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CARBON DIOXIDE SEQUESTRATION BY MINERAL CARBONATION USING ELECTRIC ARC FURNACE SLAG

OMALE SUNDAY OGAKWU

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By

OMALE SUNDAY OGAKWU

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Doctor of Philosophy

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DEDICATION

This thesis is dedicated to the Lord God Almighty whom I serve, and to my family



Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Doctor of Philosophy

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By

OMALE SUNDAY OGAKWU

June 2020

Chairman : Professor Thomas S.Y Choong, PhD Faculty : Engineering

Fossil fuel usage is the primary source of anthropogenic air pollution, where carbon dioxide (CO₂) is the most prominent agent that contributes to global climate change. The iron and steel industries are major contributors to gaseous CO₂ emission. These industries also produce solid wastes in the form of slags during operations. Steelmaking in electric arc furnace (EAF) generates between 10-15% slag wastes per ton of steel production, which can be used in mineral carbonation to capture and store CO₂. In this study, the EAF slag from an iron and steelmaking factory in Klang, Malaysia, was utilized for the CO₂ sequestration through both direct aqueous and indirect mineral carbonation method in a batch reactor. The direct aqueous carbonation investigation was at room temperature, and different solid/liquid ratio, pressure, and time. The indirect carbonation was performed, after the extraction of essential metallic ions from the EAF slag at a different temperature, solvent concentration, and solid/liquid ratio.

The direct aqueous mineral sequestration capacity was 58.36 ± 5.84 g CO₂/kg of slag under room temperature after 3 hr, solid/liquid ratio of 1:5, and using < 63 µm particle size. The sequestration efficiency was 28.11 %, and the degree of carbonation was 23.30 % at the pressure of 5 bars. The shrinking core model shows that the direct aqueous carbonation was by the ash layer product phase-controlled, with the regression coefficient (R²) of 0.97.

In the dissolution of essential metallic ions like Ca, Mg, and Fe, the slag from EAF was the source. The dissolution efficiency was affected by temperature, solvent concentration, solid/liquid ratio, and reaction time. At the temperature of 75 °C, Ca ion was extracted from the slag with 86.46 % efficiency and Mg ion of 30.13 % after 1 hr of using 0.22 M HCl. However, the dissolution in the solid/liquid ratio of 10 g/l

was higher than 20 g/l and 30 g/l. The dissolution of Ca ion was 80.27 %, 61.33 %, and 50.53 %, and that of Mg ion was 26.63 %, 24.20 %, and 22.16 %, respectively, after 1 hr in 0.22 M HCl. The acid concentration of 0.44 M extracts more Ca, Mg and Fe ions than 0.33 M and 0.22 M. The efficiency after 1 hr.; at 35°C from 20 g/l was 68.84 %, 65.63 %, 61.33 % for Ca, and 27.52 %, 26.40 %, 24.20 % of Mg, while that of Fe from 20 g/l was 10.09 %, 8.76 %, and 5.18 % respectively.

Meanwhile, in the indirect carbonation, the dissolved Ca ion was used for the formation of calcium carbonate through CO_2 sequestration. The formed precipitate calcium carbonate (PCC) of 98.61 ± 1 % purity, and the sequestration capacity of 0.4105 ± 0.195 kg of CO_2 /kg of CaCO₃ within 1 hr was achieved. This shows that at moderate conditions (0.22 M HCl, 35°C, and 1 hr), both CO₂ sequestration and calcium carbonate of high purity were realized.

In a reaction of a heterogeneous solid-liquid mixture, the modified shrinking core model was appropriate. The modified shrinking core model best interpreted the kinetics behavior for all the parameters studied in the dissolution of Ca ion from the EAF slag. From the regression coefficient (\mathbb{R}^2), the dissolution was controlled by the product layer phase.

