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Mechanical Properties of High-Volume Fly Ash Mortar Modified by Hybrid Carbon Nano Tube and Graphene Oxide

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Abstract

The effect of combined nano materials on mechanical properties of high-volume fly ash mortar (HVFAM) was carried out in this investigation. The characteristics of HVFAM mixes were first evaluated using slump flow tests. Then, the mechanical properties of HVFAM including the measurement of setting time and the development of early-age compressive strength were performed. Structural changes during hydration were analyzed using FTIR analysis. The study utilized fly ash (FA) as a 60% replacement for cement, with the addition of 0.01% carbon nanotubes (CNT) and graphene oxide (GO) with five different dosages ranging from 0.01% to 0.05%. Results of study showed that the incorporation of hybrid CNT and GO significantly affected the mechanical properties of HVFAM. Specifically, increased of CNT and GO contents lead to significant reduction in both workability and setting time of HVFAM, with a more pronounced reduction in final setting time compared to initial setting time. The development of early-age strength of HVFAM improved by 15.8% with the highest 28 day-strength increasing by approximately 23% at 0.01% CNT and 0.03% GO dosages. FTIR analysis confirmed that the improved early-age strength was attributed to the accelerated hydration of cement caused by the seeding effect. The outcome of the study provides a suitable approach for the development of eco-friendly materials with improved mechanical properties that could be effectively used for HVFAM.

Keywords

Hybrid, CNT, GO, Mechanical property, Cement mortar, High-volume fly ash

1. Introduction

High-volume fly ash, characterized by the replacement of 50% or more of Portland cement with fly ash, is now emerging as a highly sustainable and eco-friendly material to conventional cement that could reduce the demand for Portland cement and lessens the environmental impact associated with cement production. Incorporating high volumes of fly ash can improve the long-term performance of concrete, including increased resistance to sulfate attack and reduced permeability, thereby increasing the durability and longevity of the material. The use of high-volume fly ash also promotes energy efficiency in construction by utilizing a byproduct that would otherwise contribute to environmental waste. With global fly ash production exceeding 900 million tons (Fakhry et al., 2020; Xi et al., 2016; Zhou et al., 2021), managing this substantial amount of byproduct poses significant challenges, including potential air and water pollution risks. Recent advances have transformed fly ash from a waste product into a valuable construction material, often used as a partial cement replacement in amounts ranging from 15% to 25%. Optimal use of fly ash can enhance the mechanical strength and durability of concrete. Compared to other supplementary cementitious materials such as slag, metakaolin, silica fume, and rice husk, fly ash is particularly recommended for its benefits in strength, workability, and durability (Herath et al., 2020).

High-volume fly ash in cement-based composite has been the focus of extensive research. Gunasekera et al. (2019) studied high volume fly ash concrete with up to 80% replacement of fly ash and found significant reductions in compressive strength. Specifically, concrete with 65% and 80% fly ash exhibited strength decreases of 62% and 82%, respectively, at 3 days compared to ordinary Portland cement concrete. Huang et al. (2013) similarly observed that high volume fly ash concrete with 60% fly ash resulted in a 54% and 35% reduction in compressive strength at 1 and 3 days, respectively. When the fly ash replacement increased to 80%, the reductions were 56% and 52% at the same ages. These findings were observed similarly in self-compacting concrete, where high fly ash dosages affected mechanical strength development. Additionally, Huang et al. (2013) reported a decrease in flexural strength as the fly ash percentage increased from 60% to 80%, highlighting the substantial effect of high fly ash replacement on both compressive and flexural properties (Huang et al., 2013).

The low early-age strengths observed in concrete with more than 50% fly ash replacement is primarily due to the slower pozzolanic reaction between fly ash and calcium hydroxide compared to the reaction in ordinary Portland cement hydration (Miah et al., 2023). This pozzolanic reaction occurs when reactive siliceous react with calcium hydroxide in water, producing additional calcium silicate hydrate. Calcium silicate hydrate is the most important compound responsible for the durability and strength of concrete. The low early-age strengths of high-volume fly ash concrete compared to normal concrete can lead to significant delays in reaching the desired strength, potentially causing problems in construction (Soutsos et al., 2016; Szostak & Golewski, 2018). Thus, a study to improve the early-age strength of high-volume fly ash in concrete and cement mortar is essential.

