

Investigating on a Process Method of Beneficiating and Increasing the Graphite Purity to 99% Grade

Hilda Vimbai Kaitano, Stanford Mudono

Department of Chemical Engineering, Faculty of Engineering, National University of Science and Technology, Bulawayo, Zimbabwe

Email: stanford/mudono@nust.ac.zw

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Abstract

The main aim of this project was to come up with an efficient method for the purification of graphite to at least 99%. There has been an increasing demand for high-grade graphite products with up to 99.99% carbon that has resulted in the development of various approaches to remove impurities even to parts per million range. Removal of impurities from 94% graphite is important to achieve a high-purity graphite product. Microwave irradiation was used to prepare high-purity graphite from 94% graphite concentrate. The results showed that microwave irradiation could enhance the fixed carbon of flake graphite to a higher level. Under the optimum conditions selected of 4 minutes' reaction time and 100% microwave output (800 W), a graphite product with a fixed carbon content of 98.845% was obtained from flake graphite concentrate. According to XRD, FTIR and Handheld XRF analysis, impurities mainly composed of Fe, Co, Sr and Zr were present before treatment. After treatment under optimum conditions Fe, which was the major impurity in the sample was reduced from 3.566% to 1.031%. The ash content of graphite under optimum conditions was 1.55%. The crystal structure of flake graphite showed no change. It can be concluded from this study that graphite purification using microwave irradiation increases the carbon content of graphite.

Keywords

Crystal Structure, Fixed Carbon, Graphite, Graphene, Microwave, Irradiation

1. Introduction

As the world moves towards decarbonisation, electric vehicles (EVs) and clean

energy technologies offer a path toward a sustainable future. However, these technologies are mineral-intensive, and the minerals they use are becoming increasingly valuable. Graphite is one such mineral and demand for high purity graphite has markedly increased in the last decade (forecast to rise by 10% - 12% per year). One of the key roles of graphite is in the Green Technology Economy (Nuclear Power Industry, Fuel Cell Industry, Battery Industry, Power Tools Industry and Consumer Electronics Industry) as a key component of lithium-ion batteries (energy storage devices) [1]. Traditionally, energy storage devices included lead acid batteries and zinc manganese batteries. However, lithium-ion batteries have higher capacity and higher circulation efficiency and are fast replacing these traditional batteries. Though the lithium-ion battery is lightweight, rechargeable and relatively affordable more research is needed to further reduce its cost, weight and size. The lithium-ion cell consists of a carbon anode material such as graphite, a cathode material made up of lithium liberating compounds such as Lithium Cobalt-oxide (LiCoO₂), Lithium Manganese-oxide (LiMn₂O₄), and Lithium Nickel-oxide (LiNiO₂), a separator and an electrolyte [2] [3]. The key factor of the performance of lithium batteries is the power storage capacity of its anode material. A metal anode can be used though its performance is limited due to some irreversible phenomena such as electrochemical dissolution reactivity. Graphite, either natural or synthetic, is therefore currently used as a negative electrode material. The advantages of using graphite as an anode material include low cost, high electrical conductivity, mature production process and abundant resources [2].

The purification of natural graphite is required to achieve +99.9% carbon content with minimum metallic impurities for use in lithium-ion batteries. Currently, synthetic graphite is a dominant component for anode material in batteries. However, the high cost of synthetic graphite production acts as a key economic impetus to the development of new natural graphite sources for energy conversion and storage devices in general. Removing any impurities within the lattice structure helps to attain a highly purified graphite material for high-end technological applications [4] [5]. Separation and purification techniques that are currently employed for graphite mineral beneficiation can upgrade graphite to about 95% carbon by flotation, but further upgrading by physical methods is challenging. After flotation, the main impurities in the concentrate are minute silicate mineral grains and chemical compounds of sulphur (S), potassium (K), sodium (Na), magnesium (Mg), calcium (Ca), iron (Fe), and aluminium (Al) disseminated in graphite scales. Metallic impurities present can have a negative impact on products of graphite such as graphene. These impurities persist even after the chemical reduction of graphite oxide, which is employed to fabricate graphene materials. Such metallic impurities are extremely active as catalysts and the metallic impurities dramatically alter the electronic, electrochemical, redox, catalytic, and toxicological properties of the carbon materials. Important implications of impurities include the possibility of altering the electrochemical and electronic behavior or toxicity of graphenes since these materials are being used as electrode surfaces or tracers in cancer imaging [6]. The most abundant impurities, including Fe, Zr, Sr, cobalt (Co) and nickel (Ni) which were observed by an X-Ray Fluorescence (XRF) in our study, display prominent catalytic effects with consequently possible devastating effects in toxicological events.

