

Sequestration of carbon dioxide using ground granulated blast furnaces slag and kaolin mixtures

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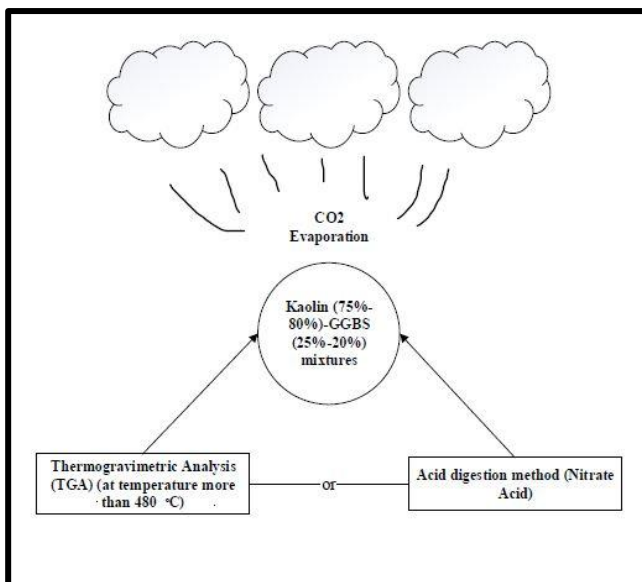
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Received: 16/12/2020, Accepted: 07/01/2021, Available online: 15/01/2021

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<https://doi.org/10.30955/gnj.003487>

Graphical abstract



Abstract

Mineral carbonation has been utilised extensively worldwide as the most important way of permanently sequestering carbon dioxide (CO₂). This study objective includes sequestering of CO₂ gas from atmosphere via carbonation of magnesium and calcium. Waste materials such as ground granulated blast furnaces slag (GGBS) extracted through iron manufacturing as well as brown kaolin mixtures were employed. Acid digestion method as well as thermogravimetric analysis were employed to quantify CO₂ sequestered by GGBS-kaolin mixtures. The outcomes indicated that the acid digestion technique is more dependable compared to TGA in measuring CO₂. Nonetheless, both methods deduced that the quantity of CO₂ sequestered is about 5% from the total dry mass of the mixtures. Thus, GGBS-kaolin mixtures effectively sequester a substantial quantity of CO₂.

Keywords: Kaolin clay, ground granulated blast-furnaces slag, carbon dioxide, acid digestion method, thermogravimetric analysis.

1. Introduction

Global warming is considered a key ecological issue affecting the socio-ecological condition of the Earth. Persistent action is required for eliminating the factors that give rise to global warming. Otherwise, the climate's impact would reach a severe and dangerous level, which will affect both nature and humans (Rajanen and Rajanen, 2019). Changes in the environment include raised sea level and temperatures, acidification of oceans, and global reduction in ice thickness. The core cause of global warming is the rise in greenhouse gas concentration (GHG) in the air. Carbon dioxide (CO₂) is one of the dominant gases in a GHG (Galina *et al.*, 2019). The allowable amount of CO₂ in the atmosphere is 350 ppm (Rahman *et al.*, 2017). About 77% of greenhouse gas concentration originates from CO₂, while its composition in the air is about 400 ppm (ppm=part per million). This indicates that CO₂ has to be reduced.

Globally, the annual release of CO₂ increased to 32.1 billion tonnes in 2015 from 17.78 billion tonnes in 1980. The rise is about 100% in 35 years, and it is expected to double by 2035. In Malaysia, CO₂ release increased by 69% during 2000 to 2020 to about 285.7 million tonnes (Jorat *et al.*, 2018). Cement is termed the largest source of CO₂ release, led by the disintegration of carbonates. It is also associated with the release of nitrogen oxide, which causes air pollution (Latifi and Meehan, 2017). The overall manufacturing of cement worldwide is accountable for about 7% of the total release of CO₂ (Abdullah *et al.*, 2018). Production of one tonne of cement leads to a release of 0.8 to 1.0 tonnes of CO₂ (Abdullah *et al.*, 2018; Latifi and Meehan, 2017).