Incorporating nano materials could be a practical way to improve the strength of high-volume fly ash mortar without adversely affecting its properties. Carbon nanotubes (CNT) and graphene oxide (GO) are among the popular nano materials due to their beneficial effects in nano-modified cement composites. Using nano technology, cement composites are treated at the nano scale to develop concrete with improved properties and performance. Even small amounts of nano materials can substantially enhance mechanical properties of concrete such as compressive and flexural strength (Konsta-Gdoutos et al., 2010).

GO is a form of graphene modified with oxygen-containing functional groups which have similar properties and structure to graphene. These oxygen-containing functional groups can interact with other functional groups and organic molecules (Du et al., 2020). Due to these advantages, GO is a promising material for cement based nano modification. The effect of admixed GO on compressive strength of high-volume fly ash concrete has been investigated. The addition of GO at dosage up to 0.05% increased the compressive strength of high-volume fly ash concrete from 7% to 30% compared to normal concrete (Wang et al., 2019).

In contrast, CNTs are characterized as graphite sheets rolled into a one-dimensional tubular shape. Their primary advantage in cement mortar is their ability to improve the tensile strength due to their highly ductile properties that effectively bridge pores and cracks within the mortar (Makar & Chan, 2009; Sanchez & Sobolev, 2010). However, CNTs typically exhibit weak interfacial bonding with cement hydrates. New CNTs with oxygen-containing functional groups (f-CNTs) can interact well with calcium silicate hydrate and other cement hydrates. Incorporating CNTs would considerably improve mechanical properties and microstructure of fly ash concrete and mortars. For example, adding 1% CNTs to a mix with 20% fly ash replacement can increase compressive strength by around 20%. Additionally, CNTs act as fillers, leading to a denser microstructure and increased strength of the mortar when mix with fly ash (Selim et al., 2020).

Advanced studies have explored the combined effects of two types of nano materials, such as GO and CNT in cement-based composite. The results indicate that the combination of GO and CNTs significantly improves both the hydration process and the mechanical properties of cement paste (Kaur & Kothiyal, 2019; Lee et al., 2018; Li et al., 2016; Liu et al., 2018). Wang et al. (2019) found that hybrid GO and CNT dosages at 0.05% resulted in an improvement of both compressive strength and tensile strength by approximately 25% and 30%, respectively. The improvement of mechanical properties by GO and CNT has also been studied by Selim et al. (2020). They observed that incorporating 0.01% CNT with 0.05% GO increases approximately 18% in compressive strength and 22% in tensile strength of the mortar. Recent studies further indicate that the hybrid GO and CNT demonstrate effective interaction to significantly improve dispersion, mechanical and durability properties of cement mortar compared to other combinations such as nano silica with carbon nanotubes (NS and CNT) or nano silica with graphene oxide (NS and GO). The GO can help in homogenous dispersing and strong bonding CNTs in the cement matrix, leading to improved reinforcement of the cement mortar. This synergistic effect has shown outperforms compared to the other hybrid combinations. In contrast, combinations NS and CNT or NS and GO may not provide the same level of synergistic effect. While NS can improve mechanical strength and reduce porosity, its combination with either CNT or GO cannot achieve the same degree of dispersion and bonding effect as hybrid CNT and GO. To date, there has been limited research on strengthening high-volume fly ash mortar by using hybrid combinations of two types of nanomaterials. Therefore, it is essential to evaluate the effects of incorporating the hybrid CNT and GO on the mechanical strength of high-volume fly ash mortar. Previous studies have demonstrated significant effects from the use of individual nanomaterials CNT and GO, highlighting the need to explore the potential benefits of combining them in HVFAM.

