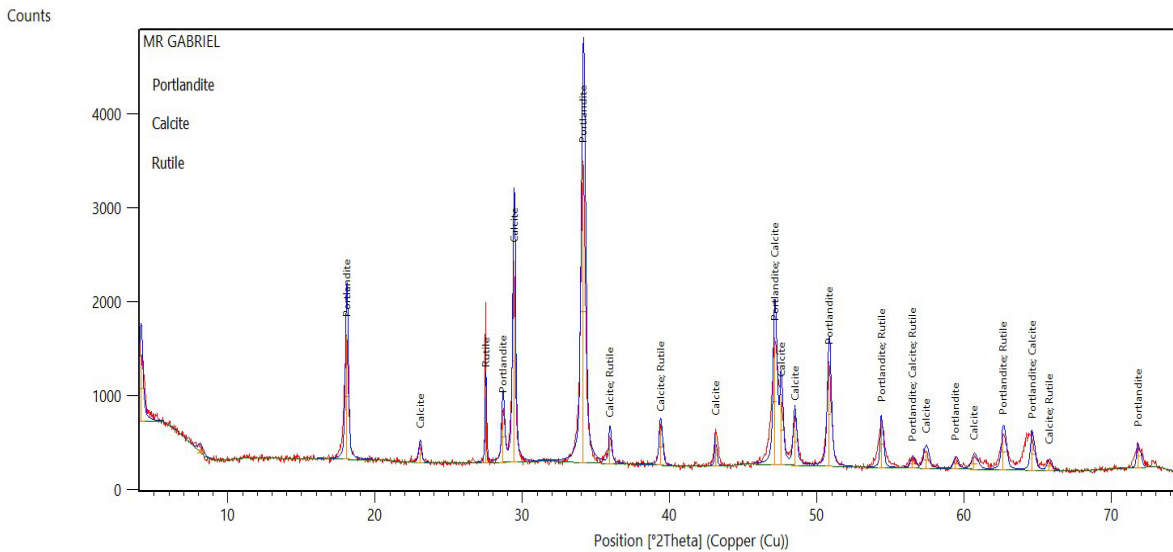
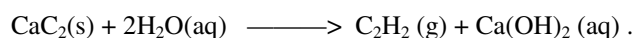


Figure 1: The generated diffraction pattern superimposed on the matching stick patterns from the powder diffraction file database. the major

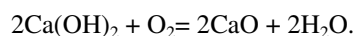
compounds in the discarded waste are calcium hydroxide, (portlandite) $\text{Ca}(\text{OH})_2$, calcium oxide, (calcite) CaCO_3 , and titanium dioxide, (rutile) TiO_2 , respectively.

From the result of the x-ray diffraction analysis, it can be seen that

From stoichiometric considerations, calcium carbide reacts with water to release acetylene gas according to the equation,



The calcium hydroxide residue from the above reaction, when exposed to air, will combine with atmospheric oxygen, to form calcium oxide according to the equation



Other oxide impurities in the discarded waste, including titanium dioxide, are possibly contaminants associated with the lime source used during the production of the calcium carbide, as reported by [6].

V. CONCLUSION and RECOMMENDATIONS

From the results of the x-ray analyses discussed above, it can be concluded that impurities in the lime source used during the production of calcium carbide could remain in the discarded residue after carbide use.

These impurities are found in association with the major constituents of the waste material which are calcium hydroxide and calcium oxide. Thus the titanium oxide and other trace elements such as silver, yttrium, niobium, etc. are possibly contaminants associated with the lime source used to produce the calcium carbide.

It is important to note that while some components of the discarded waste could be converted to useful purposes, some of the heavy metal impurities could pose potential public health danger as they could easily contaminate the ground water.

ACKNOWLEDGEMENT

The authors heartily acknowledge the Tertiary Education Trust Fund (TETFUND) of the federal government of Nigeria for sponsoring this research endeavor. We also acknowledge the contributions of members of the polytechnic community, especially the Rector, OgbonnayalbeEnwo.

REFERENCES

- [1].P. Pradyot. Handbook of Inorganic Chemical Compounds. New York. McGraw-Hill Education, 2003
- [2]. B. Langhammer. Calcium Carbide. Ullmann's Encyclopedia of Industrial Chemistry. 1st Online Ed. New Jersey USA. Wiley Interscience. June 2000
- [3]. J.VWayne. Risk Management for Hazardous Chemicals. Florida USA. C.R.C Press, 1996.

- [4]. N.NGreenwood. Chemistry of the Elements. 2nd ed. Oxford U.K. Butterworth-Heinemann, April 1997.
- [5]. J.W Frye. "Calcium Carbide Furnace Operation". In Proc .Electric Furnace Conference. American Institute of Mechanical Engineers, New York .1970. Pp. 163[4].
- [6]. J.T Christensen, S.L Svanestipp, L. Lakshtanor, J. Baker. "Rare Earth Element Incorporation in Natural Calcite: Upper Limits for Actinide Uptake in a Secondary Phase". *RadiochimicaActa*. Vol. 94 (9). Pp. 523-528.Jan. 2006.
- [7]. MAsif. "Physico-Chemical And Toxic Effect Of Fruit Ripening Agent Calcium Carbide". *Annals of Tropical Medicine and Public Health*. Vol. 5, Issue 3. Pp. 150- 156.July 2012.
- [8]. D. Michaud. (2017). Principles of X-Ray Fluorescence and X-Ray Diffraction [Online]. Available: [http:// www.911metallurgist.com](http://www.911metallurgist.com).
- [9]. A.KChatterjee. X-Ray Diffraction. Handbook of Analytical Techniques in Concrete Science and Technology. Norwich New York William Andrew Publishing, 2001.
- [10].B.LDutrow. "X-Ray Powder Diffraction". *Journal of Geoscience Education*. Vol. 52. No.1.Feb. 2020.
- [11].S.ASpeakman. (2013). Introduction to X-Ray Powder Diffraction Data Analysis. *Publication of the Center for Materials Science and Engineering*. Massachusetts Institute of Technology.[Online]. Available: <http://prism.mit.edu/x-ray>.