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Modelling and Simulation of Graphite Synthesis *via* Hydrochloric Acid Leaching

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ABSTRACT

In this study, a simulation model of the synthesis of graphite from industrial waste *via* acid leaching was developed in MATLAB. The results obtained from this simulation will help contribute towards the preliminary understanding of large scale production of graphite. The acid leaching of industrial waste was carried out with the aid of HCl as the leaching agent based on 4 factors namely the concentration of HCl (A), temperature (B), solid to liquid ratio (C) and leaching time (D). Next, a statistical analysis was done *via* Response Surface Methodology (RSM) which is commonly used for modelling and optimization studies. Twenty-seven experimental runs were done based on the 4 factors on 3 levels of CCD. The ANOVA model and response surface plots were generated. ANOVA analysis was used to study the importance of each factor for the percentage of metal removal. The F-value and p-value of 68.17 and <0.0001 respectively were significant with the leaching model. The R_2 value of 0.99 and Adj-R_2 value of 0.97 were used to prove the fitting of the model. From the ANOVA model, a quadratic equation was attained and this equation was used for the simulation model. The simulation model was then done *via* Simulink in MATLAB and based on the optimum parameters the simulation was run. From the findings, variable A did not show any changes in the metal removal percentage. As for variable B, C and D the highest metal removal percentage was obtained at temperature of 70°C, a solid to liquid ratio of 10:1 and a leaching time of 60 mins, respectively.

1. Introduction

Graphite is made up of carbon atoms which are arranged closely whereby they are stacked in parallel in the form of planar honeycomb-lattice sheets [1]. Due to its distinctive physical properties, graphite has attracted a lot of attention in chemistry, physics and material science in terms of research. In 2004, Andre Geim and Konstantin Novoselov discovered graphene by peeling off a graphite layer with the aid of a tape from its block. The initially cleaved flake was continuously peeled off for any more layers until it was only a few atoms thick [2].

For many years, graphite has been an important material for the usage of refractory and high-technology applications. Fastest predicted growth portion of the market has been represented

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by the graphite used for high technology in which they are applied in lithium-ion batteries for electric motor vehicles, electric energy storage devices which are used at large scale and graphite derivatives namely grapheme [3,4], spherical graphite, expanded graphite and graphite foil. In early 1990s when lithium-ion batteries were first commercialized, they were known due to graphite's high energy storage capacity and stable cycle life [5,6]. Paint and pigments also use graphite because of its water repellent quality. Furthermore, the pencil industry uses up to 8 % of the graphite production in the world [7].

Graphite exhibits many distinctive properties which makes it suitable to be used for electronics, lubricants and metallurgy. For example, thermal stability, lubricity, high electrical conductivity and chemical inertness [1]. Chemical inertness towards most chemicals is showcased by graphite and it is a good heat and electricity conductor due to its structural feature of parallel stacking of layers onto each other and the presence of loose Van der Waals forces. Graphite can be used for arc lamp electrodes as it is a good conductor of electricity [8]. This weak Van der Waals forces among the layers has made graphite physically soft and slippery and therefore it is good lubricant in dynamos and electric motors [9,10]. It also has a great regular stiffness and strength. All these properties are maintained even when the temperature is reaching ~3600°C and that too without experiencing thermal expansion. Therefore, graphite is able to be used for mechanical, nuclear, aerospace and chemical applications [11].

There are three forms of commercial natural graphite namely flake, vein and amorphous graphite. Flake graphite is crystalline graphite flakes whereby their coarseness is >150 µm and fineness is <150 µm. Vein graphite has coarse graphite crystals aggregate interlock whereby their size is usually >4 cm. As for amorphous graphite, it is made up of soft earthy graphite and micro-crystalline which has a size of <40 µm. The flake, vein and amorphous graphite ore contains 2 - 30 %, >90 % and >70 % graphite respectively [12].

Graphite has an electrical conductivity ranging 2-3 × 10^4 Sc m^{-1} and a thermal conductivity of 1500-2000 $Wm^{-1}K^{-1}$. Apart from that, graphite is flexible but not elastic and it has a specific surface area at the range of 10-20 m^2g^{-1}. Graphite usually forms laminated or scaly clumps but it can also be granular or even scattered. It has an opaque black colour with a metallic shiny but it can also be lacklustre and soil-like [13].

Usually, graphite is acquired *via* mining natural resources namely coal and ores but due to the rapid overconsumption of the natural resources, an alternative method has to be looked up to produce graphite. Therefore, in this research industrial solid waste is used as an alternative resource to produce graphite.

Since Malaysia produces a lot of industrial waste annually, it would be a wise decision to make use of that waste to produce something beneficial. Graphite can be produced through the acid leaching of waste and this technique is not only an inexpensive alternative of graphite production but it can also be a solution for all industrial waste that is produced. High efficiency of leaching can be obtained if the operating conditions are optimal and important process variables are acquired from a simple parametric model of a conventional leaching process which involves multifaceted variables and complex dynamic behaviour. Therefore, the possible solution to tackle this issue of using the industrial waste (metal scrape) to produce graphite *via* hydrochloric acid leaching [14] using modelling is required to predict the performance of graphite synthesis in order to ensure that high leaching efficiency is acquired.

2. Methodology

The flowchart of the research is shown in Figure 1. In this work, industrial solid waste was used as the raw material to produce graphite. The raw material underwent grinding, sieving and characterization to recognize its elemental composition and the compound structure. Once the preparation was completed, the leaching experiment using HCl was carried out. Next, a statistical analysis was carried out to obtain the metal removal percentage against the leaching parameters *via* the RSM method. Lastly, based on the ANOVA equation, a simulation model was developed in MATLAB. In this work, experimental data and ANOVA equation was directly adopted from the study conducted by Siaw *et al.,* [14]. This work was extended from the existing study conducted by Siaw *et al.,* [14] with the model and simulation analysis performed in MATLAB.

