POTENTIAL OF MINE WASTE MATERIAL FOR MINERAL CARBONATION PROCESS IN CARBON CAPTURE AND UTILIZATION APPLICATION

By

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

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Mining activities may pose risks to its surrounding environment and population due to contaminant release and mine waste generation, while mining industry itself is considered as one of the carbon-extensive industries that contributes to the increasing carbon dioxide emission to the atmosphere. Carbon sequestration through mineral carbonation is one of the ways to mitigate these problems. Mineral carbonation can be explained by the reaction of carbon dioxide with silicate minerals forming stable carbonate. Mine waste has variety of potential minerals which can be utilized as the source of silicate minerals for carbonation. Therefore, this study focuses on the potential of the alkaline mining waste for carbon sequestration which involves the process of mineral carbonation. Throughout this study, characterization of the samples from three sites (i.e. gold mine, iron mine and limestone quarry) were performed and this includes particle size distribution (PSD) analysis, pH analysis, and chemical, mineralogical and morphological analyses. Mineral carbonation experiment was carried out under low pressure-temperature conditions using a designated stainless steel reactor. Results of pH analysis showed that most of the samples have an alkaline nature which shows its suitability for undergoing mineral carbonation reaction. Moreover, particle size distribution analysis for fine particles reveals the presence of large amounts of small-size particles (silt fraction) in gold mine waste which makes it suitable for this process. On the other hand, the iron mine waste consists of a large amount of large-sized particles (sand) indicating that pre-treatment needs to be done in order for the carbonation process to be optimized. Based on mineralogical analysis performed, all mine waste samples from the three mining sites contain minerals needed for the formation of carbonates where all of these minerals contain the important oxide or silicate minerals of calcium, magnesium and iron to enable the carbonation process. The chemical composition of all samples from three sites shows the presence of magnesium oxide (MgO) and iron oxide (Fe_2O_3) i.e. 1.74%-2.72% and 3.04%-11.79%, respectively in gold mine waste, iron oxide (Fe_2O_3) and calcium oxide (CaO) i.e. 39.58%-62.95% and 7.19%-15.24%, respectively in iron mine waste, and calcium oxide (CaO) and magnesium oxide (MgO) i.e. 72.12%-82.88% and 3.49%-4.36%, respectively in limestone waste rocks with high percentage showing high potential in sequestering and capturing carbon dioxide.

Finally, the carbonation efficiencies (ranging from 2.11% to 3.97%) and carbon uptake results of 56.09-363.33 g CO_2/kg reveals that smaller particle size of less than 38 µm, pH 8-12 in low temperature (80 °C) is ideal for the carbonation process to occur with maximum uptake capacity obtained of 87.66 g CO_2/kg and 363.3 g CO_2/kg for iron mine waste and limestone waste, respectively. From the mineral carbonation process, 0.46 g $FeCO_3$/kg and 1.65 g $CaCO_3$/kg have been successfully sequestered from the iron mine waste and limestone waste, respectively. Presence of carbonation product was confirmed by its morphological structure as needle-shaped crystal which was identified as aragonite in limestone waste. Statistical analysis shows that there was a statistically significant difference ($p<0.05$) in mean ranks between 38 µm and 75 µm, and a statistically significant, negative correlation between conversion efficiency (%) and particle size used (r= -0.487, p = 0.006). This indicates that particle size fraction is a crucial parameter in the carbonation process, and that using smaller particle size fraction can increase the carbonation efficiency. Findings show that these waste material have high potential to act as carbon sinks via mineral carbonation process. In line with the sustainable development goals in combating climate change, this study proposes a sustainable way towards low-carbon industry while making profit with the value-added carbonate produced. Therefore, this study is important to help tackle the issues of carbon emissions and strategy for carbon dioxide reduction in the future.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

