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Evaluation of the influence of accelerated carbonation on the microstructure and mechanical characteristics of coconut fibre-reinforced cementitious matrix

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Abstract: In this study, two methods were used to measure the carbonation depth of coconut fibre-reinforced mortars that were prepared from Portland cement (PC) and calcium sulfoaluminate cement (CSA cement), including traditional phenolphthalein reagent and thermogravimetric analysis (TGA). Accelerated carbonation was conducted at 4% CO₂ concentration, temperature of 20°C and 65% relative humidity (RH) within 12 weeks. Complete carbonation was detected in the CSA cement-based mortar while PC-based mortar was carbonated partially according to both methods used. The difference in carbonation depth, however, is observed as soon as the reagent and TGA measurement were applied. The significant effectiveness of the carbonation process on the microstructure changes and mechanical characteristics of the cementitious matrix is highlighted through analysis of the change in the quantity of calcium carbonate (CC) and portlandite (CH) on the different mortar profiles and comparison with natural cured samples.

Keywords: coconut fibre, mortar, carbonation, phenolphthalein, TGA measurement.

1. Introduction

Green materials are investigated because of their eco-friendly advantages. Utilization of natural fibre in reinforced composite can reduce not only the dependency on conventional concrete-making materials but also the impact on the environment. Natural fibre selection is in conjunction with the concept of sustainability as the sources are abundant over the world, the main part in tropical regions. In addition, the use of natural fibre in the construction industry could partially reduce the amount of agricultural waste that usually ends up in landfills. In fact, the use of natural fibres for reinforcement in construction materials allows decreasing 65% of the greenhouse gas footprints, generates less carbon emission and gives the lowest energy consumption compared with cement and lime [1,2]. In another way, the use of agriculture products could bring substantial gain, reducing resource consumption and waste production.

However, it is not easy to recommend and employ such material for construction because of problems to solve as long-term properties and durability. The significant reduction in the long-term mechanical performance has been examined and identified in the natural fibre-reinforced composite in aging due to deterioration of the incorporated natural fibres under cementitious environment [3–5]. In recent years, carbonation was well indicated to achieve mitigation of degradation of natural fibre and to improve the durability of fibre-based cement composite [6–10]. In nature, the reaction between carbon dioxide CO₂ and cementitious composites occurs very slowly due to the CO₂-poor environment, ranging from 0.03-0.04 % [11,12]. Therefore, it takes at least one year in accordance with XP P 18-458 French standard or even four years [13] to evaluate the influence of carbonation on resistance of such composites. Experimentally, accelerated carbonation was highly recommended to significantly decrease the time period of carbonation curing [14]. This accelerated method consists of creating an environment in which the concentration of CO₂ is considerably higher than the natural environment with selected experimental conditions, *i.e. environmental conditions*.

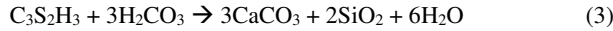
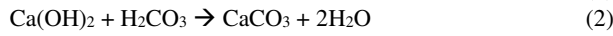
Ambient environmental conditions, including CO₂ concentration, temperature, relative humidity (RH), and the duration of exposure as well, play crucial role in the carbonation process and have significant effects on the carbonation of cementitious composite materials. Parameters considered in several previous studies on accelerated carbonation are listed in Table 1 with their experimental conditions and objectives.

Table 1. A literature review concerning accelerated carbonation.

Reference	Binder(s)	Concentration of CO ₂ (%)	Testing conditions		Max. exposure time	Properties investigated
			Relative humidity RH (%)	Temperature (°C)		
XP P 18-458		50 ± 5	65 ± 5	20 ± 3	1 week	Carbonation depth
Accelerated carbonation test – French standard EN 12390-10 – European standard		0.040 ± 0.001	65 ± 2	20 ± 2	1 year	Carbonation depth
EN 13295 – European standard		1	60 ± 10	21 ± 2	56 days	Carbonation depth
Lee et al.[11]	PC, fly ash	5	60	20	1 year	Carbonation degree
Georget et al.[12]	PC	0.040	70	Not mentioned	6 months	Micro-structure characterisation
RILEM Committee CPC-18 [13]		0.03	65	20	4 years	Carbonation depth
PERFDUB project [15]		3 ± 0.5	65 ± 5	20 ± 2	70 days	Carbonation depth
EN 12390-12 – European standard		3 ± 0.5	57 ± 3	20 ± 2	70 days	Carbonation depth
Tang et al.[16]	42.5 PC	20 ± 2	70 ± 5	20 ± 5	4 weeks	Carbonation depth
Qin et al.[17]	42.5 PC, fly ash, GGBS, limestone powder	20	60 ± 5	20 ± 3	4 hours	Mineral composition, mechanical properties,
De Weerd et al.[18]	PC, fly ash	1	60	20	9 weeks	Carbonation depth, moisture content, mineral composition
Ashraf et al.[19]	Calcium silicates	15	94	35, 45 and 60	145 hours	Carbonation kinetics
		100	94	60	120 hours	Microscale mechanical performance
		100	94	60	145 hours	Macroscale mechanical performance
Leemann et al.[20]	PC, fly ash, slag	4	57	20	9 weeks	Mechanical properties, carbonation coefficient
Zhu et al.[21]	PC	100	70	20	24 hours	Mechanical properties, mineral composition
Romildo et al.[22]	OPC	9.8	64.3	26.7	109 days	Durability
Tonoli et al.[23]	PC	100	75	20	7 days	Physical and mechanical properties
Tomography et al.[24]	PC	20 ± 3	70 ± 5	20 ± 5	28 days	Carbonation degree, crack distribution, pore structure
Neves Junior et al.[25]	High initial strength and sulfate-resistant PC	20	60	25	24 hours	Mechanical and porosity properties
Mi et al.[26]	Cement	0.04	70	20	28 days	Distribution of carbonation

Note: (O)PC = (Ordinary)Portland cement.