The order of reaction for acid concentration and S/L ratio was 0.31419 and -1.02459 respectively. The activation energy of the process over the temperature range was, calculated to be $E_a = 3.881 KJ/mol$. From the results of the two sequestration methods, the indirect route was better with a higher sequestration capacity and calcium carbonate of high purity. The EAF slag demonstrated the potential and available material for both CO₂ sequestration and economic purposes, instead of being landfilled.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PENGASINGAN KARBON DIOKSIDA MELALUI KARBONISASI MINERAL MENGGUNAKAN RELAU PEMBAKARAN ELEKTRIK TERAK

Oleh

OMALE SUNDAY OGAKWU

Jun 2020

Pengerusi : Profesor Thomas S.Y Choong, PhD Fakulti : Kejuruteraan

Penggunaan bahan api fosil adalah sumber utama pencemaran udara anthrapogenic, di mana karbon dioksida (CO₂) merupakan agen yang paling terkenal yang menyumbang kepada perubahan iklim global. Industri besi dan keluli adalah penyumbang utama kepada pengeluaran gas CO₂. Industri-industri ini juga menghasilkan sisa pepejal dalam bentuk terak semasa operasi. Penghasilan keluli di dalam relau pembakaran elektrik (EAF) menjana antara 10-15 % sisa terak setiap tan proses pengeluaran keluli yang boleh digunakan dalam karbonisasi mineral untuk menangkap dan menyimpan CO₂. Dalam kajian ini, terak EAF dari kilang pembuatan besi dan keluli di Klang, Malaysia telah digunakan untuk pengasingan CO₂ di dalam reactor kumpulan melalui karbonisasi mineral akueus secara langsung dan tidak langsung. karbonisasi mineral akueus secara langsung telah diperiksa pada suhu bilik dan nisbah pepejal/cecair yang berbeza,tekanan dan masa. Manakala karbonisasi mineral secara tidak langsung berlaku selepas pengekstrakan ion logam penting dari terak EAF pada suhu, kepekatan pelarut, dan nisbah pepejal / cecair yang berbeza.

Kapasiti pengasingan cecair mineral secara langsung didapati 58.36 ± 5.84 g CO₂ / kg terak di bawah suhu bilik selepas 3 jam, nisbah pepejal / cecair 1: 5, dan menggunakan saiz zarah <63 µm. Kecekapan pengasingan adalah 28.11 % dan tahap karbonisasi adalah 23.30 % pada tekanan 5 bar. Model teras penyusutan menunjukkan bahawa karbonisasi akueus secara langsung dikawal oleh fasa penghasilan lapisan abu. dengan pekali regresi (R2) 0.97

Sementara itu, dalam pembubaran terak dari EAF adalah sumber untuk ion logam penting seperti Ca, Mg, Fe dan lain-lain. Kecekapan pembubaran dipengaruhi oleh suhu dan kepekatan pelarut, nisbah pepejal/cecair dan juga masa tindakbalas. Pada suhu 75°C, hampir semua Ca ion telah diekstrak dari terak dengan kecekapan 86.46%

dan Mg sebanyak 30.13% selepas menggunakan 0.22M HCl selama 1 jam. Walau bagaimanapun, pembubaran oleh nisbah pepejal/cecair 10g/L adalah lebih tinggi daripada 20g/L dan 30g/L. Pembubaran Ca ion adalah 80.27 %, 61.33 %, dan 50.53 %, manakala Mg ion adalah 26.63 %, 24.20 %, dan 22.16 %, mengikut turutan nisbah pepejal/cecair masing-masing selepas menggunakan 0.22M HCl selama 1 jam . Kepekatan asid 0.44 M telah mengekstrak lebih banyak Ca, Mg and Fe ion daripada kepekatan asid pada 0.33 M dan 0.22 M. Kecekapan selepas 1 jam pada suhu 35°C pada 20g/l adalah 68.84 %, 65.63 %, 61.33 % untuk Ca ion, dan 27.52 %, 26.40 %, 24.20 % untuk Mg ion, manakala untuk Fe ion pada 20g/L nisbah pepejal/cecair adalah sebanyak 10.09 %, 8.76 %, dan 5.18 % mengikut turutan kepekatan asid masing-masing.