POTENSI SISA PERLOMBONGAN UNTUK PROSES KARBONASI MINERAL DALAM APLIKASI PENANGKAPAN DAN PENGGUNAAN KARBON

Oleh

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Kegiatan perlombongan boleh menimbulkan risiko terhadap alam sekitar dan populasi sekitarnya kerana pembebasan pencemaran daripada penghasilan sisa lombong, sementara industri perlombongan itu sendiri dianggap sebagai salah satu industri yang menyumbang kepada peningkatan pelepasan karbon dioksida ke atmosfera. Pemencilan karbon melalui proses karbonasi mineral adalah salah satu cara untuk mengurangkan masalah ini. Karbonasi mineral boleh dijelaskan dengan tindak balas diantara karbon dioksida dan mineral silikat sehingga terhasilnya karbonat yang stabil. Sisa perlombongan mempunyai pelbagai jenis mineral yang berpotensi untuk digunakan sebagai sumber mineral silikat dalam proses karbonasi. Oleh itu, kajian ini memberi tumpuan kepada potensi sisa perlombongan yang bersifat alkali untuk penangkapan karbon yang melibatkan proses karbonasi mineral. Sepanjang kajian ini, pencirian sampel dari tiga lokasi (iaitu lombong emas, lombong bijih besi dan kuari batu kapur) dilakukan dan ini termasuk analisis taburan saiz partikel, analisis pH, dan analisis kimia, mineralogi dan morfologi. Eksperimen karbonasi mineral dilakukan dalam keadaan suhu-tekanan rendah menggunakan reaktor keluli tahan karat yang telah disesuaikan. Hasil analisis pH menunjukkan bahawa kebanyakan sampel mempunyai sifat alkali yang menunjukkan kesesuaiannya untuk menjalani reaksi karbonasi mineral. Lebih-lebih lagi, analisis taburan saiz partikel untuk partikel halus mendedahkan adanya sejumlah besar partikel bersaiz kecil (pecahan kelodak) dalam sisa lombong emas yang menjadikannya sesuai untuk proses ini. Sebaliknya, sisa lombong bijih besi terdiri daripada sebilangan besar partikel bersaiz besar (pasir) yang menunjukkan bahawa pra-rawatan perlu dilakukan agar proses karbonasi dapat dioptimumkan. Berdasarkan analisis mineralogi yang dilakukan, semua sampel sisa lombong dari tiga lokasi perlombongan mengandungi mineral yang diperlukan untuk pembentukan karbonat di mana semua mineral ini mengandungi mineral oksida atau silikat penting iaitu kalsium, magnesium dan ferum untuk memungkinkan berlakunya proses karbonasi. Komposisi kimia semua sampel dari tiga lokasi yang berbeza menunjukkan adanya magnesium oksida (MgO) dan ferum oksida (Fe_2O_3) dengan komposisi 1.74%-2.72% dan 3.04%-11.79%, masing-masing bagi sisa lombong emas, ferum oksida (Fe_2O_3) dan kalsium oksida (CaO) dengan komposisi

39.58%-62.95% dan 7.19%-15.24%, masing-masing bagi sisa lombong bijih besi, dan kalsium oksida (CaO) dan magnesium oksida (MgO) dengan komposisi 72.12%-82.88% dan 3.49%-4.36%, masing-masing bagi batuan sisa batu kapur dengan peratusan yang tinggi menunjukkan potensi yang tinggi dalam penyerapan dan penangkapan karbon dioksida. Akhirnya, keberkesanan karbonasi (antara 2.11% hingga 3.97%) dan hasil penangkapan karbon sebanyak 56.09-363.33 g CO_2/kg mendedahkan bahawa saiz partikel yang lebih kecil iaitu kurang daripada 38 µm, pH 8-12 dalam suhu rendah (80 °C) adalah sesuai untuk proses karbonasi berlaku dengan kapasiti pengambilan maksimum yang diperolehi sebanyak 87.66 g CO_2/kg dan 363.3 g CO_2/kg, masing-masing bagi sisa lombong bijih besi dan sisa batu kapur. Daripada proses karbonasi mineral, 0.46 g $FeCO_3$/kg dan 1.65 g $CaCO_3$/kg telah berjaya dihasilkan, masing-masing daripada sisa lombong bijih besi dan sisa batu kapur. Kehadiran produk karbonasi telah disahkan oleh struktur morfologinya di mana kristal seperti jarum yang dikenal pasti sebagai aragonit telah dikenal pasti dalam sisa batu kapur. Analisis statistik menunjukkan bahawa terdapat perbezaan yang signifikan secara statistik ($p<0.05$) dalam kedudukan min antara 38 µm dan 75 µm, dan korelasi negatif yang signifikan secara statistik antara keberkesanan karbonasi (%) dan saiz partikel yang digunakan ($r=-0.487$, $p=0.006$). Ini menunjukkan bahawa saiz partikel adalah parameter yang penting dalam proses karbonasi, dan bahawa menggunakan saiz partikel yang lebih kecil boleh meningkatkan keberkesanan karbonasi. Dapatan kajian menunjukkan bahawa bahan buangan ini berpotensi tinggi untuk bertindak sebagai penyimpan karbon melalui proses karbonasi mineral. Sejajar dengan matlamat pembangunan yang lestari dalam memerangi perubahan iklim, kajian ini mengusulkan cara yang mampan ke arah industri rendah karbon sambil menghasilkan keuntungan dengan nilai tambah karbonat yang dihasilkan. Oleh itu, kajian ini penting dalam membantu menangani isu pelepasan karbon dan strategi pengurangan karbon dioksida pada masa akan datang.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Master of Science. The members of the Supervisory Committee were as follows:

