The Future of Engineering Education
2024 Annual Conference & Exposition
Oregon Convention Center
Portland, OR . June 23 - 26, 2024
ASEE
Paper ID #41496

Board 249: Effect of Carbon Nanomaterials on the Compressive Strength of Cement Mortar: Research at Marshall University's 2023 REU Site

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I am an Associate Professor at Marshall University, focusing on environmental engineering and nanotechnology. My research involves the fate and transport of contaminants in engineered systems and sustainable developments for cementitious materials. I have published impactful articles and secured several external grants for projects from the EPA, NSF, and DoD. As a mentor and educator, I strive to adapt to each student's needs and foster a collaborative environment.

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Since 2018, Dr. Sukjoon Na has been an assistant professor of Civil Engineering at Marshall University in West Virginia. With a Ph.D. in Civil Engineering earned from Drexel University, Dr. Na specializes in developing innovative and sustainable construction materials, coupled with expertise in failure analysis. His notable contributions include presentations at prestigious journals and conferences dedicated to materials engineering and fracture mechanics, reflecting his active engagement and expertise in the field. Dr. Na received the Best Paper Award in Failure Analysis and Prevention at the Society of Plastic Engineers (SPE) annual technical conferences in 2013 and 2016.

Effect of Carbon Nanomaterials on the Compressive Strength of Cement Mortar: Research at Marshall University's 2023 REU Site

1. Abstract

This paper describes the experience and outcomes of a non-engineering major who participated in a 10-week Research Experience for Undergraduates (REU) program at Marshall University. The main objective of the research project was to investigate the effects of carbon nanomaterials on the mechanical properties and durability of cement mortar. The non-engineering major was involved in manufacturing and testing cement mortar cubes with different concentrations of carbon nanotubes and graphene using an ASTM standardized procedure. The paper reflects on the benefits and challenges of conducting quantitative research in an engineering field, such as learning how to use laboratory equipment, analyze data, and write technical reports. The paper also discusses how the interdisciplinary nature of the project helped to broaden the perspective and enhance the problem-solving abilities of the non-engineering major, who applied concepts and methods from forensic anthropology to engineering materials. The paper concludes that the REU project was a valuable opportunity to learn about engineering research and education, despite the inconclusive results that are possibly due to experimental errors.

When mixed with water and aggregate, cement is useful in the construction industry due to its strength, versatility, and durability, and additives are often incorporated to improve these properties. This research integrates carbon nanomaterials including graphene and carbon nanotubes (CNTs) into Type 1 cement mortar cubes to investigate their effect on the compressive strength of the resulting concrete. Researchers have previously investigated this topic and the current study seeks to find the optimum amount of carbon nanomaterials to maximize the compressive strength. A water-to-cement (w/c) ratio of 0.45 and sand-to-cement ratio of 2.75 were used to mix fresh cement mortar. The sand was oven-dried and sieved by a No. 10 standard sieve (2 mm). A non-ionic surfactant, Igepal Co-630 combined with ultrasonic dispersion was applied to disperse the carbon nanomaterials before incorporating them into the cement mortar. The tested graphene-to-cement ratios include 0.1%, 0.5%, 1.0%, 1.5%, and 2%. For CNTs, 0.5% and 0.1% of CNTs were tested. Cement mortar cube specimens with dimensions of

2 in × 2 in × 2 in were molded, and all specimens were cured in water at room temperature until compression strength testing at 7, 14, 21, and 28 days. The experimental results show that adding the tested amount of carbon nanomaterials had negative effects on the compressive strength, and the 28-day strength generally decreased as the amount of the content increased, although there were a few enhanced cases at early-stage strength. These controversial results could be derived from the high content of carbon nanomaterials or improper preparation of test samples. Further research will be conducted to conclude the effect of carbon nanomaterials.