Carbon Capture and Utilization (CCU) intends not just to decrease the quantity of emissions into the atmosphere but also to attain a gain by using CO₂ in different kinds of industrial processes, substituting traditional raw materials (Mohsin *et al.*, 2020). CCU has more and more concentrated on transforming CO₂ into carbon-based, economically feasible fuel and feedstock. This is not only due to economic reasons but also because such products can decrease the usage of their equivalents based on fossil

fuels (Mohsin *et al.*, 2020). The sequestered CO₂ can be utilised for several applications like feedstock for manufacturing plastic items (Arning *et al.*, 2020), drawing out of compounds with supercritical CO₂ (scCO₂), water treatment, dry cleaning, and food industry applications. For the food sector, CO₂ is used as a substitute for the toxic elements utilised for food decontamination. In biological applications, it is utilised for fast growing biomass. CCU technologies are split into four classes: Chemicals from CO₂, CO₂ as a solvent, Fuels from CO₂, and Enhanced Oil Recovery (EOR) & Enhanced Coal Bed Methane (ECBM) (Baena-Moreno *et al.*, 2019).

Carbon capture and storage (CCS) technologies are deployed for reducing the quantity of CO₂ in the atmosphere using three different methods (Saran *et al.*, 2018). The first is ocean storage with a projected volume of 1000 Gt. However, its ecological effect (alteration of ocean chemistry, pH reductions) has been a drawback. The second is geological stowage with a capacity of 500 to 3000 Gt. The key drawback is the likely leakage of CO₂ and the fact that nations such as Korea lack adequate area for geological sequestration (Mun and Cho, 2013).

The third technique is mineral carbonation. This is termed as a substitute for geological storage. In mineral carbonation, CO₂ gas is allowed to react with magnesium as well as calcium silicates. The products related to this reaction are silica and carbonate (Prigobbe *et al.*, 2009). Under ambient conditions, magnesium and calcium carbonates remain thermodynamically stable when they are not contacted with any of the acids (Eloneva *et al.*, 2008). A permeant reaction occurs between calcium or magnesium with CO₂ and needs an extended time (Mun and Cho, 2013). This process is fast-tracked by raising the concentration or pressure of CO₂ as well as raising the temperature (Mo and Panesar, 2012). One more benefit of calcium and magnesium carbonation is improvement in soil strength (Cai *et al.*, 2015; Cai and Liu, 2017; Cao *et al.*, 2017; Guang-hua *et al.*, 2015; Ho *et al.*, 2017; Liu *et al.*, 2017; Nakarai and Yoshida, 2015; Song-yu *et al.*, 2017; Yi *et al.*, 2013a; Yi *et al.*, 2013b; Yi *et al.*, 2013; Yi *et al.*, 2016).

To sequester CO₂, calcium- or magnesium-rich materials are combined with various soil types while also improving soil strengths. To store CO₂ in the form of minerals carbonate, reactive magnesia (magnesium-rich material) are combined with various types of soils like sandy soil (Yi *et al.*, 2013) and silty soil (Cai *et al.*, 2015; Guang-hua *et al.*, 2015). A magnesium-rich material is olivine, which is combined with clayey soil for strength improvement when sequestering CO₂ (Fasihnikoutalab *et al.*, 2016). Cement was blended with silica sand (Nakarai and Yoshida, 2015). Majority of the earlier findings overlooked the amount of CO₂ sequestered within such combination, as these focussed only on the strength gained because of carbonation. Apart from Liu *et al.* (2017), when various types of soil were combined with 20% reactive magnesia (from soils dry weight), the amount of CO₂ that has been sequestered within those mixture was found to lie in the range of 8%–16% from the mixture dry weight.

For materials that contain sufficient amount of magnesium and calcium for mineral carbonation, the by-product material, ground granulated blast furnaces slag (GGBS), from steel manufacturing contains high CaO content as well as a considerable amount of MgO. GGBS enjoys a benefit over other material (cement, reactive magnesia, and olivine) since it is a sustainable material which does not cause the release of high quantity of CO₂ during production. This paper concentrates on the amount of CO₂ sequestered within the ground granulated blast furnaces slag-kaolin mixtures that have been carbonated under various conditions. Kaolin is regarded to possess low strength along with relatively high compressibility feature; therefore, it entails stabilisation prior to construction. Two methods are employed to quantify the amount of CO₂ that has been sequestered in GGBS-kaolin mixtures; the acid digestion and thermogravimetric analysis (TGA).

2. Materials and methods

The soil taken in use was somewhat acidic and brown kaolin, which was brought from Kaolin (Malaysia) Sdn Bhd plant situated in Tapah, Perak, Malaysia. The GGBS was brought from a local factory situated in Johor Bahru, Johor, Malaysia. The physical characteristics of kaolin and GGBS are displayed in Table 1, whereas the chemical structure of both is shown in Table 2.