2. Materials and Method

The materials used for preparation cement mortar specimens in this study consist of: OPC, FA, fine aggregates, superplasticizer, GO, and CNT. Ordinary Portland cement (OPC) type I produced by PT Semen Andalas Indonesia was used. This OPC has a specific gravity of 3.10 and satisfies the requirements of ASTM-C150 standard. FA was obtained from PLTU Nagan Raya, in West Aceh Indonesia. The chemical compounds of FA were examined using XRF test as shown in Table 1. This FA can be classified as class F due to its calcium oxide (CaO) content is less than 10% and the main oxide content (silica, alumina, and iron oxide) exceeding 70% of the total mass (ASTM C618-00, 2001). Local sand from quarry mines was used as fine aggregates in this study. This sand has a specific gravity of 2.65. The water absorption of sand was 0.5%. The gradation curve of fine aggregates conformed to ASTM-C33 standard as shown in Fig. 1. The obtained fineness modulus of the fine aggregate is 3.82.

Table 1. Chemical composition of FA from PLTU Nagan Raya, in West Aceh Indonesia

Oxide Compounds	Percentage (wt. %)
CaO	6.67
SiO_2	46.4
Al_2O_3	28.0
Fe_2O_3	7.17
MgO	3.12
Na_2O	1.93
K_2O	1.85
TiO_2	0.743
P_2O_5	0.455
SO_3	2.90
Cl	0.415
MnO	0.0979
NiO	0.0138
CuO	0.0134
ZnO	0.0129
Rb_2O	0.0110
SrO	0.135
Y_2O_3	0.0066

The CNT was supplied by the Maxlab laboratory in Jakarta Indonesia and GO was obtained from the NRE laboratory in Medan Indonesia. The GO has 99% high purity, dark brown color and pre-dispersed in water with a solid concentration of 10 g/L. The multi walls CNTs were also pre-dispersed in water-based with a solid concentration of 1 g/L. This CNT has 90.9% purity and a length of 10–50 μm with an outside diameter of 8–15 nm. A Sika ViscoCrete 8045P type of high range water reducer superplasticizer (SP) obtained from PT Sika Indonesia was used in this study. It was a colorless liquid with a density of 1.06 kg/L. The materials used for specimens preparation are shown in Fig. 2.

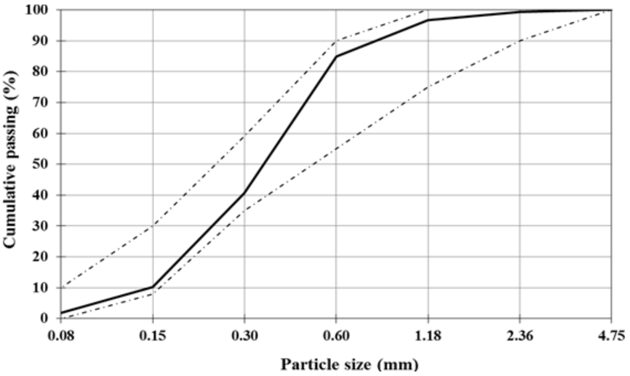


Fig. 1 Gradation curve of fine aggregates

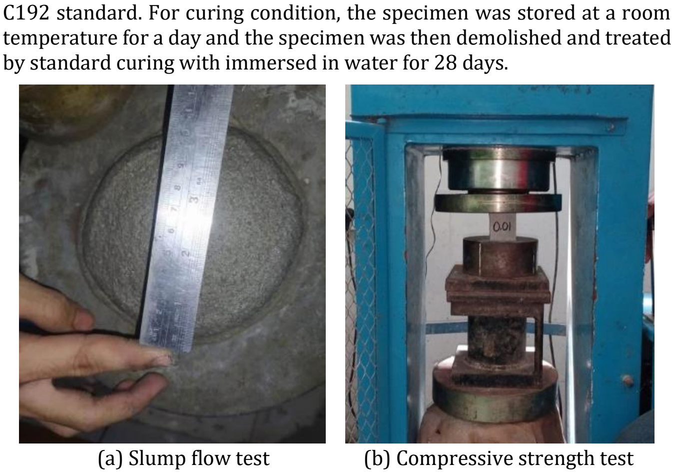
Fig. 2 Materials used for specimens preparation