All these studies have suggested that the accelerated carbonation is a useful means to the sequestration of CO₂ and an effective procedure to diminish the alkaline environment prematurely in the cementitious composite. This is due to the process of carbonation reaction in cementitious matrix that carries out as follows:



The degree of carbonation is the main concern of some previous studies with inconsistent observations depending on the binder type and test condition. After carbonation storage, the surface of sample could be divided into three different zones depending on CO₂ absorbed amount in theory and CO₂ uptake in sample, including the non-carbonated zone, the partially carbonated zone and the fully carbonated zone [27]. Complete carbonation was observed (i) for CEM III-based concrete under natural carbonation after 3.7 years [28], (ii) for portlandite at temperature of 20°C and 91% RH and 1% CO₂ concentration [29]. Whereas partial carbonation was detected (i) for a polypropylene fibre-cement composite with 15% of CO₂ concentration, at 60°C and with 90% relative humidity [30], (ii) for an autoclaved aerated concrete under conditions of 20°C, 90% relative humidity and 3% of CO₂ concentration after 20 days [31] or (iii) for OPC-based concrete with the conditions of 23°C, 70% relative humidity and 20% of CO₂ concentration within 16 weeks [25].

Several methods for the measurement of carbonation depth are applied, from simple to cutting-edge technology methods. However, the traditional and most popular parameter used to measure carbonation depth is the use of a suitable indicator [32]. Some reagent solutions were highly recommended like phenolphthalein [33,34] or thymolphthalein [11,35]. These indicators are sprayed on the tested surface of samples. The change of the pH value in samples leads to the change in colour of the indicator. But more recent conclusions [36,37] believed that even when the reduction of the pH value due to the carbonation process could be made visible clearly by the colour change of phenolphthalein pH reagent, that area can still be considered as only underwent partial carbonation. Therefore, in order to limit the drawbacks of the reagent solution, it is necessary to use simultaneously other methods for improving the accuracy of carbonation depth measurement. Some methods were suggested in previous studies such as thermogravimetric analysis [6,17,18,38], images analyses [24,39], X-ray diffraction [27,29], infrared spectroscopy [29] or using fibre optic chemical sensors [40]. Among them, digital image analysis was believed to be the most quickly and inexpensive technique for determination maximum carbonation depth [41].

Among accelerated carbonation testing conditions, the CO₂ concentration is considered as a main factor for the laboratory carbonation test [32,42]. It should be noted that CO₂ concentration is the largest difference between natural and accelerated carbonation conditions, inducing the laboratory process used to replace natural carbonation. With the CO₂ concentration of under 5%, Neves et al. [43] have found that a linear relationship between carbonation depth and CO₂ concentration was observed. Otherwise, when the CO₂ concentration was beyond 5%, this relation differed significantly since the carbonation depth - CO₂ concentration curve could be described by a square root function [44]. Moreover, Auroy et al. [45] reported that 3% CO₂ concentration was the most suitable content for accelerated carbonation in the laboratory to investigate and compare the consequences of the natural environment and accelerated carbonation on the properties of cementitious matrix. Statistically, most of the research was carried out with relative humidity range of 50 - 75 % to get a higher carbonation rate [29,46,47]. Ceukelaire et al. [46] determined the role of relative humidity condition in the carbonation process of concrete by using a series of different relative humidity levels from 40 to 90 % with an increment of 10%. They indicated that the increase in relative humidity induces an increase in the carbonation resistance. In detail, with relative humidity of 50%, a maximal rate of carbonation was obtained, expressed by maximal carbonation depth of 8.5 mm after 21 days of exposure in 10 % CO₂ while only 1.4 mm was recorded for the same time exposure in natural carbonation. Otherwise, these values with the relative humidity level of 90% dropped to only 4.9 mm and 0.5 mm for accelerated and natural carbonations, respectively. The presence and abundance of free water caused by high relative humidity could diminish the rate of CO₂ diffusion and leads to a slow carbonation rate [48]. As well as, Elsalamawy et al. [47] reported that relative humidity and carbonation depth relationship corresponds to a polynomial function, showing a carbonation depth peak obtained with an approximately relative humidity of 65% for all four samples tested, regardless of the types of cement and water/cement ratio used. This seems to be corroborated by the result in ref. [49] which found that the highest carbonation rate reaches at relative humidity level of 65%, proving the ability of increase the rate of carbonation process in moderately saturated moisture conditions.

Meanwhile, in the report of Weerdt et al. [35], the higher carbonation rate was observed in mortar based on Portland-fly ash cement (30 wt.% fly ash) compared to PC-based mortar at similar controlled conditions. The observed increase in carbonation rate could be interpreted as being a consequence of the fly ash reaction resulting in the reduction of portlandite and formation of mono-carbonate, which made lower pH value in Portland-fly ash cement-based mortar [50,51]. The dependence of water/binder ratio on the carbonation degree was also investigated in the literature [11,52]. The increase in water/binder ratio enhances carbonation depth for all the types of binder used. The higher water/binder ratio provokes the

formation of larger pores that contributes to the carbon ion penetration deeply into sample. This is a convenient environment to promote the carbonation process.

Concerning the relationship between the mechanical strength and accelerated carbonation of composites, the carbonation process could make the compressive strength of cementitious matrix higher in comparison with the samples cured under natural conditions whatever the types of added minerals, especially at the early age [53]. This is mainly owing to the formation of CaCO_3 from the transformation of calcium hydroxide Ca(OH)_2 that fills pores to mitigate the porosity in the cementitious matrix. These results provided additional support for previous findings in the literature [54–58]. In contrast, some other studies [25,37,47,49,52,59] believed that the compressive strength of the cementitious composite was decreased at the higher carbonation degree levels. They believed that the reduction of the pH value during CO_2 curing process has negative effects on the reinforcement and leads to decrease in the mechanical performance of composite. The carbonation process causes a structural change in the CSH phase of cementitious composite, which could induce both the increase in strength and carbonation cracking. This complicated change leads to the prediction effects of carbonation on the mechanical performance of cementitious materials become very difficult [60].