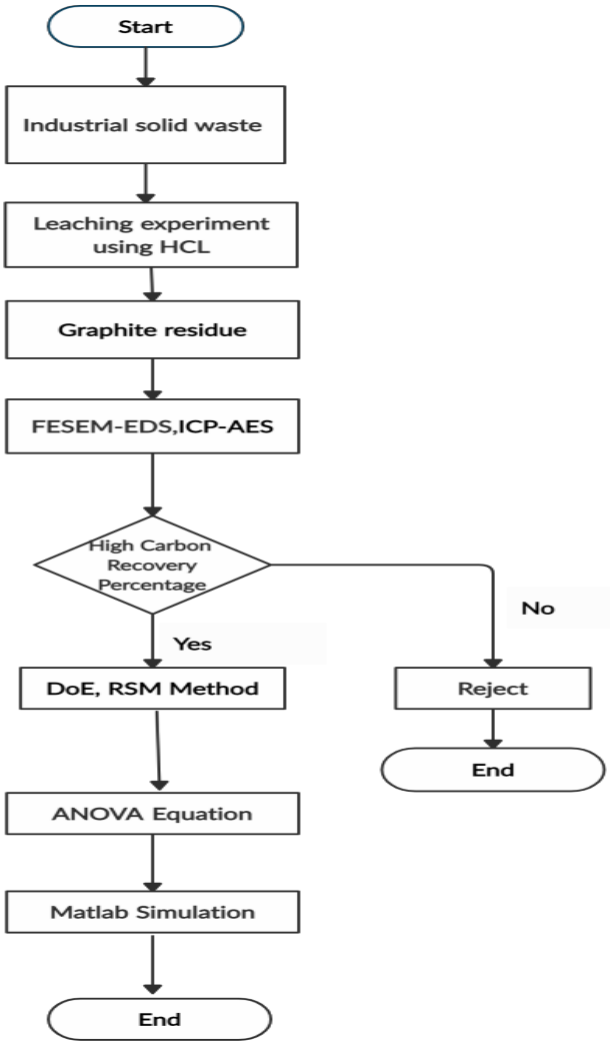

Fig. 1. Simulation and modelling of graphite from industrial waste

2.1 Chemical and Materials

In this study, the raw material that was used is the untreated industrial solid waste which can be obtained from metal scrap. Grinding *via* a mechanical grinder (Vigor mix 700 Swing Type Dry Grinder) was done to prepare a 106 µm of powder slag from industrial solid waste. The power slag

was then sieved using the GILSON 106 µm stainless steel test sieve. In the leaching experiment, a R&M brand acid (37 % HCl) which was supplied from Evergreen Engineering & Resources was used.

2.2 Leaching Process

Based on Siaw *et al.,* [14], the leaching process was done *via* RSM and the graphite residue was characterized. First of all, industrial solid waste of 20.0 g was leached in a solution which contained 3.0 M HCl at 30.0 °C for 120.0 min as tabulated in Table 1. Next, the graphite residue and the leachate were separated with the aid of centrifugation *via* Sigma 3-18K centrifuge. In order to obtain the dry graphite residue, a Binder drying oven at a temperature of 50.0 °C was used. After that, FESEM-EDS (elemental analysis) was done. As for the leachate solution, it was collected and stored in a waste bottle.

Table 1
Condition of leaching process with HCl [14]

Solvent	HCl
Concentration, M	3.0
Temperature, °C	30.0
Time, min	120.0
Stirring speed, rpm	50.0

2.2.1 Leaching experimental procedure

In Siaw *et al.,* [14], HCl was used to carry out the leaching process *via* the standard leaching apparatus set. A round-bottomed flask (750.0 mL nominal capacity) was equipped with a rotary evaporator and a water bath (Heidolph Hei-VAP Platinum 2 Rotary Evaporator). Rotation speed of 200.0 rpm was used to agitate the contents of leaching and the experimental set-up is shown in the Figure 2.

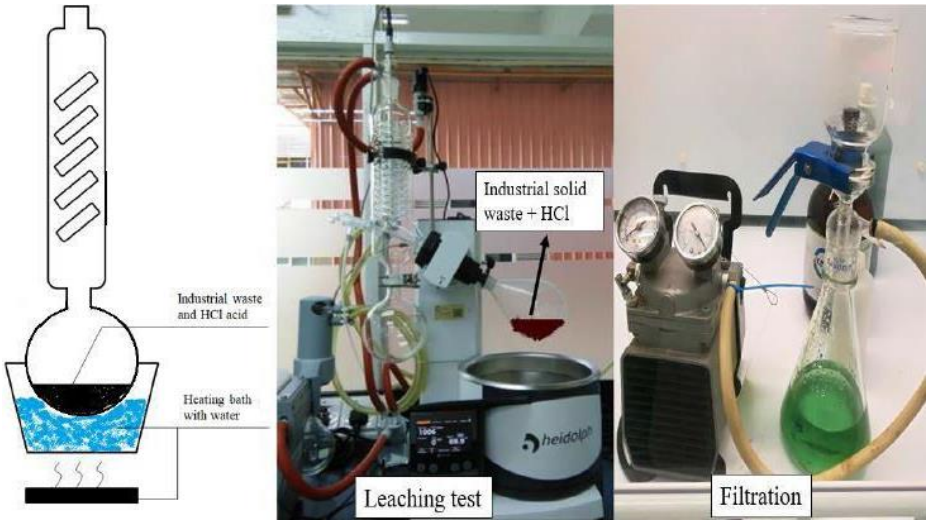

Fig. 2. Experimental setup for leaching process [14]

Vacuum filtration was used to separate the leachate and the graphite residue after the leaching process was done. Next, elemental compositions of impurities in the leachate were identified with the help of ICP-AES and with this, the leaching efficiency was calculated. In order to remove the excess Cl^-, the solid residue obtained was rinsed with distilled water. Then, the residue was filtered with

Whatman filter paper (125.0 mmΦ) in a glass funnel and the Binder drying oven was used to dry the residue at 60.0°C for 24.0 h.

2.2.2 Response surface methodology

Modelling and optimization studies are usually done *via* RSM which is a mixture of numerical and statistical techniques. In order to investigate the key interaction of the factors on the response in the leaching process, this method comprises of 3 levels namely CCD whereby four variables were chosen as factors which were HCl concentration, M (A), temperature, °C (B), solid to liquid ratio (C) and leaching time, min (D). The selection of the factors was made based on the factors that showed the greatest importance in the leaching process studies found in the literature. Based on the studies conducted in the past, the values of for the upper and lower levels of each input factor were selected which is tabulated in Table 2 [15-19].

Table 2
The lower and upper values of input factors

	Factor	Level			References
		Low	Centre	High	
		-1	0	+1	
A	[HCl], M	2.0	4.0	6.0	[15-19]
B	Temperature, °C	30.0	50.0	70.0	
C	Solid to liquid ratio	10.0	20.0	30.0	
D	Leaching time, min	60.0	180.0	300.0	

2.2.3 ANOVA

For statistical analysis of the experimental data, an ANOVA model was generated. In the CCD design however, 3 techniques of optimization were proposed namely, numerical, graphical and point prediction optimization. For the numerical optimization, you need to set up the goal in order to attain a response whereas for the graphical optimization, you need to set the maximum and minimum limits to get a response. The point prediction optimization predicts the response value once the desired operating condition is keyed-in.

2.2.4 Elemental analyses

Field Emission Scanning Electron Miscopy (FESEM) with Energy Dispersive X-Ray Spectroscopy (FESEM-EDS) and Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES) was applied to determine the elemental composition in the leachate and graphite residue.