Sementara itu, dalam karbonisasi mineral secara tidak langsung Ion Ca terlarut digunakan untuk pembentukan kalsium karbonat melalui pengasingan CO₂. Pembentukan mendapan kalsium karbonat (PCC) adalah 98.61 \pm 1% tulen dan pengasingan berkapasiti 0.4105 \pm 0.195 kg CO₂/kg kalsium karbonat dalam masa satu jam telah dicapai. Ini menunjukkan bahawa pada keadaan yang sederhana (0.22 M HCl, 35°C, and 1 hr) kedua2 pengasingan CO₂ dan kalsium karbonat ketulenan yang tinggi telah direalisasikan.

Dalam tindak balas campuran pepejal/cecair heterogen, model teras penyusutan yang diubah suai adalah sesuai. Model teras penyusutan adalah terbaik untuk menaksirkan tingkah laku kinetik untuk semua parameter yang dikaji dalam pembubaran ion Ca dari terak EAF. Dari pekali regresi (R^2), pembubaran ion logam dikawal oleh fasa lapisan produk. Turutan tindak balas untuk kepekatan asid dan nisbah pepejal/cecair masing-masing adalah 0.31419 and -1.02459. Pengaktifan tenaga untuk proses ini dalam julat suhu dikira sebanyak Ea= 3.881 KJ/mol. Daripada hasil kedua-dua kaedah pengasingan kaedah tidak langsung lebih baik dengan kapasiti pengasingan yang lebih tinggi dan kalsium karbonat dengan ketulenan yang tinggi, Terak EAF telah menunjukkan ianya bahan yang berpotensi dan senang digunakan untuk kedua-dua pengasingan CO₂ dan untuk tujuan ekonomi bukannya untuk pelupusan.

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Declaration by graduate student

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Signature: Name of Chairman of Supervisory Committee:	Professor Dr. Thomas S.Y. Choong	
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Signature: Name of Member of Supervisory Committee:	Dr. Shamsul Izhar Siajam,	
Signature: Name of Member of Supervisory Committee:	Associate Professor Dr. Yip Mun Wai	

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LIST OF ABBREVIATIONS

BHD	Baghouse dust
BOF	Basic oxygen furnace
B.F.	Blast furnace
BET	Brunauer-Emmet-Teller
CO_2	Carbon Dioxide
CuO	Copper Oxide
CCS	Carbon capture and storage
EDX	Energy Dispersive X-ray Spectroscopy
EAF	Electric arc furnace
EAFS	Electric arc furnace slag
FTIR	Fourier Transform Infrared Spectroscopy
GHG	Greenhouse Gas
H.M.	Hydrothermal method
ICP-OES	Inductively coupled plasma-optical emission spectrometry
L.F.	Ladle furnace
M.W.	Molecular weight
PCC	Precipitated calcium carbonate
SEM	Scanning electron microscope
SCM	Shrinking core model
TGA	Thermogravimetric Analysis
XRD	X-Ray Diffraction

CHAPTER 1

INTRODUCTION

1.1 Research Background

Carbon is essential to a living organism, but the ability to control the large release of carbon dioxide is a major challenge that needs urgent attention (Styring et al., 2011). A large amount of flue gas, containing a substantial amount of carbon dioxide, comes out of the smokestacks of industrial plants in power, iron and steel industries, cement production, oil refineries, biogas sweetening, and chemical sectors (Gassnova, 2013). The CO₂ released from these industrial plants is a significant contributor to global warming and climate change (Styring et al., 2011; Mun and Cho, 2013). The carbon dioxide (CO₂) concentration in the atmosphere had increased from about 277 ppm in 1750 when the industrial era began, to more than 400 ppm (Quéré et al., 2013; Ukwattage et al., 2017). It was, projected that the emission of CO₂ needs to be reduced from its present concentration to maintain the worldwide temperature to less than 2°C in years ahead (Rosa et al., 2015).