Regarding thermogravimetric analysis, several studies [9,61] also confirmed that when samples are subjected to the carbonation process, the conversion of Ca(OH)_2 into CaCO_3 could result in mitigation of the pore size in the cementitious system. Therefore, at higher carbonation degree levels, samples show a better resistance to high temperature due to the change in microstructure of the carbonated sample. For instance, the amount of Ca(OH)_2 diminishes as the quantity of CaCO_3 enlarges along the carbonation process and decreases the porosity of composite. Additionally, the disintegration of Ca(OH)_2 occurs at the temperature range from 450°C to 550°C while the weight loss at the temperature above 550°C only concerns CaCO_3 . The reaction of Ca(OH)_2 to acquire CaCO_3 could lead to the reduction in alkalinity of the cementitious composite, create a less aggressive environment to protect natural fibres in better condition [22]. Therefore, thermal, and chemical stabilities are considered as two main advantages of carbonated products.

In present study, the influences of incorporating natural fibre and cement type on the carbonation development of mortars were determined by means of carbonation depth measurement, mechanical performance, and pyrolysis behaviour. With the usage of two different types of cement and four replacement amounts of coconut fibres, the effects of the accelerated carbonation on the microstructure change and mechanical characteristics of mortars were investigated experimentally. The outcomes of this present study could contribute to having a better understanding in carbonation resistance of fibre-reinforced mortar and the influence of accelerated carbonation on behaviour performance of the cement-based materials.

2. Materials and experimental methods

2.1. Materials

Coconut fibres from Vietnam were used in present study as raw fibres. Firstly, pith, coir dust, and undesirable materials were manually removed to select raw fibres. These fibres were then cut into 10 - 20 mm length, as shown in Fig. 1. The specifications of fibres were identified in previous study [62] and given in Table. 2.



Fig. 1. Appearance of fibres before mixing.

Tab. 2. Fundamental properties of coconut fibre [62].

Parameters	Value
Diameter (mm)	0.250
Length (mm)	10 - 20
Absolute density (g/cm^3)	1.41
Water absorption (%) (after immersion of 48 hours)	133
Tensile failure (MPa)	123.6
Failure strain (%)	26.9
Water content (%)	7.60

Mortars were manufactured based on two types of cement, CEM I 52.5 N type I Portland Cement (PC) and Calcium Sulfoaluminate cement (CSA Cement). CSA cement, containing approximately 55% of calcium sulfoaluminate, is considered as a green binder due to its environmentally friendly characteristics, i.e., 37% lower of CO_2 emission emitted than PC [63]. Indeed, CSA cement is produced at a lower calcined temperature and less energy requirement for grinding

procedure than PC due to the easier grinding of CSA clinker [64]. In addition, CSA cement is appraised as a special cement with lower alkali content, i.e., pH range of 10.5-11 and 12 – 13 for CSA cement and PC, respectively. This pH value range for CSA cement could result in reducing the degradation of natural fibres in the mortar composite. The main physical properties and oxide compositions of the two binder types and sand are presented in Table 3. CHRYSO Fluid Optima 352 EMx was added with a ratio of 1% by mass of cement as a superplasticizer for a significant water reduction and improvement of the workability of the fresh mixture in accordance with the suggestion of Yan et al. [65].

Table 3. Chemical composition, fineness, and density of raw materials.

	PC	CSA	Sand
Loss on ignition	2.2	3.37	0.3
SiO ₂	19.4	10.55	99.6
Al ₂ O ₃	4.6	18.28	-
CaO	64.3	45.07	-
Fe ₂ O ₃	3.9	-	-
MgO	1.2	0.76	-
K ₂ O	0.86	-	-
Na ₂ O	0.06	-	0.0051
TiO ₂	0.3	-	-
P ₂ O ₅	0.3	-	-
SO ₃	2.6	16.53	-
S ²⁻	<0.02	-	-
Cl ⁻	< 0.007	0.04	< 0.001
Blaine fineness (cm ² /g)	4200	5100	-
Density (g/cm ³)	3.16	2.96	2.64

2.2. Mortar mix design

In present study, the mortar mix design was produced according to EN 196-1 standard. The incorporation of fibres consists of substituting a volume of sand by a corresponding volume of fibre with a constant total mass of fibres and sand of 1350g. The fibres incorporation ratios were 0% (without fibres), 1%, 2% and 3%, percent expressed in volume of mortar. A mixing procedure was carefully applied to achieve the best homogeneous dispersion of fibres in the mixture.

With the usage of two different cement types and four replacement levels of fibres, eight mixtures were performed. The detailed compositions with the mixture references (cement symbols are followed by the ratios of fibres) are presented in Table 4.

Tab. 4. Mix proportion of coconut fibres cementitious composite.

Mortar reference	PC (control)	PC1 (1% fibre)	PC2 (2% fibre)	PC3 (3% fibre)	CSA (control)	CSA1 (1% fibre)	CSA2 (2% fibre)	CSA3 (3% fibre)
PC (g)	450	450	450	450				
CSA cement (g)					450	450	450	450
Water (g)	225	225	225	225	225	225	225	225
Sand (g)	1350	1339.20	1328.41	1317.61	1350	1339.20	1328.41	1317.61
Superplasticizer (g)	4.5	4.5	4.5	4.5	4.5	4.5	4.5	4.5
Raw fibres (g)	0	10.80	21.59	32.39	0	10.80	21.59	32.39

2.3. Experimental methods

2.3.1. Samples under accelerated carbonation

After the period curing of 14 days, mortar samples were placed in the carbonation chamber, as showed in Fig. 2. Based on XP P 18-458 – French standard, present study proposes to use the carbonation chamber installed at 4% CO₂ concentration, the temperature of 20°C and 65% relative humidity (RH). The time in the storage chamber is 12 weeks.