Table 1. Properties of kaolin and GGBS

Material	Kaolin	GGBS
Liquid limit (%)	40.5	36.6
Plastic limit (%)	22.5	-
Plasticity limit (%)	18	-
Clay (%)	0.65	1.876
Silt (%)	92.6	98.124
Sand (%)	6.75	0
Specific gravity	2.52	2.83
Optimum moisture content (%) (OMC)	18	-
Maximum dry density (kg/m ³) (MDD)	1640	-
UCS (kPa)	115	-
pH	4.33	10.6

The GGBS content in the blend was 20% and 25% of total dry amount of kaolin. The mixtures were properly mixed to confirm a consistent mixture, and further 18% of distilled water was mixed with this blend (OMC). The samples were blended again and then put into a cylinder mould having 38 mm diameter and 76 mm height. All of the samples were compressed to attain the MDD (the samples were formulated with the MDD and OMC for raw kaolin). Two groups of samples were made; the first one was put in a humidity chamber having a temperature range (of 27 ± 2°C) for 7 days before carbonation, therefore it allowed GGBS to hydrate and separate more CO₂. Another set was put in the carbonation cell right after demoulding; the working details of the experiment are shown in the Table 3.

The process of carbonation begins after filling the carbonation cell with the sample (triaxial test cell which is attached to a gas cylinder of 99% CO₂). The cell was fastened properly to makes sure that there is no leakage.

The gas regulator was kept at 200 or 100 kPa. The inflow was released to allow CO₂ to enter the cell, the outflow was released for 5 minutes to take out the air present inside the cell and let the cell get fully saturated with CO₂. After 5 minutes, the valve of the outflow was closed whereas the valve of the inflow was left open for a definite period (1, 3, 6, 24 hours). Following the process of carbonation, the

samples were taken out of the cell, put in the kiln for 24 hours, then compacted and sieved through 2mm openings. Only particles having diameter less than 2 mm were utilised for acid digestion and TGA (thermogravimetric analysis) methods. The trial samples for both the techniques are shown in Table 3.

Table 2. Chemical composition of GGBS and kaolin

Chemical composition	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	K ₂ O
GGBS	30.5%	10.4%	0.30%	47.6%	4.88%	0.317%
Kaolin (Saeeda et al., 2015)	49.5%	30.31%	1.02%	0%	0%	8.78%

Table 3: Experimental work details for the carbonation process

Content of GGBS (%)	20				25			
	0		7		0		7	
Curing period (days)	0		7		0		7	
CO ₂ pressure (kPa)	100	200	100	200	100	200	100	200
Carbonation period (hours)	-	-	-	-	-	-	-	-
1	√	√	√	√	√	√	√	√
3	√	√	√	√	√	√	√	√
6	√	√	√	√	√	√	√	√
24	√	√	√	√	√	√	√	√

Solution of 10% nitrate acid was used for the acid digestion method. Approximately 40 grams of the selected soil was placed in a 600 ml mug, prior to which the weight of empty beaker with glass rod was recorded. 300 ml of 10% nitrate acid solution was put in another beaker whose weight was measured and logged. The weighed solution was mixed with the soil; CO₂ bubbles then emerged on the outside. The concoction was mixed together for ten minutes using the glass rod (until the bubbles vanished). Last of all, the weight of soil, beaker, glass rod, and the HNO₃ solution was logged, and the following equation was applied to calculate the amount of CO₂ sequestered in the processed kaolin.

$$CO_2 = \frac{m_1 + m_2 - m_3}{m_1 / (1 + w_f)} * 100 \quad (1)$$

where m_1 =sieve carbonated kaolin (40 gm approximately), m_2 =nitrate acid solution (300 gm approximately), m_3 =mixture weight after 10 minutes of continuous stirring, w_f =the gravimetric water content of carbonated soil.

TGA analysis was performed based on ASTM E1131 – 08. Graphs of both the DTA (differential thermal analysis) and TGA were employed for evaluation. The assessment was carried out in nitrogen (N₂) environment, the first sample mass was logged, and then it was heated at the rate of 20°C /Min. The weight of the sample was logged at the end of every minute.

