Development and characterization of novel biochar-mortar composite utilizing waste derived pyrolysis biochar

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Abstract— Industrialization and urbanization have increased exponentially over the decade. This have caused serious impact on the environment due to the vast consumption of energy and eco-unsustainable production. Therefore, scientists and engineers are placing more effort of designing better environmentally sustainable systems. To achieve sustainability, one potential solution is to use renewable resources to produce construction materials. In this research, biochar, a carbonaceous solid material produced from the waste source poultry litter, is utilized as a renewable resource to replace cement content while making mortar which is being used in the construction industry. The mechanical properties of mortar was investigated through different tests. Three major tests were used to analyze cement-biochar composite. The tests are compressive strength test, Fourier transform infrared spectroscopy (FTIR) and water absorbance test. A total of 60 samples were prepared for mechanical testing of biochar-mortar composites. The compression test revealed that with greater biochar replacement in mortar, compression strength was reduced. However, the 28 days cured biochar cement composites exhibited higher compressive strength than 12.5MPa, which is the minimum compressive strength requirement of mortar for structural use. The FTIR analysis showed that with increasing biochar replacement, less calcium silicate hydrates were formed in the biochar cement composites. The water absorption test showed that with increasing biochar replacement, more water is retained in the mortar-biochar composites. From the results, it can be concluded that biochar can be a viable alternative of cement, up to certain percentage, while making mortar for specific applications.

Index Terms— Biochar, Renewable resource, Mortar, Compression test, FT-IR analysis, Cement, Composites.

1 Introduction

Environmental sustainable production is one of the concern to environmental scientists and environmental engineers as industrial activities have increased exponentially over the decade. In New Zealand and many other countries, Portland cement is one of the most used construction material due to its low cost and raw material availability. However, the production of Portland cement is extremely resource and energy intensive. It is reported that every ton of cement requires about 1.5 tons of raw materials, simultaneously one ton of carbon dioxide is released into the environment as a result of the production. The two main points which contributes to the greenhouse gas emission during cement manufacture is fossil fuel combustion and calcination. Burning Fossil fuel is required during the manufacturing process to thermally decompose raw materials into calcium oxide, which is a key ingredient of making cement. Klee [1] has concluded that the annual production of concrete has crossed over 25 billion tonnes. Stolaroff et al. [2] has shown that cement is used as a binding element in concrete along with fine aggregate, water and coarse aggregate. Portland cement is made up of four main compounds: tricalcium silicate ($3CaO \cdot SiO_2$), dicalcium silicate ($2CaO \cdot SiO_2$), tricalcium aluminate ($3CaO \cdot Al_2O_3$), and a tetracalcium aluminoferrite ($4CaO \cdot Al_2O_3 Fe_2O_3$).

The two main points which contributes to the greenhouse gas emission during cement manufacture is fossil fuel combustion and calcination. Chemical equation of calcination is given below:

$$CaCO_3(s) \xrightarrow{heat} CaO + CO_2(g) \quad (1)$$

Burning Fossil fuel is required during the manufacture process to thermally decompose raw materials into calcium oxide, which is a key ingredient of making cement. The carbon dioxide emission from the cement industry alone, contributes about 7% of the global CO_2 emission annually (Oh et.al. [3]). According to Yang et al. [4], only cement is responsible for approximately 90% of the total emission of CO_2 in concrete. Therefore, it is necessary to find an effective method to alleviate the impact of cement production. Fly ash (Kabay et al. [5]), silica fume (Zhang et al. [6]), ground granulated blast furnace slag (Divsholi et al. [7]), waste glass (Aliabdo et al. [8]) has been used in concrete to partially replace the cement. Babu

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and Neeraja [9] investigated the effects of hen eggs shell inclusion in mortar with replacing cement by fly ash and recommended that the use of 0.25% hen shell and 55% fly ash improved compressive strength in a cost effective way.