The possible means for CO_2 reduction are as follows: (i) decreasing carbon usage; (ii) reducing the amount of CO_2 produced; (iii) switching to less carbon-intensive fuels (iv) increasing the use of renewable energy; and (v) capturing and storing CO_2 (Mikkelsen et al., 2010; Yang et al., 2008). In light of this, the fifth approach, which includes developing a new and less costly method of carbon capture and storage (CCS) techniques, will be of interest. The burning of fossil fuels will continue until the use of new carbonless sources of energy is developed (Voutsinos, 2018). However, in recent times, CCS technology with "utilization" has become an attractive choice for reducing CO_2 emissions globally (Pan et al., 2012).

Some of the steps taken to help toward getting to this target for mitigating CO_2 are the introduction of carbon capturing and storage (CCS), using alkaline wastes from the industries instead of the geological, oceanic, and natural methods. Therefore, the need to use solid wastes that have CaO and MgO chemical composition from these industries that also produce the greenhouse gas (CO₂) has become attractive to used solid wastes to mitigate the excess gaseous waste.

The carbon dioxide utilization to form carbonates is part of the accelerated carbonation technology. The primary objective of carbon accelerated technique is to imitate the natural weathering process, in which metal oxide reacts with CO₂ to form stable and insoluble carbonates. Calcium or magnesium oxide is the most favorable metal oxide material for this reaction (Revathy et al., 2016).

The mineral carbonation from the metallic oxide of calcium and magnesium is as follows in Equation (1.1) and (1.2)

$$(Ca, Mg)O + CO_2 \to (Ca, Mg)CO_3 \tag{1.1}$$

The aqueous carbonation can be shown as in equation (1.2)

$$CO_2 + H_2O \rightarrow H_2CO_3 \rightarrow H^+ + HCO_3^- \rightarrow (Ca, Mg)^{2+} + HCO_3^- \rightarrow$$

 $(Ca, Mg)CO_3 + H^+$

(1.2)

Industrial residues like combustion residues, steelmaking slags, waste concrete, fly ashes, etc. are alkaline and appear to be raw materials possibly for CO₂ sequestration, because these materials are generally rich in metal oxides (Pan et al., 2012). The possible advantages of accelerated carbonation of applied industrial alkaline solid wastes comprise: (a) its inherent properties may reduce energy consumption and cost because its carbonation is exothermic in reaction; (b) high availability of deposits offers its great sequestration capacity; (c) in the absence of acidification, carbonation products like calcium or magnesium carbonates are thermodynamically stable under ambient conditions; (d) since transportation is not, required at sites within steel-works, it makes it cost-effective; (e) products may be reused as materials in a variety of application, like in Construction, and (f) it could neutralize the pH of the solution as carbonate precipitations are, formed if alkaline wastewater is, used as the liquid agents (Huijgen et al., 2005; Lim et al., 2010; Pan et al., 2012).

Mineral sequestration, which is also called mineral carbonation, is used for the option of storage into permanency (Bobicki, 2012).

Through careful control of greenhouse gas (GHG) emissions, the industry is moving toward environmental sustainability and the proper management of steel manufacturing residues products. With this background, a combined method aimed at capturing carbon dioxide (CO₂) with improving the environmental and mechanical properties of steelmaking slags will be highly needful (Pan et al., 2016). Hence, carbon dioxide (CO₂) capture by carbonating the steelmaking slags may perhaps be an interesting alternative for reducing CO₂ emissions from the steel plant. Assessing the CO₂ capture potential of these materials by accelerated carbonation of steelmaking slags have been progressively tested in recent times (Bonenfant et al., 2009; Chang et al., 2015; Cho et al., 2011; Shao et al., 2010)



1.2 Hypothesis

The huge emission of anthropogenic greenhouse gas (GHG)s, especially CO_2 , has an impact on global climate change. And it is a major challenge to human health and the ecosystems. Therefore, the need to reduce this gas is a serious concern and a pledge by world leaders to effect (Bobicki et al., 2012).

