NanoWorld Journal

https://doi.org/10.17756/nwj.2023-s5-001

Research Article

Open Access

# Pull-out Test Behavior for Graphene Nanoengineered Concrete

**Suehail Aijaz Shah and Manzoor Ahmad Tantray***

*Department of Civil Engineering, National Institute of Technology, Srinagar, Jammu and Kashmir, India*

***Correspondence to:**
Manzoor Ahmad Tantray
Department of Civil Engineering,
National Institute of Technology, Srinagar,
Jammu and Kashmir, India.
**E-mail:** matantray@nitsri.ac.in

**Received:** October 11, 2023
**Accepted:** December 18, 2023
**Published:** December 21, 2023

**Citation:** Shah SA, Tantray MA. 2023. Pull-out Test Behavior for Graphene Nanoengineered Concrete. *NanoWorld J* 9(S5): S01-S06.

Published by United Scientific Group

## Abstract

Cement concrete is the most commonly employed material in infrastructure development. The introduction of reinforcement with a higher tensile strength makes up for the relatively low tensile strength of cement in reinforced cement concrete. The effective load transmission between concrete and steel reinforcement by bonding action is one of the most crucial factors in the design of bar-reinforced concrete members. Being the material of the twenty-first century, graphene possesses several qualities, including high flexibility, Young's modulus, and tensile strength. It is a suitable material for reinforced cementitious composite because of its exceptional qualities. Impermeable carbon-based nanoparticles with a high surface-to-volume ratio are known as graphene nanoplatelets (GNPs). GNPs are highly outstanding since they have greater mechanical, thermal, and barrier potential, attributed to their high aspect ratio and plate-like structure. GNPs with exceptional mechanical properties have been widely used as reinforcing materials in a variety of sectors. This study analyzes how the addition of graphene reinforcement to cement concrete can enhance its qualities. The compressive strength and bond strength of steel with concrete composite have been proven to be improved more effectively by GNPs. This research offers a fresh perspective on how GNPs have improved the cement composite.

## Keywords

Graphene nanoplatelets, Superplasticizer, Workability, Strength, Bond strength

## Introduction

The most often utilized building material worldwide is concrete. It doesn't exist as an independent construction material as it constitutes of different ingredients. Each constituent contributes its specific property leading to the development of this marvellous material. With the advances in technology, research, and development, concrete has also developed from basic normal concrete to high-performance concrete. It has completely revolutionized the traditional mixes used in ancient times. The composite materials made of reinforcement with better tensile or durability strengths compensate for the relatively poor ductility and strength of concrete. It is evident from the research that fracture, unlike plastic yielding, causes failure to concrete. Numerous studies on this subject have been conducted, and the results suggest that to restrict the growth of micro-level cracking, nanoscale cracks must be halted to stop them from spreading to the micro-scale [1].

The development of industrial technologies for use in strengthening concrete has also advanced rapidly. Traditional cementitious composites are fragile and prone to fracture. Research and innovations in science and technology that began in the 21st century gave rise to nanotechnology, this innovation developed material in control dimensions between 1 and 100 nm. For the past decade, cement-based products have frequently used nanomaterials, such as nanoparticles or nanofibers. These nanoparticles control crack initiation and development at the microscopic level. For the optimum design of reinforced cement concrete members, the dependable transmission of forces between reinforcement and concrete is desired. By creating a strong relationship between the two parties to transfer stresses between concrete and the strengthening reinforcing steel, strain compatibility and composite action can be created.

As a result of the growth of nanotechnology in the field of construction industry, numerous nanoparticles developed are being used within the cement matrix. Various carbon allotropes at the nanoscale such as carbon nanotubes, graphene oxide, and GNPs have found their place in the construction industry. Because of its outstanding chemical, mechanical, electrical, optical, and thermal capabilities, graphene has been employed in cement-based nanomaterials. Graphene has emerged with a new possibility as a nanosized cementitious additive. The origin of all graphitic forms is graphene and possesses versatile properties as enlisted in table 1 [2]. Due to easier synthesis from graphite or graphene oxide, multi-layer GNPs are more often used in practical applications. Future infrastructure can have a significant improvement in damage resistance and service life attributable to cementitious materials with improved performance.