2.2.5 FESEM-EDS

Industrial solid waste and the residue obtained from leaching was analyzed for their atomic elemental composition with the aid of Energy-dispersive X-ray spectroscopy JOEL JED-2300 (EDS). Figure 3 shows that EDS is attached to the FESEM whereby it was used to obtain the elemental and surface morphology analysis [14].

Siaw *et al*., [14] even mentioned that element peaks were generated when the X-ray excitation in EDS interacts with the sample and this was fulfilled with the quantification of the elemental compositions. After that, a gold sputter coater was used to coat a 1.0 mg of sample with Au, it was

then cleaned *via* a blower before it was moved into a chamber to refrain from contamination. Next, before the clean sample was put into the sample holder, a copper tape was placed on it. Lastly, the sample was then shifted to the FESEM specimen chamber where it was observed at an accelerating voltage of 15.0 kV.

Fig. 3. FESEM-EDS [14]

2.2.6 ICP-AES

The metal compositions in organic and inorganic materials were analyzed *via* a distinctive technology namely the PerkinElmer NexION 2000 B ICP Atomic Emission Spectrometer (ICP-AES) which is depicted in Figure 4. To attain a data of high accuracy, a standard preparation was done to acquire the calibration curve for each metal element. Organic compounds in the 1.0 g of sample were eliminated by dissolving a mixture of HCl and HF solution into the sample. Due to the sensitivity of ICP-AES which is up to 0.01 ppb, the sample was diluted in distilled water for 10 times. With a continuous injection of Ar, ICP-AES was performed under 26.0°C so as to obtain elemental composition data generated from the Single Particle ICP-AES Software [14].

Fig. 4. ICP-AES

2.2.7 Development of regression model in MATLAB

A regression model was developed *via* Simulink in the MATLAB R2019a software. Simulink was used for modelling, simulating and analyzing multi-domain dynamical systems. With the aid of the quadratic equation from the ANOVA model, a regression model was developed. In Simulink, there are various types of blocks available in the system such as math operation blocks, source blocks or even sink blocks which are drawn based on the elements required to run the simulation. For each block, parameters can be set according to our preference. Once all the parameters were set, the simulation was activated and the graphite yield obtained.

3. Results and Discussion

As mentioned earlier, this work was extended from a study conducted by Siaw *et al.,* [14]. The details from the previous study have been re-explained in this study so as to provide a better understanding for the readers. Here, the experimental results including ANOVA analysis were briefly explained to give an overview on how the ANOVA model was generated from the synthesis of graphite *via* HCl leaching. With the aid of the CCD design in RSM, twenty-seven experimental runs were carried out for the leaching process. The method used the four major factors to carry out these experimental runs and the factors were HCl concentration(M) (A), temperature (°C) (B), solid-to-liquid ratio (C) and leaching time (min) (D). The experimental factors in coded and actual units are portrayed in Table 3.

Table 3
Experimental factors in coded and actual units [14]

Exp. run	Independent factor in coded form				Independent factor in actual form				Metal removal (%)
	A	B	C	D	A (M)	B (°C)	C	D (min)	
1	+1	-1	+1	-1	6	30	30	60	60.60
2	+1	0	0	0	6	50	20	180	69.34
3	-1	-1	+1	+1	2	30	30	300	42.10
4	-1	-1	-1	+1	2	30	10	300	78.30
5	+1	-1	-1	-1	6	30	10	60	82.57
6	0	0	+1	0	4	50	30	180	49.30
7	0	0	0	0	4	50	20	180	60.70
8	+1	+1	-1	+1	6	70	10	300	86.94
9	+1	+1	-1	-1	6	70	10	60	88.20
10	0	+1	0	0	4	70	20	180	69.70
11	-1	+1	-1	-1	2	70	10	60	74.70
12	-1	-1	+1	-1	2	30	30	60	39.71
13	+1	+1	+1	-1	6	70	30	60	58.81
14	-1	0	0	0	2	50	20	180	56.48
15	0	-1	0	0	4	30	20	180	61.51
16	+1	-1	+1	+1	6	30	30	300	55.27
17	0	0	0	+1	4	50	20	300	61.32
18	0	0	0	0	4	50	20	180	60.30
19	-1	-1	-1	-1	2	30	10	60	61.40
20	0	0	0	0	4	50	20	180	57.70
21	0	0	0	-1	4	50	20	60	60.50

22	+1	+1	+1	+1	6	70	30	300	45.90
23	+1	-1	-1	+1	6	30	10	300	86.70
24	-1	+1	+1	+1	2	70	30	300	41.79
25	0	0	-1	0	4	50	10	180	76.27
26	-1	+1	-1	+1	2	70	10	300	77.90
27	-1	+1	+1	-1	2	70	30	60	44.50

Note: Coefficient terms are represented as A for HCl concentration, B for temperature, C for solid to liquid ratio and D for leaching time

Based on Siaw *et al.,* [14], ANOVA analysis to study the importance of each factor for the percentage of metal removal was applied. The F-value and p-value of 68.17 and <0.0001 respectively were significant with the leaching model. The R_2 value of 0.99 and Adj-R_2 value of 0.97 were used to prove the fitting of the model. From the results, the experimental data and the predicted value from CCD design of the model have portrayed good agreement between the two values.

3.1 Simulink Model Development

Based on the ANOVA model, Eq. (1) [14] which is shown below was attained and with that a Simulink model was developed. The model was used to understand the behavioural performance based on the four leaching factors, HCl concentration, temperature, solid to liquid ratio and leaching time. From the ANOVA model, the highest metal removal percentage, 88.20 % was acquired at the conditions of 6M (A), 70°C (B), solid to liquid ratio of 10:1 (C) and 60 mins (D). Therefore, these conditions are the optimum conditions to analyze the behavioural performance of the leaching factors,

$$Y = 62.22 + 6.63A + 0.69B - 15.44C + 0.041D - 0.19A^2 + 2.29B^2 - 0.19C^2 - 0.19D^2 - 1.67AB - 0.64AC - 2.24AD - 1.55BC - 1.32BD - 2.35CD \quad (1)$$

where, A is the HCl concentration, B is the temperature, C is the solid to liquid ratio, D is the leaching time and Y represents the metal removal percentage.

The Simulink model is portrayed in Figure 5 to 8. In these figures, the step block was used to introduce disturbance in which a step was provided between two definable levels at a specified time, 60 mins whereby only one of the inputs were swapped at a time while the rest remained unchanged.

