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An Introduction to High Performance Graphene Concrete

T. Ginigaddara[a*], J.C. Ekanayake[b], P. Mendis[a], P. Devapura[c], A.D. Liyanage[c], P. Vaz-Serra[a]

[a] *University of Melbourne, Australia*
[b] *University of Moratuwa, Sri Lanka*
[c] *Ceylon Graphene Technologies, Sri Lanka*
**Corresponding Author: tginigaddara@student.unimelb.edu.au*

ABSTRACT: Developments in nanomaterial technology have generated a strong research interest in the construction industry aiming at enhancing the properties of concrete. Many studies have explored the use of engineered nanomaterial such as nano-silica, carbon nanotubes (CNT) and nanofibers in cementitious composites. Recently, nanomaterial studies have focused on Graphene and Graphene Oxide (GO). Graphene is the single atomic layer thick two-dimensional form of graphite and GO is the oxidized form of graphene which is synthesized by oxidation of graphite. Investigations have demonstrated that the use of GO in cementitious composites can enhance their performance. This paper outlines the development of a high-performance graphene-based concrete. Moreover, the paper presents a brief review of previous studies conducted on GO induced cementitious composites and remarkable performance enhancement enabled by GO. The findings of this study contribute towards establishing how GO can be adopted as a nanomaterial additive for concrete.

Keywords: graphene oxide, nanomaterial, high performance concrete

1 INTRODUCTION

Concrete deliver high compressive strength, better durability at a relatively low cost when compared with other construction materials such as timber and structural steel. However, the inherent weaknesses of concrete include low tensile strength induced brittleness, ductility, low strength to weight ratio and propensity for cracking which have limited many applications of concrete structures. Moreover, the continuous development of global construction standards demands advanced construction materials, which can facilitate the needs of modern infrastructure. The properties of cement-based construction materials primarily depend on calcium silicate hydrate (C-S-H) nanoplatelets which is the key product of the hydration process of cement. Moreover, the most common defects and failures in concrete are related to the cement binder. Investigations on these factors have revealed that nanoengineering and nanomodification of cementitious composites can facilitate macro scale performance improvements. Consequently, countless studies have been conducted in the last few decades with the addition of various nanomaterials such as nano-silica, nano-alumina, nano calcium carbonate, nano titanium dioxide, nano ferric oxide, carbon nanofibers (CNFs), and carbon nanotubes (CNTs) in cementitious composites (Bjegović, Serdar, & Štirmer, 2018; Chuah, Pan, Sanjayan, Wang, & Duana, 2014; Cwirzen, 2010; Debia et al., 2016; Silvestre, Silvestre, & De Brito, 2016; Sobolev, 2016).

Nanomaterial products used in concrete can be categorized as nanoparticles, nanofibers and nanosheets. The applications of nanoparticles include nano-silica and nano-titanium oxide while the applications of nanofibers include carbon nanofibers (CNF) and carbon nanotubes (CNT). Zhou, et al. reports that between the discovery of CNTs in 1991 and 2017, more than 135,000 scientific studies have been conducted on CNTs (Zhou, Wang, & Wang, 2017). Most construction related nanomaterial studies include nanomodifications to improve mechanical properties and durability performance of concrete and other cementitious composites. These nanomodifications aim to enhance the performance of concrete through phenomena such as chemical reactions with C-S-H. The fundamental mediums by which the nanomaterials deliver improved performance for cementitious composites can be identified as the filling effect and seeding effect (Shah, Hou, & Konsta-Gdoutos, 2016). Through the filling effect of nanomaterials, a compact and dense microstructure is generated by filling the pores in the nanoscale. On the other hand, the seeding effect facilitates strength improvement through the additional surface area provided for an enhanced hydration process. Regardless of decades of investigations, the construction industry is yet to absolve the use of nanomaterials such as CNFs and CNTs in a global scale. Li Zhao et al. argues that the cost intensiveness, difficulty of dispersion and challenge of scaled up production are the key rea-sons behind the lack of applications of nanomaterials in construction industry (Zhao et al., 2020). However, in the last decade of nanomaterial technology and research, scholars have carried out extensive investigations into Graphene. Studies have demonstrated that the use of graphene-based nanomaterials can deliver remarkable performance enhancements for cementitious composites such as mortar and concrete.