The use of fossil fuels as the main energy source in the world will be there for a longer time because of its dominance (Pan et al., 2014).

It is therefore agreed by many that a meaningful reduction in GHG by especially capturing and storing the CO₂ gas will help. However, the sequestration of carbon dioxide in solid form at a low cost for either commercial quantity for economic purpose or be disposed of is appealing. The whole target is to reduce the volume of CO₂ emission to between 50 - 85 %, thereby minimizing the increase in global temperature to 2°C (Olajire, 2013).

Mineral carbonation of CO_2 is an attractive method of capturing carbon dioxide to mitigate climate change (Chang et al., 2011; Eloneva et al., 2012). The presence of calcium/magnesium in raw material can store CO_2 as Ca/MgCO₃ (Teir et al., 2007).

Waste materials from industries, such as iron and steelmaking slags, are by-product for CO₂ reduction through mineral carbonation (Eloneva et al., 2008).

Both direct aqueous carbonation and indirect carbonation are methods of CO_2 sequestration (Jo et al., 2017; Revathy et al., 2016).

1.3 Problem Statement

Reducing industrial wastes disposal is a concern to environmental regulators across countries of the world (Meyer, 2009). Therefore, encouraging and mandating the reuse of these wastes is important (Meyer, 2009; Pan et al., 2016) because these nations are facing a big challenge from some of these waste materials or by-products.

Enhancing the quality and encouraging citizens to live in a healthy environment, which thus improved the economic and social life of the people. It is sustainable development for both the present and generations to come (Meyer, 2009).

In this case, the raw materials transportation, extraction, processing, and transformation with the maintenance and use of the product to either finally eliminate or recycle it must be, giving consideration (Meyer, 2009). For steelmaking, among the

objective of sustaining steelmaking is to minimize both hazardous waste and materials used to landfill (Meyer, 2009; Pan et al., 2016).

Steel slag utilization in another industry as a raw material has a prospect in the environmental feature, because the recovery or reuse of the slag offers related benefits to the environment (Pan et al., 2016; Yuksel, 2016)

Worldwide production of steelmaking slag is about 50 million tons annually. It also depends on the steel composition and the process involved (I. Altuna & Yılmazb, 2002; Pan et al., 2016). About 120–150 kg of slag is produced per ton of steel from the processes of basic oxygen furnace (BOF) and electric arc furnace (EAF). Slags comprise of recoverable and reusable essential metals and are generally rich in Ca compound (Chang et al., 2011; Yuksel, 2016)

Some advanced countries are recycling some percentages of the steelmaking slag while high portions are being discarded directly in many countries.

In 2012, the European Slag Association (EUROSLAG) members steel slag (S.S) product was almost 24.7 million tons. With BOF slag production of 46 %, while EAF slag was 38% and secondary (S.S) 17%. Meanwhile, 24.7 million tons of steel slag was reused by EUROSLAG members, out of which 43% was in 2012 for road construction. At the end of 2009, nearly 5 million tons of slag are produced annually. Still, 87 % of steel slag is being stored by the iron and steel industry within the plants (Yuksel, 2016).

Now, in Europe, this slag is being reused in road construction, interim storage, internal recycling, fertilizer, hydraulic engineering, and cement production 45%, 17%, 14%, 3%, 3%, and 3% respectively. With these whole recycling methods in place, still, more of the slag is dumped to landfills otherwise stockpiled at steel plant for a long time. There are heavy metals present in the slag, and this could be hazardous. So, using it for landfilling or recycling will need the slag metal content to be reduced by further retreatment. (Hucheng et al., 2014)

Based on slag to steel output ratios, the worldwide yearly steel slag output was about 170–250 million tons in 2014 (Yuksel, 2016). It makes it a major environmental challenge for countries and the world at large (Yuksel, 2016). The harmful effects on the environment from the steelmaking industry can be minimized by developing technology that is environmentally friendly (Yuksel, 2016).