Further refining using the caustic roasting-acid leaching technology can be employed. However, the caustic roasting-acid leaching technology is costly and also causes environmental pollution as well as workplace health and safety impacts [7]. Thermal purification of graphite at temperatures over 2500°C can be used but such high-temperature furnaces are expensive to build and operate. Alternative technologies such as microwave irradiation produce high-quality graphite. Microwave technology is energy-efficient and environmentally friendly and it is in great demand. Advantages of microwave irradiation include rapid and selective heating of materials, shorter reaction times, and low energy consumption, fast switch on and off, flexible modular design, higher product yields and high-energy efficiency while also being environmentally benign [8].

The differences in dielectric properties of the mineral matter content during microwave irradiation increase the amount of defects and cracks in the graphite structure. These cracks will increase the accessible area in the graphite structure for leach liquors and decrease its strength [9]. Yu-Feng li, Shi-fu Zhu and Lei Wang leached natural graphite under microwave irradiation by acid mixtures containing 12 molar hydrochloric acid (HCl) (12 M) and nitric acid (HNO₃) (16 M). It was found that the purification efficiency was affected by microwave power, reaction pressure, reaction time and the acid ratio. The carbon content of the graphite increased from 95.84% to 99.43% under optimized leaching conditions with an acid volume ratio of 1:1, pressure of 1.0 MPa, reaction time of 25 min and microwave power of 800 W. Scanning Electron Microscope (SEM) indicated that the morphology remained unchanged and X-Ray Diffraction (XRD) indicated that there was no apparent change in the 2λ peak position, peak height and full width at half maximum after the leaching. This is of particular importance to many applications of graphite [7]. Microwave heating is independent of thermal conductivity of the surrounding materials, thereby providing an instant on-and-off heating mechanism [10]. In our study, we demonstrate that microwave irradiation of graphite can be considered an effective pretreatment prior to the chemical leaching of graphite. However, further purification using acid leaching is required to obtain 99.9%.

2. Experimental Procedure

2.1. Materials and Equipment

Carbolite Muffle Furnace type ELF 11/6, Sartorius Electronic balance, Silica Crucibles, Desiccator, Gold star multi-wave Microwave, X-Ray Diffraction (XRD), TrueX handheld XRF analyzer and Fourier Transform Infrared (FTIR) Spectroscopy.

2.2. Determination of the Ash and Carbon Content of Graphite

1 g of ground sample (<500 μ m) was weighed and placed in a silica crucible. The sample was dried for 1 hour at 105°C and weighed accurately (to 4 decimal places). Graphite was completely oxidized in a furnace at 900°C for 2 hours. The sample was cooled in a desiccator and weighed. The ash content was determined by calculating the difference between the initial weight of the sample at 105°C and the final weight at 900°C over the initial weight as a percentage. Ash content was calculated from the following Equation (1):

Percentage ash content =
$$\frac{105^{\circ}C_{weight} - 900^{\circ}C_{weight}}{105^{\circ}C_{weight}} \times 100$$
 (1)

The carbon content of the 1 g sample was determined by calculating the weight at 900°C over the initial weight as a percentage.