The specimen preparation consists of mixing the dry materials with pre-dispersed water-based nano materials, casting, and curing. Since GO composition has significant effect on the synergistic of hybrid nano materials, GO was selected as a variable in this study. Specimens were produced, replacing part of the cement at 0.01% for CNT and five different levels for GO: 0.01%, 0.02%, 0.03%, 0.04%, and 0.05%, respectively. A water-cement ratio of 0.46 was taken for specimens' preparation. To enhance the workability, SP was added in the mixture at 1.5 % by weight of cement before mixing the mortar. Detailed proportions of nano materials in cement mortar are shown in Table 2.

Table 2. Proportions of materials in mortar specimens

Specimen	Cement (g)	Sand (g)	Water (g)	Fly Ash (g)	CNT (%)	GO (%)	SP (%)
Pure HVFAM	87	533	100	130	-	-	1.5
CNTGO-01	87	533	100	130	0.01	0.01	1.5
CNTGO-02	87	533	100	130	0.01	0.02	1.5
CNTGO-03	87	533	100	130	0.01	0.03	1.5
CNTGO-04	87	533	100	130	0.01	0.04	1.5
CNTGO-05	87	533	100	130	0.01	0.05	1.5

For mortar mixing, solid materials (OPC and sand) were firstly mixed in a rotary mixer for approximately one minute to obtain a homogenous mixture. Then, pre-dispersed water-based nano materials GO and CNT were put into the mixture for approximately 3 minutes at a velocity of around 140 rpm. Finally, the mixing was conducted by a rotary mixer at 285 rpm for around 5 minutes. The casting of mixtures followed ASTM-C192 standard. For curing condition, the specimen was stored at a room temperature for a day and the specimen was then demolished and treated by standard curing with immersed in water for 28 days.



(a) Slump flow test (b) Compressive strength test

Fig. 3 Mechanical properties tests of mortar specimens

To assess effects of hybrid CNT and GO on mechanical properties of HVFAM, a series of compressive strength measurements were performed using UTM compression machine. Specimens have a dimension size of 5cm×5cm×5cm tube following the ASTM-C109 Standard. Each mixture consists of three specimens, and it is therefore the compressive strength is determined as the average value of the specimen's compressive strength. All mixtures compressive strengths were carried out at the ages of 3, 7, and 28 days as shown in Fig. 3(b).

3. Results and Discussion

3.1 Workability

The workability of mortar specimens was carried out using mini cone slump test as shown in Fig. 3(a), with the results presented in Fig. 4. It can be seen that the increase contents of GO could generally lead to a significant decrease in workability of HVFAM specimens. For example, when CNT 0.01% with GO up to 0.05%, the workability of specimens could be reduced by 21.2%. The decrease in workability can be attributed to the content of nano materials. The main reason for this result is associated with an increase in the surface area of the mixture that absorbs part of mixing water (Adhikary et al., 2020; Bagheri et al., 2022). When content of nano materials increases, the mixture has less water and becomes stiffer. This result indicates that the addition of a small amount of GO has a great impact on the flow performance of HVFAM mixes.