2. Personal Narrative

The first part of this paper delineates the personal narrative about the experience of a 10-week Research Experience for Undergraduates (REU) focusing on civil engineering materials at Marshall University in 2023 (NSF grant# 2149891). Marshall's REU recruited a diverse group of non-engineering participants with varying academic backgrounds, including the featured individual majoring in forensic science. The narrative was given from the point of view of the forensic science student who uses any pronouns.

The participant's initial interest in Marshall's REU stemmed from the prospect of gaining practical experience in surveying or ground-penetrating radar (GPR), aligning with their future pursuit of a doctoral degree in forensic anthropology. The participant was admitted to the REU despite submitting a late application, stepping in as a replacement for a student who withdrew at the last minute. For this reason, the participant was offered a research topic of carbon nano-reinforced cementitious materials which was not their first choice. Despite the project's misalignment, they embraced the opportunity driven by a passion for interdisciplinary research. The REU experience, albeit not aligning precisely with their research interest, proved to be a valuable opportunity for the participant to immerse themselves in a distinctive research environment.

Prior to their involvement in the REU, the participant's research pursuits primarily revolved around osteology, with a year and a half of collaboration with a professor at their home institute, a public university in West Virginia designated as M3: Master's Colleges and Universities – Small programs. While this work was relevant to the participant's future career objectives, it confined their research exposure to a single laboratory and mentor. Participation in Marshall's REU expanded their horizons by providing exposure to research in a distinct discipline, fostering collaboration with diverse research mentors, and allowing them to work in a different institutional setting. Furthermore, the experience of being part of a cohort of students with diverse backgrounds and interests significantly contributed to a robust and enriching collaborative learning experience.

In the REU project, an intrinsic aspect of considerable value lies in its full-time commitment coupled with inherent schedule flexibility owing to its independent nature. The participants, encompassing all eight participants in the cohort, were afforded the liberty to engage in their daily research activities at any given time, underscoring the independent character of the research framework. This unique structure provided an inaugural exposure to the realm of full-time research without imposing stringent expectations on participants' presence within the laboratory confines. Commencing the project with an absence of prior experience in cement or related domains, the initial week was dedicated to an exhaustive exploration of pertinent literature. The participant diligently compiled these sources into an annotated bibliography, facilitating the acquisition of knowledge about the topic and presenting a challenge to seek information with limited foundational understanding guiding the research.

In addition, the REU experience presented an opportunity for exposure to quantitative research. In this independent laboratory setting, the focus was on consistency and repetition, employing a uniform recipe to produce cementitious test cubes. The previous research involvement in forensic anthropology leaned heavily towards qualitative evaluation, making this experience a valuable simulation of the more quantitative aspects typical in a professional laboratory environment. This multidisciplinary approach contributed to the participant's skill development, enabling them to adapt to the quantitative requirements of a professional laboratory context.

As a respite from the research-intensive activities, participants were treated to a series of mid-week group excursions, meticulously orchestrated by faculty and fellow participants. These excursions encompassed visits to the Huntington Art Museum, exclusive behind-the-scenes tours of the Blenko Glass Company and the Robert C. Byrd Locks and Dam, and a particularly memorable excursion to the New River Gorge National Park. The highlight of this experience was a bridge walk on a catwalk beneath the New River Gorge Bridge. This curated selection of activities not only served as a welcome break from the research routine but also infused a diverse blend of educational and entertainment elements. Significantly, this was particularly enriching for participants unfamiliar with the locale, offering both historical context and enjoyable components.

While the research experience boasted numerous positive facets, participants collectively identified meal options as an area necessitating improvement. Residing in dormitories on Marshall University's campus, the dining hall emerged as the primary source for meals. Regrettably, during the summer period, meal choices were limited, particularly for those with dietary restrictions, and the perceived quality of the food fell short of expectations. This concern was communicated to the organizers during the experience, prompting a clear commitment to exploring alternative meal provisions in subsequent years, informed by the constructive feedback provided by the participants.