The carbon content of the sample was determined using the following Formula (2):

Carbon content of the sample =
$$\frac{900^{\circ}C_{\text{weight}}}{105^{\circ}C_{\text{weight}}} \times 100$$
 (2)

2.3. XRD Analysis

X-ray diffraction (XRD) analysis was used to identify the crystalline structure of graphite. The interplane distance d for the sample was determined using Bragg's law [11].

$$d = \frac{\lambda}{2\sin\theta} \tag{3}$$

where λ is the radiation wavelength (1.5405); θ is the reflection angle for the reflex (00*l*) (*l* = 2, 4, or 6).

The packing density of the layers was also calculated using the following Equation (4):

$$p = \frac{0.762}{d} \tag{4}$$

The thickness of layers and the number of layers of graphite were calculated using the following Debye-Scherrer Equations (5) and (6):

$$t = \frac{0.89\lambda}{\beta\cos\theta} \tag{5}$$

$$n = \frac{t}{d} \tag{6}$$

where *t* is the total thickness of layers

 β = full width at half maxima (FWHM)

n = number of layers

d =interlayer spacing

 θ = diffraction angle

 $\cos\theta$ = value in radians

XRD analysis was used to determine if the morphology (shape) remained un-

changed during microwave heating.

2.4. Microwave Treatment of Graphite by Varying Time

Graphite samples were microwave-treated at varying times of 2, 3, 4, 5, 10, 15, 20 and 30 minutes at a constant microwave power output of 800 W. An XRD analysis of the treated graphite was carried out. An XRF analysis was also carried out to determine the carbon content of graphite.

2.5. Microwave Treatment of Graphite-Varying Microwave Power

Silica crucibles were thoroughly cleaned to remove any impurities that may interfere with the treatment procedure. 5 g of 94% graphite was weighed in a silica crucible and placed in a microwave. The graphite was heated at varying microwave power from 20%, 40%, 60%, 80% and 100% of 800 W at constant time. An XRD analysis of the treated graphite was carried out to determine if the morphology (shape) remained unchanged during microwave heating.

2.6. Chemical Analysis of Graphite

The chemical composition and the respective percentage composition of both the pre-treated and treated graphite were determined using the X-ray fluorescent spectrometer.

2.7. Determination of Functional Groups Using an FTIR Spectrometer

FTIR spectroscopy was used to investigate the different types of functional groups present in the samples and **Table 1** aided in identifying the locations and intensities of absorptions produced by typical functional groups.

3. Results

3.1. X-ray Diffraction (XRD)

X-ray diffraction (XRD) microscope was used to identify non-graphite minerals that may be present. The X-ray diffractogram of 94% graphite is presented in **Figure 1**. The sample predominantly consists of graphite with minor impurities. There is a sharp peak at $2\theta = 26.41$ which is characteristic of graphite (see **Figure 1**).

The intensity of the reflexes differs considerably. The reflexes for which $2\theta \approx 26.41^{\circ}$, 54.57°, and 87.25°, respectively, are reflections from polyarene layers. The peak at $2\theta = 26.41$ is for X-ray diffraction on a single crystal natural graphite aligned along the (002) plane. There is another peak at $2\theta = 54.57$ which is also characteristic of X-ray diffraction on a single crystal natural graphite aligned along the (004) plane. Reflexes 54.57° and 87.25° are of higher order and the reflexes in the diffraction pattern are asymmetric. That probably indicates the presence of several carbon phases (substructures) with different degrees of order



Figure 1. XRD analysis of flake graphite (94%) from Lynx mine.

Functional Group	Absorption Location (cm ⁻¹)	Absorption Intensity
Alkane (C-H)	2850 - 2975	Medium to strong
Alcohol (O-H)	3400 - 3700	Strong, broad
Alkene (C=C)	1640 - 1680	Weak to medium
(C=C-H)	3020 - 3100	Medium
Alkyne		
(C≡C)	2100 - 2250	Medium
(C≡C-H)	3300	Strong
Nitrile($C \equiv N$)		
Aromatics	1650 - 2000	Weak
Amines (N-H)	3300 - 3350	Medium
Carbonyls (C=O)		Strong
Aldehyde (CHO)	1720 - 1740	
Ketone (RCOR)	1715	
Ester (RCOOR)	1735 - 1750	
Acid (RCOOH)	1700 - 1725	