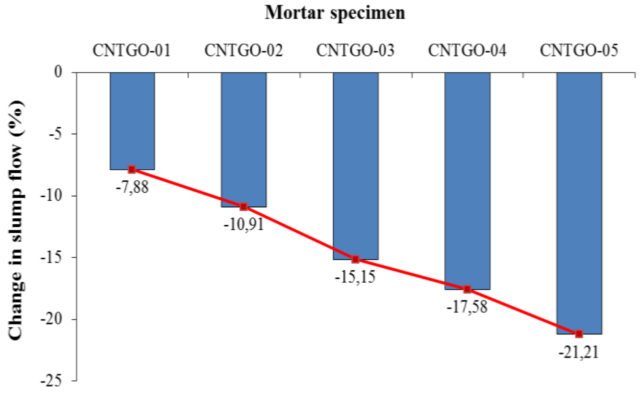


Fig. 4 Changes in slump flow of mortars with hybrid GO and CNT compared to Pure HVFAM

3.2 Setting Time

The addition of hybrid CNT and GO may result in significant reduction of the setting time (initial and final setting times) of HVFAM. As seen in Fig. 5, the influence of hybrid CNT and GO in all specimens with the increase dosage of GO on initial and final setting times of HVFAM compared to Pure HVFAM. For example, the incorporation of 0.01% GO in the CNTGO-01 specimens could result in around 55 minutes and 97 minutes reduction of both the initial and final setting times, respectively. In the CNTGO-05 specimens, the increase dosage of GO up to 0.05% could lead to significant reduction in the initial setting time and final setting time of about 96 minutes and 177 minutes. The effect of adding hybrid nano materials in HVFAM specimens has significantly higher reduction in both initial and final setting times compared with those of pure HVFAM. Further, the reduction in final setting time is more significant as compared to initial setting time. This effect is caused by the presence of nano materials in cement composites with a higher heat hydration that could accelerate cement hydration process, leading to shorter setting and hardening times (Indukuri et al., 2020; Zhong, 2023). Therefore, adding hybrid nano materials can be considered as an effective way to improve a significant delay in the required concrete strength achievement such as in pre-cast composites production.

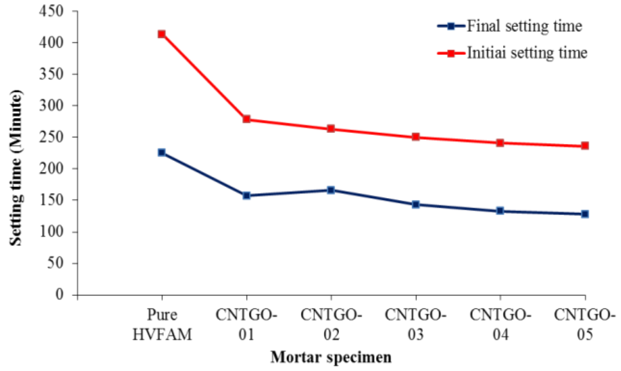


Fig. 5 Setting time of HVFAM specimens

3.3 Compressive Strength

Fig. 6 showed the compressive strength of the specimens at the ages of 3, 7, and 28 days. As seen in Fig. 6, pure HVFAM specimens have the smallest compressive strength. It indicates that the addition of a higher quantity of FA led to a reduction in compressive strength. This is due to the pozzolanic reaction of FA is much slower as compared to the hydration of HVFAM with hybrid nano CNT and GO. To improve the concrete strength of pure HVFAM, hybrid CNT and GO were added into the mixes. In all specimens, the compressive strength of HVFAM increased with hybrid CNT and GO mixes when GO was given from 0.01%-0.03% and further decreased when GO was added from 0.04% to 0.05%. The early age strength of HVFAM with hybrid CNT and GO mixes was found to have increased when GO increased from 0.01%-0.03%. The compressive strength of HVFAM with hybrid CNT and GO mixes at 3 and 7days age increase by 15.8% compared to pure HVFAM specimens. The CNTGO-03 specimens achieved the highest compressive strength, reaching 23% of the compressive strength of pure HVFAM at 28 days age.

The improved compressive strength of HVFAM specimens is mainly affected by two ways, filler effect and hydration seeding effect of both CNT and GO. The filler effect is related to the ultrafine nano materials that refine microstructure of mortars become denser and the pores formed become smaller. In such a way, the filler effect has successfully reduced the growth and formation of cracks at nano scale. Through seeding effect, nano materials act as seeds during the precipitation process, accelerate hydration rate, and stimulate cement to produce additional CSH to react with excess CH at the early ages. As a result, mechanical strength of HVFAM at the early ages were improved (Gunasekara et al., 2019; Makar & Chan, 2009).