One of the potential solution to reduce CO_2 emission in the cement industry is to add supplementary materials to replace the content of cement. These materials are usually industrial by-products like fly ash and steel slag, which helps to reduce the impact of greenhouse gases. Spiesz et al. [10] used waste glass, fly ash and ground granulated blast furnace slag in concrete to reduce the effect of Alkali-silica reaction (ASR) and also recommended the use of washed glass to have better strength and durability properties. Ling and Nor [11] used waste tyres in concrete paving blocks to improve the skid resistance. On the other hand, Modarres et al. [12] used different percentages coal waste ash to replace cement in concrete pavements and concluded that 5 to 20% replacement of coal waste powder improved the compressive strength of pavements. Recently the use of agricultural waste in concrete is becoming increasingly popular. Agarwal et al. [13] used bamboo to replace steel reinforcement in concrete and concluded that bamboo can be used in concrete without compromising the tensile strength of concrete. However, the raw inclusion of biomass can emit carbon dioxide which leads to the climate change. (Kriker et al. [14]). As a consequence, it would be perfect to have an environmentally sustainable material that can reduce the ratio of cement and on the other hand, enhance the properties of mortar and concrete.

There are different types of organic waste such as: municipal waste, industry waste, and agricultural waste etc. These are mostly non-recyclable. Therefore it is very important to either decompose this type of waste or to produce useful composites mixing with these waste products, which has potential application. Pyrolysis is one of the methods to convert organic waste into biochar. Biochar, a biomass-derived carbonaceous solid material obtained from thermochemical conversion of biomass in an oxygen-limited environment (Shackley et al. [15]) Use of biochar in soil improvement in agricultural lands and horticulture is well established by Zhu et al. [16] and Vaughn et al. [17]. Biochar can also be used in waste water treatment as studied by Lee et al. [18]. According to Zhao et al. [19], biochar can be used as an effective asphalt binder as it contains higher surface area. There are three main advantages of using biochar as a supplementary material:

1. The production is clean and does not impose carbon footprint on the environment
2. It is not combustible as it is thermos, chemically stable.
3. It can be produced by utilizing waste such as poultry litter that would otherwise go to landfill or waste plants. Thus, making it an environmentally sustainable material to use.

To date, however, very limited research has been done to investigate the feasibility of using biochar in mortar and its potential for carbon storage. Therefore, the aim of this work is to investigate the effect of biochar as a replacement of cement in mortar, on its strength properties and determine the optimal percentage, while making mortar for specific applications.

2. Methodology

2.1 Test requirements of mortar samples

To be able to utilise biochar cement composite in the construction industry, the strength and durability must comply with the structural standards. In New Zealand concrete masonry manual, it is specified that the minimum required compressive strength of mortar in structural masonry is 12.5 MPa at 28 days of curing [20]. In order for biochar cement mortar to meet the compressive strength requirement, proper sample preparation and testing are essential.

In New Zealand concrete masonry manual, it is highlighted that proper proportion of mortar materials is important as it influences the durability of mortar. Common problems of incorrect material proportion can result in cracking, absorption and reduced bond strength. A guidance of sand to cement ratio is given in the manual and is showed in table 1. The mortar ingredient values in table 1 are measured by volume instead of weight (CCANZ, New Zealand concrete masonry manual: mortar and mortar joints [20]). Ordinary Portland cement (OPC) is one of the most common binders used to develop construction materials. The three key elements of OPC are Lime (CaO), Silica (SiO_2), and Alumina (Al_2O_3). CCANZ [20] provides an estimate of the oxides content limits of OPC as shown in Table 1.

Table 1: Oxide content present in the ordinary Portland cement used in this study

Oxide	Content (mass %)
Lime (CaO)	60 – 67
Silica (SiO_2)	17 – 25
Aluminia (Al_2O_3)	3 – 8
Iron Oxide (Fe_2O_3)	0.5 – 6
Magnesia (MgO)	0.1 – 4.5
Alkalis (Na_2O+ K_2O)	0.5 – 1.3
Titania (TiO_2)	0.1 – 0.4
Phosphorous (P_2O_5)	0.1 – 0.2
Gypsum (expressed as SO_3)	1 – 3

2.2 Curing methods

Manufacturing techniques can also change the mechanical properties of the mortar. In particular, curing time and curing methods would have the most significant effect on the compressive strength of mortar.

Ban and Ramli [21] compared the effects of curing for 7 days versus curing for 28 days. The results indicated that samples cured for 28 days yield a higher compressive strength to those cured for 7 days (See Figure 1). Thus by having a longer curing time, the mortar is able to settle and harden more. Furthermore, this also develops greater binding which in turn increases the compressive strength of the mortar.