Table 1. IR Absorptions of common functional groups.

and structural characteristics. **Table 2** presents the basic structural parameters and calculation results of the graphite on the basis of the initial reflexes $2\theta \approx$ 26.41°, 54.57°, and 87.25° (that is, the reflexes before resolution into components). The interplane distances calculated from the initial reflexes $2\theta \approx$ 26.41°, 54.57°, and 87.25°, are similar. The calculated interplane distances are 3.373, 3.360 and 3.349 Å. These values are typical of natural graphite. Different graphite

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1					0	1		
2 <i>0</i>	Intensity	θ	Sin θ (deg)	n	ho (kg/m ³)	nλ	2sin $ heta$	d (Å)
12.7	5039	6.35	0.110602	5	0.021883	7.7025	0.221203	34.8209
26.45	25293.2	13.225	0.228776	1	0.226325	1.5405	0.457551	3.366836
44.81	5037.7	22.405	0.381151	6	0.062845	9.243	0.762302	12.12511
54.57	5803.5	27.285	0.458417	2	0.226753	3.081	0.916834	3.360478
59.13	5039.9	29.565	0.493411	6	0.081354	9.243	0.986821	9.366438
83.81	5032.9	41.905	0.667898	7	0.094392	10.7835	1.335795	8.072721
87.25	5041.6	43.625	0.689935	3	0.227515	4.6215	1.379871	3.349226

 Table 2. Basic structural parameters and calculations of graphite.

samples have different carbon content and different structural characteristics. An assumption is made that the structural phase with an interplane distance of 3.340 - 3.370 Å is typical of a true graphite structure. Small differences in the parameters calculated on the basis of reflexes 26.41°, 54.57°, and 87.25° were observed. The reflexes 54.57°, and 87.25° are used as characteristics of the graphite since higher orders of reflection ensure more precise results for the interplane distance [12] [13]. There is a small peak at $2\theta = 12.50^{\circ}$. This shows the presence of an impurity. Another peak occurs at $2\theta = 44.81^{\circ}$, $2\theta = 59.13^{\circ}$, 77.77°, 83.81° and 87.14°. All these peaks are not aligned with graphite peaks.

The density of natural graphite is between 2.09 - 2.27 g/cm³ [12]. From **Table 2**, the results of XRD at 26.45°, 54.57° and 87.25° show that the densities at the respective angles are 2.26, 2.27 and 2.27 g/cm³. The values of the density of 94% graphite are close to the theoretical maximum due to the high degree of crystal-line perfection of their structure. These results are typical of natural graphite.

3.2. Microwave Treatment of Graphite-Time Optimization

5 g of graphite sample were microwave treated at varying times of 2, 3, 4, 5, 10, 15, 20 and 30 minutes. The graphite content was determined using a TrueX handheld XRF analyzer. Results were recorded and tabulated in **Table 3**. A graph showing the effect of time due to exposure to microwave irradiation on graphite is shown in **Figure 2**. The graphite content increases as time increases and at an optimum time of 4 minutes, the graphite content is 98.845%. At above 4 minutes the graphite content decreases gradually. XRD results show that as temperature increases above 4 minutes' relative intensity also decreases. This can be a result of a phase change from flake to amorphous. Another reason could be that continued heating alters the crystallinity of graphite. This is because continued heating microwave irradiation to increase the number of defects and cracks in the graphite structure. These cracks will increase the accessible area in the graphite structure for leach liquors and decrease its strength [7] [9]. Physical observation of the samples shows graphite slowly changing colour from metallic



Figure 2. Effect of time on graphite content.

Table 3. Change in relative intensity of graphite under varying conditions.

Sample	2θ	Relative intensity
40% microwave power (4 min)	26.144	157,694
80% microwave power (4 min)	26.2088	231,562
100% microwave power (4 min)	26.4515	414,741
100% microwave power (20 min)	26.4354	373,693

black to brown colour. This change can be due to the effect of graphite decomposing at high temperatures. According to literature decomposition starts at 600°C under an oxidizing environment. Graphite was microwave-treated by varying the reaction time and an optimum time of 4 minutes was observed. Analysis using an XRD at 4 minutes and another analysis at 20 minutes was conducted (**Figure 2**).