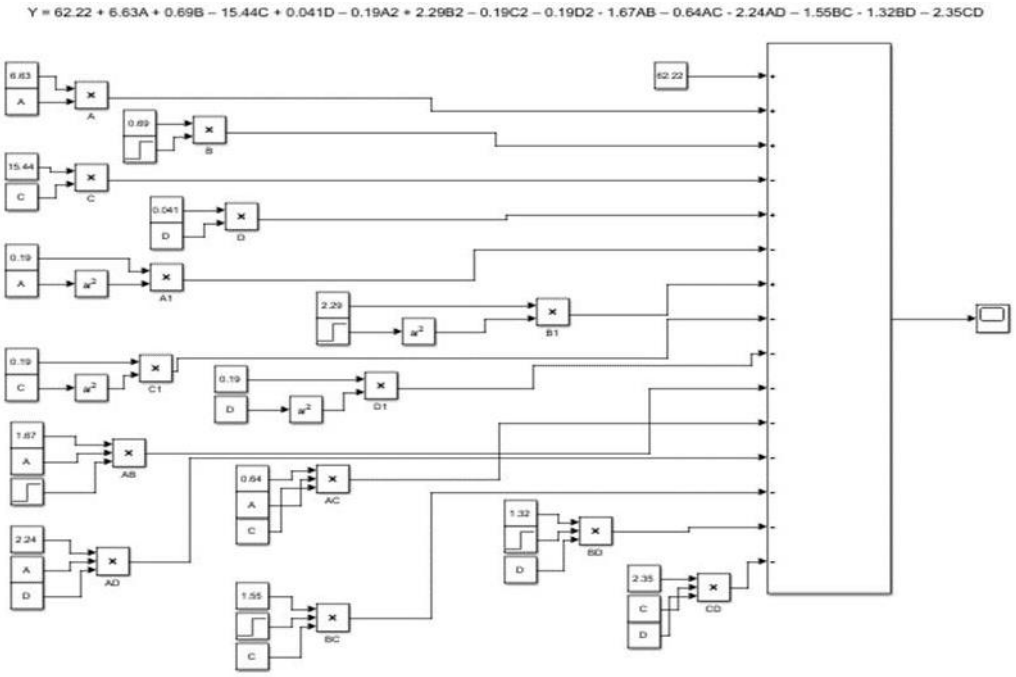

Fig. 5. Simulink model for variable A

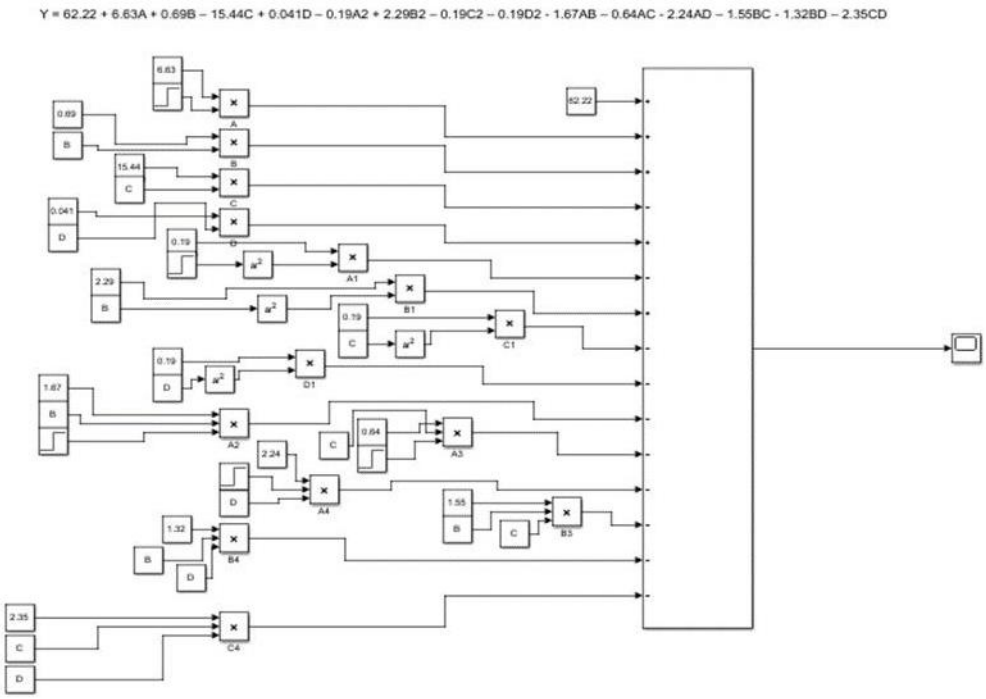

Fig. 6. Simulink model for variable B

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Y = 62.22 + 6.63A + 0.69B – 15.44C + 0.041D – 0.19A2 + 2.29B2 – 0.19C2 – 0.19D2 - 1.67AB – 0.64AC - 2.24AD – 1.55BC - 1.32BD – 2.35CD

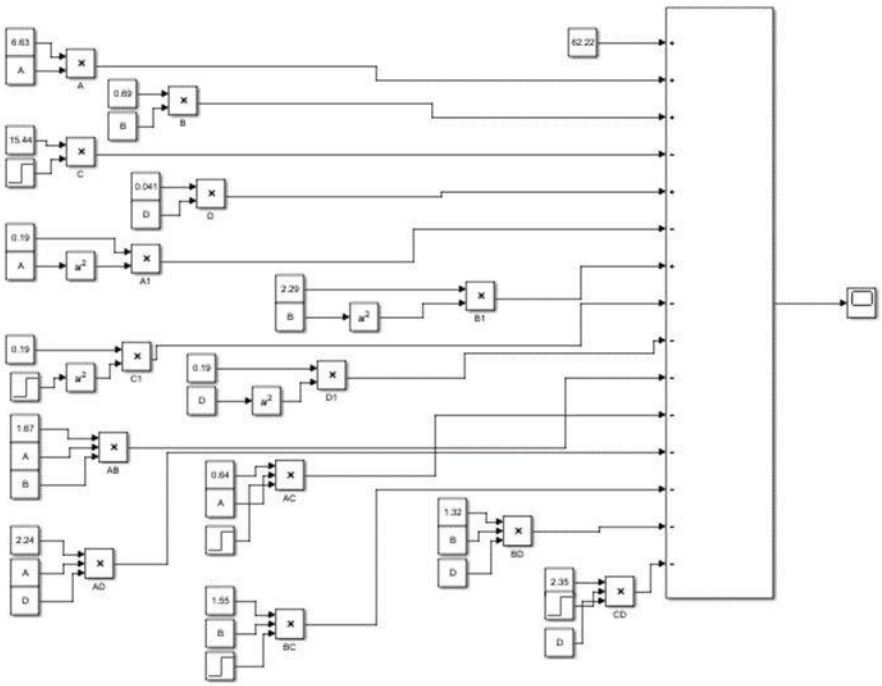

Fig. 7. Simulink model for variable C

Y = 62.22 + 6.63A + 0.69B – 15.44C + 0.041D – 0.19A2 + 2.29B2 – 0.19C2 – 0.19D2 - 1.67AB – 0.64AC - 2.24AD – 1.55BC - 1.32BD – 2.35CD