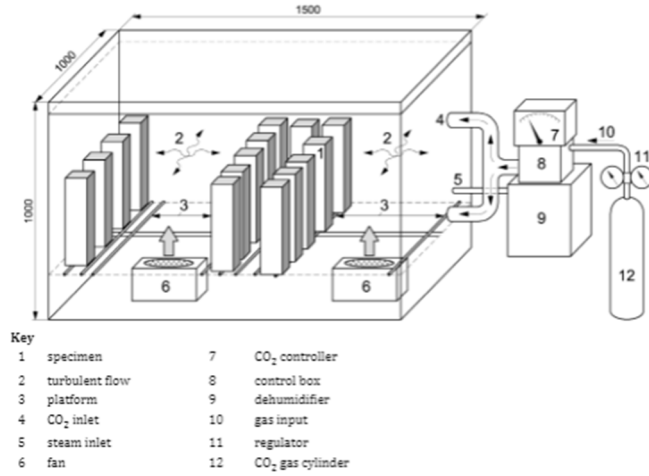


Fig. 2. Carbonation storage chamber.

2.3.2. pH indicator

1g phenolphthalein powder was dissolved in the mixture of 30 ml deionized water and 70ml ethyl alcohol. This solution was then used for the carbonation depth measurement in accordance with the guidance of RILEM Committee CPC-18 [13] and EN 13295. The clean freshly broken surface was sprayed with this solution. Due to the relative effects of the measuring time on the measured depth of carbonation after the reagent application, measurement one day after spraying was considered in present study when the limit between carbonated and non-carbonated zones was clearly demonstrated. The reagent could give a sufficiently clear colour change in sample to differentiate the neutralized areas. Phenolphthalein turns red-purple in the non-carbonated zone. Otherwise, it remains colourless in the carbonated zone (pH value in the range from 8.3 to 9.5 [11]).

2.3.3. TGA-DSC measurement

During the sample preparation process for TGA-DSC (Thermogravimetric analysis - Differential scanning calorimetry), the grinding along the profile was carried out from the external (outside) surface to the central part of sample with an increment of 2 mm. Considering the 40 mm sample height, ten profile ground sections were obtained for each sample. Thermal gravimetric analysis of unreinforced and fibre-reinforced mortars was performed on a thermal analyser (STA 449 F5, NETZSCH) in the temperature range of 20°C – 900°C in nitrogen atmosphere with the speed of 50ml/min. The rate of heating was kept constant at 20°C/min. Sample mass between 50 mg and 60 mg was put in a platinum crucible for this measurement.

Due to the thermal decomposition of portlandite (CH) at the temperature ranging between 450°C and 550°C and that of calcium carbonate (CC) at the temperature ranging of 550°C - 850°C, the amount of two minerals is expressed in following equations in accordance with refs. [6,18,66]:

$$C_{CH} = \frac{m_{450} - m_{550}}{m_{550}} \times \frac{M_{Ca(OH)_2}}{M_{H_2O}} = \frac{m_{450} - m_{550}}{m_{550}} \times \frac{74}{18} \quad (1)$$

$$C_{CC} = \frac{m_{550} - m_{850}}{m_{850}} \times \frac{M_{CaCO_3}}{M_{CO_2}} = \frac{m_{550} - m_{850}}{m_{850}} \times \frac{100}{44} \quad (2)$$

When the carbonated process occurs, CH converts gradually into CC, depending on the rate of CO₂ diffusivity. This means that the mass of CH decreases while the amount of CC increases when the carbonation process of mortar samples is happening. The carbonation degree could be determined based on the quantity of CC and CH. At the depth on which the appearance of both CH and CC is obtained, this zone is considered as carbonation zone. Otherwise, the amount of CC is negligible at the greater depth of sample from which the carbonation process almost does not occur. Hence, TGA analyses constitute a suitable and accurate method to evaluate the change in carbonation degree along the profile of sample sections.

In this study, phenolphthalein indicator and TGA-DSC measurement were performed on mortar without fibres and with 2% fibres.

2.3.4. Mechanical strength testing

Mechanical strengths of mortars were conducted based on NF EN 196-1 standard. The bending test was performed on hardened 40 × 40 × 160 mm³ prisms by means of an electromechanical press. Three samples were tested after the exposure period at a constant rate deformation of 0.05 kN/s with a capacity of 50 kN load cell.

The compression test was performed on halves of the prismatic broken samples after the bending test, i.e., six samples. The same press machine was used. However, the load cell of 250kN was installed. As recommended by the NF EN 196-1

standard, the loading rate applied was 2.5 kN/s. The half-prisms tested were placed laterally in the centre of the bearing plate of the machine.

3. Experimental results and discussions

3.1. Depth of carbonation

Reagent method

Fig. 3 and Fig. 4 show the typical images of the observed surface of samples after being covered by phenolphthalein reagent. As can be seen in these images, PC-based mortar makes the colour change in a part of sample, while CSA cement-based mortar does not present any change in colour in its whole surface. This means that PC-based mortar was only partly carbonated, while complete carbonation was observed in the mortar based on CSA cement. PC-based mortars showed relatively higher carbonation resistance but have a lower ratio of Ca/Si in comparison with CSA cement-based mortars. This behaviour could be justified in part by the difference in the amount of CaO and SiO₂ between PC and CSA cement. In fact, CaO and SiO₂ in cement convert into calcium silicate hydrate gel (CSH) and portlandite (Ca(OH)₂) due to the hydration acceleration. More CH content results in higher alkalinity in the pore solution of mortar based on PC, which results in change of its colour to red-purple in the inner part of surface after the exposure to phenolphthalein reagent. On the other hand, due to whole CH content converting into CC, remaining colourless in carbonated mortars in CSA samples was obtained as shown in Fig. 3. This result also evidences that the carbonation resistance in CSA cement-based mortar was insignificantly governed by the dense microstructure formed by ettringite [52]. Some previous studies [29,67,68] also highlighted the ratio of Ca/Si influences on the carbonation behaviour. They reported that with a higher ratio of Ca/Si, a faster carbonation degree was obtained in consequence of the rapid decalcification of calcium silicate hydrate gel (CSH). Additionally, CaCO₃ formation results from the decomposition of ettringite [69,70], which is the main phase of CSA cement, and induces to boost the depth of carbonation in CSA sample.