In its inaugural year of 2023, the program demonstrated overall success. The participants, constituting a diverse cohort, actively embraced their research responsibilities and fostered positive group dynamics. Cohesion prevailed, with no significant conflicts, and students voluntarily spent time together beyond planned activities. Feedback on research mentors was generally favorable, and despite some project complications, the overarching goals of the student research cohort were realized. Addressable issues, such as suboptimal dining choices, emerged but were considered manageable and amendable for future cohorts. The overarching aim of providing engineering exposure to non-engineering students from varied backgrounds was unequivocally achieved, culminating in a valuable and enjoyable experience for all involved.

3. Research Introduction

Concrete, a widely utilized construction material for several decades, is favored due to its low manufacturing cost and abundant raw materials. It finds application in diverse construction, transportation, and underground engineering projects [1]. However, in the contemporary era, as engineering standards evolve, there is a growing demand for elevated mechanical properties, durability, and environmental performance in concrete. To meet these heightened expectations, researchers have explored the incorporation of various additives into different types of cement-based composites, aiming to develop specialized concrete and enhance its overall performance [2]-[5].

In recent years, there has been significant interest in leveraging carbon-based nanomaterials as reinforcements in cement-based composites. These nanomaterials exhibit exceptional mechanical properties, including high tensile strength, a superior modulus, and remarkable flexibility and resilience, potentially contributing to the improvement of concrete quality and durability. For instance, carbon nanotubes (CNTs) boast a Young's modulus exceeding 1 TPa, allowing them to serve as a bridging element at crack sites in concrete. This bridging effect contributes to enhanced strength and durability [6]. Additionally, studies have reported that multi-wall carbon nanotubes (MWCNTs) can positively impact the mechanical and thermal properties of cement [7]. Investigations by Musso et al. [8] focused on CNT structures in cement composites, highlighting the favorable influence of an appropriate CNT dosage on cement mechanical properties. This study specifically centers on two distinct carbon-based nanomaterials, namely graphene and CNTs, and their effects on the compressive strength of conventional cement mortar across a broad spectrum of concentrations

4. Materials and Methods

4.1. Cement mortar composition

A commercial Portland cement (Type I) supplied by Lehigh Heidelberg Cement Group that complied with all applicable requirements under ASTM C150 [9] was used in this study. Oven-dried clean sand, characterized by its passage through a No. 10 sieve and possessing a fineness modulus of 2.38, was used as a fine aggregate. The particle size distribution of the fine aggregate in Figure 1 was determined by the standard sieve analysis. The results show that the fine aggregates used in this study conformed to the specifications defined by ASTM C33 [10].

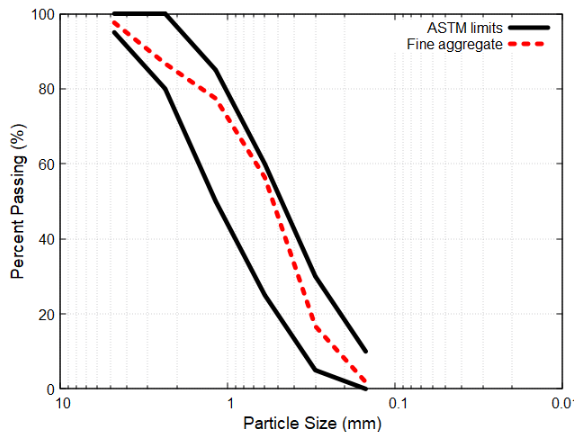

Figure 1: Particle size distribution of the fine aggregate

4.2. Graphene

The graphene used in our study, procured from TechInstro®, is an industrial-grade material tailored for scientific applications. With a bulk density of 0.24 g/cc, a thickness between 5 to 10 nanometers, and a lateral dimension of 5 to 10 micrometers with a carbon purity exceeding 99% and a specific surface area of 150 square meters per gram. Graphene was added in graphene-to-cement ratios of 0.1 wt. %, 0.5 wt. %, 1.0 wt. %, 1.5 wt. %, and 2 wt. % to the standardized mortars. The graphene was integrated into the batch water using a non-ionic surfactant, Igepal Co-630, and ultrasonic dispersion to prevent agglomeration in the mortars, which has been previously identified as detrimental to their compression strength.