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This is to confirm that:

- the research conducted and the writing of this thesis was under our supervision;
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LIST OF ABBREVIATIONS

GHG	Greenhouse Gases
CCS	Carbon Capture and Storage
CCU	Carbon Capture and Utilization
NOAA	National Oceanic and Atmospheric Administration
USGS	United State of Geological Survey
IPCC	Intergovernmental Panel on Climate Change
DOE	Department of Energy
NETL	National Energy Technology Laboratory
NRC	Norwegian Research Council
NEDO	New Energy and Industrial Technology Development Organization
WWF	World Wildlife Fund
EOR	Enhanced Oil Recovery
RCA	Recycled Concrete Aggregate
AMD	Acid Mine Drainage
BS	British Standard
ASTM	American Society for Testing and Materials
XRD	X-ray diffraction
XRF	X-ray fluorescence
UKM	Universiti Kebangsaan Malaysia
SEM	Scanning electron microscopy
EDX	Energy dispersive X-ray
TGA	Thermogravimetric analysis
DTG	Derivative thermogravimetric
OPC	Ordinary Portland cement

SCM	Supplementary Cementitious Material
CBMs	Cement-based materials
C-S-H	calcium silicate hydrate

CHAPTER 1

INTRODUCTION

1.1 Background of the study

Global warming has become the greatest environmental concern on national and international level. It has been believed that the increase in greenhouse gas (GHG) emission in the atmosphere, mainly carbon dioxide (CO_2) via fossil fuel consumption is the main contributor to the uncommon rise of the global temperature over the past several years (NOAA, 2021). Global scale climate modelling has predicted that the greenhouse gas concentration will continue to increase along with the earth average surface temperature resulting in changes in weather, rise of sea levels, disturbed ecosystem, potential loss of life and property (caused by severe flooding, intense hurricane and rain, and sea level increase) and high risk of heat strokes and deaths from frequent fires and hotter summer (Riebeek, 2010). Concern over the global warming issue on anthropogenic contribution (use of fossil fuel and carbon dioxide emission) gives rise to serious international demand on nation to lower their production of anthropogenic carbon dioxide. In an effort to overcome these issues, a large number of research has proposed sequestration of carbon dioxide or also known as carbon capture and storage (CCS) as the solution that can capture and store carbon in a stable form for a long period of time through a process namely mineral carbonation (Jacobs and Hitch, 2011).