Other studies [42,52] also indicated that carbonation depth in concrete with CSA cement as the main binder is considerably higher than that in concrete manufactured with other binders such as Portland or blast furnace cement. This value, for example, in ref. [52], was up to 15 mm after a seven-week period of carbonation exposure compared with 6 mm and 5 mm of PC-based concrete and blast furnace cement-based concrete, respectively.

Concerning the CO₂ penetration, the fibres could improve the carbonation rate of mortar due to the high air content (the fibres act an air-entraining admixture), which induces CO₂ penetration becomes easier. It can be observed from the images in Fig. 4, the carbonation degree of the control mortar is higher than that of mortar incorporating fibres, showed by a weaker colouration on the surface of reinforced mortar. When mortar with fibres addition is employed, the carbonation degree of mortar could be increased.

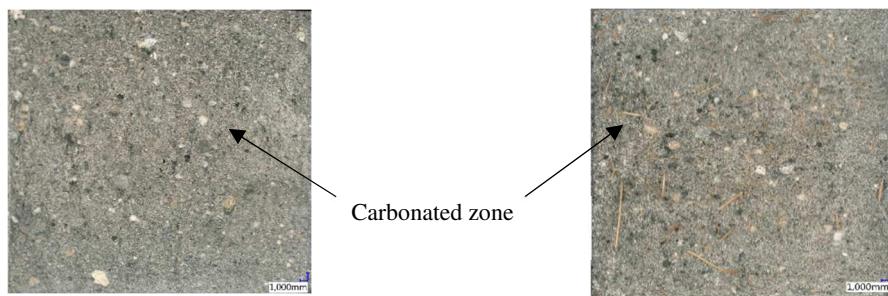


Fig. 3 Images of CSA (left) and CSA2 (right) samples surfaces after application of phenolphthalein solution.

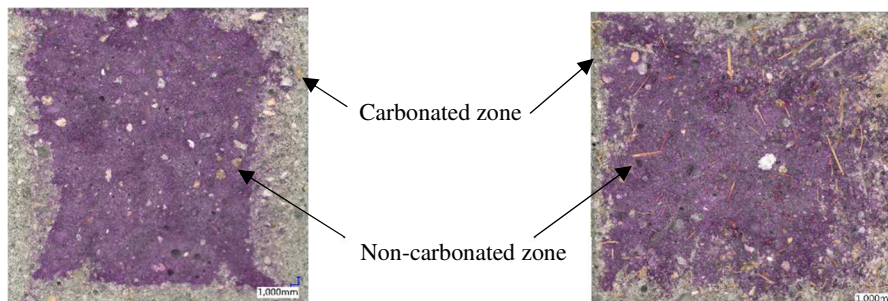


Fig. 4. Images of PC (left) and PC2 (right) samples surfaces after application of phenolphthalein solution.

To confirm the pH value reduction due to accelerated carbonation, an additional pH measurement was done with samples under natural curing. The technique used to obtain pH value of mortars bases on the recommendation of ASTM D4972 standard. A mixture of 10 g air dried mortar powders and 10 g distilled water was stirred within 12 hours at ambient temperature using a hotplate stirrer (UC125D, STUART) to facilitate lime powder in mortar to dissolve totally in water. This

solution obtained was then filtered with the filter paper of 25 μm pore size. The pH measurement of these mixtures was carried out by means of pH meter. The results of the pH value for mortars based on two types of cement are given in Fig. 5. It is observed that the pH of mortar based on CSA cement is significantly smaller than that of PC-based mortar due to the difference in pH of CSA cement and PC. In addition, the pH value of mortar seems to be not governed by fibre content.

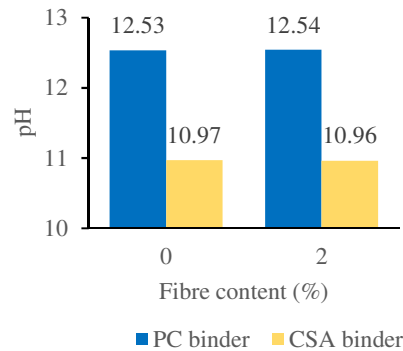


Fig. 5. Effect of type of cement on pH values for PC and CSA cement-based mortars.

TGA-DSC measurement

Fig. 6 illustrates the measured CC and CH contents using TGA-DSC measurement, depending on the depth from the surface to centre of sample. For PC2 sample, where a part of area was exposed by carbonation, a clear decrease in CC content was detected while the amount of CH measured was increased gradually. This means that the exposure to carbonation has significant effects on the degree of clinker reaction. This induces the two opposite trends in CC and CH contents from the outer surface to the middle part of the carbonated samples observed. More deeply, the CO_2 penetration was more complicated and almost restricted in the outer sections due to the dense microstructure impeding. On the contrary, the concentration distribution of CH and CC in CSA2 sample, which was carbonated completely, was almost constant over the sample whole depth.

Additionally, the amount of CC in the carbonated area of PC2 sample was higher than that of CSA2 sample, but lower in the non-carbonated area. A stronger carbonation degree for PC2 sample compared to CSA2 sample in outer sections was obtained. This observation could be explained by CaO content in the two types of binders. The higher content of CaO in PC could induce the higher content of CC in the carbonation area. Also, because of the low pH value and low porosity of CSA cement-based mortar [63], the CO_2 diffusivity becomes more difficult even in the outer sections. Nevertheless, the difference happened upon the non-carbonated area and the lower content of CC in PC2 sample was observed. The cause of this phenomenon is that clinker reaction under carbonation curing during 12 weeks increases the amount of CC created, which makes the higher content of CC in CSA2 sample than in PC2 sample within non-carbonation sections. Otherwise, due to the low CH content [71], the consumption of CH in CSA samples were almost completed after 12 weeks of accelerated carbonation.

