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PROPERTIES OF OPTIMIZED CEMENTITIOUS COMPOSITES INCORPORATE PRECIPITATED $CACO_3$ PARTICLES

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A B S T R A C T

Calcium carbonate and its derivatives are used broadly in the cement and construction industry. It hereby provides a reasonable and active solution for reducing the earth's carbon footprint. In the current work precipitated $CaCO_3$ (PCC) particles are prepared by precipitation technique, characterized, and added to cement at different concentrations (0.076-0.92) wt. % of cement, and w/c ratio: 0.36-0.50. The fresh cement composites were tested for setting time and temperature rise during the early hydration stage, and the hardened composites for compressive strength, density, and water absorption. The results obtained showed that at a constant w/c ratio increasing the content of the PCC up to 0.25 leads to an increase in the initial setting time, density, and compressive strength. However, more PCC content has negative effects on the properties. Increasing the w/c ratio increased the setting time and water absorption of the composites. Low concentrations of PCC lead to a decrease in the tendency of water absorption, while high concentrations showed diverse effects. At a constant w/c ratio, samples with PCC content (0.92 and 0.076 wt. %) showed temperature rising at about 25 and 75 minutes respectively after batching, while the control samples showed the temperature rising at about 105 minutes. The observations confirmed that the hydration reaction of both the control and cement composite samples incorporated with PCC are exothermic, and the PCC particles have a positive impact on the hydration rate. Overall, the incorporation of PCC in cement at appropriate concentration and w/c ratio contributes to obtaining strong and durable cement composite for structural applications.

1. INTRODUCTION

Compared to stone and steel, concrete is considered a newer construction material. It has been worldwide used for more than one century. Nevertheless, a broad range of cementing materials including Pozzolans (Robalo et al., 2021) and nanomaterials have been used to improve concrete mechanical properties and durability characteristics (Kamal et al., 2021), (Goel et al., 2022). Calcium Carbonate ($CaCO_3$) obtained directly by

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grinding limestone has been previously put under examination as a significant partial replacement for cement (Ali et al., 2015), (El Mendili & Benzaama, 2022), (Abed et al., 2023). However, powdered limestone is not a pozzolan and reduces compressive strength at higher doses**.** On the other hand, precipitated calcium carbonate of high purity and a specific crystal morphology could be used in the production of sustainable cement that provides swift comprehensive municipal development without the related emissions of cement manufacturing (Sawara et al., 2002), in addition, nano-calcium carbonate showed more significant effect on the hydration process, workability, strength, and durability properties of cementitious composites (Erdogan & Eken, 2017), (Muhsin & Fawzi, 2021). In addition, in massive concrete structures, the temperature alterations within the structure are substantial. Principally, the maximum temperature differential between the interior and exterior concrete should not surpass 20°C to avoid crack growth (Van Tran et al., 2023).

The current work aims to investigate the effect of the incorporation of nano and micro $CaCO_3$ on setting time and temperature rise in fresh cement paste and on compressive strength, density, and water absorption of the hardened cement.

2. EXPERIMENTAL PART

2.1 Materials

The ordinary Portland cement used is with chemical and physical properties conformed to Iraqi specifications and matched Iraqi Standards (Iraqi Standards, 2019). $CaCl_2$, Na_2CO_3, ethanol, and ethylene glycol were purchased from Merk.

2.2 Preparation of the precipitated CaCO3 and the experimental design

The precipitated $CaCO_3$ was prepared by precipitation technique following the method reported by Thapa et al. (2017) with some modifications. Table 1 shows the experimental design (10 experiments) with the actual levels of the operating variables used to prepare the cement composites. Other additional experiments were carried out to achieve the required plots. The operating variables investigated are the precipitated $CaCO_3$ content (% wt./ cement): 0.076-0.92, and the w/c ratio: 0.36-0.5.