Another study conducted by Sajedi and Razak [22] analysed the effects of different curing methods on the compressive strength of OPC mortars. Their research provided insight into

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Durability	Portland Cement	Hydrated Lime	Mortar Sand
M4 Very High	1	0-0.25	3
M3 High	1	0.5	4.5
M2 Medium	1	1	6

the difference in compression strength of OPC mortars that were air cured (under room temperature) and water cured for 7 days to 90 days; results are shown in Figure 2. However due to the constraint of having a low water-cement ratio, a super plasticizer (SP) was used in the process.

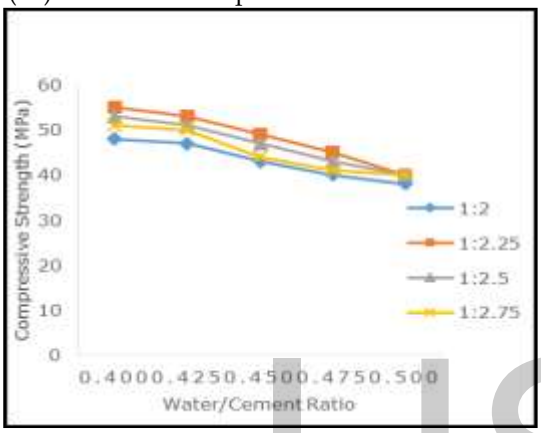


Figure 1: Effect of water to cement ratio and cement/sand mix configurations on compressive strength (Reproduced from Ban and Ramli [21])

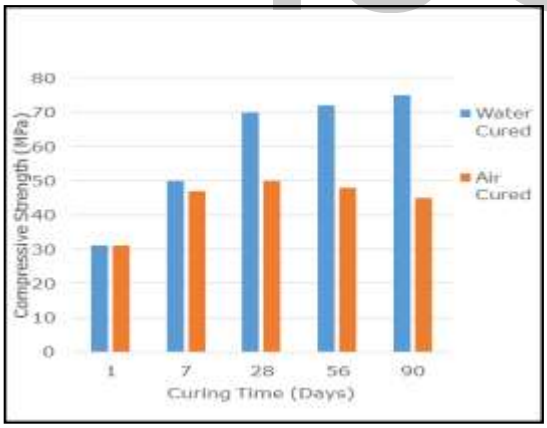


Figure 2: Comparison of water cured and air cured for ground OPC mortar (Reproduced from Sajedi and Razak [22])

2.3 Mix design

According to ASTM C39 [23], a guidance of sand to cement ratio is given in the Table 2. The mix design ratios considered in this study, were varying in both cement-sand ratios and also water-cement ratios. The cement-sand ratio was 1: 2.25 and the water cement ratios ranged from 0.46. This is shown (Ban and Ramli [21]) to be the optimal mix ratios to achieve desirable compressive strength of high performance mortar.

Table 2: Mortar mix ratio according to ASTM C39 [23]

3 Experimental Investigation

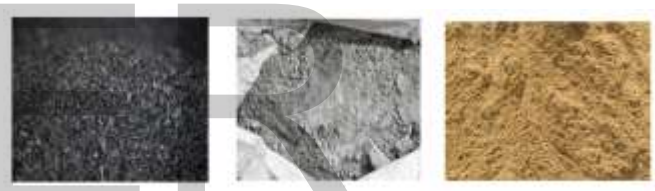
Experimental investigations has been conducted in the following phases:

3.1 Sample preparation

Materials

Biomass origin used to make biochar was gathered from poultry litter (PL). The process involved a thermochemical conversion of the waste under an oxygen limited environment (pyrolysis). A slow pyrolysis technique was conducted under 450°C. An alternative enhanced poultry litter (EPL) variation of the biochar was also produced which included bentonite clay as an additive. EPL biochar was primarily developed for comparison purposes against ordinary PL biochar.

The remaining materials used for our mortar samples were cement and sand (fine aggregates). General purpose Golden Bay Portland cement, available in New Zealand was used as the binder. The chemical compositions along with oxide contents present in ordinary Portland cement used in this research are shown in Table-1. Bio-char mortar ingredients are shown in Figure 3. Also shown in Table-3, are the density of biochar, cement and sand used to make the mortar samples in this study.



(a) Biochar (b) Cement (c) Sand

Figure 3: Biochar-mortar ingredients

Table 3: Biochar-mortar material characteristics

Biochar	Cement	Sand
Density= 700 kg/m³	Density=2080 kg/m³	Density = 2090 kg/m³

3.2 Moulding and Curing

Cylindrical moulds, as shown in Figure 4, (102 mm × 200 mm) were used to cast all mortar samples. This is in compliance with most regulatory test standards [20, 23].