It may be viewed in Fig. 6, the different depth of carbonation according to two methods, i.e., one using phenolphthalein reagent and the other, the thermogravimetric analysis for sample powder profiles, was observed. The ratio between depth on which both CC and CH were detected, and the maximal depth of colourless zone was approximately three. A reasonable explanation for this difference may be the heterogeneity of carbonation front depth in the entire sample induced by the differences in the internal structure of composite and diffusivity of CO_2 and the powder sampling approach [35]. Another possible cause is the carbonation degree. While phenolphthalein indicator remained colourless only in the fully carbonated zone, where the carbonation degree was above 50%, the appearance of both CH and CC was detected by TGA technique even in partly carbonation zone, i.e. where the carbonation degree is less than 50% [27]. The restricted use of the traditional method is to focus only on the change in the pore solution alkalinity of the carbonated sample, and apparently, it lacks the correlation with other criteria [49]. Therefore, the depth of colourless zone is mostly underestimated [32,41] and smaller considerably than the carbonation depth identified by other measurements. However, phenolphthalein method could offer a simple way to investigate the carbonation-induced difference concerning alkalinity that occurred at the surface of sample applied. In contrast, TGA method shows a sufficient level of measurement accuracy in relation to CH and CC concentrations distribution of CH and CC in each profile section.

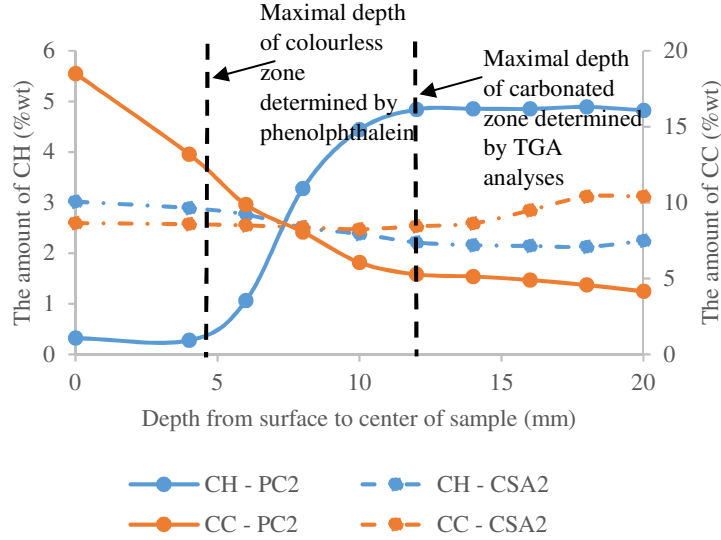


Fig. 6. CH and CC concentrations distribution over the depth of PC2 and CSA2 samples.

3.2. Mechanical properties

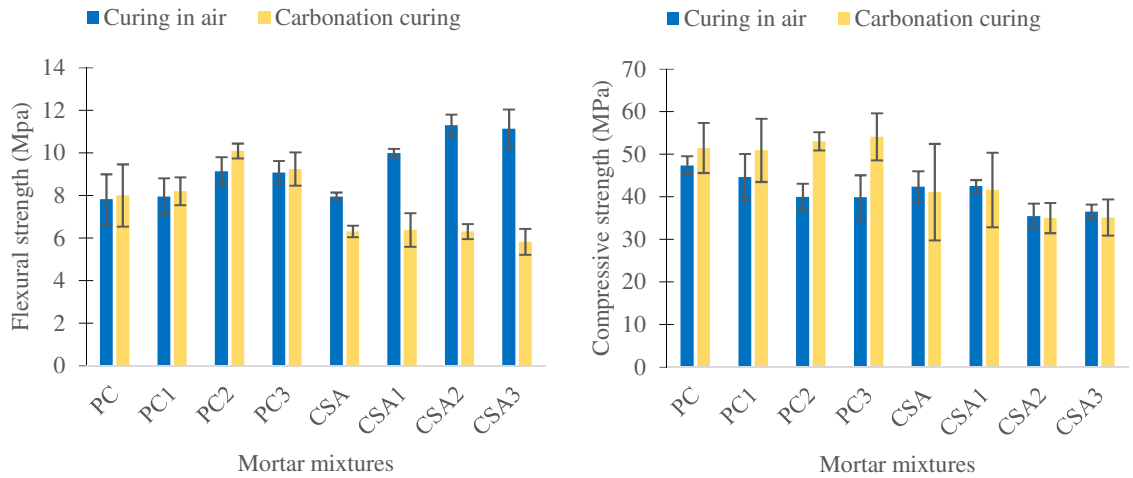


Fig. 7. Mechanical strengths of mortar samples cured in two different conditions.

Concerning the PC mortar samples with and without coconut fibres, the 12-week carbonation reaction process improved the compressive strength by 8.7% and 32.7%, respectively. For CSA cement-based mortars, which were higher carbonation depth, the compressive strength of control and fibre-reinforced samples decreased slightly by 3% and 1.3%, respectively. The carbonation process of CSA cement-based mortars occurred on complete area tested, and then led to the degradation of ettringite in a short time. This could result in a slight decrease of compressive strength in CSA samples after 12 weeks of storage in carbonation chamber as shown in Fig. 7. Chi et al. [54] also indicated a logical relationship between carbonation depth and mechanical strength since they concluded that the compressive strength decreased at higher carbonation depth. This could be contributed by the pore structure of composite, which gains remarkable changes after accelerated carbonation, controlling both the carbonation degree and compressive strength totally. It is clearly seen that this relation depends significantly on the type of binder used in composite [20,47,72]. Thus, compressive resistance should not be used as a main determinant of carbonation resistance since samples contain different binder types.