After CCS has been introduced, CCU which stands for carbon capture and utilization has emerged to add value to the end product of carbon capture. This means that instead of only storing carbon, it can be utilized in for instance, green material product development for commercialization. The aim of CCS and CCU is similar; to capture carbon emission and to prevent it from being released back to the atmosphere, however, the last destination of the carbon dioxide captured is different. CCS transfers the captured carbon dioxide to a waste storage for a long period of time, while CCU produces useful product by capturing and storing carbon dioxide such as in cementitious materials in construction industry, and in other sectors as synthetic fuels and many more depending on the type of materials used (Huang et al., 2021; Alshalif et al., 2021; Othman et al., 2021; Zhu et al., 2021). Among the potential CCS and CCU technology, mineral carbonation was commended as the most ideal approach where the carbon dioxide in the atmosphere can be captured as stable carbonate which can further be used and commercialized as partial cement replacement, brick production, paint etc. (Lackner et al., 1995; Jacobs & Hitch, 2011; Bobicki et al., 2012). Mineral carbonation can be regarded as both CCS and CCU but the latter has more potential where it offers additional revenue in addition to removing carbon dioxide from the air.

Mineral carbonation is the reaction of basic minerals with carbon dioxide to form non-toxic solid carbonate. Mineral carbonation can occur naturally but a little modification to accelerate this natural process can reduce the level of excess carbon dioxide in the atmosphere, thus reducing the global temperature. Mineral carbonation can be explained by the reaction of carbon dioxide with metal oxide bearing minerals forming insoluble carbon as shown in equation 1.1 (Sanna et al., 2014):

$$\text{Metal oxide} + CO_2 \rightarrow \text{Metal Carbonate} + \text{Heat} \quad (1.1)$$

Generally, mineral carbonation reaction can occur with different starting materials including Mg-silicate minerals and Fe- or Ca-rich silicates. Equation 1.2, 1.3, 1.4, and 1.5 show the mineral carbonation processes with Mg, Fe and Ca-rich silicates (Sanna et al., 2014).

$$Mg_2SiO_4 + 2CO_2 + 2H_2O \rightarrow 2MgCO_3 + H_4SiO_4 \quad (1.2)$$

$$Mg_3Si_2O_5(OH)_4 + 3CO_2 + 2H_2O \rightarrow 3MgCO_3 + 2H_4SiO_4 \quad (1.3)$$

$$Fe_2SiO_4 + 2CO_2 + 2H_2O \rightarrow 2FeCO_3 + H_4SiO_4 \quad (1.4)$$

$$CaSiO_3 + CO_2 + 2H_2O \rightarrow CaCO_3 + H_4SiO_4 \quad (1.5)$$

Mineral carbonation can be achieved either in-situ or ex-situ. The in-situ carbonation is when the carbon dioxide is stored underground when reaction takes place between carbon dioxide and the material trapping carbon dioxide as carbonate materials, whereas ex-situ is the same reaction as with the in-situ but occurs in above ground in a chemical processing plant (Gerdemann et al., 2004). Mineral carbonation proposes a promising solution for carbon sequestration where the carbonate formed at the end of the reaction are non-hazardous to the environment and to human, and it is in a stable form (Lackner et al., 1995). In addition, the abundance of the raw materials used adds to one more of the advantages of using waste materials for mineral carbonation. Various types of waste materials can be used as feedstock for mineral carbonation process such as mining waste, steel slag, fly ash and many more. Among the waste materials, utilization of mine waste has been a reliable choice of feedstock material, as this application may help overcome the commonly known issue of waste production at most mining sites across the globe. Thus, it is the aim of this study to assess the potential of mine waste material in capturing carbon dioxide, as well as to evaluate the influential factors on the carbonation reaction by determining the carbonation conversion efficiency and carbon uptake capacity. The carbon capture capacity will be compared based on different influencing parameters which will further determine the ideal condition for the carbonation process to occur.

1.2 Problem statement

As reported by NOAA (2017), carbon dioxide concentration has increased from 280 ppm in the 1970s to 402.9 ppm in 2016 mainly due to the increasing earth population and increasing activity of fossil fuel combustion. Until recently, the monthly average of carbon dioxide concentration has reached 419.13 ppm in May 2021 compared to the monthly average of 417 ppm in May 2020, and this was announced as the highest carbon dioxide level since 1974 (NOAA, 2021). The atmospheric strain of carbon dioxide is now similar to 4.1-4.5 million years ago (Pliocene Climatic Optimum) where carbon dioxide level was measured approximately 400 ppm. Within that time, the average temperature was about 4 °C warmer than in pre-industrial times, and global mean sea level was 23.5 m above current level (Dumitru et al., 2019). Study also reveals that large forest area in the Arctic are now Tundra (Bringham-Grette et al., 2013). The fact that the level of carbon dioxide has become on par with one of the historic time on earth is alarming as these changes can affect everything and this includes extreme weather that can implicate the surrounding environment, human and other living things.