Based on the dimensions of the mould and mix ratios, quantities of biochar, cement and sand, were calculated. Mix design ratios considered in this study are shown in Table-4. Mix ratios involved one sample as the control which did not contain any biochar, followed by increasing quantities of biochar replacement. Three samples for each mix were produced and subjected to water curing for 7 and 28 days (See Figure 5), to achieve greater accuracy in the results. Water curing was used in this study as it is more effective than air curing in terms of compressive strength [22].

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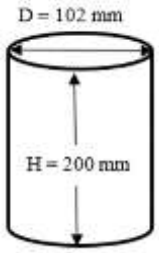

Sample	Cement (% volume)	Biochar (% volume)	Sand (% volume)
Control	30	0	70
PL/EPL1	29	1	70
PL/EPL5	25	5	70
PL/EPL10	20	10	70

Figure 4: Mould dimension
Table 4: Mix design ratios by volume for all samples

Figure 5: Sample preparation and curing

3.2 Compressive strength

A conventional static loading compression test was carried out on all samples to evaluate the compressive strength of biochar-mortar. Standard testing procedure mentioned in ASTM C39 [23], was followed. Figure 6 shows, photograph of the test rig where cylindrical concrete specimens (102 mm × 200 mm) were loaded with an increasing loading rate of 1 kN/S. Failure load was recorded to calculate the compressive strength of the samples.Three specimens were tested for each of the mix design after 7 and 28 days of water curing.

Figure 6: Photograph of compression testing machine

3.3 Fourier transformed infrared spectroscopy (FT-IR)

Fourier transformed infrared (FT-IR) spectroscopy had been well acknowledged in building material science for the last few decades (Ghosh and Handoo [24]). It involves scanning of the sample to output an absorbance spectrum. Based on the peaks in absorbance on particular wavelengths, relevant compounds and functional groups can be identified. The higher the absorbance level indicates greater number of bonds and or functional groups.

This research focused only on the attenuated total reflection (ATR) method for FT-IR as it does not involve altering the sample before scanning. Other FT-IR testing methods involve chemically transforming the samples for enhanced spectra. (Chollet and Horgines [25]). By performing the FT-IR test, functional groups and chemical compounds present in the mortar samples, can be identified. These in turn would help to explain the mechanical properties of the composites such as the compressive strength. Ring mill machine was used to convert the samples into powdered form to maintain the homogeneity of the samples. Figure 7 shows the FT-IR apparatus used in this research. The spectra was obtained and analysed in the range of 800 cm^{-1} to 4000 cm^{-1}wave numbers at a scan rate of 64 with a spectral resolution of 4 cm^{-1}.

Figure 7: FT-IR apparatus

3.4 Water absorption

A standard water absorption test was also conducted as per ASTM C642 [26], to find the water retention capabilities of the mortar samples. The samples of dimensions 75 × 75 × 250 mm were prepared for water absorption tests and specimens were water cured for 28 days. The dry weight of the specimens was initially recorded and then moist weight was recorded after 28 days.

4. Results and Discussion

4.1. Compressive strength

The compressive strength of PL and EPL samples after 7 and 28 days is compared in Figure 8 and 9 respectively. The

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trend in both the figures showed the compressive strength of all EPL samples are higher than the PL samples. It was expected as EPL has the addition of bentonite clay to improve biochar's mechanical strength. Both figures also showed that with increasing biochar percentage, the compressive strength of samples decreases. As biochar amount reaches 10%, compressive strength is significantly reduced in comparison to the control sample.

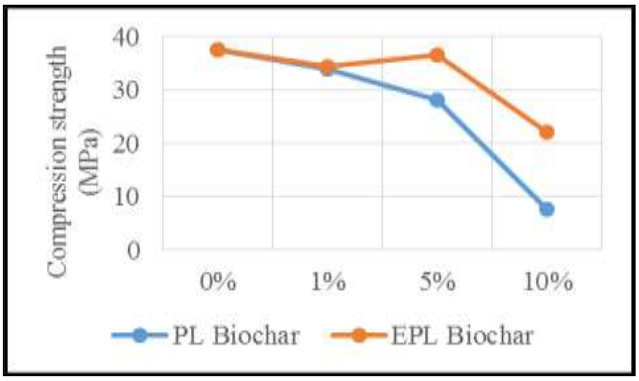

Figure 8: Compressive strength comparison of PL and EPL biochar-mortar after 7 days of curing