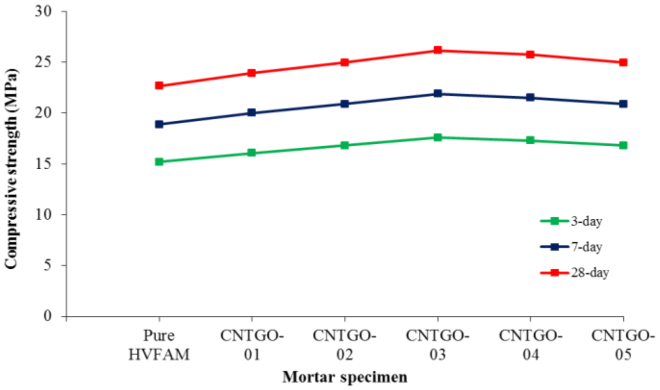


Fig. 6 Compressive strength development of pure HVFAM and HVFAM with hybrid CNT and GO

The incorporation of CNT and GO into the HVFAM mix design offers significant advantages in both strength and sustainability. The hybrid effect of CNT and GO could lead to increased strength and durability, making it more resistant to cracks and improving overall performance. Using a high-volume fly ash in the mortar not only reduces reliance on Portland cement but also minimizes the carbon footprint associated with cement production. Moreover, the incorporation of CNT and GO may improve sustainability by potentially reducing the overall binder content required while still maintaining strength. However, the use of CNT and GO in the HVFAM presents several issues such as higher initial cost due to relatively expensive of CNT and GO compared to conventional materials. This may cause difficulties for large-scale applications. In addition, the mixing process for incorporating CNT and GO requires precise control to ensure uniform dispersion which may result in complicated manufacturing compared to ordinary mortar production. Therefore, posing these advantages and challenges is essential for the future of HVFAM in sustainable construction practices.

3.4 Fourier Transform Infrared (FTIR) analysis

The application of Fourier Transform Infrared (FTIR) technique has been widely used to determine molecular vibrations and predict the structure of chemical compositions. In cement mortar, structural changes occurring during hydration reaction can be obtained from FTIR results using major peaks in FTIR spectra. Fig. 7 shows results of FTIR testing of HVFAM specimens with hybrid nano materials. It can be seen that FTIR wave bands pattern in all specimens are almost the same. The broad peaks from 3000 cm^{-1} to 3500 cm^{-1} correspond to the O–H stretching vibrations due to the presence of CNT and GO. The FTIR band intensity at wave number of 1640 cm-1 shows C-H bending mode which reacts with calcium hydroxide to form more CSH (Rada et al., 2023). The presence of CO3 was found at two stretching bands at around 782 cm^{-1} and 1435 cm^{-1}, respectively. The stretching band at 1017 cm-1 corresponds to vibration of C-O due to the presence of CNTs with carboxyl groups. The stretching vibration due to the formation of CSH was observed at peak at

980 cm-1. The characteristic bands of FTIR spectra observed in this study indicated that the effect of hybrid CNT and GO on the formation of CSH in HVFAM is significant. Therefore, it confirmed that hybrid CNT and GO could accelerate hydration of cement through seeding effect similar to adding other nano materials in cement composites (Gunasekara et al., 2019; Makar & Chan, 2009).