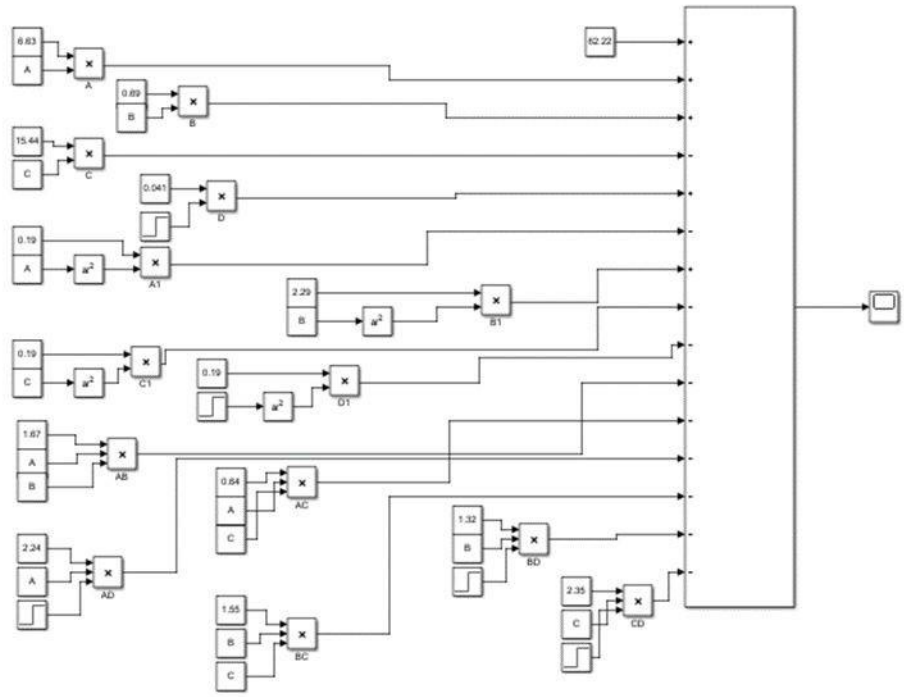

Fig. 8. Simulink model for variable D

3.2 Step Changes in HCl Concentration

The performance of the HCl concentration at a range of 2M to 6M was illustrated *via* conducting independent step tests with respect to the metal removal percentage with the

assumption of the temperature (B), solid to liquid ratio (C) and the time taken (D) were at a constant value at 70°C, 10:1 and 60 mins respectively. From Figure 9 and 10 which are the positive and negative step changes of variable A, we can note that there are no visible changes in step in both of the figures and the metal removal percentage was constant at 100 % from a concentration of 2M to 6M and *vice versa*. Generally, the increase in acid concentration aids in the anion generation and these anions are essential in order to form soluble compounds as they dissolve the metal ions. This then increases the metal removal efficiency. However, in this simulation, no change was shown as the high temperature at 70°C could have affected the results. The temperature does influence the leaching rate. Therefore, we can deduce that the concentration of the HCl acid does not affect the metal removal percentage.

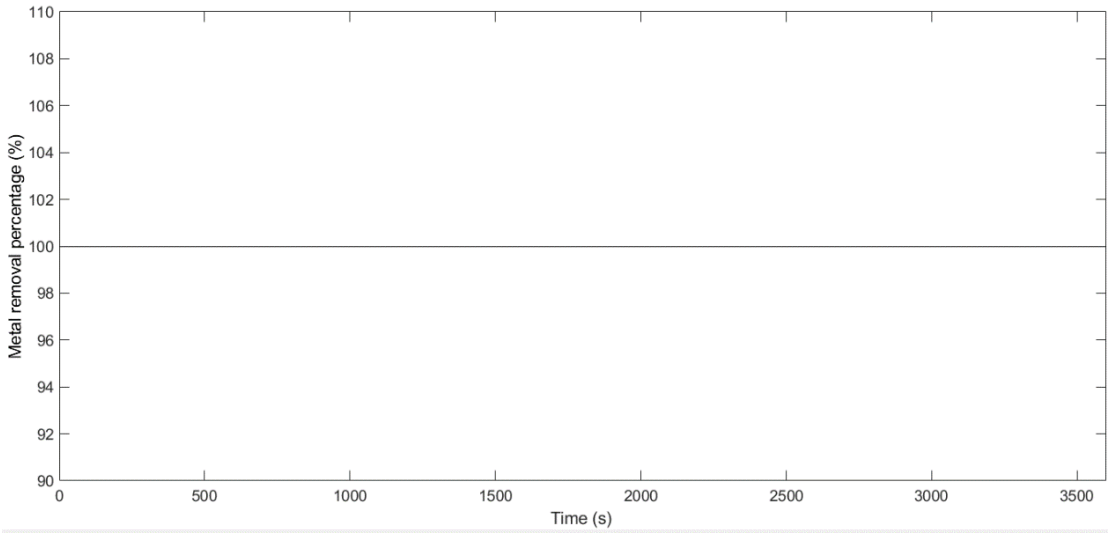

Fig. 9. Positive step change for variable A

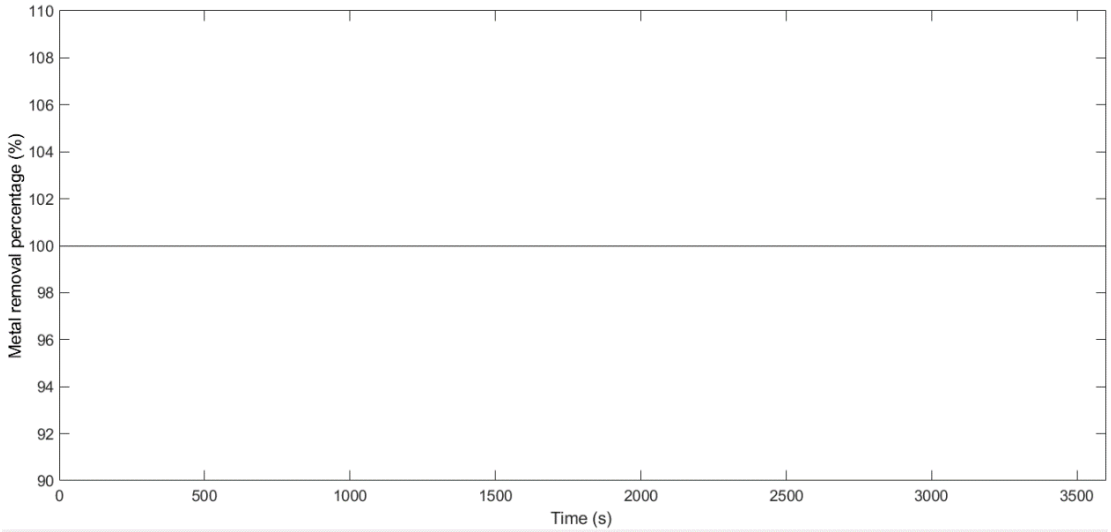

Fig. 10. Negative step change for variable A

3.3 Step Changes in Temperature

The analysis of the temperature performance was carried out to determine the effect of temperature on the metal removal percentage. Figure 11 depicts the positive step change of temperature ranging from 30 - 70℃ in which an increase in step is shown from 0 - 100 %. As for Figure

12, the negative step change, the metal removal percentage declined from 100 % to 0 from 70 - 30°C. The rate of reaction of the leaching process increases with the inclination in temperature thus increasing the leaching efficiency. This shows that the leaching process is preferred to be done at a higher temperature compared to a lower temperature as there was a significant difference in percentage of metal removal at a temperature of 30°C and 70°C.