3. Results and discussion

3.1. Acid digestion method

The method of acid digestion consists of the chemical reaction between magnesium or calcium carbonate yield and acid wherein the CO₂ evaporates and returns to the atmosphere. An exothermic reaction takes place which includes the formation of magnesium or calcium nitrate, water, and there was also the formation of CO₂ gas as

foams. The 10% nitrate acid (HNO₃) solution was used in this study.

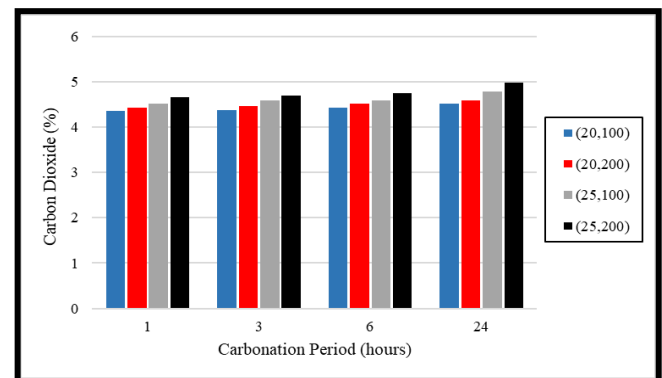


Figure 1. Amount of CO₂ sequestered by kaolin-GGBS mixture at 0 days curing carbonated under different conditions using acid digestion method

Figures 1 and 2 show the CO₂ amount isolated by the processing of kaolin with GGBS for durations of 0-day and 7-days carbonation process under separate conditions using the method of acid digestion, respectively. The tag (A, B) represents the kaolin processed with A GGBS amount and under B CO₂ applied pressure.

Results from Figure 1 state that the CO₂ amount isolated from the processed kaolin with GGBS 20% and 25% ranges from 4 to 5% in comparison to its original weight. In such cases, a sample (20,100) processed for 0 days and given carbonation treatment for 1-hour isolate approximately 4.35% CO₂ which rises to 4.42% after 6 hours of carbonation and keeps rising until it reaches 4.51% after one day of carbonation process. The amount of CO₂ for sample (20,200) with the same carbonation durations is slightly higher in comparison to the sample (20,100).

Sample (25,100) separates about 4.51% CO₂ at the end of 1 hour of carbonation, and thus increases to 4.59% following 6 hours carbonation; further reaches 4.79% after 24 hours of carbonation.

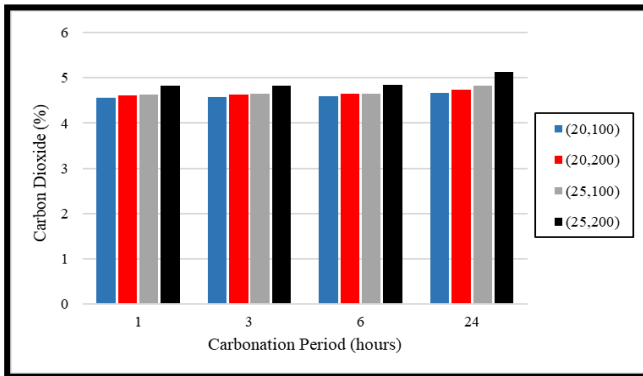


Figure 2. Amount of CO₂ sequestered by kaolin-GGBS mixture at 7 days curing carbonated under different conditions using acid digestion method

Figure 2 shows the amount separated by the processing of GGBS-kaolin mixture for 7 days and carried out carbonation process with 100 and 200 kPa CO₂ pressure for a duration of 24 hours. The result resembles that of Figure 1 wherein the CO₂ amount in the soil increases as time progresses and with the increase in the applied pressure amount. For example, in the sample (20,100), there is formation of about 4.56% CO₂ after an hour's carbonation which keeps increasing slightly and gets 4.6% and 4.67% after 6- and 24-hours carbonation, respectively. Then again, for sample (20,100), there is a slight increase in the values as it reaches 4.61%, 4.65%, and 4.73% after 1, 6, and 24 hours of carbonation, respectively.

There is essentially no noteworthy difference between the amount sequestered at the 1st hour, and that sequestered after 24 hours; most of the CO₂ sequestration occurs during the 1st-hour of the carbonation process. The outcome resembles earlier results (Cai and Liu, 2017; Yi *et al.*, 2016) in which the CO₂ amount increases gradually. The result showed a pattern similar to the UCS outcome; the rate of the isolation of CO₂ is proportional to the strength development.