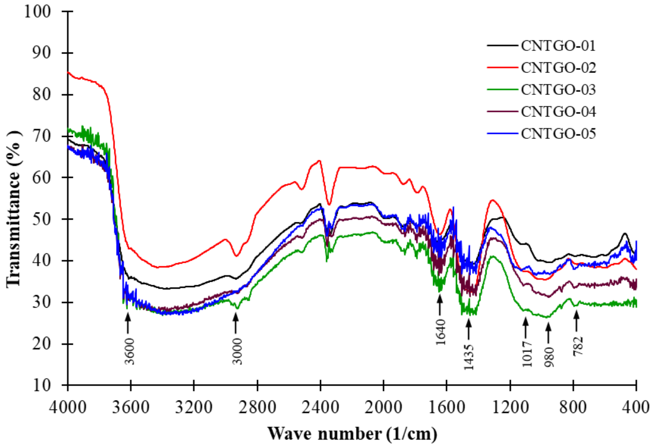


Fig. 7 FTIR testing of HVFAM with hybrid CNT and GO

3.5 CO2 Emission Analysis

In this study, the CO2 emissions associated with the production of the cement mortars were estimated based on the raw materials required for the preparation of 1 m^3 of mortar specimens. According to previous literature, the emissions for each material were determined as follows: cement production contributes 860 kg of CO2 per ton, sand 0.649 kg/ton, fly ash 207 kg/ton, GO 357 kg/ton, CNT 600000 kg/ton, SP 600 kg/ton, and water 0.8 kg/ton (Licht et al., 2019; Sun et al., 2021; Wee, 2013). Fig. 8 shows a comparison of CO2 emissions between OPC mortar and HVFAM with the addition of CNT and GO. It can be seen that HVFAM mixes result in a substantial reduction, approximately 42% less CO2 emissions, compared to OPC mortar. This reduction is significant, especially considering that HVFAM with CNT and GO also demonstrates higher compressive strength. These results also indicate that HVFAM with the addition of CNT and GO not only enhances the performance of cement mortars but also promotes sustainability by reducing their carbon footprint, thus promoting eco-friendly construction materials.

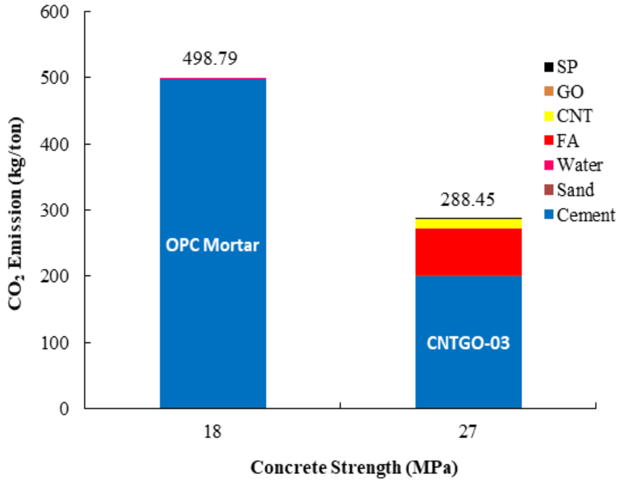


Fig. 8 CO2 emissions from mortar specimens estimated based on the raw materials used

4. Conclusion

The effect of hybrid CNT and GO to improve the mechanical performance of HVFAM was investigated. HVFAM with hybrid CNT and GO developed in this study is more sustainable and eco-friendly for cement mortar production. Based on the analysis, some major findings are summarized as follows:

(1) The incorporation of hybrid CNT and GO can improve the early-ages compressive strength of HVFAM. At the ages of 3 and 7 days, the compressive strength of HVFAM with hybrid CNT and GO increased by 15.8% as compared to pure HVFAM specimens. The HVFAM specimens achieved the highest 28 day-strength by around 23% at 0.01% CNT and 0.03% GO dosages.

(2) The addition of hybrid CNT and GO reduced the workability of mortar specimens. The workability of HVFAM specimens could be reduced by 21.3% when the dosage of GO increased from 0.01% to 0.05%.

(3) Hybrid CNT and GO may lead to a reduction in setting time of HVFAM specimens. Further, the reduction in final setting time is more significant compared to initial setting time.

(4) The observed band intensity at wave numbers of 1435 cm-1 and 1640 cm-1 increases with the increasing hybrid CNT and GO content. It clearly demonstrates a significant increase in peaks due to the presence of hybrid CNT and GO.

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