Bankole et al. (2011) reported that Malaysia, with five major steelmaking industries, would be producing a lot of slag wastes as a by-product. As a result of these enormous slags product and their environmental impact, it has become necessary to find more ways of utilization of the slag to minimize its negative effect on the environment and to salvage the natural resources. Malaysia has more than 100 steel manufacturing

industries, with five major processing and producing factories, i.e., Megasteel, Amsteel, Ann Joo Steel, Southern Steel, and Antara Steel (Monitor, 2018). Most of the produced slag as a by-product is landfilled (Bankole et al., 2011).

It necessitates the calls for further studies to see other additional and effective ways to use these slag wastes. Mineral carbonation for the mitigation of CO_2 is an interesting option (Styring et al., 2011). Both direct aqueous and indirect carbonation have yet to be studied for steel slags produced in Malaysia. Therefore, there is a need to investigate factors that affect the performance of mineral carbonation. It is also important to identify the rate-controlling step in the mineral carbonation, involving the fluid-solid reaction of a heterogeneous phase (Ajemba, Regina O and Onukwuli, 2012; Jo et al., 2017; Teir et al., 2007).

1.4 Research Objectives

The objective of this work, is the sequestration of carbon dioxide through mineral carbonation, using the EAF slag from iron and steel industry in Klang, Malaysia.

The sequestration is through direct aqueous carbonation and indirect mineral carbonation.

The specific objectives are: -.

- 1. To examine the physio-chemical properties of the EAF slag particles sizes of $<63\mu m$, before and after carbonation. Also, the characterization of the slag residue will be performed.
- 2. To evaluate the performance of the slag in CO_2 sequestration through
 - (i) Direct aqueous mineral carbonation and
 - (ii) To examine the physio-chemical properties such as BET, FTIR, EDX, SEM, XRD, and TGA of the carbonated slag.
 - (iii) To evaluate the effects of solid/liquid ratio, pressure, and time on direct aqueous carbonation to ascertain the extent of sequestrated CO₂.
 - (iv) To investigate the kinetics of the reaction rate mechanism on the direct aqueous mineral carbonation using the (SCM).
- 3. To evaluate:
 - (i) The influences of temperature, solvent (HCl) acid concentration, solid/liquid ratio, and time on Ca, Mg, and Fe extraction from the slag through ICP-OES analysis.
 - (ii) And examine the physio-chemical properties such as BET, FTIR, EDX, SEM, XRD of the slag residue.
- 4. To evaluate the performance of the CO₂ sequestration through(i) Indirect mineral carbonation techniques

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BIODATA OF STUDENT

Omale, Sunday Ogakwu was born on 5th August 1963 in Abejukolo Ife, Omala Local Government of Kogi State, Nigeria. He attended Kaduna Polytechnic, Kaduna State, Nigeria. He graduated with Higher National Diploma (HND) in Chemical Engineering in 1992, and He obtained a Master of Technology (M.Tech.) degree in Chemical Engineering in 2010 from Rivers State University of Science and Technology (RSUST), Rivers State, Nigeria. He joined Universiti Putra Malaysia in 2016 to pursue a Ph.D. in Chemical Engineering. He is presently a lecturer at the Department of Chemical Engineering, Kaduna Polytechnic, Kaduna, Kaduna state, Nigeria. He is a registered member of the Council for the Regulation of Engineering in Nigeria (COREN) and the Nigerian Society of Chemical Engineers.

S.O. Omale research interest, is focused on recycling of slag and other solid wastes for recovery of Calcium, Magnesium and Iron using environmentally friendly lixiviates. The impact of his research interest will unlock the supply of Calcium, Magnesium and Iron. These are essential elements for human health and precipitated calcium carbonated (PCC) of high purity for the paper, iron and steel industries, focusing on green environmental future.

PUBLICATION

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