3.2. Thermogravimetric (TGA) analysis

TGA assessment was conducted alongside acid digestion technique in order to quantify the weight loss caused by heating. Also, computation of the amount of CO₂ was done. Weight loss as well as differential thermal analysis (DTA) with increase in temperature have been shown in Figures 3 to 6.

The decomposition was carried out for well-crystallised CaCO₃ and amorphous CaCO₃ in the temperature range of 780–990°C and 550–780°C, respectively. However, due to the stabilisation, distinguishing between the actual carbonated material from the raw material versus the carbonated material is not possible. Thus, we can overlook the amount of carbonated material from the raw material. Post 480°C, the sample's weight loss could be due to the

decarbonation of calcium and magnesium (Cai and Liu, 2017; Liu *et al.*, 2017).

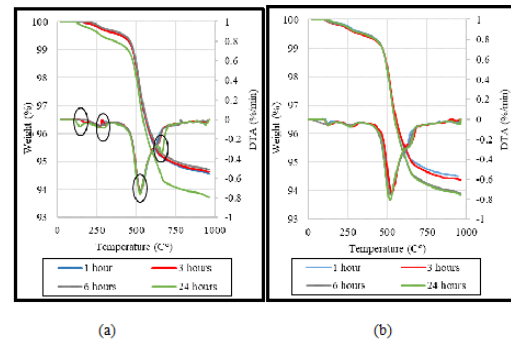


Figure 3. TGA and DTA curves of carbonated 20%GGBS treated kaolin at 0 days curing carbonated with (a) 100 kPa (b) 200 kPa

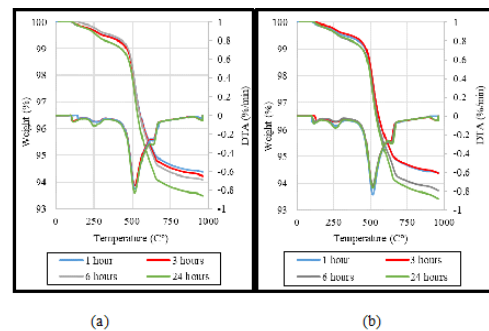


Figure 4. TGA and DTA curves of carbonated 25%GGBS treated kaolin at 0 days curing carbonated with (a) 100 kPa (b) 200 kPa

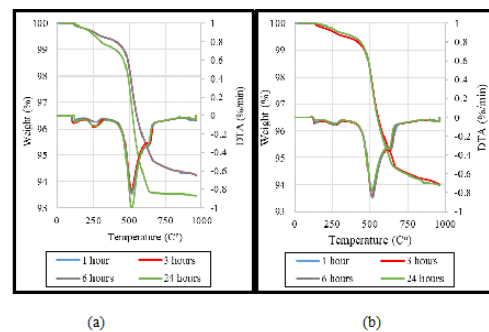


Figure 5. TGA and DTA curves of carbonated 20%GGBS treated kaolin at 7 days curing carbonated with (a) 100 kPa (b) 200 kPa

Figures 3 to 6 display the curves for TGA and DTA pertaining to carbonated GGBS with different CO₂ pressure processed kaolin that has been treated for 0 and 7 days. As per the TGA assessment, a decrease in the weight is caused by the increase in temperature. Once the temperature reaches 850°C, every sample loses around 6%–6.5% weight versus its original weight. At temperatures 450–850°C, majority of the weight loss occur. For all samples, the pattern of weight loss is almost similar, and this could be clearly seen bases on the DTA assessment.

As displayed in Figure 3a, the DTA graphs have been represented with four peaks, along with the change in temperature (0°C to 850°C). Due to loss of bound water, at 0°C to 250°C, the first small peak is seen to appear. This may also show the nesquehonite's transformation to dypingite or hydromagnesite, since at temperatures ≥ 50 C, nesquehonite loses stability. Occurrence of a small peak is

seen when the UCS sample was kept in the oven for 24 hours to measure the water content, and due to the presence of small amount of nesquehonite in the sample. The second peak, which exists between temperatures of 250°C and 480°C, represents the dehydroxylation of calcium hydroxide ($\text{Ca}(\text{OH})_2$) and brucite ($\text{Mg}(\text{OH})_2$) that are yet to be carbonated (Unluer and Al-tabbaa, 2013). The second peak's size as well as the considerably small weight loss specify that most of the calcium and magnesium have been carbonated as well as a reaction with CO_2 , that results in just a small amount of uncarbonated residue.