4.3. CNTs

The multi-walled carbon nanotubes employed in this study were sourced from CTI Materials. These nanotubes exhibited characteristics essential for our research, including a purity exceeding 95wt%, an outer diameter ranging from 5 to 15 nanometers, and lengths varying between 10 to 30 micrometers. With an ash content below 1.5 wt%, the nanotubes demonstrated minimal impurities. Their specific surface area surpassed 110 m^2/g, indicating a highly reactive surface. Initially, CNT ratios of 0.1 wt. %, 0.5 wt. %, 1.0 wt. %, 1.5 wt. %, and 2 wt. % were planned. However, preliminary graphene break test results indicated higher ratios were performing poorly, so instead ratios were started at 0.05 wt. %. Due to time constraints, only batches of 0.05 wt. % and 0.1 wt. % CNT ratios were completed.

4.4. Mix design and sample preparation

Cement mortar specimens incorporating varying proportions of graphene and carbon nanotubes (CNTs) were formulated to assess their impact on the compressive strength of conventional cement mortar. Adhering to ASTM C109 [11], the cement-to-fine aggregate ratio was maintained at 1:2.75 by weight, while the water-to-cement ratio (w/c ratio) was fixed at 0.45. The mixing procedure, as outlined in ASTM C305 [12], was followed for all cement mortar samples. To ensure a uniform distribution of carbon nanomaterials, they were pre-mixed with water, incorporating a surfactant, and subjected to a 30-minute sonification process at room temperature before mixing with cement and fine aggregates. Cubical cement mortar specimens, measuring 2 in x 2 in x 2 in, were subsequently molded. The graphene/cement composites were created by blending cement with weight fractions of 0%, 0.5%, 1%, 1.5%, and 2%, chosen systematically to identify the optimal range of graphene contents. In the case of CNTs, due to time constraints in the REU project, weight fractions of 0%, 0.5%, and 1.0% were exclusively examined. All cube specimens underwent curing in water at room temperature, with subsequent compression strength testing conducted at intervals of 7, 14, 21, and 28 days. Twelve specimens for each composite mixture were tested after the completion of each designated curing duration. The mix design details for the cement composite mortars are presented in Table 1.

Table 1: Mix design of carbon nanomaterials/cement composites

Material	**Weight Ratio**
Portland Cement (Type I)	1
Dry Graded Sand (-No. 10)	2.75
Water	0.45
Graphene or Carbon Nanotubes	0.005, 0.01, 0.015, and 0.02

5. Experimental Results

5.1. Compressive Strengths of Test Specimens

The compression strength test results for graphene/cement composites are presented in Table 1 and illustrated in Figure 2. It is important to highlight that the 7-day control group underwent complete remaking and retesting due to unexplained and significant variations, attributed primarily to an unfamiliarity with the procedure. The findings substantiate the augmentation of compressive strength in normal cement mortar with increased curing time. A parallel observation is made in the graphene composite samples, where strength generally escalates with extended curing periods. Notably, at the early 7-day stage, a definitive correlation between strength and graphene content was not discerned. Specifically, 0.5% and 1.5% graphene exhibited lower strength than normal cement mortar, while 1.0% and 2.0% showed marginally higher strength. However, a discernible trend emerges at the 28-day mark, indicating that the addition of graphene to normal cement generally diminishes strength, with a more pronounced effect at higher content levels. The results underscore that a modest amount of graphene (e.g., 0.5%) marginally enhances strength, while excessive amounts exceeding 1.0% result in a decrease in strength. Notably, 2.0% graphene led to a substantial -25% reduction in strength.