Recently, the use of GNPs has attracted a lot of interest, which are made up of a few layers of graphene sheets, as a nano reinforcement in cement-based composites. Figure 1 illustrates the schematic representation of GNPs [3]. GNPs have a larger surface-to-volume ratio, a significant specific surface area, and a two-dimensional structure. GNPs are an excellent material choice for the development of nano-reinforced cementitious composites. Many researchers have been exploring the use of graphene in cement and concrete in recent years. The present work carried out an investigation involving the study of bond characteristics developed between the reinforcement and the concrete doped by GNPs.

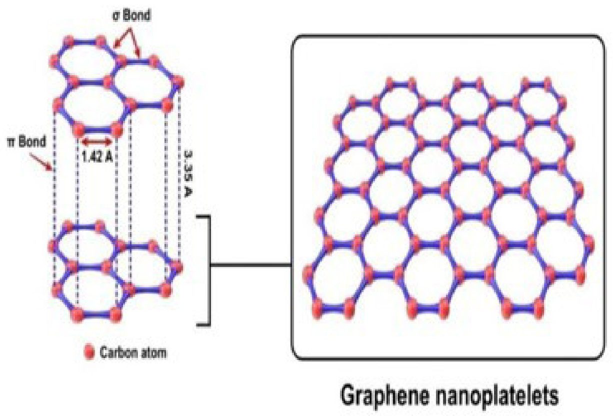

**Figure 1:** Schematic representation GNPs.

**Table 1:** Properties of GNPs [2].

| Property | Value |
|---|---|
| Young's modulus | 1.0 TPa |
| Intrinsic strength | 130 GPa |
| Electrical resistivity | 50 mΩcm (in-plane) |
| Thickness | ~30 nm |
| Surface area | ~2630 $m^2.g^{-1}$ |
| Aspect ratio | 50 - 300 |
| Thermal conductivity | 5.30 x $10^3$ $W.(mK)^{-1}$ |

Krstek et al. who shared the 2010 Nobel Prize in Physics for their work, discovered graphene in 2004. Graphene is pure carbon, and the carbon atoms are organized in a single-atom-thick sheet called a gingle. GNPs was produced from natural graphite, usually via chemical exfoliation, thermal stress, and shear, or in a plasma reactor. There are several different types of graphene, including sheets, powder, ribbons, and flakes. Depending on the size of the flake, it can be monolayer, multilayer, or nanoplatelet in powder form and is stable. The utilization of graphene in industrial applications has been made possible by several technological breakthroughs. Graphene, on the contrary side, was manufactured via high-shear liquid exfoliation of graphite powder, and it was utilized in concrete, in which it was found to significantly boost compressive and flexural strength with dense matrix formation [4].

Due to their widespread application, high efficiency, and enhanced compressive strength, cementitious materials are extensively used in the building sector. Low strain capacity, poor crack resistance, and low tensile strength are considered as the main limitations of this material. To solve the problem, researchers have concentrated on using alternate steel bars, fibers, and nanoparticles as reinforcing material. Nanomaterials offered more effective and efficient reinforcement by reinforcing the material at the nanoscale. The integration of steel bars or fibers increases the tensile strength of the material by delaying the development of microcracks (at the initial stage), leading to crack control at the nanoscale (before these micro levels develop into macro size cracks. Large-surface-area nanoparticles (particularly GNP) have been carefully examined for their capacity to serve as pore fillers plus seeding sites to create dense microstructure. As a potential replacement for the nanoscale reinforcement of cement-based composites and other structural materials, GNPs are now attracting a lot of attention.

According to Shuang and Baomin [5], incorporating graphene can produce a cement mix which is less viscous. Nanoparticles may adsorb calcium ions more effectively and have wider surfaces for overcorrosion, which results in free ion exchange. Adding well-dispersed graphene flakes greatly reduced the direction index of calcium hydroxide and stopped cracks from forming in a cement slurry, according to Liu et al. [6]. Tong et al. assert that the alteration of cement paste's microstructure and enhancement of the interfacial interaction between it and the surrounding precipitated C-S-H gel depend on graphene [7]. According to Qi et al. [8], highly dispersible graphene has been found to increase the cement mortar's toughness, including their impermeability and resistance to chloride penetration. Wang and Zhao [9] discovered that by incorporating small amounts of modified grapheme nanosheets into the cement paste, the penetration depth and coefficient of chloride ions could be reduced by 37% and 42%, respectively. The laboratory observations from the literature, which are presented in table 2, show that adding very small weight

% levels of GNP significantly improves the cementitious materials' strength qualities.