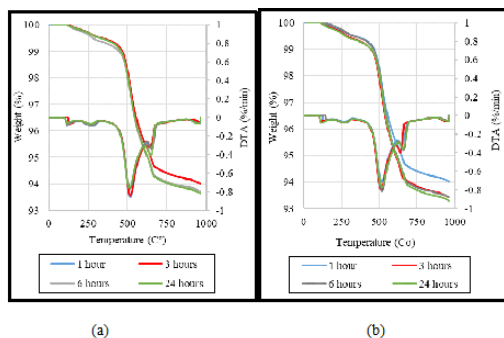


Figure 6. TGA and DTA curves of carbonated 25%GGBS treated kaolin at 7 days curing carbonated with (a) 100 kPa (b) 200 kPa

Between 480°C and 850°C, the relatively large fourth and third peaks are an outcome of decarbonisation of Ca and Mg carbonated products (here, CO_2 is sent back to the atmosphere). These peaks can be clearly distinguished versus the first and second peaks. The amount of CO_2 segregation is displayed in the graph based on the weight loss observed at temperatures of 480°C and 850°C.

Figures 7 and 8 display the amount of CO_2 that has been sequestered by the GGBS-kaolin mixture via TGA. The label (20,100) specifies 20% GGBS treated kaolin that has been carbonated at 100 kPa CO_2 applied pressure. Here, identical behaviours were observed in both the figures. The mixture composition includes 4.3% to 5.2% CO_2 pertaining to the soil's overall dry mass. This is almost identical to the estimated amount based on the acid digestion technique.

The amount of CO_2 sequestered in the soil is affected by several factors, such as amount of calcium and magnesium available, CO_2 applied pressure for acceleration, curing period before to the carbonation process as well as carbonation time. In this study, the impact of these four factors has been analysed. Extension in the carbonation period was found to result in increase in CO_2 amount. Eventually, at a certain point, the soil achieves its maximum capacity, thus halting the sequestration.

As shown in Figures 7 and 8, on employing 200 kPa CO_2 for identical condition, the amount of CO_2 sequestered was found to be marginally higher versus on employing 100 kPa CO_2 . Applying higher CO_2 pressure led in an increment in the pore solution acidity. This resulted in release of additional magnesium and calcium ions for carbonation (Fasihnikoutalab *et al.*, 2016; Mo and Panesar, 2012), which consequently led to more CO_2 absorption. Furthermore, a longer curing period (i.e., 7 days) enabled sequestration of

CO_2 as it allowed a higher amount of CO_2 . The magnesium and calcium hydrates due to 7 days of curing helped form $\text{Mg}(\text{OH})_2$ and $\text{Ca}(\text{OH})_2$, respectively, which resulted in formation of additional carbonation products.

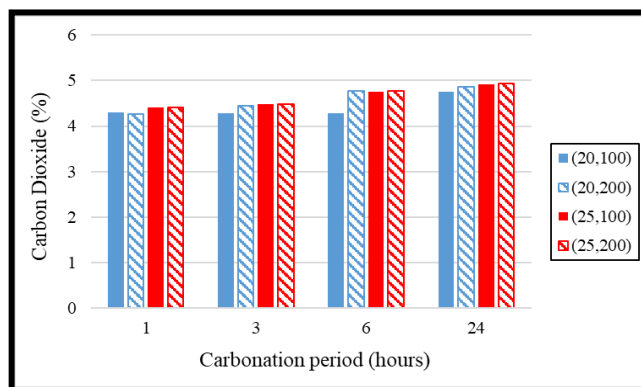


Figure 7. Amount of CO_2 sequestered by kaolin-GGBS mixture at 0 days curing carbonated under different conditions using TGA analysis

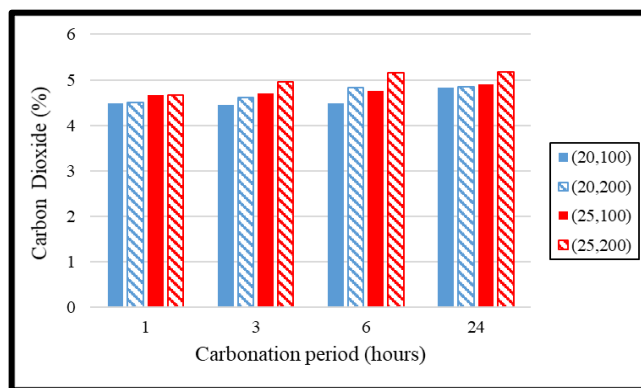


Figure 8. Amount of CO_2 sequestered by kaolin-GGBS mixture at 0 days curing carbonated under different conditions using TGA analysis