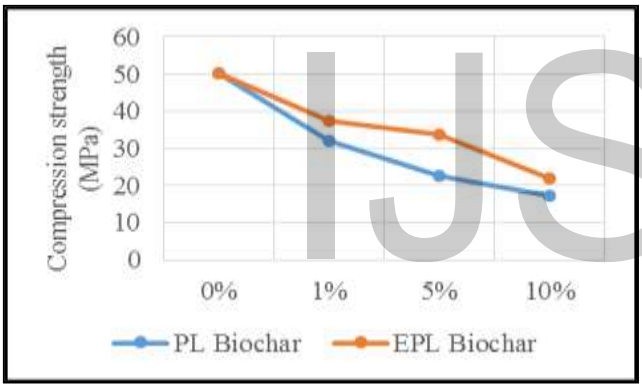

Figure 9: Compressive strength comparison of PL and EPL biochar-mortar after 28 days of curing

Also can be seen in Figure 8, when EPL is increased from 1 to 5 %, the compressive strength of EPL-biochar mortar is increased. The compressive strength of EPL 5% is slightly lower than the control. This is because of enhanced poultry litter biochar acting as a self-curing agent. In Figure 10, the change in compressive strength over different curing periods are plotted for all samples. All the specimens, including 10% biochar replacement, are able to achieve a compressive strength higher than the minimum requirement of 12.5 MPa [20] thus being fit for purpose of mortar in the structural industry. The primary source of strength in mortar comes from cement hardening. With increasing biochar quantities, the amount of cement reduces, which explains the lower strength of the composites compared to the control. Compressive strength results shown in Figure 11 are all subjected to 28 days water curing. Also from Figure 11, it can be observed that biochar composites reached maximum compressive strength quicker than the control sample. The control sample gained an additional 10 MPa between 7 to 28 days whereas almost all the biochar composites reached the peak compressive strength within 7 days.

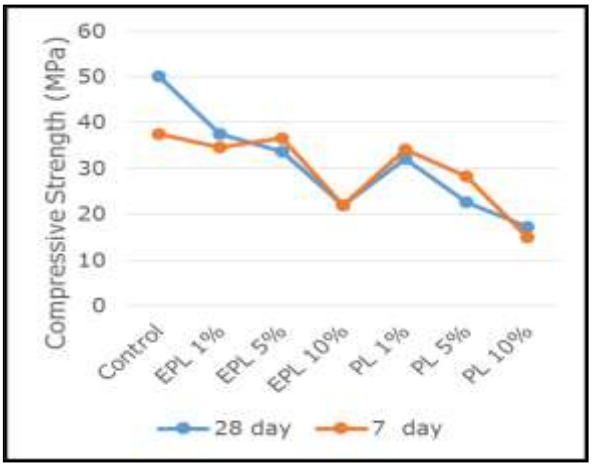

Figure 10: Change in compressive strength over different curing periods

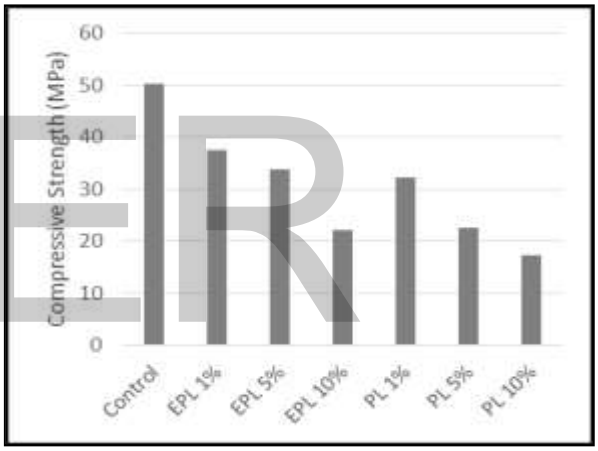

Figure 11: Compressive strength after 28 days curing for different samples

4.2. Fourier transform infrared spectroscopy

Looking at the absorbance spectra of the mortar samples of the FT-IR tests can provide reasoning for the compressive test results. Figure 12 shows the absorbance spectra of three samples that achieved the largest compressive strength, control, EPL 1% and PL 1%. The primary indicator of strength can be linked with the alkyne bonds (C ≡ C) in the 2300 cm^{-1} region. Control sample had the largest absorbance for this wavelength, thus it had greater amount of alkyne bonds resulting in higher strength compared to EPL and PL 1% samples.

When considering the strength difference between EPL and PL samples, firstly it can be observed that the presence of bentonite clay additive in EPL, increases its compressive strength. However, it is also important to note that EPL 1% had greater calcite and silica bonds than PL 1%. Calcite and silica are the sources of strength during hydration (Bediako et al. [27]) which can also be attributed to the

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higher compressive strength of the EPL samples.

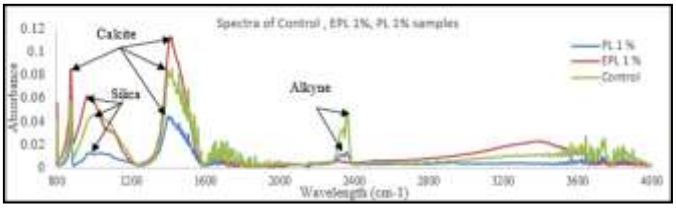

Figure 12: Absorbance spectra of Control, EPL 1% and PL 1% samples

In Figure 13, it showed clear sharp peaks at between 800 to 1600 cm^{-1} and at 2500 cm^{-1}. As can be seen, Ca-O and Ca-Si-H bonds are present in the control sample. The bonds developed in the control sample are expected as Portland cement contains silica (SiO2) and lime (CaO).

In order to analyse the new bonds present in the samples due to the addition of biochar, comparison can be made using the control sample and the biochar samples. In Figure 14, comparison is made between control, PL1% and EPL1%, it shows different absorbance values but have peaks at similar wavelength number. This indicates both PL and EPL biochar samples have the same type of bonds developed like the control sample.

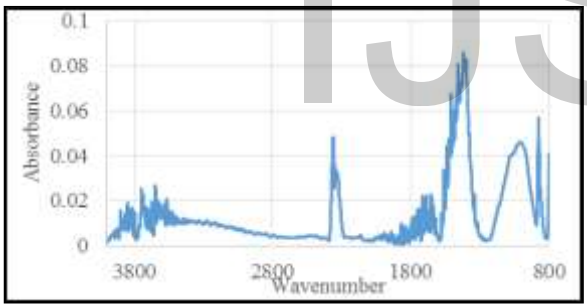

Figure 13: FTIR scan of the control sample

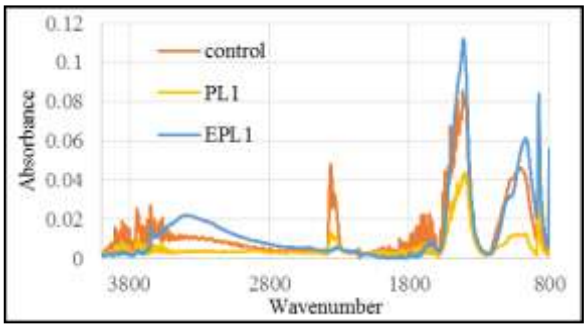

Figure 14: Comparison of FTIR scan of control, PL1 and EPL1

In Figure 15, a comparison of the control sample with PL10% and EPL10%, is shown. It is evident that the peak at 974 cm^{-1} are diminished for both biochar samples when compared to the control. The peak at 974 cm^{-1} represents the bond of Ca-Si-H bonds. For concrete and mortar samples, Ca-Si-H bonds are responsible for compressive strength development during the curing phase. This means at 10% biochar addition, the formation of Ca-Si-H bonds are significantly interrupted. Thus, it can be concluded that overdose of biochar can be detrimental to the compressive strength gain of mortar.