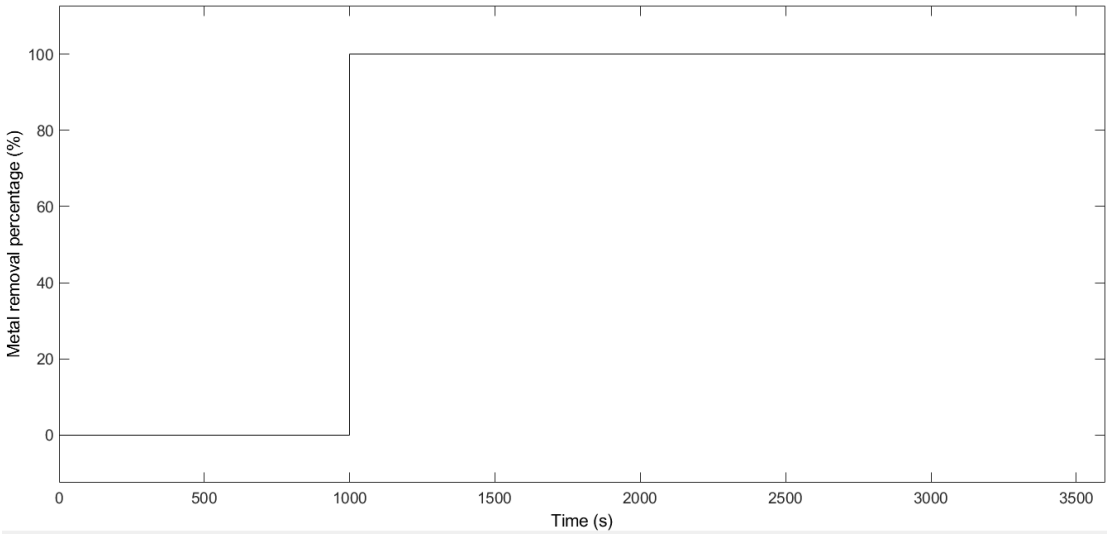

Fig. 11. Positive step change for variable B

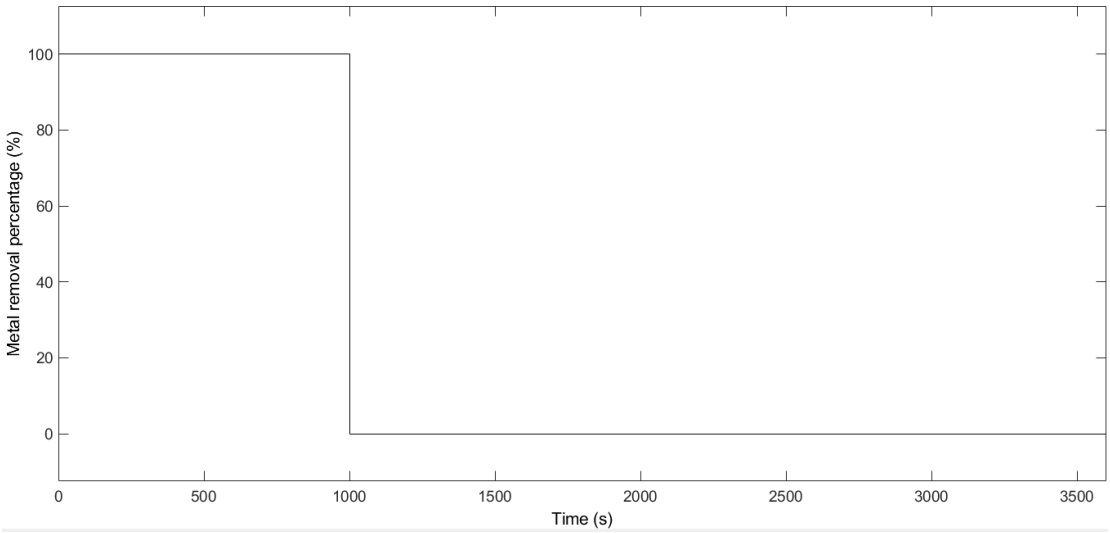

Fig. 12. Negative step change for variable B

3.4 Step Changes in Solid to Liquid Ratio

Solid to liquid ratio plays an important role in the leaching process. Hence, the percentage of metal removal is affected by the solid to liquid ratio. The positive step change is portrayed in Figure 13 whereby the percentage of metal removal decreases from 100 % to 0 at a ratio ranging from 10:1 to 30:1 whereas the opposite is shown in the negative step change which is displayed in Figure 14. So, an increase in the metal removal percentage can be seen when the solid to liquid ratio is reduced due to the decrement in the solution viscosity which led to an effective dispersion of the metal impurities. This effective dispersion is a result of the increase in the velocity of leaching which

then increases the leaching efficiency. Therefore, a solid to liquid ratio of 10:1 is best suited for the metal removal process in order to obtain a higher metal removal percentage.