GNPs have evolved into one of the leading nano-material for enhancing the mechanical properties of modified concrete with a meagre substitution percentage. Moreover, by this combination positive results have been achieved in reinforcement. However, based on findings from earlier work, the significant task to address is the effective and efficient dispersion of nanomaterials. Ineffective dispersion techniques influence the surface properties of the nanomaterial in the solution which have an effect on the degree of agglomeration.

## Materials and Methods

### Materials

#### Cement

Khyber brand, ordinary Portland cement grade 43 cement confirming to IS 8112:2013 was used.

#### Fine aggregate

River sand was utilised as the filler material for the concrete matrix. Sand confirming to Zone II, was obtained from river Sindh, from its middle course near free from clay coatings and other debris.

#### Coarse aggregate

Crushed river bed boulders with nominal size of 20 mm, available with the local stone crusher zone were utilised. The obtained batch of aggregates was crosschecked for the nominal size requirement and for other deleterious materials.

#### Water

For the production of concrete, available portable tap water was utilised. The average pH of water was tested to be 6.7 to 7.0 and free from other undesired salts and materials.

#### Admixture

Superplasticizer (SP) Auramix-400 manufactured by Fosroc was used in our mixes. Auramix-400 is a advanced Low viscosity high performance SP, based on polycarboxylic technology.

#### Graphene

Hexorp (Mumbai, Maharashtra, India) supplied the graphene used in our experimental study [16]. According to the technical datasheet included with the product, table 3 lists the details of the GNPs that were used.

### Mix proportions

Table 4 displays a detailed mix proportion of specimens made in compliance with Indian standards. By varying the replacement ratios of GNPs, new design mixes with different replacement levels (0.025, 0.05, 0.10, 0.15, and 0.20%) were produced. The ratio of cement to water was regulated at 0.40. A SP dosage was required to create a true slump with a slump of 75 ± 10 mm for both standard and modified concrete. The application of ultrasonic dispersion of GNPs was utilized in order to reduce the agglomeration of nanoparticles in the mix. The partial fraction of SP and water batched for mixing was added with the GNPs followed by sonication for a period of 27 min in order to break particle agglomerates followed by the addition of same quantity to the designed mix.

### Methodology

#### Workability

By performing a slump test, the workability of the GNP-imbibed cement composite and plane concrete is assessed and compared. By adjusting the SP content in the corresponding modified concrete, a true slump of 75 ± 10 mm was maintained. The modification in each mix was made according to the demand to attain the true slump state.

**Table 3:** Properties of GNPs (as per TDS).

| Property | Value |
|---|---|
| Color | Black |
| Material form | Powder |
| Bulk density | 0.30 g/cc |
| Average length | 5 to 10 microns |
| Average thickness | 3 to 8 nm |
| Number of layers | 3 to 6 layers |
| Surface area | 180 $m^2/g$ |

**Table 2:** Summary of literature.

| Matrix | GNPs type | GNPs content %wc | Water/ Binder | Dispersion technique | Performance improvement | Ref. |
|---|---|---|---|---|---|---|
| Paste | GONPs | 0.03 | 0.3 | Mixer | 41% tensile strength at 28 days | [10] |
| | GNPs | 0.05 | 0.35 | Ultrasonication, SP | 15 - 24% compressive strength; 3 - 8% flexural Strength | [11] |
| Mortar | Multi-layer grapheme | 1 | 0.6 | Ultrasonication, SP | 21% flexural strength | [12] |
| | GNPs | 0.4 | 0.5 | Ultrasonication | 1700% fracture energy | [13] |
| UPHC | GNPs;<br>Diameter = 25 nm;<br>SSA = 300 $m^2/g$ | 0.30 | 0.2 | Ultrasonication, SP | 59% flexural strength;<br>40% tensile strength;<br>187% energy absorption capacity | [14] |
| | GNPs;<br>Diameter = 30 nm;<br>SSA = 150 $m^2/g$ | 0.30 | 0.2 | Ultrasonication, SP | 203% flexural strength;<br>45% tensile strength;<br>153% energy absorption capacity | [15] |

**Table 4:** Mix proportions.

| Mix proportion | GNP % | Cement (kg/$m^3$) | GNP (g/$m^3$) | FA (kg/$m^3$) | CA (kg/$m^3$) | Water (kg/$m^3$) | SP (g/$m^3$) |
|---|---|---|---|---|---|---|---|
| 0GNP | 0 | 385 | 0 | 738 | 1175 | 154 | 2118 |
| GNP025 | 0.025 | 384.90 | 96.25 | 738 | 1175 | 154 | 2176 |
| GNP05 | 0.05 | 384.80 | 192.50 | 738 | 1175 | 154 | 2208 |
| GNP10 | 0.10 | 384.61 | 385.00 | 738 | 1175 | 154 | 2311 |
| GNP15 | 0.15 | 384.42 | 577.50 | 738 | 1175 | 154 | 2419 |
| GNP20 | 0.20 | 384.23 | 770.00 | 738 | 1175 | 154 | 2475 |