In addition, even though carbon dioxide removal methods have been found where carbon dioxide can be stored, e.g. using the terrestrial ecosystem through the natural pathway of carbon cycle, there is still risk of carbon leakage presence over time. Leakage causes the sequestration to be ineffective and this happens when carbon dioxide returns to the atmosphere quickly. This may affect the environment badly causing not only human health and other living things to be affected but also their welfare and well-being. In order to prevent this from happening, other methods need to be studied where the carbon dioxide can be kept permanently without a risk of leakage over time. One of the options is through the adoption of carbon capture and storage technology via mineral carbonation.

On the other side, mining industry is considered as a carbon-intensive industry which has contributed to the increasing level of carbon dioxide in the atmosphere. Despite its role as one of the main industries in Malaysia, several other mining-related problems arise such as contamination of surface and ground water leading to acid mine drainage formation (Affandi et al., 2018). Another critical issue of most mining sites is due to the accumulation of mining waste that have little to no market value, often being regarded as mine waste. Utilization of such waste material which has been known to be able to sequester carbon dioxide can be seen as a feasible approach that benefits not only the mining industry but also the environment as a whole. Therefore, in an effort to overcome these issues, sequestration of carbon dioxide or CCS/CCU method can be an advantageous solution that can sequester and capture carbon dioxide for long term which can also be developed as value-added product that brings revenue to the industry. This can be achieved by means of mineral carbonation using the mining waste as the feedstock for the process, which has long term benefits in mitigating the global carbon emission.

1.3 Significance of study

The use of mining waste in capturing carbon dioxide can be regarded as a beneficial approach as this can help in solving the problem of increasing carbon dioxide emission from a carbon-intensive industry (e.g. quarries, mining facilities) as well as the increasing amount of accumulated waste in many mining waste lands. By reusing mine waste such as for the feedstock in mineral carbonation process, the size and area of the waste lands can be reduced, thus reducing the environmental footprint due to mining activities. The mining industries can also benefit from the utilization of its waste where it requires less expenditure for them to close the mine for rehabilitation, closure and long-term monitoring.

Moreover, mineral carbonation is considered as a permanent and safe method for carbon dioxide disposal because the reaction of carbon dioxide with the minerals form a carbonate product which is not hazardous to the environment, humans and other living things and there will be no risk of carbon dioxide leakage over time. Clearly, the method tackles not only the carbon emissions issue but also the waste production in most mining sites, often associated with contamination issues. Thus, this will help solve many problems associated with mining activities, which ultimately leading to the reduction of carbon dioxide footprint of mining industries.

In addition to having a permanent storage and stable nature, the carbonate product formed from the carbonation process has great potential for commercialization in the construction sector. Obviously, it can be used to produce cement-based products such as bricks and block, concrete, fibreboard and non-cementitious products such as fire extinguisher, paint and many other. This will become an additional revenue-generating product that will give a positive impact to the industries (mining and construction sectors) and the country's economy. In other words, the carbonated product from mineral carbonation can be turned into usable or saleable product which can bring revenue to the mining industry that benefits the construction sector as well. This is also in line with the concept of circular economy in mining industries.

This study will provide a better insight of the carbonation potential of mine waste materials (i.e. iron mine waste, gold mine waste and limestone waste), thus making use of it so that it can be applied in a larger scale such as carbon sequestration plant or integrating it into the mining operation. Consequently, this will successfully reduce the level of carbon dioxide in the atmosphere through long term storage via the process of mineral carbonation.