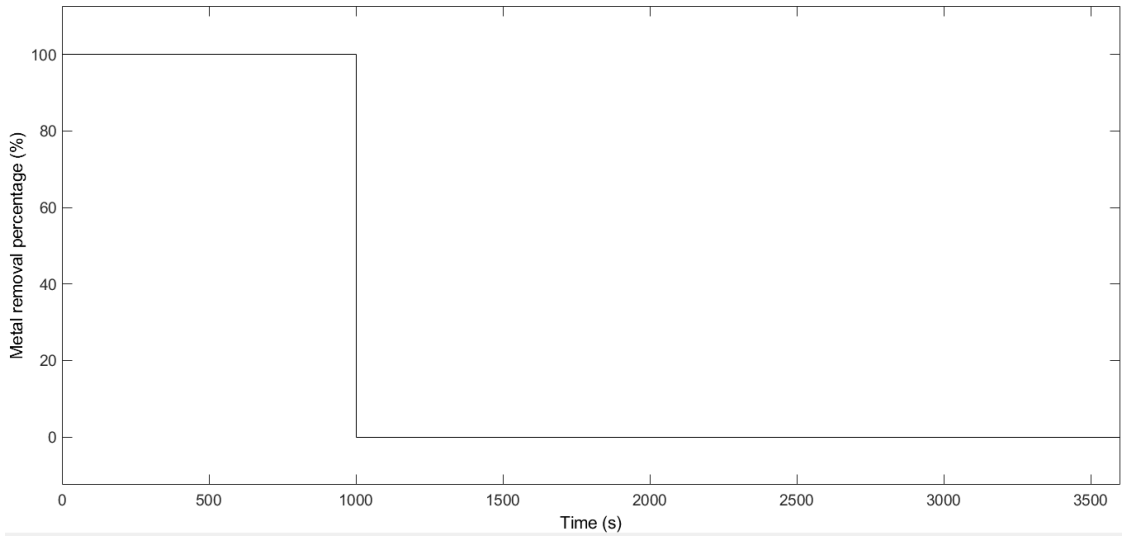

Fig. 13. Positive step change for variable C

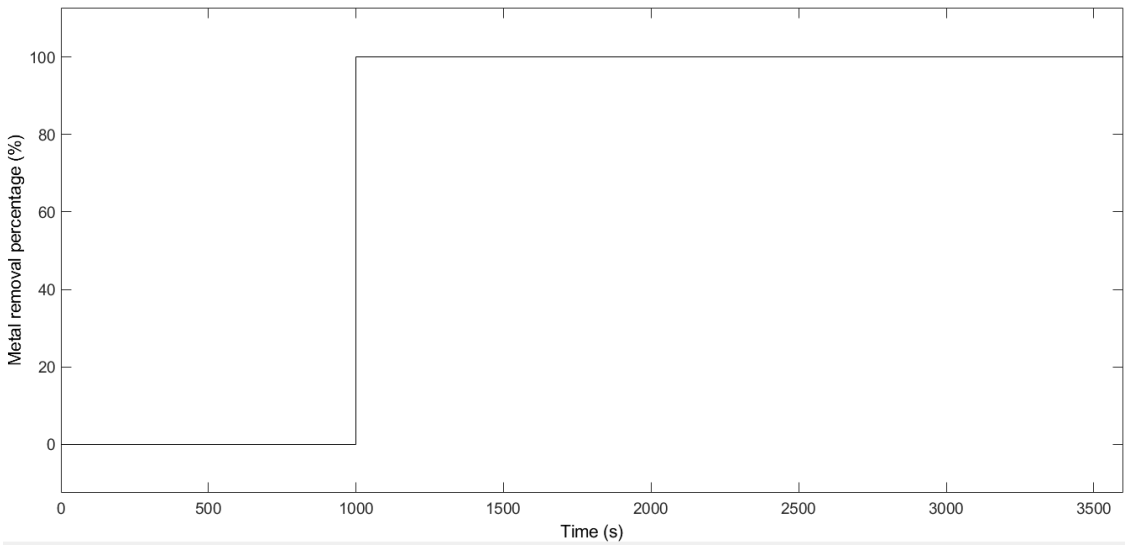

Fig. 14. Negative step change for variable C

3.5 Step Changes in Leaching Time

In the leaching process of graphite, the leaching time was taken into account as it is a crucial factor. On that account, the performance of leaching time was analyzed so as to increase the metal removal percentage. The leaching time ranged from 60 – 300 mins was input into the positive and negative step change function whereby for the positive step change in Figure 15, a decline in the percentage of metal removal was noted and for the negative step change in Figure 16, an incline in the percentage was shown. Hence, the best metal removal percentage can be attained at 60 mins.

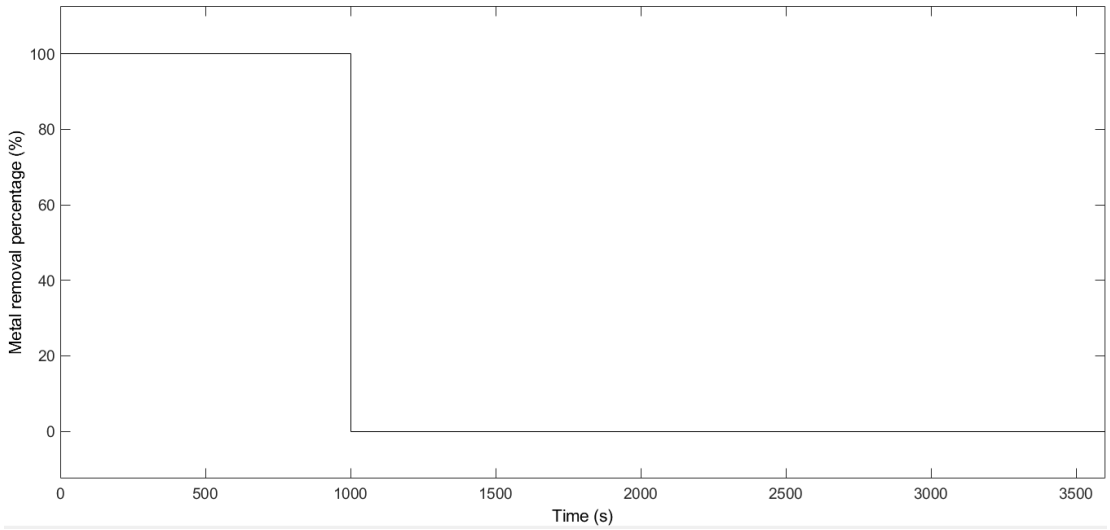

Fig. 15. Positive step change for variable D

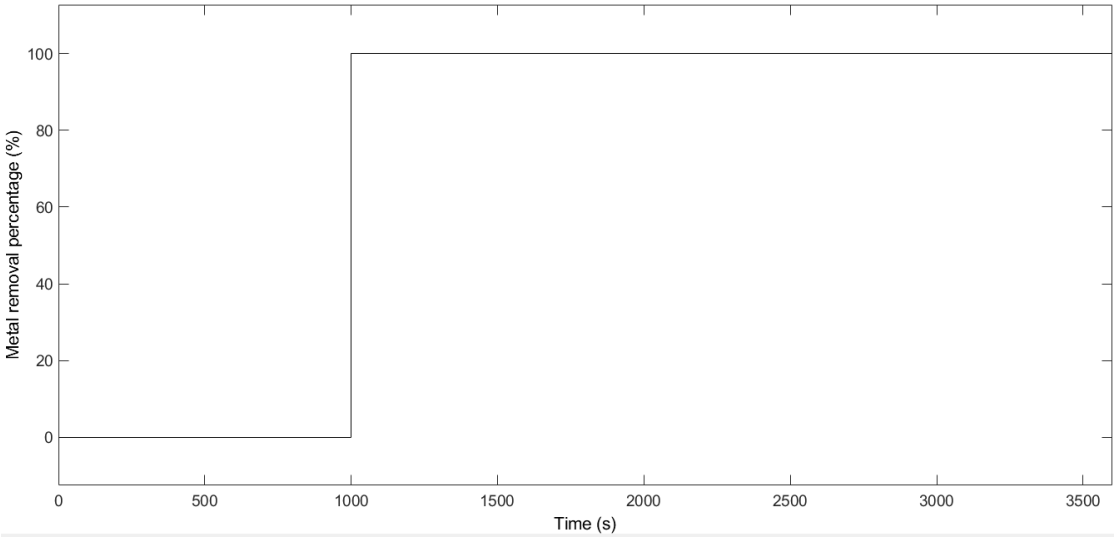

Fig. 16. Negative step change for variable D

4. Conclusions

In this paper, the acid leaching process used to synthesize graphite from industrial waste was studied whereby a simulation model was developed in MATLAB to provide a better understanding on the factors that affect the leaching process. These factors namely the HCl concentration, temperature, solid to liquid ratio and leaching time were analyzed based on their performance thus providing a preliminary understanding on the large scaled production of graphite. The simulation model in MATLAB was built based on the ANOVA model from a previous study [14] and from this study, the optimum parameters were attained.