The effects of carbonation on flexural strength are different for both CSA cement and PC-based mortars. A relative drop in flexural resistance was observed in mortar containing CSA cement after carbonation storage. Accelerated carbonation-induced flexural strength of CSA cement-based mortar was only approximately 6 - 6.5 MPa, either incorporating fibres or

without fibres. Flexural strength of CSA mortar samples incorporating fibres decreased significantly by nearly a half. This may be due to the cumulated effects of incorporating fibres and tensile strength of cementitious matrix (loss of ettringite), which affects remarkably the bending behaviour. Otherwise, flexural strength of PC-based mortar showed a slight upward trend, regardless of fibre content.

Comparison with PC based-mortar without fibres, PC based-mortar incorporating fibres showed a higher development in both compressive and flexural strengths. This phenomenon can be caused by the higher porosity in fibre-reinforced mortar, which is a convenient environment for CO₂ to penetrate deeply into sample, and the carbonation degree occurs more rapidly than in reference mortar. It is reported that the carbonation reaction seems to be governed considerably by CO₂ diffusion process. Porosities in sample are filled and the sample becomes denser after accelerated carbonation is applied in mortar samples [9,61]. It is clear that the presence of fibres plays a critical role in the change of mechanical characteristics of mortar samples. The establishment of carbonation could result in the decrease of the alkaline environment prematurely in cementitious matrix. This critical advantage provides more chemical stability to the cellulose fibres due to a less aggressive environment [61]. In addition, after the accelerated carbonation is applied, the bonding between natural fibres and cementitious matrix is improved, resulting in a drop of the voids surrounding fibres. Due to this, mortars at higher fibre content could give better mechanical performance.

3.3. Thermogravimetric analyse

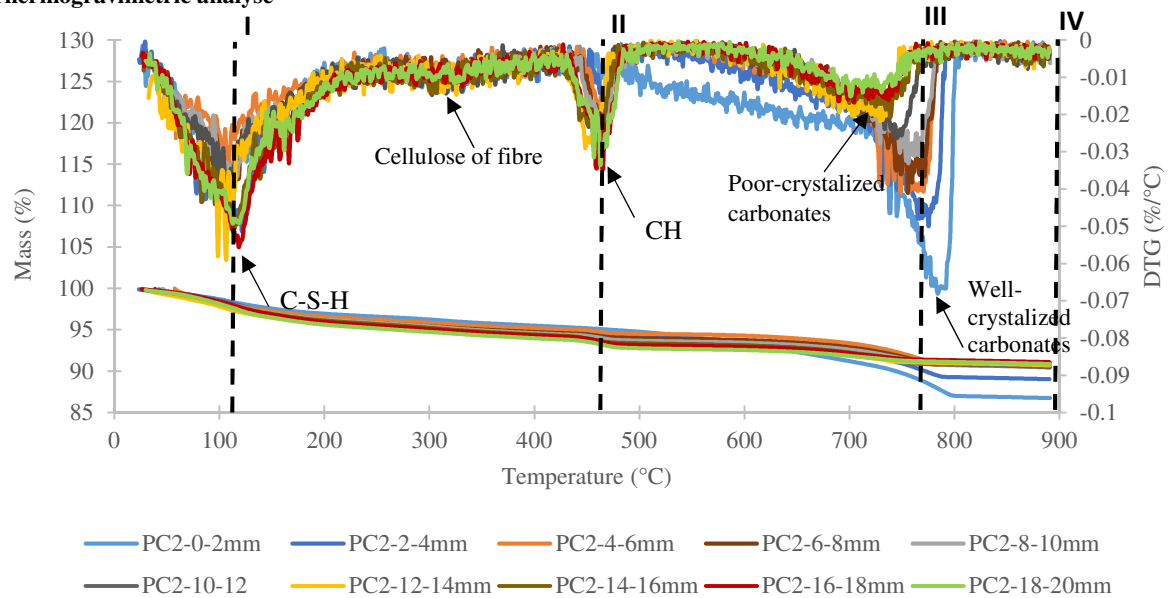


Fig. 8. TGA and DSC graphs for different profiles of PC2 sample after accelerated carbonation.

The results of thermogravimetric analysis and differential scanning calorimetry (TGA-DSC) from the surface to centre part of PC2 sample used in the carbonation test are presented in Fig. 8. With these graphs, the weight loss is identified in Fig. 9. Concerning the derivative of the thermogravimetric curve, three decomposition peaks were obtained, including CSH, CH and CC. The first two peaks indicate that CSH and CH content in the inner (inside) profile ground sections are higher than in outer ones (near the surface) while the last peak concerns the decomposition of CC, showing the amount of CC in outer sections is higher than that in inner ones. Due to the negligible mass of fibre powder compared to cementitious matrix powder, the decomposition peak regarding cellulose of fibre, which occurs in the temperature range from 300 to 360°C [30,62], was almost observed. When the coconut fibres are employed in reinforcement, these fibres are the lightest components in the mortar mixture, and the influence on the powder components is difficult to recognize. These quantity changes result from the reaction between HCO₃⁻ ions, which dissociated by the reaction of CO₂, and Ca²⁺ ions, and released by dissolution of CH, to form CC within the carbonation curing. The decomposition peaks of CSH and CH mainly occurred in non-carbonated and part-carbonated sections in the temperature range from 100 – 150°C to 400 – 500°C, respectively, and the strongest in PC2-16-18mm and PC2-18-20 mm profiles. While CC peaks almost occurred in carbonated areas in the temperature range from 700 – 800°C, the strongest peaks were found in PC2-0-2 mm and PC2-2-4mm profiles.