3.3. Microwave Treatment of Graphite-Varying Microwave Power

The microwave power output varied from 20%, 40%, 60%, 80% and 100%. For this experimental procedure, the time was maintained at 4 minutes. The maximum power output of the microwave was 800 W. Figure 3 shows the effect of power on the carbon content. From the graph, it is observed that the carbon content increases as the power output also increases. Maximum carbon content is obtained at 100% power output. The maximum carbon content was 98.845%.

3.4. XRD Analysis of Microwave Treated Graphite at Various Microwave Power Output

Samples at 40%, 80% and 100% power output were analyzed using an XRD (Figure 4). Table 3 shows how the relative intensity increases as the microwave power output increases. At 40% microwave power output, a relative intensity of



Effect of power output on the carbon content

Figure 3. Effect of power output on the carbon content.



Figure 4. XRD analysis of microwave-treated graphite under varying conditions.

157,694 was obtained, and there is a shift of the diffraction angle from 26.41° to 26.144°. XRD plot of sample 2 (40% microwave power) shows the presence of an impurity at 12.32. Relative intensity of 231,562 was obtained at 80% microwave power output. There was a shift of the diffraction angle to 26.2088°. When graphite was treated using the maximum power output (800 W) the relative intensity of 414,741 was obtained therefore treating graphite at maximum power gives the best results. This is according to literature which states that the higher the relative intensity the sharper the peak and the more crystalline (pure) graphite becomes. Plots of sample 1, sample 3 and sample 4 show the absence of any impurities. The results show that maximum treatment is obtained at 4 min since a maximum intensity was obtained.

3.5. FTIR Analysis

FT-IR spectroscopy was used to investigate the different types of functional

groups present in 94% natural graphite. The FT-IR spectra of graphite are shown in **Figure 5**. Firstly, the broad and intense peak at 3430 cm⁻¹ arises from stretching vibrations of O-H group, and the peak observed at 1639 cm⁻¹ is due to stretching vibrations of the C = C (aromatic ring). There is a peak at 1108. Another peak is at 1280 cm⁻¹ stretching vibrations of epoxy C-O groups. **Table 4** gives a summary of the functional group present in the graphite.

Figure 5 shows the FTIR spectra for 94% graphite before microwave treatment. FTIR spectroscopy is regarded as an important tool for the characterization of functional groups. The characteristic peaks at 3448, 1637, 1400, 1116, and 613 cm⁻¹ in the 94% sample were due to O-H stretching, C = O stretching, aromatic C = C stretching, O-H deformation, epoxy C-O stretching, and fingerprint region.

3.6. Chemical Analysis of Graphite

The chemical compositions of 94% graphite and microwave-purified graphite obtained by X-ray fluorescence are given in Table 4. Table 5 shows chemical



Figure 5. FTIR spectra of 94% natural graphite.

	T	able	4.	Summar	v of functional	groups in	94%	graphite
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Wavelength number (cm ⁻¹)	Functional Group	Absorption Intensity
3448	(O-H) group	Strong, broad stretching
1637	Alkene (C=C)	Weak to medium
1400	Fingerprint region	Weak
1108	Epoxy C-O	Blank
615	Fingerprint region	Blank

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Time (min)	LE	Fe	Со	Sr	Zr
0	96.346	3.566	0.044	0.018	0.015
2	98.346	1.56		0.025	
3	98.821	1.033	0.017	0.014	0.029
4	98.845	1.031	0.009	0.011	
5	98.668	1.213		0.015	
10	98.566	1.264	0.01	0.011	
15	98.782	1.095		0.01	
20	98.538	1.312		0.02	0.011
30	98.716	1.204	0.013	0.02	

Table 5. Chemical composition (wt %) of raw graphite and microwave-treated graphite obtained using an XRF Spectrometer.

composition when elements with less than 0.1% concentrations were neglected. The microwave purification process enhanced the purity of the samples from 96.346% to 98.845%. The increase in carbon content is accompanied by a net decrease of the other chemical elements (Fe, Co, Sr, Zr) which are associated with graphitic carbon as impurities. It is worth noting that the studied graphite does not contain any heavy or toxic metals; the heaviest detected metal is iron. Therefore, the impurities are composed of non-toxic materials. The initial iron content was 3.566% and it was reduced to 1.031% at 4-minute microwave treatment. Neglecting elements with 0.1% concentrations, the chemical composition of graphite becomes as shown by **Table 6**.