From the findings, variable A, the HCl concentration ranging from 2M to 6M showed no changes in the metal removal percentage for both positive and negative step changes. This was due to the temperature selection which was kept constant at 70℃ as the temperature affects the leaching efficiency. Variable B, the temperature, portrayed at metal removal percentage of 100 % at 70℃ whereas at 30℃ the metal removal percentage was 0 thus explaining the fact that the higher the temperature, the higher the metal removal percentage. Next, at a solid to liquid of 10:1, variable C portrayed a metal removal percentage of 100 % because the smaller the particle size, the higher

the velocity of leaching. Hence, more metal was able to be removed at a ratio of 10:1 compared to 30:1. Lastly, the percentage of metal removal was the highest at 60 mins whereby a percentage of 100 % was attained in comparison to the leaching time of 300 mins whereby the percentage of metal removal was 0.

References

[1] Materials, S. (2015) 'Properties and Characteristics of Graphite for Industrial Application', Industrial production intro, (January), pp. 13.1-13.2.
[2] Novoselov, Konstantin S., Luca Colombo, P. R. Gellert, M. G. Schwab, and K. A. J. N. Kim. "A roadmap for graphene." *nature* 490, no. 7419 (2012): 192-200. https://doi.org/10.1038/nature11458
[3] Sadasivuni, Kishor Kumar, Deepalekshmi Ponnamma, Sabu Thomas, and Yves Grohens. "Evolution from graphite to graphene elastomer composites." *Progress in Polymer Science* 39, no. 4 (2014): 749-780. https://doi.org/10.1016/j.progpolymsci.2013.08.003
[4] Dickson, James Sean. "Talga eyes graphene potential." *Industrial minerals* 567 (2014): 19.
[5] Kim, Na Rae, Hong Joo An, Young Soo Yun, and Hyoung-Joon Jin. "Li-ion battery anodes from ginkgo leaf-derived nanoporous carbons rich in redox-active heteroatoms." *Carbon letters* 22 (2017): 110-114.
[6] Evarts, Eric C. "Lithium batteries: To the limits of lithium." *Nature* 526, no. 7575 (2015): S93-S95. https://doi.org/10.1038/526S93a
[7] Bhushan, Bhan, and H. N. Roy. "A Note on Properties and Uses of Graphite." *Transactions of the Indian Ceramic Society* 14, no. 1 (1955): 11-15. https://doi.org/10.1080/0371750X.1955.10877668
[8] Robinson Jr, Gilpin R., Jane M. Hammarstrom, and Donald W. Olson. *Graphite*. No. 1802-J. US Geological Survey, 2017. https://doi.org/10.3133/pp1802J
[9] Kamal, Afiqah Samsul, Noor Hafidzah Jabarullah, and Rapidah Othman. "Catalytic graphitization of Oil Palm Frond using iron and silica." *Materials Today: Proceedings* 31 (2020): 211-216. https://doi.org/10.1016/j.matpr.2020.05.138
[10] Nasir, Salisu, Mohd Zobir Hussein, Zulkarnain Zainal, and Nor Azah Yusof. "Carbon-based nanomaterials/allotropes: A glimpse of their synthesis, properties and some applications." *Materials* 11, no. 2 (2018): 295. https://doi.org/10.3390/ma11020295
[11] Ko, Seunghyun, Yeon Ju Kwon, Jea Uk Lee, and Young-Pyo Jeon. "Preparation of synthetic graphite from waste PET plastic." *Journal of Industrial and Engineering Chemistry* 83 (2020): 449-458. https://doi.org/10.1016/j.jiec.2019.12.018
[12] Mitchell, C. J. "Industrial minerals laboratory manual: flake graphite." (1992).
[13] Keeling, John. "Graphite: properties, uses and South Australian resources." *MESA J* 84, no. 3 (2017): 28-41.
[14] Siaw, W. C., MR Musram Rakunman, N. Kamaruzaman, T. Tsuji, and N. Abdul Manaf. "Metal Removal from Industrial Waste by Hydrochloric Acid." In *IOP Conference Series: Materials Science and Engineering*, vol. 808, no. 1, p. 012008. IOP Publishing, 2020. https://doi.org/10.1088/1757-899X/808/1/012008
[15] Golpayegani, Mohammad Hasan, and Ali Akbar Abdollahzadeh. "Optimization of operating parameters and kinetics for chloride leaching of lead from melting furnace slag." *Transactions of Nonferrous Metals Society of China* 27, no. 12 (2017): 2704-2714. https://doi.org/10.1016/S1003-6326(17)60299-1
[16] Qiu, Tingsheng, Huashan Yan, Jifu Li, Qingsheng Liu, and Guanghua Ai. "Response surface method for optimization of leaching of a low-grade ionic rare earth ore." *Powder technology* 330 (2018): 330-338. https://doi.org/10.1016/j.powtec.2018.02.044
[17] Liu, Hong, Yi-min Zhang, Jing Huang, Tao Liu, Nan-nan Xue, and Qi-hua Shi. "Optimization of vanadium (IV) extraction from stone coal leaching solution by emulsion liquid membrane using response surface methodology." *Chemical Engineering Research and Design* 123 (2017): 111-119. https://doi.org/10.1016/j.cherd.2017.05.001
[18] Bohlouli, A., M. Reza Afshar, M. R. Aboutalebi, and S. H. Seyedein. "Optimization of tungsten leaching from low manganese wolframite concentrate using Response Surface Methodology (RSM)." *International Journal of Refractory Metals and Hard Materials* 61 (2016): 107-114. https://doi.org/10.1016/j.ijrmhm.2016.07.012
[19] Salehi, Soghra, Mohammad Noaparast, and Seyyed Ziaodin Shafaei. "Response surface methodology (RSM) for optimization of chalcopyrite concentrate leaching with silver-coated pyrite." *Physicochemical Problems of Mineral Processing* 52, no. 2 (2016): 1023-1035.