In terms of weight loss, at the first of the decomposition period, both non-carbonated and carbonated areas have very similar thermal behaviour. The pyrolysis behaviours of ten profiles were almost the same. As can be seen from the data results, carbonated sections present higher resistance to temperature than other ones from ambient to 550°C. This could be explained because carbonated sections have higher CC content decomposed only after 550°C. A significant difference in the pyrolysis behaviour was observed from 550°C when the weight loss after this temperature level was characterized by carbonation. The steeper curves after 550°C for the outer profiles thus clearly indicate the attack of CO₂ and the higher rate of weight loss due to accelerated carbonation [73]. The endothermal effects could be divided into four main steps: (I) 20 – 110°C (evaporation and CSH), (II) 110 – 460°C (CSH and cellulose), (III) 460 – 760°C (portlandite and poor-crystallized carbonates) and (IV) 760 – 900°C (well-crystallized carbonates). In detail, at the first step from ambient temperature to 110°C, decomposition of sections occurs slowly since the mass loss was about 2% for all profiles and mainly concerned free water content. The weight

loss changes at the temperature range between 110 and 200 °C significantly relate to the consumption of calcium silicate hydrate gel (CSH) advanced by the carbonation process with the CO₂ absorption capacity of the cement matrix. However, the degradation process happens mainly between 110°C and 760°C that a mass loss of 8% was observed. The step, happening between 350°C and 400°C, relates to cellulose degradation, and nearly no change was found between the tested profiles. Then the development of endothermal peaks from 450 to 550 °C correlates with the decomposition of CH. From 550°C, the changes were observed between the carbonated areas. The degradation process continues slowly at the last stage with the weight loss of about 2% for outer profiles and approximately 1% for inner ones. It gets a stable value of residue left at 900°C. At the end of the decomposition period, residual weight at 900°C of outermost profiles ranged of 86-90% is against slightly more than 90% of most in-depth profiles. A possible explanation for this could be that at the high temperature, the decomposition was in regarded to CC formed from the carbonation process in carbonated areas. Otherwise, the decomposition at this temperature in uncarbonated areas is mainly due to the amount of calcium-carbonated filler, which is already in the component of conventional cement to decrease the cost in cement production industry [61]. As a result of thermal analysis, it is observed that carbonated areas have a poorer ability to resist high temperature than other ones due to the strong CC decomposition in outer profile ground sections.

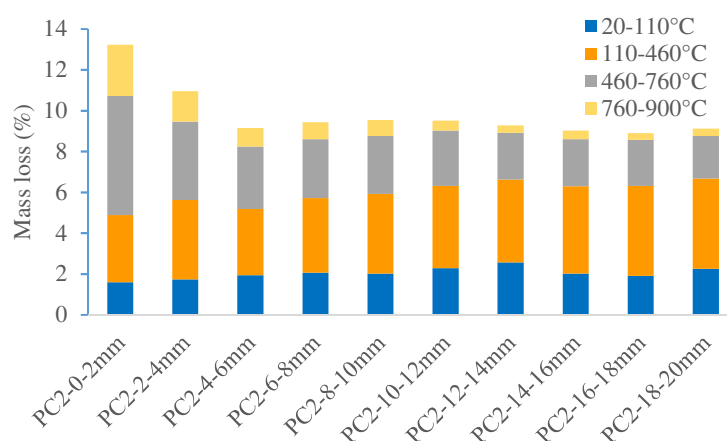


Fig. 9: Mass loss for different profiles of PC2 sample and transition temperature ranges.

4. Conclusion

Coconut fibre-reinforced mortar was prepared using two different cement types, *i.e.*, *PC* and *CSA* cement. The carbonation resistance was investigated in laboratory conditions. The comparison in terms of carbonation degree and mechanical performance between types of mortars was carried out. According to the test results, several conclusions can be summarised:

- Using *CSA* cement with lower alkali content could lead to a notable reduction in carbonation resistance due to the lower content of CaO compared to conventional cement. While the large inner surface area of *PC*-based mortar presents in red-purple, all broken surface of *CSA* cement-based mortar shows in colourless when they are sprayed by phenolphthalein reagent after the carbonation exposure period of 12 weeks.
- Incorporating natural fibres into mortar could lead to an increase in the carbonation degree due to the additional presence of air entrainment that supports strongly for CO₂ penetration into fibre-reinforced mortar.
- A difference in carbonation degree was obtained between two measurement methods used: phenolphthalein reagent and TGA measurement. The depth of colourless zone determined by phenolphthalein is nearly three times smaller than the carbonation depth obtained by TGA technique. Several drawbacks of the conventional method by the reagent were reported, but this simple method still suits to evaluate the change in alkalinity of pore solution in samples after the carbonation exposure. Meanwhile, TGA technique could give accurate results even if in the case powdered samples are representative only of different small areas, not of the entire sample.
- The carbonation has different effects on the mechanical performance of mortar. While carbonation-induced compressive strength of *PC*-based mortar increases to 32.7%, that of all mortars based on *CSA* cement decreases insignificantly. However, in bending, the flexural strength ratio of *CSA* cement-based mortar before and after carbonation storage is nearly two due to the cumulated effect of fibres added and loss of ettringite of *CSA* cement-base samples during the carbonation process. It should be noted that for composites containing different binders, mechanical behaviour could be not a key factor in evaluating carbonation resistance.
- Three decomposition peaks were clearly observed corresponding three decompositions, namely of CSH, CH and CC in the *PC2* sample, respectively. Meanwhile, inner profile (centre part of sample), are concerned by the two first decomposition peaks and outer (near surface) ones are responsible for the last decomposition peak of CC. This means that from the external surface to centre part of *PC2* sample, the increase in CSH and CH contents and decrease CC content were obtained.
- The thermal stability of the non-carbonated area is slightly higher than that of the carbonated area. For instance, at 900°C, the mass loss of the carbonated area is approximately 10 – 14%, while this value of the non-carbonated area is less than 10%. This could be due to the formation of CC, which is decomposed at the higher temperature during the carbonation process in the carbonated area. Other, the decomposition at this temperature mainly depends on the calcium-carbonated filler content in the production of conventional cement.

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