### Compressive strength

The resulting mixture was casted into cube moulds of standardised size. The molds were removed after 24 h, followed by curing for 28 days at a lab temperature of 25 °C.The molds were casted in 3 layers and vibrated adequately. To create a smooth surface, the tops of the specimens were levelled. Using a digital compressive testing machine with a 200 kN capacity, the comprehensive strength test of the cube was carried out. For the compressive strength test, an average of 3 cube samples were tested after 7 and 28 days of age.

### Pullout testing

A 200 kN capacity universal testing machine was used to conduct the pullout test. In this test, monotonic loading speed was used, and force was supplied up until the steel bar yielded or slipped out of the specimen. Equation 1 was used to calculate the concrete's ultimate bond stress.

$$\tau = P/\pi L\phi \qquad (1)$$

Where, $\tau$ = Ultimate bond stress of concrete; P = Peak force in the bar; L = Depth of rebar penetration; and $\phi$ = Diameter of the bar.

For each concrete cylinder, a single reinforcing bar was positioned vertically in the centre. In order to grip the specimen in the testing instrument adequate length to be gripped is provided. One end of the bar was inserted into the cylinder and the other end was protruded 500 mm from the top of cylinder. Cylindrical molds of 102 mm in diameter and 203 mm in height were used to fabricate all of the test specimens. Each time, the depth of rebar penetration was kept and noted in line with the fixed proportion to its diameter. These moulds were casted in three layers and were properly vibrated on the vibrating table together with the implanted steel bar. The moulds were removed after 24 h and maintained at room temperature for a 28-day curing period. The specimens were submerged for the curing all the way up to the top level in the water curing tank. Figure 2 displays the test configuration, loading direction, and specimen shape.

## Results and Discussion

### Workability

Workability is a broad technical property used to maintain the efficiency of each stage in construction and to achieve stable performance of fresh concrete. A true slump of 75 ± 10 mm was to be maintained as specified in our design mix. In fact, the smoother the concrete will flow without segregation, the better the workability. Additionally, because workability of concrete is related to its strength, it must be effectively managed. According to experimental findings, the workability of the concrete matrix decreased as GNPs in the matrix increased. Thus, in order to maintain the true slump value for different concrete matrix the SP content was increased proportionally. Figure 3, illustrates the SP demand per metre cube in order to maintain a true slump state.

### Compressive strength

Figure 4 reports the compressive strength of the samples indicated in table 4. It is evident that the comprehensive strength of GNPs modified samples is significantly influenced by the percentage of GNPs. The pattern established shows that when GNPs content increased, the overall strength initially moved up and then decreased. Figure 4 shows that there is a wt.% at which the best overall strength may be reached. With respect to reference concrete mix in the current mix proportion, adding 0.025%, 0.05%, 0.10%, and 0.15% of GNPs, there was an increase of 9.44%, 17.66%, 9.49%, and 1.24% in respective compressive strength. This rise in concrete's compressive strength may be attributable to the matrix phase's enhanced connections between the GNPs and the surrounding concrete. Due to their higher contact surface and surface transmission, GNPs can therefore be seen as the primary load-bearing components. However, it is clear that concrete loses 1.68% of its compressive strength when GNPs are added at a rate of 0.02%. Maximum increase of 17.67% in comprehensive strength was observed at the incorporation of 0.05% GNPs in the concrete mix.