The findings pertaining to CNT-reinforced cement mortar are depicted in Figure 3. It is pertinent to note that the range of CNT contents was adjusted based on the results of graphene/cement composites, with 0.1% CNT content newly introduced, and 2.0% omitted. Analogous to graphene, elevated CNT contents generally resulted in decreased strength across all curing durations. However, a noteworthy observation is that 0.1% CNT exerted a significant strengthening effect. This minimal content incrementally increased the 7-, 14-, 21-, and 28-day strength by 46%, 10%, 30%, and 55%, respectively. These outcomes underscore that the incorporation of CNT beyond the optimal range detrimentally impacts the strength of normal cement. The optimal range for CNT, conducive to enhanced compressive strength, was observed to be below 0.5%.

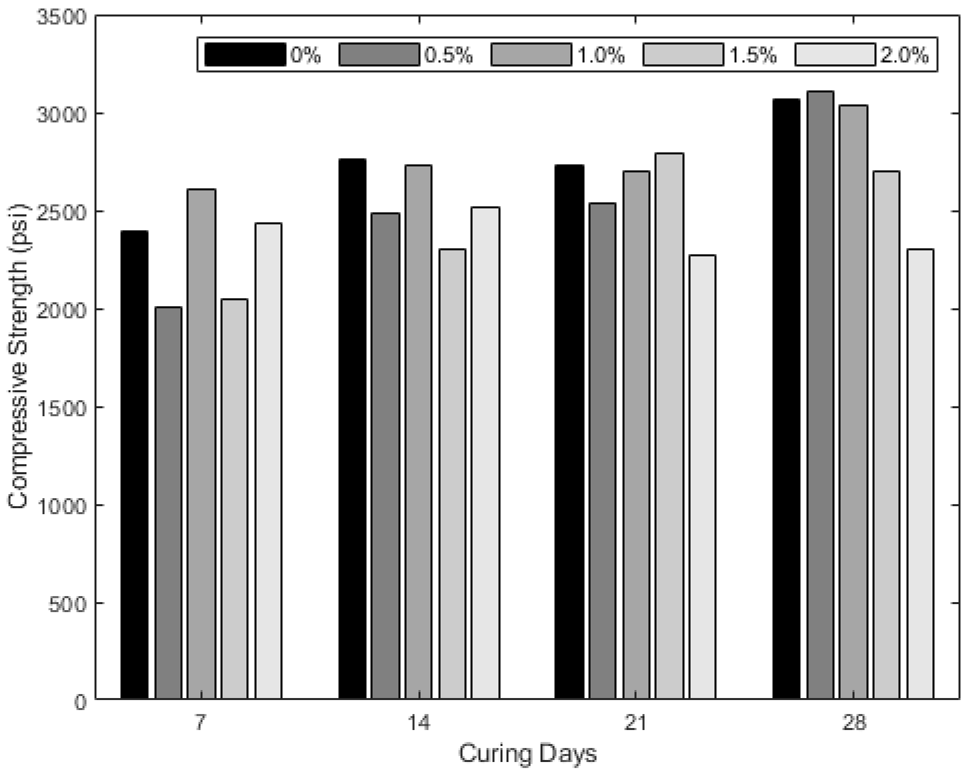

Figure 2: Measured compressive strength of graphene/cement mortars

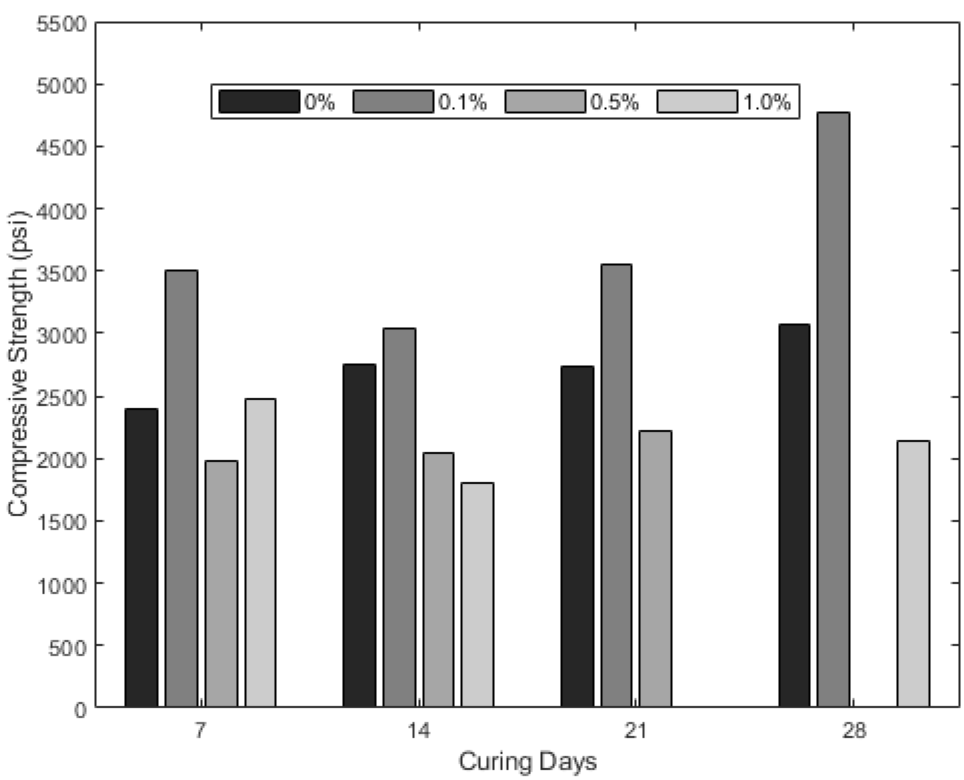

Figure 3: Measured compressive strength of CNT / cement mortars

5.2. Additional Qualitative Observations

Produced mortars with graphene and CNTs both appear a noticeably darker color, CNT cubes more so than graphene, and the darkness increased with the percentage of the additives as shown in Figure 4. Graphene dispersed in water noticeably better, however, it also had a higher tendency to spread and become stuck to other surfaces than CNTs. The graphene mortars also noticeably shed nanomaterials, especially those with higher percentages of the nanomaterial. Upon completion of the cement mortar mixture, the CNT-containing mixtures underwent a noticeably significant texture change. Although initially, these mixtures seemed physically wetter and more easily displaced in tamping, they also seemed to set more quickly. As such, the mixture in the molds went from difficult to tamp down due to quick displacement to difficult to tamp due to solidity very quickly. This could be a major factor in hindering the usability of CNT additives in larger-scale projects. Overall, although CNTs were easier to work with before the addition of a cement mixture due to their tendency to adhere rather than cohere, graphene was easier to work with and more similar in consistency to the control samples after integration.

Figure 4: Cement mortars with 0, 0.5, 1.0, 1.5, 2.0% graphene

6. Discussion

This research does appear to support that adding less than 0.5 % of graphene or carbon nanotubes to Type 1 cement mortars can positively impact their resultant compressive strength. However, the higher standard deviations present for groups with integrated nanomaterials over the control indicates that this integration may result in higher variability of the resulting mortars, likely due to dispersion variations. In addition, the integration of the nanomaterials increases the preparation time and may not be easily scalable due to the limitations of ultrasonic dispersion in larger mediums. Potential health hazards to individuals working directly with the nanomaterials or due to graphene shedding after curing could also be problematic. As such, further research should focus on replicating these results in larger batches, improving dispersion methods, and testing with various smaller ratios to determine an ideal ratio of cement to graphene. In addition, before having any commercial use tests regarding the potential consequences of leached nanomaterial from any resultant cement or concrete or sealant methods.

7. Conclusion

Incorporating less than 0.5% of graphene or carbon nanotubes has demonstrated the potential to enhance the 28-day compressive strength of cement mortars. To ascertain an optimal ratio and assess the impact of lower contents on variability, additional research is recommended. This exploration is crucial for determining the viability and practicality of these mixtures for commercial applications. If these findings prove scalable and methods are devised to mitigate variability, carbon nanomaterials could emerge as advantageous additives for larger-scale applications in cementitious materials.

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