The most critical parameter that regulates the CO_2 amount in the soil is the amount of available calcium and magnesium in the chemical composition of the mixture. It also helps control other factors as discussed previously. As per the findings, it was found that more CO_2 can be captured with 1 gram of magnesium versus 1 gram of calcium. In addition, as large amounts of magnesium and calcium are available, more CO_2 is captured by 25% GGBS-kaolin mixture versus 20% GGBS-kaolin mixture (Cai and Liu, 2017).

As per this study, about 5% CO_2 was captured by kaolin-25% GGBS mixture; however, this is not regarded as the maximum capacity of the mixture. In addition, the chemical composition of kaolin did not include any traces of calcium or magnesium, and hence the presence of GGBS causes sequestering of CO_2 . Thus, in conclusion, GGBS with 4.9% magnesium and 47.5% calcium can capture around 20% CO_2 from its dry weight.

When this method of acid digestion was compared with the thermogravimetric analysis, identical volume of the CO_2 that has been sequestered in the treated soil was found.

However, compared with TGA analysis, such type of acid digestion method is regarded to be more reliable when it comes to quantification of CO₂. This could be due to the following two reasons: first, usage of inhomogeneous powder in TGA analyses, which may not signify each sample, and second, high volatility of nitrate acid could have resulted in the release of large quantities of heat during the process of digestion amongst the acid as well as treated Kaolin (Liu *et al.*, 2017). In this method of acid digestion, the employed mass of the soil was found to be around 40 g, or 25% of the UCS sample mass in total, compared to just 10 g pertaining to the sample used for TGA analyses.

The CO₂ sequestration based on carbonation of GGBS-kaolin mixtures is regarded to be significant. For future work, greater than 25% GGBS can be added to kaolin as it is regarded to sequester additional CO₂. Mixing of other various types of soil could be done with GGBS in order to establish the role played by the soil during the carbonation process. An alternative approach (by-products and waste material rich with calcium and magnesium) to substitute GGBS can be identified as this would also be beneficial for recycling and managing of waste.

4. Conclusion

This study objective includes determination of GGBS' ability to stabilise soil as well sequester carbon dioxide simultaneously. The following conclusion can be drawn:

1. As per the TGA analysis as well as acid digestion method, it was revealed that the quantity of CO₂ that was sequestered within the GGBS-kaolin carbonation mixture was in the range of 4.3%–5% with regards to the mix's dry mass.
2. With rise in the content of GGBS in the mixture, the amount of CO₂ that has been sequestered within the kaolin-GGBS mixture was also found to increase.
3. More CO₂ was yielded with the samples that were left for curation for 7 days prior to carbonation in order to allow hydration of GGBS storage versus the samples that were placed directly under the carbonation cell.
4. Applying 200 kPa CO₂ pressure results in sequestration of additional CO₂ when compared with applying 100 kPa CO₂ pressure. This can be attributed to the increment in the pore solution acidity when applying greater CO₂ pressure. This results in release of additional magnesium and calcium ions for carbonation, thus resulting in sequestering of additional CO₂.
5. Compared with TGA analysis, the acid digestion method is regarded to be more reliable when it comes to quantification of CO₂. This can be attributed to the following reasons: high volatility pertaining to nitrate acid that results in the release of large quantities of heat during the digestion process, the inhomogeneous powder employed in TGA analysis and the higher amount of sample employed in acid digestion method (10 grams for TGA and 40 grams for acid digestion method), which help achieve more accurate results.
6. Previous study (Liu *et al.*, 2017) demonstrated that 20% reactive magnesia, when mixed with various soil type, would capture almost 8%–16% CO₂ from the mixture dry mass. This was found to be higher versus the current study (4.3%–5%), because of larger content of MgO in reactive magnesia (80%–90%) versus GGBS (CaO=47% and MgO=4.88%), which results in additional CO₂ sequestration. There are two benefits associated with GGBS when compared with reactive magnesia: first, it being a sustainable material, and second, reactive magnesia production results in higher CO₂ emission versus GGBS.

Acknowledgements

The authors are grateful for the support offered by MOE and UTM with Vote no. FRGS/1/2019/TK10/UTM/02/21 R.J130000.7851.5F197.

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