**Figure 2:** The setup for the tension pullout test.

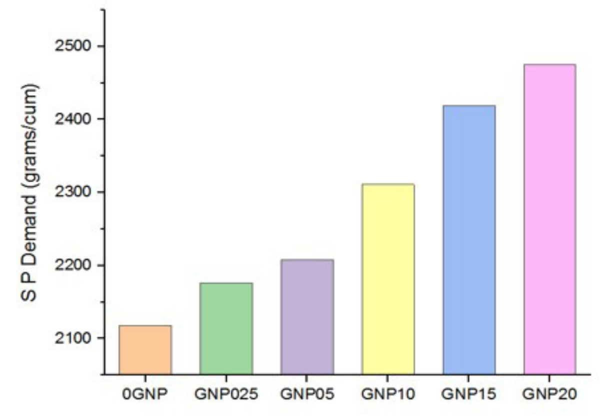

**Figure 3:** SP demand per cubic metre.

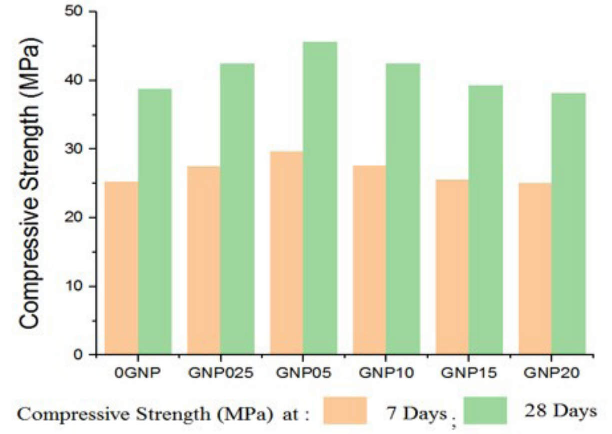

**Figure 4:** Comprehensive strength of various design mixes.

### Pullout testing

Table 5 displays the results of the bar pullout test. Peak measured forces have been tabulated along with associated nominal bond stresses. In order to calculate the peak nominal bond stress, divide the achieved pullout force by the curved surface area of the bar's of thesubmerged part as per useig equaiton 1. Failure in the tested samples was noted as a combined process of pulling out and splitting conical cracks due to lower bearing capacity between the concrete and bars and subsequent deformation. Crushing and radial crack growth was observed close to the bar that propagated to the top portion of the sample, marking their presence along with the failure. The outcomes reported above demonstrate that adding GNPs to the concrete improved the cohesiveness of the steel bar and concrete, causing a pullout force that is higher than the steel failure load. The findings showed that adding 0.05% of GNPs raised the bond tension by 16.8% for bar of 12 mm diameter with an embedded length of 108 mm (calculated as $9\phi$).

## Conclusions

- GNPs changed the concrete's slump value when they were introduced. Workability decreased when GNPs content increased.
- In order to ensure working performance of GNPs modified concrete, appropriate amount of water-reducing agent should be incorporated into the mix.

**Table 5:** Pullout test results.

| Mix proportion | Φ (mm) | L | P (kN) | Bond strength (MPa) | % variation with respect to reference |
|---|---|---|---|---|---|
| 0GNP | 12 | 108 | 37.55 | 9.23 | - |
| GNP025 | | | 41.21 | 10.13 | + 9.72 % |
| GNP05 | | | 43.87 | 10.78 | + 16.80 % |
| GNP10 | | | 40.86 | 10.04 | + 8.78 % |
| GNP15 | | | 37.96 | 9.33 | + 1.06 % |
| GNP20 | | | 36.88 | 9.06 | - 1.81 % |

- In comparison to neat concrete, GNPs modified concrete had a slight change in its overall strength. The total strength of GNPs in GNP05 increased by a maximum of 17.67% compared to GNPs in 0GNP.
- The ideal replacement percentage value, as measured by GNPs, was found to be around 0.05%. Up to the optimum point, the trend showed that the comprehensive strength increased linearly as GNPs content increased. The comprehensive strength however, declined with additional incremental substitution once the maximum had been reached.
- Due to the bridging effect, developed crack arresting mechanism induced by nano-reinforcement of GNPs, has enhanced the bond stresses.
- Due to improved adhesiveness, the use of GNPs reduced the early slippage of the inserted steel bar.

## Acknowledgements

The authors acknowledge the National Institute of Technology Srinagar for providing the necessary resources and facilities for conducting this research. We are also grateful to laboratory staff of the Structural Engineering section, for their assistance during the sample preparation and experimental technical support.

## Conflict of Interest

None.

### References

1. Stynoski P, Mondal P, Marsh C. 2015. Effects of silica additives on fracture properties of carbon nanotube and carbon fiber reinforced Portland cement mortar. *Cem Concr Compos* 55: 232-240. https://doi.org/10.1016/j.cemconcomp.2014.08.005
2. Alkhateb H, Al-Ostaz A, Cheng AH, Li X. 2013. Materials genome for graphene-cement nanocomposites. *J Nanomech Micromech* 3(3): 67-77. https://doi.org/10.1061/(ASCE)NM.2153-5477.0000055
3. Madhad HV, Mishra NS, Patel SB, Panchal SS, