Proceedings of the 10th International Conference on Civil Structural and Transportation Engineering (ICCSTE 2025)
July, 2025 | Imperial College London Conference Center, London, United Kingdom
Paper No.217
DOI: 10.11159/iccste25.217

Directional Effects of Sustainable Graphene Derivatives on the Flexural Strength of 3D-Printed Cement Composites

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Abstract – This study investigates the anisotropic properties of a novel and sustainable graphene derivative, specifically a date syrup-based graphene-coated sand hybrid (D-GSH), incorporated into 3D-printed cement mortar (3DPC). The flexural strength of 3D-printed beams was determined after 7 days of curing by varying the loading directions, i.e. parallel and perpendicular to the printing direction for the mixes containing D-GSH, and the results were compared with a mix containing silica fume. The flexural strength increased when the force was applied parallel to the printing direction, which is due to better load distribution and stronger bonding of the 3DPC. In contrast, when the load was applied perpendicular to the printing direction, the strength was reduced due to weaker interlayer bonding. For example, a mixture with 5% silica fume showed a 25% increase in flexural strength when the load was applied parallel to the printing direction as opposed to perpendicular. On the other hand, mixes with 0.3 wt% D-GSH and 0.5 wt% D-GSH showed improvements of 11.6% and 9.5% respectively. As a result, adding D-GSH reinforced the layer interface and reduced the variance in flexural strength between the two loading orientations, thereby enhancing interlayer bonding in the 3D-printed structures.

***Keywords*:** Additive manufacturing, Graphene, Nanomaterials, Flexural strength, Directional dependence.

1. Introduction

3D printing is a process that enables the production of three-dimensional objects from a variety of materials using computer-aided engineering [1]. Compared to traditional construction, 3D-printed cement mortar (3DPC) requires a shorter construction time, less labor, does not require abandoned formwork, and has the ability to allow more freedom in the design of buildings, especially with curved surfaces [2]. The most widely used form of 3DPC is extrusion-based 3D printing, which refers to the layer-by-layer production of structural components utilizing printable materials [3]. In contrast to cast concrete, which is isotropic, 3DPC is anisotropic due to the special extrusion and layer-by-layer printing process [4].

Extrusion-based 3DPC presents several challenges, including determination of mix design, nozzle clogging, curing conditions, and directionality of mechanical strength. The integration of nanomaterials in cement mortar offers the possibility to overcome these challenges by minimizing pores and voids and improving mechanical properties [5, 6]. Notably, the physical properties of nanomaterials highly depend on their concentration and degree of dispersion [7]. Among the various nanomaterials, research on incorporating a new sustainable graphene derivative called date syrup-based graphene-coated sand hybrid (D-GSH) into 3DPC is in its early stages. The D-GSH is derived from date syrup, a renewable carbon source that is abundant in the United Arab Emirates and surrounding areas. This is achieved through pyrolysis, a process in which graphene nanosheets are formed by graphitizing natural date syrup directly on the dune sand [8]. Ali et al. [9] investigated the effect of adding D-GSH to 3DPC and found significant improvements in the printability, buildability, rheology, and mechanical properties. For example, the addition of 0.5 wt% D-GSH improved the compressive strength, modulus of elasticity, and flexural strength by 62%, 40%, and 118%, respectively, compared to a mix with silica fume. In their study, the load was applied parallel to the printing direction. In this study, however, the load was applied perpendicular to the printing direction. This is the first study to investigate the directional effect of D-GSH on flexural strength. The aim of this study is to examine the anisotropic nature of 3DPC by adding D-GSH. A three-point bending test was conducted on 3D-printed beams after 7 days of curing to evaluate the early-age mechanical properties of the material under different loading directions. This early testing phase is crucial for assessing the structural performance of 3DPC. The results were compared with a mix containing silica fume to highlight the influence of the D-GSH additive on flexural strength development at early ages, particularly for applications that demand rapid strength gain.

2. Methodology

2.2. 3D printing setup and materials

A Delta WASP 3D printer was used to print cement mortar. The setup includes a custom-designed extruder, a WASP motor and a 5 mm circular nozzle, as illustrated in Figure 1(a). The extruder was designed for suitable cement mortar extrusion. The design of the extruder consists of a hopper with a maximum capacity of 400 g for the mix, a 12 cm long screw with a pitch of 5 mm to propel the substance through the nozzle, the nozzle affecting the material output, and a WASP motor driving the screw [10].

Cementitious materials consist of cement, sand, water and additives. Standard Portland cement (Type I, CEM-1 42.5 N) was used as the binder for the mixture. The sand grain size is between 0.2-0.5 mm. The micro-material used in this study was silica fume from Micro-Silica Elkem. In addition, D-GSH, which was produced in an environmentally friendly way, was used as an additive. The manufacturing process of D-GSH is shown in Figure 1(b). First, dune sand and date syrup were mixed in a ratio of 2:5 and stirred for 1 hour. The mixture was then incubated overnight at 80 °C to remove excess moisture. The substance was then carbonized in a tube furnace with atmospheric nitrogen. The mixture was heated to 200 °C to convert the date syrup into carbon-like graphene and then to 750 °C to complete the graphitization. Finally, after cooling, the D-GSH compound is produced [8]. In contrast to conventional graphene production, which requires extreme temperatures (e.g. 1100 °C for chemical vapor deposition) and hazardous chemicals, the D-GSH synthesis process, which involves one hour of stirring and overnight incubation at 80 °C, is energy efficient and sustainable. In addition, the use of renewable date syrup and low-energy processing reduces the environmental impact.

D-GSH must be properly dispersed to avoid agglomeration in the cement matrix in order to improve the mechanical strength of 3DPC. In this study, D-GSH was dispersed in water at concentrations of 0.3% and 0.5% of the weight of the sand using an ultrasonic wave mixer for approximately 40 minutes. The dispersed solution was then mixed with the cement and sand to obtain a cement mortar.

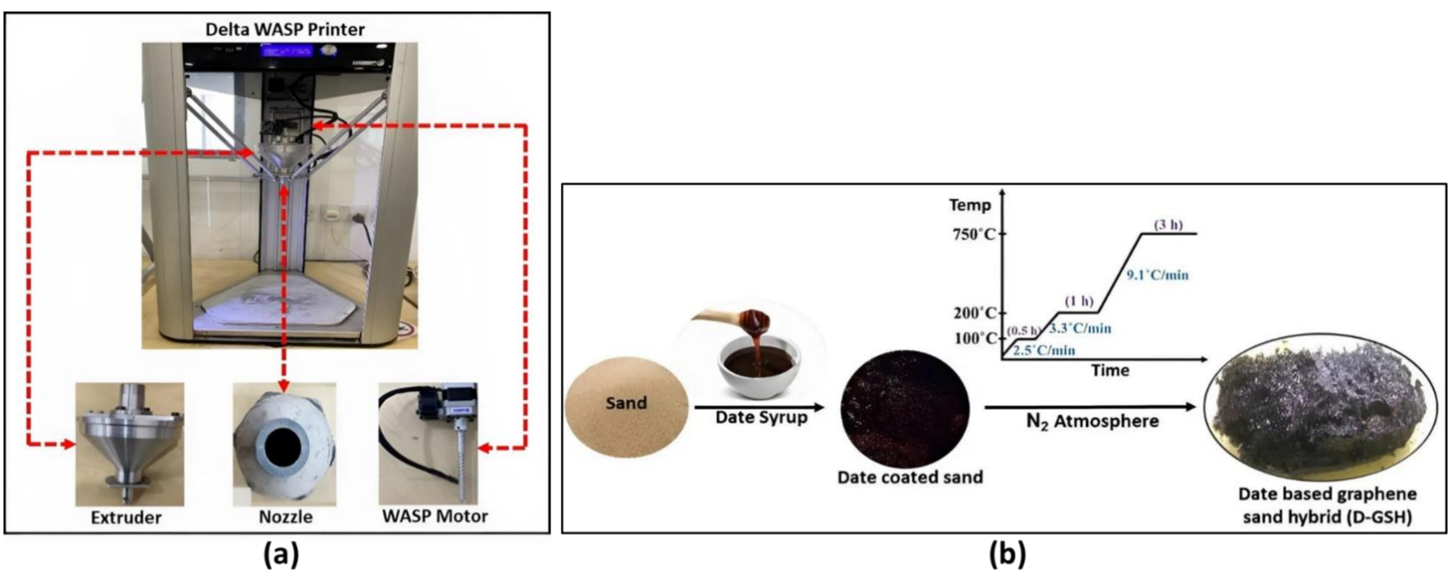

Fig. 1: (a) Delta WASP 3D-printer system with a custom-designed extruder; and (b) D-GSH synthesis process [8].

2.2. Mix design and Experimental procedure

The optimal mix ratios containing D-GSH at concentrations of 0.3 wt% (mix-D03) and 0.5 wt% (mix-D05) were considered based on the study by Ali et al. [9] to compare the results with a mix containing 5 wt% silica fume (mix-S5). Table 1 shows the mix ratios expressed in mass percentage. It should be noted that the concentration of D-GSH is expressed as a percentage by weight of sand; however, if expressed by weight of cement, the corresponding D-GSH contents would be approximately 0.08% for mix-D03 and 0.14% for mix-D05. The water/cement ratio (w/c) of mix-S5 is 0.40, while it is 0.39 for the mixes containing D-GSH. For the mixtures containing D-GSH and silica fume, a beam with the dimensions 150×40×40 mm was printed. The average geometric variation between 3D-printed beams and design was less than 5%. To investigate the anisotropic behavior of 3DPC, three-point bending tests were performed on the 3D-printed specimens after 7 days of curing using the INSTRON tester, varying the loading directions with respect to printing direction as shown in Figure 2. In direction 1 the load was applied parallel to the printing direction and in direction 2 perpendicular to the printing direction. Three samples were considered for each mix, and the mean flexural strength was determined using Equation (1).

$$Flexural\ strength = \frac{3PL}{2wh^2} \tag{1}$$

where P, L, w, and h are peak load, length, width, and depth of the 3D-printed beams.

Table 1: Mix proportions [9].

Mixes	Cement (%)	Sand (%)	Water (%)	Silica fume (wt%)	D-GSH (wt% by sand weight)	Superplasticizer (wt%)	w/c
S5	55.00	18.00	22.00	5.00	-	-	0.40
D03	59.90	16.30	23.40	-	0.30	0.10	0.39
D05	59.90	16.10	23.40	-	0.50	0.10	0.39

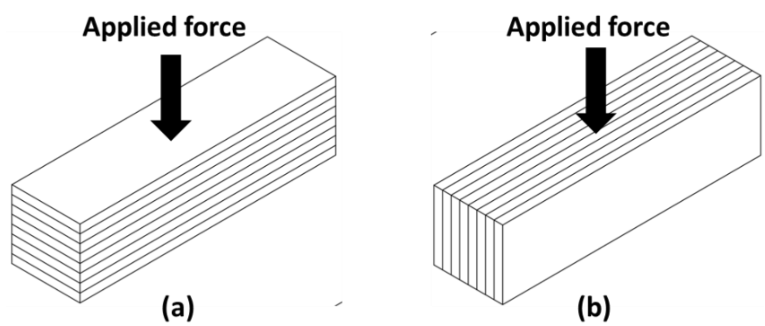

Fig. 2: Loading orientations for three-point bending test: (a) Direction-1 (parallel to printing direction) and (b) Direction-2 (perpendicular to printing direction).

3. Results and discussion

Figure 3(a) shows the 3D-printed beams of the printable mixes, namely mix-S5 (5 wt% silica fume), mix-D03 (0.3 wt% D-GSH), and mix-D05 (0.5 wt% D-GSH). Figures 3(b) and (c) show the maximum load and flexural strength of these mixes in the two different load directions. The values in direction- 1 for mixes containing silica fume and D-GSH are indicated by green color bars, while red color bars are used for direction 2. The standard deviation is shown in the form of error bar. The flexural strength in direction-1 was higher than in direction-2 for all printable combinations. Mix-S5 showed a 25% higher flexural strength in direction-1 compared to the other directions. Similarly, the strengths in direction-1 for mix-D03 and mix-D05 were 11.6% and 9.5% higher than those of direction-2, respectively. The improvements in direction-1 for all mixes were due to the higher load-carrying capacity of the 3D-printed beams, as shown in Figure 3(b). In contrast, the weaker interlayer filaments of 3DPC resulted in lower flexural strength in direction-2. However, the layer interfaces of the printed structures were improved by the addition of D-GSH, as evidenced by a 109% and 137% increase in the flexural strength of mix-D03 and mix-D05 in direction-2 compared to mix-S5 in the same direction. The addition of D-GSH also reduced the variation in flexural strength between the two directions. These improvements emphasize the positive effects of D-GSH on overall strength.

Researchers have reported that the orientation dependence of 3DPC helps to bridge micro-cracks, resulting in increased flexural strength, especially under perpendicular loading conditions [11]. Ali et al. [9] conducted a microstructural investigation and found that D-GSH effectively fills voids, bridges nano-sized cracks and develops strong bonds with the hydration products in the cement matrix. The addition of carbon nanotubes also leads to a nanoscale bridging effect in the cement matrix, leading to enhancement in strength [10, 12]. According to Ding et al. [13], the bending performance of 3D-printed structures is influenced by the anisotropy caused by the layered structure and the thin filament surfaces. Thus, when a force is applied in direction-2, the stress is perpendicular to the weakened layer interfaces, resulting in a decreased flexural strength. Panda et al. [14] studied the flexural strength of glass fiber-reinforced geopolymer under three loading directions: perpendicular to the printing layer, parallel to the layer, and perpendicular to the printing direction. The highest strength was recorded when loading was applied perpendicularly to the printed layer, attributed to better compaction and stronger interlayer bonding. Lower strengths in other directions were due to weaker layer-to-layer adhesion. On the other hand, Ziejewska et al. [15] investigated the flexural strength of 3D-printed concrete-geopolymer hybrids and discovered it to be anisotropic, with samples bent parallel to the printing direction exhibiting 5-16% higher strength than those bent perpendicular to it. However, in this study, the addition of D-GSH strengthened the interfaces between the layers, minimizing the differences in flexural strength between the loading orientations.