1.4 Objective of study

This study aims to investigate the possibility of reusing mine waste material as a potential carbon sinks via mineral carbonation process. The underlying research objectives are as follows:

1. To evaluate the potential of raw mine waste materials as feedstock for mineral carbonation based on its physical, mineralogical, morphological and chemical characteristics.
2. To assess the influence of different parameters, i.e. particle size, pH and reaction temperature on mineral carbonation reaction based on the carbonation conversion efficiency and carbon uptake capacity.
3. To elucidate the formation of carbonate product from the mineral carbonation process and its potential application.

1.5 Research questions

To investigate the possibility of reusing mine waste material as a potential carbon sinks via mineral carbonation process, the underlying research questions are as follows:

1. Does raw mine waste material have the potential to be used as feedstock for mineral carbonation based on its physical, mineralogical, morphological and chemical characteristics?
2. How does particle size, pH and reaction temperature affect mineral carbonation reaction in terms of its carbonation conversion efficiency and carbon capture capacity?
3. Does raw mine waste material have the potential to form carbonate product via carbonation process?

1.6 Scope of study

With the increase of carbon dioxide concentration year by year, there has been an increase in the demand for a more ‘green’ approach in carbon-intensive industries. Following this situation, this research explores the possibility of reusing mine waste material as a potential carbon sinks via mineral carbonation process. The use of this waste can be seen as a feasible approach that benefits not only the mining industry but also the surrounding environment. This study covers the physical, mineralogical, morphological and chemical characteristics of the mine waste materials (i.e. limestone, gold mine and iron mine waste) to evaluate its potential to be used as carbon sinks. Then the mine waste materials will be selected based on its initial characterization to undergo mineral carbonation process, in which the factors that can influence the carbonation process will be assessed afterwards. The factors that will be looked into in this study are particle size (i.e. 38 µm, 63 µm and 75 µm), pH condition (8-12) and reaction temperature (i.e. 80 °C, 150 °C and 200 °C). Furthermore, the carbonation conversion efficiency (%) and carbon uptake capacity of mine waste materials will be determined in relation to the different parameters applied. The effect of the various parameters will be discussed in view of its carbonate product formation to suggest the possible operating parameters optimal for carbonation reaction to proceed. To this end, the potential application of the mineral carbonation will be discussed based on the carbonate product formed through the process.

1.7 Thesis structure

Following this chapter, the remaining chapters of the thesis are outlined below:

Chapter 2: Literature Review

Overview of the topics covered throughout the study; background of carbon capture and storage (CCS) and carbon capture and utilization (CCU) technology focusing on mineral carbonation technology which discussed the process route involved, the potential feedstock that can be used in carbonation process with mine waste material as the main focus, explanation on the parameters influencing the mineral carbonation process (i.e. temperature, particle size and pH condition), summary of previous related studies on mineral carbonation, and the recent development of mineral carbonation technology.

Chapter 3: Methodology

Details of the methods undertaken during the laboratory works throughout the study; laboratory works include field sampling, sample preparation, sample characterization, mineral carbonation experiment, the carbonation product characterization followed by the calculation of carbonation efficiency, carbon uptake capacity and carbonate product formed.

Chapter 4: Results and Discussion I: Characteristics of Mine Waste Material

Results and discussion on the characteristics of mine waste material form three different sites (i.e. limestone quarry, gold mine site and iron mine site). The pH, particle size distribution, mineralogical, chemical composition and morphological structure were discussed in association with its potential as feedstock for mineral carbonation.

Chapter 4: Results and Discussion II: Influence of Different Parameter on Mineral Carbonation Process

Results and discussion on the influence of pH, particle size and temperature on the carbonation process based on the carbonation efficiency and carbon uptake capacity achieved. This include the statistical analysis to determine the differences in carbonation conversion efficiency using different parameters.

Chapter 4: Results and Discussion III: Production of Mineral Carbonation Product and its Application

Results and discussion on the products of the mineral carbonation process (i.e. amount of $CaCO_3$ and $FeCO_3$ formed). These include the amount of carbonate product formed via carbonation and its significance in the brick making industry.

Chapter 5: Conclusions and Recommendations

Summary of the major contribution and findings of this thesis and the summary of the work done in the fulfilment of the research aims and objectives. Recommendations for future research directions follow.

APPENDICES

Related information to support the thesis contents.

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