2 GRAPHENE

Graphene is a 2D nanosheet which translates to the two-dimensional form of graphite. Pristine graphene presents a honeycomb framework with an approximate thickness of 0.335 nm, having the ability to wrap into 0D fullerenes, into 1D nanotubes or stack into 3D graphite (Giannazzo, Sonde, & Raineri, 2011). Figure 1 illustrates the schematic structure of carbon allotropes including pristine graphene, fullerenes, nanotubes and graphite.

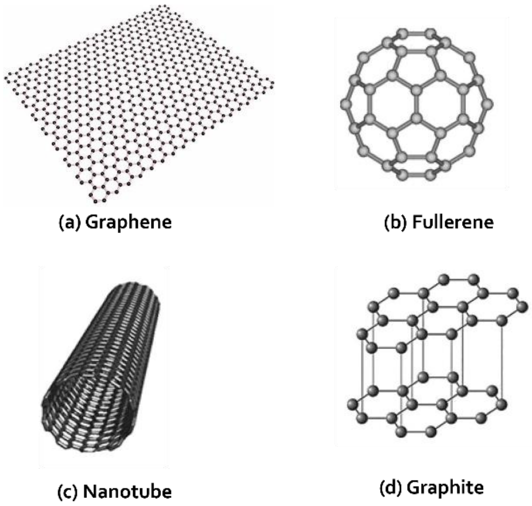


Figure 1. Schematic Structure of (a) Graphene, (b) Fullerene, (c) Nanotube and (d) Graphite

Graphene received strong research momentum and widespread attention due to its remarkable properties and applications. As per the International Standards organization (ISO), graphene is defined as one atom thick layer of graphite (Nanotechnologies, 2017). Graphene was a known material for scientists and many studies such as graphene-based fullerene, CNT and CNF were carried out for a long period time. However, it was not until the findings of Novoselov and Geim in 2005, the successful fabrication and identification of graphene was reached (Novoselov et al., 2005). The most noteworthy property of graphene is its strength and the lightweight. Graphene has a tensile strength of 130 GPa and an elastic modulus of 2.4 TPa. The electrical conductivity of graphene is 100 MS/m and it demonstrates great potential in the energy sector. Although many of these findings were unravelled in the last decade, graphene applications have not reached the international market yet. The key reason behind this is the challenge of graphene sheet manufacturing in the industry scale. Although some studies have presented manufacturing of small size graphene sheets in laboratory scale, the challenge of production of pristine graphene in industry scale persists.

There are two key methods to produce graphene and they are known as the “Top-down method” and the “Bottom-up method” (Biswas et al., 2012; Poniatowska, Trzaskowski, & Ciach, 2019). The bottom-up method includes chemical vapour deposition (CVD) and the top-down method involves the synthetization of graphene from its three dimensional form; graphite (Arole & Munde, 2014). Previous studies that have been carried out include both these methods and graphene nanomaterial research have many applications of graphene-based materials such as graphene nanoplatelets, graphene oxide (GO) and reduced graphene oxide (rGO) (Yang et al., 2012). GO is the oxidized product of graphite having few graphene layers. GO consist of several layers of wrinkled two-dimensional carbon sheets with various oxygen containing functional groups such as hydroxyl, carboxyl, and epoxy on its surface or between the layers (Thickett & Zetterlund, 2013). The following section provides a brief review of existing studies on the use of GO in cementitious composites.

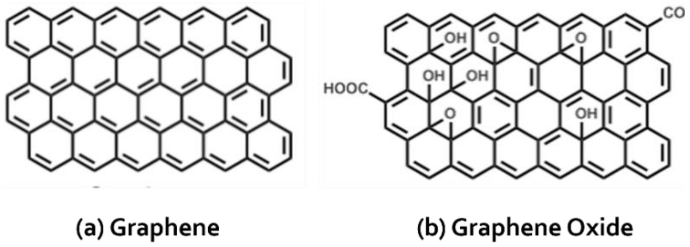
3 USE OF GO IN CEMENTITIOUS COMPOSITES: A REVIEW OF PREVIOUS STUDIES

GO is known for its potential to improve the mechanical performance of cementitious composites such as the compressive strength, tensile strength and flexural strength. Further, GO presents functionalities of self-cleaning, self-sensing, thermal interfacing, and electromagnetic shielding. The widespread research interest in GO is mainly due to its ability to deliver performance enhancement for cementitious composites even when added in very low ratios. Similar to other nanomaterial such as nano-titanium and nano-silica, GO has been incorporated into construction materials with the aim of nanomodifications to materials (Bjegović et al., 2018; Chuah et al., 2014; Silvestre et al., 2016).

In comparison to the abundantly researched areas such as CNTs and CNFs, only a limited number of studies have been carried out on GO applications in cementitious composites. Thus, it can be identified that the development GO reinforced cement composites is a relatively new research direction. The first article published on GO reinforced cement was published in 2011 and very few studies have been published before 2014. However, after 2015 a sudden surge in GO reinforced cementitious composite based research can be identified. The multiple forms of GO such as nanosheets, nanoplatelets and aggregates are currently being studied on. Although studies are extensively carried out in this area at present, many hindrances limiting the real-world applications are yet to be addressed such as agglomeration, stating that GO based research is still at its preliminary stages. Moreover, GO and graphene concrete shows promise to be manufactured in large batches with higher reproducibility, and it poses less hazards to health when compared to other carbon-based nanomaterials.

Derived form more mature research carried out on carbon-based nanomaterials such as CNTs and CNFs, GO incorporated cementitious composites have illustrated remarkably enhanced performances. Previous studies have shown that GO modified cementitious mixtures deliver compressive strength, tensile strength and flexural strength improvements of 77.3%,78.6% and 78.3%, respectively (Wang, Xu, Wu, Zhang, & Hu, 2020). These remarkable findings show promise on transforming the engineering design of concrete structures, new building forms and designs. In addition to the mechanical performances in graphene and GO incorporated mixtures, studies report improvements in sulphate attack resistance with a decrease of transport properties. Moreover, studies show that the microstructure of the GO modified mixtures have improved degree of hydration, pore structure refinement, higher rate of hydration and reduced crack propagation (Mohammed, Sanjayan, Duan, & Nazari, 2015).

The key areas of GO concrete research include the workability of GO based cement composites, mechanism and formation of the microstructure of hydration products, mechanical properties of these cementitious composites, volume stability and durability (Pillay et al., 2020). Therefore, the mechanism by which the remarkable mechanical properties are gained have been extensively studied by researchers. Researchers have carried out nanoscale investigations to determine the causations behind the correlation between improved performance of cementitious composites and GO. The 2D nature of GO provides a higher specific surface area (SSA). The hydrophilic nature of GO makes it a better candidate in cementitious composites when com-pared hydrophobic derivatives of graphene such as rGO and pristine graphene (Liu et al., 2021). GO mainly include hydroxyl, carboxyl and epoxide functional groups attached to the basal and edge planes of the nanoscale structure. Figure 2a and 2b illustrates the microstructure and functional groups of Graphene and GO respectively.



(a) Graphene **(b) Graphene Oxide**

Figure 2. a) Microstructure of Graphene, b) Microstructure of GO

This enables GO to exhibit higher reactivity and high dispersibility within the cement matrix. Graphene with lower number of functional groups than GO, is called rGO, thus resents intermediate properties between GO and Pristine graphene. Therefore, the incorporation of GO in cementitious composites have given better results over mixing rGO into cementitious composites (Qureshi & Panesar, 2019).

The hydration process of cement (reaction with water) generates products such as C-S-H, ettringite, and monosulfates. However, the degree of hydration in normal concrete typically varies between 65% - 75%. Thermogravimetric analyses and heat of hydration calorimeter tests carried out on graphene concrete have revealed that the degree of hydration of graphene concrete is approximately 89%. These investigations provide strong evidence that GO has increased the degree of hydration and delivered effective and efficient use of cement which is otherwise unhydrated in normal concrete. Moreover, the studies show that the rate of hydration is also improved by incorporation of GO. Zhao, et al., 2020 demonstrates an early peak under heat of hydration of a cement sample which contained 0.04% GO by weight of cement (bwoc).

Regardless of the significant strength improvements, studies report that GO addition can reduce the fluidity of the cement mixtures, increasing its viscosity and reducing the workability. According to Wang et al. when 0.05% GO (bwoc) was added to the cementitious material, the fluid loss of the slurry was at 70% with a 1.850% increase of viscosity. Since GO is hydrophilic, GO incorporated cement composites tends to have increased viscosity. Moreover, it is supported by the 2D planer honeycomb lattice microstructure and high specific surface area of GO. Studies have been carried out to improve the rheological properties of these cement-based composites but the compromise on workability continues to be a leading challenge. In concrete, the addition of coarse aggregates further increase the resistance due to internal friction, reducing the workability. Hence, further studies are required to improve the workability and slump loss of concrete, by examining the feasibility in implementing novel molecular cutting and grafting technology and the application of GO in multifunctional chemical admixture.

Exploring the formation mechanism of cement hydration products are required to determine the performance of these cement composites. Studies have shown that GO has a substantial effect in the hydration of cement. According to Lv, et al., 2013 with the presence of GO in the mixture, the hydration products develop as a well ordered crystalline or into petal like shapes. Further, the pores in the cementitious composite becomes more uniform and diminish in size as GO sheet layers have higher strength and toughness, enhancing the growth of the hydration products of cement (the pores were more uniform and smaller as GO had the sheet layers with higher strength and toughness, and hence enhancing the growth of cement hydration products). The findings of Lv, et al., 2013 was further supported by Jiang and Wang, et al., 2015 Contrastingly, the study conducted by Horszczaruk, et al., 2015 argues that the addition of GO causes no significant difference in the shapes and formation of the cement hydration products. Cui et al. further disapproving Lv, et al., 2013, Cui, et al., 2017 states that the petal shaped, or crystalline substances are not hydration products but the results of carbonation reactions (formation of Calcium carbonate) that takes place during cement sample preparation. Further studies were conducted by Lv, et al., 2013 to comprehend the crystallization and shape of hydration products through controlling the shapes of the hydration products via GO nanosheet doping. However, altering the microstructure of GO based cement composites to obtain the desired performance is a promising future direction.

The volume stability of a cement composite affects the cracking sensitivity of a concrete structure related to autogenous shrinkage, drying shrinkage, plastic shrinkage, creep and deformation due to temperature. The changes within the cement composite microstructure, change of hydration product density and viscous flow are related to the aforementioned deformations affecting the volume stability. Although this is the case, only few studies have addressed the volume stability of GO based cement composites. It’s noteworthy that most prevailing studies have employed GO based cement slurry or mortar with limited studies employing GO in concrete.

Comparative to the mechanical properties, the durability of GO incorporated concrete based research is still in the primitive phase [17]. A wide scope of causes including reinforcement corrosion due to chloride penetration or carbonation, freeze-thaw cycles and alkali aggregate reaction affects the durability of cementitious composites. Studies have shown that GO shows direct correlation with corrosion resistance of GO reinforced concrete. Studies conducted by Mohammed, et al., 2015 concluded that GO can reduce the depth of penetration of Chloride ions. This reduction in the depth of penetration can be explained by the sponge–shaped structure formed by the interconnected GO layers that traps the chloride ions. Tong et al concluded that anti-freezing performance of GO incorporated mortar was far less superior than standard mortar, conceivably due to the adsorption and desorption occurring in nano level pores of the mortar. According to Lv, et al., 2013, GO incorporated cementitious composites can decrease the depth of permeability by 72%, reduce the 28-day depth of carbonation by 66% and improve the dynamic elastic modulus by 70% after 100 freeze-thaw cycles. The orderly formation of crystal-like hydration products that reduces the racking and hazardous pore formation within the microstructure due to the addition of GO nanosheets can improve the durability of the composite. Studies shed concern on the reduction of long-term strength of cement composites under high temperature settings due to the delayed formation of Calcium alum. It has raised attention towards the effects of high temperature to the properties of GO incorporated concrete. Only a limited number of studies so far address the issue of whether GO could prevent the long-term strength loss in concrete. A widely concerned issue in construction is material fatigue. Hence, the ability of GO to improve the fatigue resistance of cement composites is a major concern.

A large number of studies are found in existing literature regarding the mechanical properties of GO incorporated cement composites in which GO have been mixed in varying proportions. Despite the promising results on strength enhancements, the reported improvements are still inconsistent. Existing studies emphasize a wide range of strength improvements in GO reinforced concrete, ranging from 15% to 160% in compressive strength gain and 18% to 185% gain in tensile strength (Anwar, Mohammed, Wahab, & Liew, 2020; Chuah et al., 2014; Xu et al., 2018). These inconsistencies of strength gain can be partially explained by the mix percentage of GO into the cement composite, ranging from 0.02% to 4%, different methods of GO synthesis and different sources of graphite (Chuah et al., 2014; Xu et al., 2018). However, most studies have incorporated the modified hummer's method to obtain GO from natural graphite. Apart from the different mix percentages of GO, factors such as size, charge, surface chemistry and the defects of the graphene structure can also affect the resultant mechanical properties of these composites.

Moreover, most of existing studies are limited to the use of GO in cement paste and cement mortar, whereas only a handful of studies have investigated the use of GO in concrete. The lack of systematic scientific studies on the use of GO in concrete in industry scale is one of the key reasons why the GO induced concrete applications have not reached the construction industry yet. The following section of this paper presents the findings of a systematic scientific study of a graphene concrete in which the induced GO was synthesized using an industry scale manufacturing facility.

4 EXPERIMENTAL PROGRAMME

4.1 *Materials*

The modified Hummer's method has been adopted to synthesize GO in an industry scale manufacturing facility. The GO was synthesized from the purest form of graphite, known as vein graphite which has more than 98% of carbon purity. The source of graphite is Kahatagaha mine, Sri Lanka and GO was synthesized as an aqueous solution of 10g/l concentration. The concrete mix design was developed using grade 43 Ordinary Portland Cement (OPC), river sand for fine aggregate and crushed natural coarse aggregate of 20 mm maximum nominal size. Moreover, a polycarboxylate based superplasticizer (PCE) was used in the concrete mix.

4.2 *Mix Proportions and Casting*

In order to conduct tests on fresh concrete and hardened concrete, a total of four mixtures were developed. The water/binder ratio of all mixes were maintained at 0.45 with 456 kg/m^3 cement content and 205 kg/m^3 of water. The amount of coarse and fine aggregates in 1 m^3 of concrete were 1175 kg and 579 kg respectively. Detailed mix proportions for each mix are illustrated in Table 1.

Table 1. Concrete Mixture Proportions (Kg/m^3)

Mix ID	**Water**	**Cement**	**Coarse Aggregate**	**Fine Aggregate**	**GO (% bwoc)**	**PCE**
M1	205	456	1175	579	0 (0%)	0
M2	205	456	1175	579	0 (0%)	5.8
M3	205	456	1175	579	0.1368 (0.03%)	5.8
M4	205	456	1175	579	0.3648 (0.08%)	5.8

As shown in Table 1, a polycarboxylic ether based superplasticizer (PCE) was used in M2, M3 and M4. M3 and M4 contained 0.03% and 0.08% GO (bwoc) respectively. In the mixing process, GO was mixed in PCE to maintain an effective dispersion of GO nanoparticles. Subsequently, the solution containing GO and PCE was mixed with water and poured into the dry concrete mix. Any sophisticated mixing methods such as ultrasonication or high shear mixing was not adopted in the process, ensuring that the method can be replicated in on site construction activities.

4.3 *Results and Discussion*

As previous studies have discussed and demonstrated, the workability of fresh concrete was affected by addition of GO. This is associated with the amount of free water adsorbed to wet the large surface area of GO nanoparticles. The workability of four fresh concrete mixes was assessed through the slump test and per BS EN 12350. However, the use of PCE in the concrete mixtures aided in maintaining adequate workability in fresh concrete.

4.3.1 *Compressive Strength Test*

The compressive strengths of specimen were tested at 7 days and 28 days periods as per BS EN 12390. Figure 3 shows the variation of compressive strength at different curing periods.

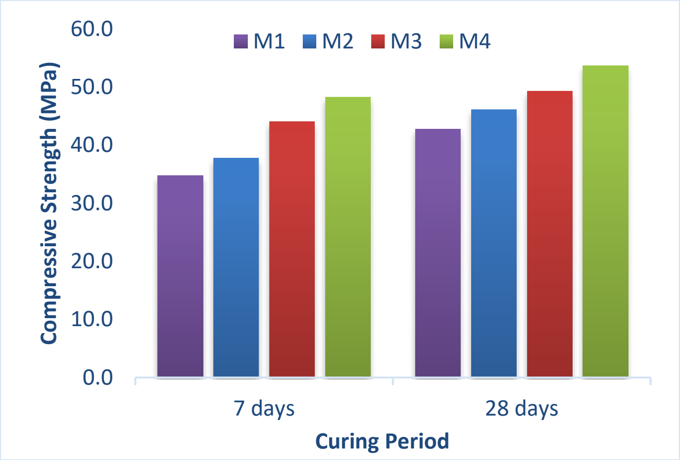


Figure 3. Compressive Strength of Concrete Mixes

Concrete containing 0.08% GO (M4) demonstrates the highest compressive strength at both 7 days and 28 days curing periods. The strength increments of M2, M3 and M4 mixes with respect to M1 mix is demonstrated in Figure 4.