Table 1. The experimental design and actual values of the operating variables

Exp. No.	1*	2	3	4	5	6	7	8	9
(wt.%) PCC	0.5	0.92	0.08	0.2	0.8	0.2	0.8	0.5	0.5
w/c	0.4	0.43	0.43	0.38	0.48	0.48	0.38	0.36	0.50

*Repetition

The fresh cement composites were tested for initial setting time and the rise in temperature during the early hydration stage (the first 4 hours directly after batching). The mixes were at adiabatic conditions during the test. The laboratory air temperature was 20-23°C. The hardened properties were tested for the dried cubes of age 28 days for compressive strength density and water absorption. Moreover, the morphology of the cement composites was evaluated using SEM.

3. RESULTS AND DISCUSSION

3.1 Characterization of PCC and the Cement Composites

The characterization of the PCC is shown in Figure 1. The XRF analysis of PCC samples is presented in Figure 1 a. The results confirmed that the PCC contains mainly CaCO3 (56.86% Cao) and small amounts of SiO2 (2.67%), Fe2O3 (0.25%), and other impurities.

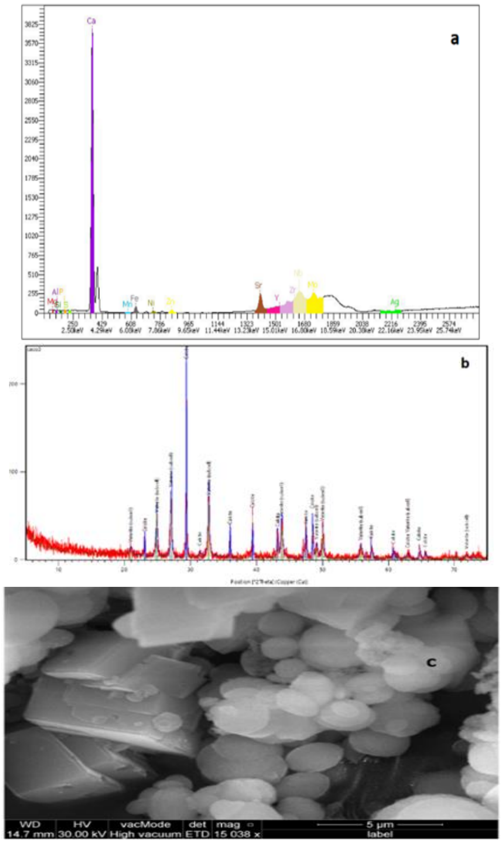

Figure 1. XRF, XRD, and SEM images of the prepared PCC

The results of X-ray diffraction are presented in Figure 1 b. The crystal structure of synthesized PCC shows that angles from 10 to 70 º . The diffraction data obtained predicted that the precipitate consists of a mixture of both vaterite and calcite were the most predominant polymorphs produced. The SEM shows that the PCC particles exhibited a spherical shape and good dispersal. diameter of about 2 µm. Meanwhile, some small nanocrystals can also be observed in Figures 1 c. The two occurring mineral phases, vaterite, and calcite, can clearly be distinguished by their characteristic morphologies. Vaterite can be recognized as framboidal spheres while calcite forms rhombohedral crystallites (Nebel et al., 2008). A considerable phase ratio for the tested samples is the framboidal spheres which are typical for vaterite (Schmidt et al., 2010).

3.2. Analysis of the experimental results

Figure 2 shows the variation of the initial setting time versus the PCC content.

The Figure showed that increasing the content of the PCC up to 0.25 leads to an increase in the initial setting time owing to the slower rate of the hydration process. Higher concentrations of the PCC exceeded 0.25 wt. % provide large reactive surfaces, which may act as a nucleation site and thereby stimulate nucleation reactions of hydration phase nuclei on their surface. Figure 3 shows the variation of the initial setting time with the w/c ratio. The setting time increases with the increase of the water/cement ratio. This may be attributed to that when the w/c ratio increases, the concentration of cementing material decreases, and the possibility of producing a greater gelling structure in the hydration reaction of slurry becomes small, so there will be a delay in cement set.

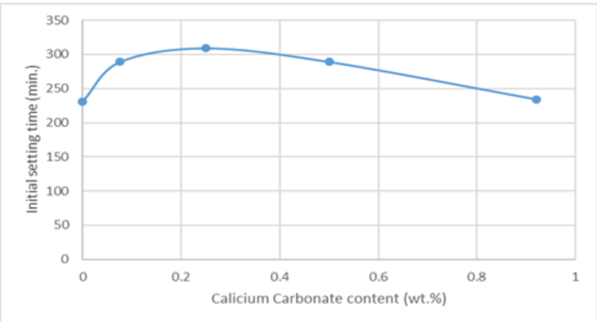

Figure 2. The variation of the initial setting time of cement composites (w/c =0.43) versus the PCC content

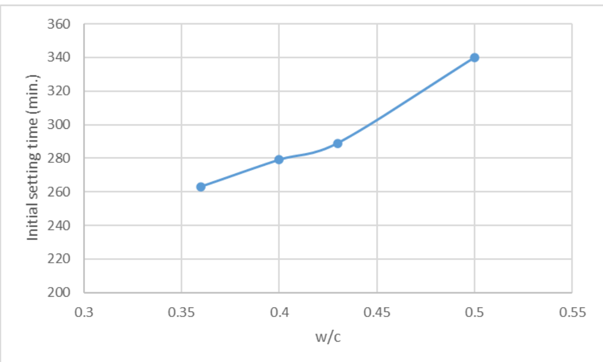

Figure 3. The initial setting time of cement composites (PCC content = 0.5 wt.%) as a function of w/c ratio

At the same time, the cement mixtures were tested for temperature variation during the early ages. Figure 4 shows the results from such a simple mock-up. It is noticed that at the early period, the temperature of the control sample showed a sharp and small rise then a steady decline stage followed by a sharp increase in temperature at about 105 minutes after batching, reaching a maximum increase at 210 minutes.

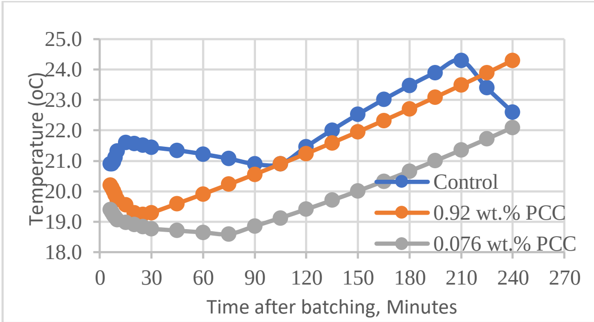

Figure 4. Temperature rises profiles for control and cement composites with different PCC content (w/c ratio is 0.43)

The cement composite with PCC showed different behaviour, samples with high PCC content (0.92 wt. %) showed the temperature rising at about 25 minutes after batching, while the samples with low PCC content (0.076 w.%) showed the temperature rising at about 75 minutes. The observations confirmed that the hydration reaction of both the control and with PCC are exothermic, and the PCC particles have a positive impact on the hydration rate and that impact became more effective with increasing the PCC content. The decline in sample temperature for the cement composites with PCC at the very early period may be related to the higher capacity of the PCC particles to dissipate the reaction temperature compared to cement constituents (Persson, 2023).

3.3. Analysis of the hardened properties

Figure 5 illustrates the trend of variation in the compressive strength of the cement composites with PCC wt.%. In the presence of water, calcium ions from PCC react with the silicate ions in cement to form calcium silicate hydrate, which is a gel-like substance that reacts with the surrounding cement particles to form a more rigid, crystalline structure that gives strength and durability to the final product.

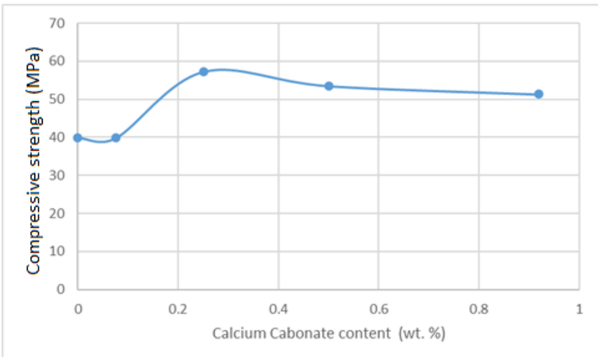

Figure 5. Compressive strength of cement composites (w/c= 0.43) as a function of PCC content

The reaction is represented in the following equation:

$$CaCO_3 + 2SiO_2 + 6H_2O \text{------>} Ca^{2+} + CO_3^{2-} + 2Si(OH)_4 + 4H^+ \quad (1)$$

However, too much calcium carbonate can have negative effects, so it is important to use the correct proportions of PCC and cement for any given application.