3.7. Ash Content Results

A furnace was used to determine the ash content. In an oxidizing environment graphite slowly decomposes at 600°C, but it can withstand 3650°C in a non-oxidizing environment and it is particularly strong against thermal shock. Ash content analysis shows a value not in compliance with the specification requirement, *i.e.* <0.2%. The ash content of graphite ranged between 1.55% - 2.669%. The lowest ash content was obtained at 4 minutes' purification. A change in colour of the graphite is observed from metallic black to brown. This is because the graphite will have oxidized. it is necessary to carry out a chemical analysis of the ash produced by LOI tests on concentrates using an ICP – AES will indicate if undesirable oxides are present. These include major elements such as SiO₂, Al₂O₃, MgO, CaO and Fe₂O₃.

From Figure 6 the lowest ash content is obtained at point 2. This is when the reaction time was 4 minutes. At points 3, 4, 5 and 6 (5, 10, 15 and 20 minutes), we observe that the ash content starts to increase again. Though we observe the lowest ash content of 1.55% at 4 minutes, the value is still high to meet the ash content specifications of pure graphite. During microwave irradiation,



Figure 6. Ash content analysis.

Table 6. The chemical composition (wt%) of raw graphite and microwave-treated graphite was obtained using an X-ray Fluorescence Spectrometer.

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Time (min)	LE	Fe
0	96.346	3.566
2	98.346	1.56
3	98.821	1.033
4	98.845	1.031
5	98.668	1.213
10	98.566	1.264
15	98.782	1.095
20	98.538	1.312
30	98.716	1.204

the organic component of graphite is a relatively weak absorber of microwave energy. Quartz and other minerals appear transparent to the radiation whereas some minerals such as pyrite decomposed as pyrrhotite or iron sulfate and water in their structures readily heat within an applied electric field [14] [15]. The bonds of sulfur–carbon in organic sulfur compounds are broken and the sulfur is released in gaseous form in response to the effects of microwave irradiation. To further lower the ash content, quartz and other minerals need to be removed by further purification by acid leaching. The choice of acid will depend on the impurities present.

4. Conclusion

Purification of graphite using a microwave increased the carbon content from 94% to a carbon content of 98.57%, however application of heat can influence the intensity of graphite. Initial characterization of 94% graphite using a XRD

Spectrometer and XRF Spectrometer shows the presence of impurities such as Fe, Sr, Zr and Co. As the temperature increases, the intensity of the graphite decreases. The initial sample had a maximum peak at 26.45° and an intensity of 25,293. Graphite was microwave-treated by varying the time from 2, 3, 4, 5, 10, 15, 20 and 30 minutes and a maximum reaction time of 4 minutes was obtained. The intensity at maximum reaction time was 414,741. This was the maximum intensity obtained from all experiments. The maximum ash content was 1.55%. The power output of the microwave was also varied from 20%, 40%, 60%, 80% and 100% and the reaction time was kept constant at 4 minutes. XRD analysis of these samples at 40%, 80% and 100% power output show an increase in the relative intensity of 157,694, 231,562 and 414,741 respectively. According to literature as the intensity increases, the purer the graphite becomes. Therefore, maximum results are obtained at 100% microwave output (800 W). It can be concluded from this study that by further purifying graphite using microwave irradiation, the impurities present are decreased thus increasing quality and purity of graphite. The main impurity found in this study is iron which had an initial value of 3.566%. After microwave treatment at an optimum time of 4 minutes, iron was reduced to 1.031%.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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