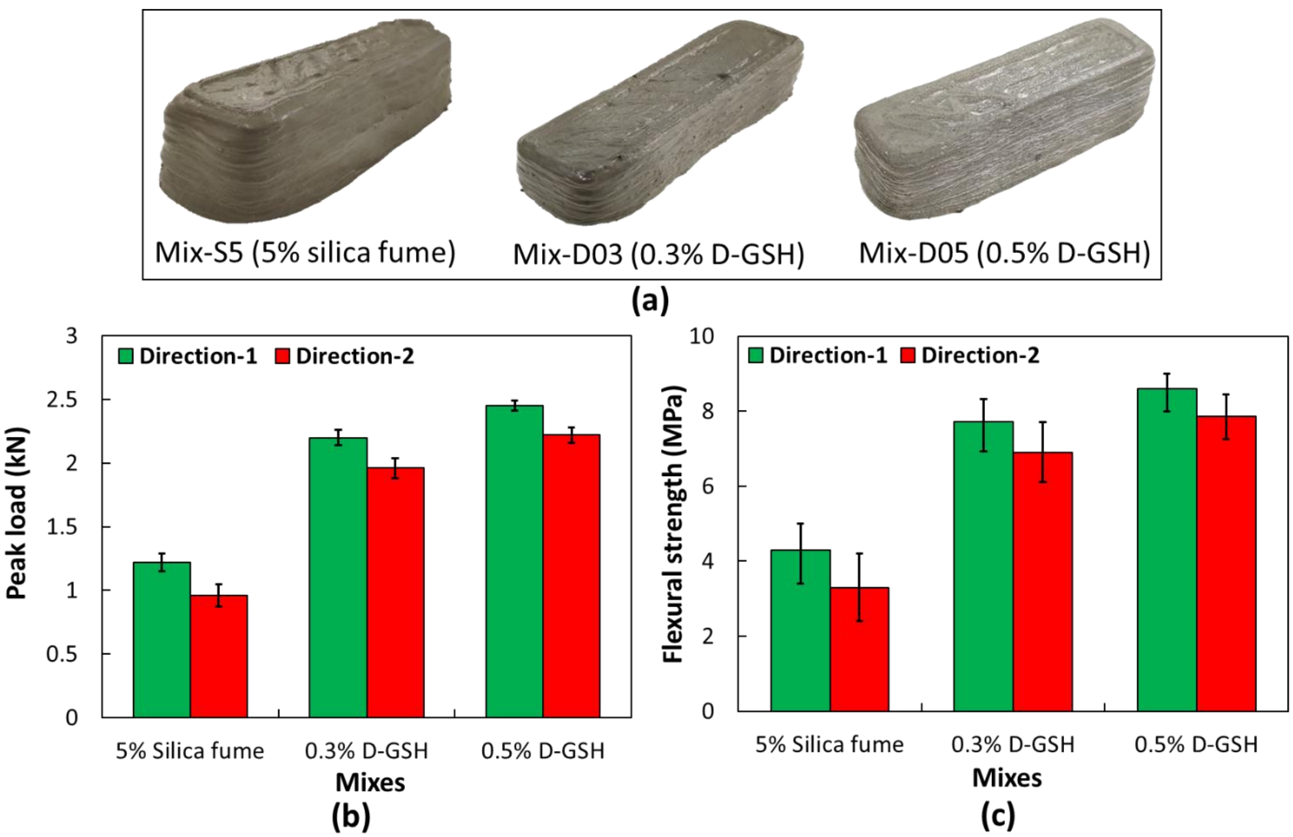

Fig. 3: (a) 3D-printed beams when vertical direction is the printing direction; (b) peak load vs. mixes: and (c) flexural strength vs. mixes in two different loading directions.

4. Conclusion

The anisotropic behaviour of 3DPC was investigated by comparing mixes containing D-GSH with a mixture containing silica fume. The flexural strength was determined by varying the direction of loading. The force applied parallel to the printing direction resulted in better load distribution and stronger bonding, which led to an increase in load-carrying capacity and flexural strength. Conversely, the strength was limited by a weaker bond between the layers when the load was applied perpendicular to the printing direction. As a result, the mix containing 5% silica fume showed a 25% increase in flexural strength when loaded perpendicular to the printed layers compared to the other orientation. In addition, a mix containing 0.5% D-GSH exhibited a 9.5% increase in flexural strength. These results suggest that the addition of D-GSH reduces the difference in strengths between the loading directions, resulting in a stronger interlayer bonding of 3DPC.

Acknowledgements

The authors acknowledge research funding of ADAM (Advanced Digital & Additive Manufacturing) Group (No. 8474000163) at Khalifa University.

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