The same trend is observed for the density variation with PCC wt.% as illustrated in Figure 6, the density increases with the incorporation of the PCC up to 0.25 wt. %, then decreases beyond that as seen in Figure 6. Increasing the concentration of the PCC may lead to agglomeration of the fine PCC particles. The large agglomerates push away the cement particles around them, increasing the void space, and thereby decreasing the compressive strength as well as the density (Al Ghabban et al., 2018).

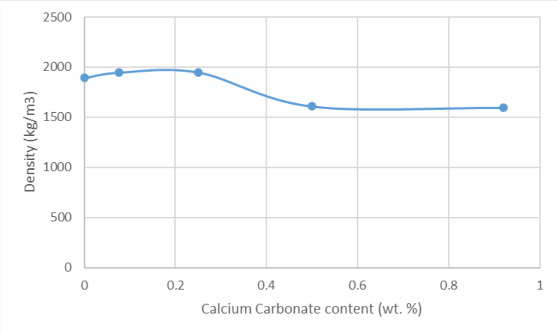

Figure 6. Density of cement composites (w/c= 0.43) as a function of PCC content

The water absorption test revealed that the cement composites incorporated with PCC generally exhibit a reduction in water absorption compared to control mixes as shown in Figure 7. The low water absorption values are attributed to the more compact and denser microstructure of the composites resulting from filling the pores with more hydration that contributes to the reduction of water absorption and hence, the durability of the composite. However, high contents of PCC particles were found to increase the water absorption capacity of the cement composites. The reason may be attributed to the agglomeration of the fine PCC particles. The large agglomerates drive away the cement particles around them, resulting in an increase in the void space, thus water absorption increases.

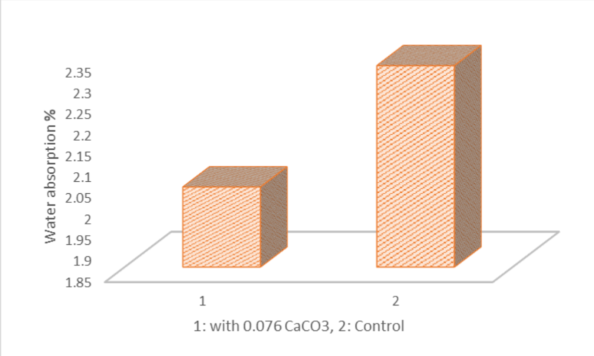

Figure 7. Water absorption % for a control sample and a sample incorporated with PCC

4. CONCLUSIONS

Increasing the earth's carbon footprint from cement and construction industry is well demonstrated. The challenge can be effectively solved by using different ways including decreasing the amount of the cement and using the appropriate type and content of additives. Calcium carbonate is one of the additives that subsidise to overcome some of the challenges face concrete sustainability. The current work used precipitated $CaCO_3$ (PCC) particles as cement additive. The results confirmed that using PCC in cement can contribute to decreasing the amount of cement used as well as modifying the cement properties. The incorporation of an optimum PCC particle content (0.25 wt.%) increased the initial setting time, density, and compressive strength and decreased water absorption. However, the development of the properties is affected by the w/c ratio. These factors showed a vital role in controlling the hydration reaction rate and the amounts of cement hydrates produced. Therefore, optimization of these factors is essential in the production of strong and durable cement composites incorporated with precipitated $CaCO_3$ particles for construction applications. Studying other durability properties and using the precipitated $CaCO_3$ particles in cement mortar and concrete is one of our upcoming research interests.