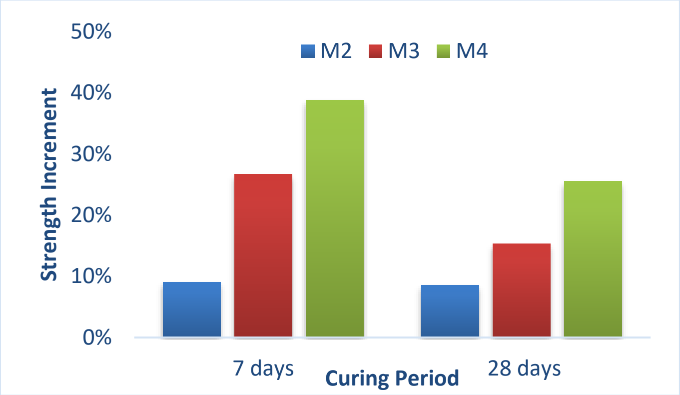


Figure 4. Strength Increment of Concrete Mixes

M4 demonstrated the highest strength gains at both 7 days and 28 days curing periods with 39% and 26% increments, respectively. M3, containing 0.03% GO demonstrated a 27% strength gain at 7 days and subsequently a 15% gain at 28 days. The results indicate a significant positive effect of GO on mechanical characteristics of concrete.

Specially, the early age strength gain of graphene concrete is a significant advantage as it is a critical element in construction processes. Many applications of the construction industry such as formwork related construction duration, post tensioning, initiation of micro cracks, early age shrinkage and thermal contraction. As graphene concrete has shown improved compressive strength at 7 days, it shows promise on achieving the required early strength in a shorter period of time that 7 days. This can be advantageous to improve the current practices in the construction industry reducing both time and cost.

Although there are limited studies on the use of GO in concrete, studies on cementitious composites argue that GO plays a catalytic role in the cement hydration process. As mentioned in the review section of this paper, the functional groups of GO facilitate H_2O rich nucleation sites for the cement particles to bond and generate hydration crystals. It can be identified that the significant early strength increment observed in this study, is closely associated with the accelerated cement hydration rate and increased 28-day compressive strength due to improved degree of hydration.

5 CONCLUDING REMARKS AND FUTURE DIRECTIVES

Graphene based concrete, introduced in this study, consist of GO synthesized from Vein Graphite. Vein graphite is the purest and the best form of graphite of the world and the only geographical location where vein graphite is found is in Sri Lanka. Vein graphite has a carbon purity of 99.5% as it is free from impurities. Moreover, it has the best crystallinity when compared to all other graphite sources in the world. The high purity and better crystallinity make vein graphite a leading candidate for industry scale manufacturing of graphene-based concrete. The crystallinity and purity of vein graphite is ideal for delivering consistent results when used in cementitious composites. The GO produced by CGT has been endorsed by experts as a very high quality material and can be sued in many applications.

It has been found that the processing method of the additive, that is how GO and PCE is mixed has an effect on the strength gain. Further improvements are expected with this on-going work at University of Melbourne and CGT.

The significant strength gain of graphene-based concrete deliver many advantages and create opportunities for a wide variety of application. For an instance, graphene-based concrete can facilitate large reductions of carbon emissions in the construction industry through the low quantity of concrete required to attain the desired strength capacities. As of now, the concrete industry is identified as the second biggest carbon dioxide emitter and the third largest industrial energy consumer of the world. Statistics show that the manufacturing of cement contributes for 6% of global carbon emission (Chuah et al., 2014). Graphene based concrete aim to provide a sustainable solution for this growing concern of carbon footprint in the construction industry.

As concrete is the most widely used construction material, future research on GO induced cementitious composites should predominantly focus on Concrete. One of such future research focuses is directed at tall buildings. The enhanced mechanical performance of graphene-based concrete enable the introduction of novel building forms and high-rise architecture. The architectural freedom for new high rise building designs and building forms are limited due to the constraints in construction materials. For an instance, the lack of tensile and compressive strength capacities of concrete have limited the span of beams. The large cross sections of long spanning beams are not only architecturally overruled, but they are also economically ineffective. Graphene based concrete may have a solution to these problems since it can provide improved strength to concrete and in turn deliver many benefits. This is part of a large research project conducted at the University of Melbourne with the help of CGT.

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