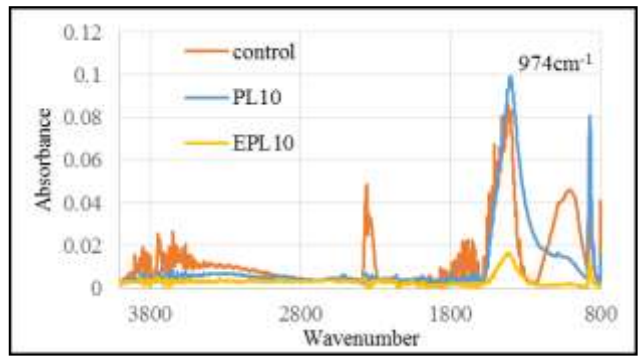

Figure 15: Comparison of FTIR scan of control, PL10 and EPL10

4.3. Water absorption

In Figure 16, it is shown that water absorption of the biochar samples is higher than the control sample. With volumetric addition of 10% biochar, the water absorbance of mortar is doubled. This is expected as biochar has great water retention and absorbance characteristics. The properties of biochar can be advantageous to withstand long term deformation of mortar due to shrinkage. Shrinkage is a phenomenon of losing moisture content in concrete or mortar over time, causing a reduction of weight and size. The water absorption and retention properties of biochar can be used to help resist moisture evaporation and shrinkage. When comparing the water absorption of PL biochar and EPL biochar, the absorbance values are approximately identical. It can be concluded that the addition of bentonite clay in EPL biochar has no influence on the water absorption of biochar-mortar.

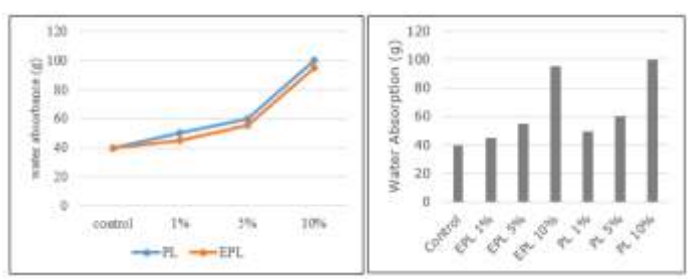

Figure 16: Water absorption capacity of control, EPL and PL biochar composite specimens after 28 days curing

4.4. Environmental benefit analysis

Cement industry is also a large contributor to the greenhouse gas(GSG) emissions with 4000 Mt of CO_2 re-

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leased into the atmosphere annually.A large benefit of replacing cement with biochar for structural materials is that the mass reduction of cement. Biochar has significantly lower density than ordinary Portland cement, the density of biochar is almost one third of cement. In this study, for every 1% increase by volume of biochar, there was a 2.2% reduction of cement by weight. Thus if we take the case of replacing 10% biochar by volume, the mix contained 22% less cement by weight compared to the control sample. If this is taken to the macro scale of the cement manufacturing industry, we can reduce the amount of CO_2 emissions by 880 Mt per annum by replacing 10% biochar on a volumetric basis. Also, the total worldwide GHG emissions levels would reduce by 1.1% which would be a significant step towards using sustainable materials.

5. Conclusions

Biochar has numerous use specifically in the environmental field such as toxin removal, water absorbance and soil amendments. However, the author adopted a novel approach of utilizing biochar for structural purposes in this research. Based on the research and the results of using biochar as a supplementary material in mortar, the following conclusions have been made.

1. According to CCANZ, the minimum required compressive strength of mortar in structural masonry is 12.5 MPa at 28 days of curing. For all biochar cement mortar cured at 28 days, all of the samples have met the minimum compressive strength requirement. Whilst the trend showed reduction in strength, biochar composites obtained up to 95% the compressive strength of ordinary mortar.

2. The lower density of biochar in comparison to cement results in a larger mass replacement. If 10% of biochar is replaced on a volumetric basis, 880Mt of CO_2 emissions can be mitigated per year reducing 1.1% of total emissions worldwide.

3. When comparing PL biochar mortar with EPL biochar mortar, EPL mortar gives higher compressive strength. This is due to the interlocking bonding properties of bentonite clay EPL biochar.

4. FTIR analysis showed that overdose of biochar can be detrimental to compressive strength gain in mortar. This is because less Ca-Si-H bonds are formed in the mortar samples. The results from compressive test also justifies this, as biochar mortar samples have less compressive strength compared to ordinary mortar. Higher absorbance peaks of silica and calcite for EPL mortar samples suggested a greater number of bonds influencing the increased strength compared to PL mortar.

5. Water absorption test showed that the biochar mortar samples exhibit great water retention and absorption compare to the ordinary mortar. Biochar mortar has potential to resist shrinkage which is a common problem for ordinary mortar. Also, the water absorption capabilities of biochar composites were significantly greater than ordinary mortar control sample. This has increased environmental benefits by reducing the runoff that damages fresh water bodies.

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