References

Abed, A. A., Kamal, I., & Mojtahedi, A. (2023). Enhancing Concrete Properties Using Silica Fume: Optimized Mix Design. *Journal of Smart Buildings and Construction Technology*, *5*(1), 84-91. https://doi.org/10.30564/jsbct.v5i1.5678

Al Ghabban, A., Al Zubaidi, A. B., Jafar, M., & Fakhri, Z. (2018, December). Effect of nano SiO2 and nano CaCO3 on the mechanical properties, durability and flowability of concrete. In *IOP conference series: materials science and engineering* (Vol. 454, p. 012016). IOP Publishing. doi:10.1088/1757-899X/454/1/012016

Ali, M., Abdullah, M. S., & Saad, S. A. (2015). Effect of calcium carbonate replacement on workability and mechanical strength of Portland cement concrete. *Advanced materials research*, *1115*, 137-141.

El Mendili, Y., & Benzaama, M. H. (2022). Investigation of mechanical and thermal performance of concrete with scallop shells as partial cement replacement: alternative binder and life cycle assessment. *CivilEng*, *3*(3), 760-778. https://doi.org/10.3390/civileng3030044

Erdogan, N., & Eken, H. A. (2017). Precipitated calcium carbonate production, synthesis and properties. *Physicochemical Problems of Mineral Processing*, *53*. doi: 10.5277/ppmp170105

Goel, G., Sachdeva, P., Chaudhary, A. K., & Singh, Y. (2022). The use of nanomaterials in concrete: A review. *Materials Today: Proceedings*, *69*, 365-371. https://doi.org/10.1016/j.matpr.2022.09.051

Iraqi Standard (IQ. S 5:2019) for CEM I (42.5 R).).

Kamal, I., Ali, A., & Sherwani, A. F. (2021). Optimization and modeling the impact of a green cementless binder and biogenic nanosilica on cement setting time. *Materials Today: Proceedings*, *42*, 2649-2655. https://doi.org/10.1016/j.matpr.2020.12.595

Muhsin, Z. F., & Fawzi, N. M. (2021). Effect of Nano Calcium Carbonate on Some Properties of Reactive Powder Concrete. *IOP Conference Series: Earth and Environmental Science, 856,* 012026. doi: 10.1088/1755-1315/856/1/012026

Nebel, H., & Epple, M. (2008). Continuous preparation of calcite, aragonite and vaterite, and of magnesium-substituted amorphous calcium carbonate (mg-acc). *Zeitschrift für anorganische und allgemeine Chemie*, *634*(8), 1439-1443. https://doi.org/10.1002/zaac.200800134

Persson, B. N. J. (2023). Heat transfer in granular media consisting of particles in humid air at low confining pressure. *The European Physical Journal B*, *96*(2), 14. https://doi.org/10.1140/epjb/s10051-023-00483-5

Robalo, K., Costa, H., do Carmo, R., & Júlio, E. (2021). Enhanced mechanical and durability performances of low cement concrete with natural pozzolan addition. *Journal of Advanced Concrete Technology*, *19*(5), 519-535. doi:10.3151/jact.19.519

Sawara, H., Yamauchi, T., Morishita, H., Katayama, S., & Takase, Y. (2002). Concrete containing aqueous slurry of ground calcium carbonate. US patent, US6402831B1.

Schmidt, M., Stumpf, T., Walther, C., Geckeis, H., & Fanghänel, T. (2010). Phase transformation in CaCO3 polymorphs: A spectroscopic, microscopic and diffraction study. *Journal of Colloid and Interface Science, 351*(1) 50-62010. https://doi.org/10.1016/j.jcis.2010.07.026

Thapa, R. K., Nguyen, H. T., Jeong, J. H., Kim, J. R., Choi, H. G., Yong, C. S., & Kim, J. O. (2017). Progressive slowdown/prevention of cellular senescence by CD9-targeted delivery of rapamycin using lactose-wrapped calcium carbonate nanoparticles. *Scientific reports*, *7*(1), 43299. https://doi.org/10.1038/srep43299

Van Tran, M., Chau, V. N., & Nguyen, P. H. (2023). Prediction and control of temperature rise of massive reinforced concrete transfer slab with embedded cooling pipe. Case Studies in Construction Materials, 18, e01817.. https://doi.org/10.1016/j.cscm.2022.e01817

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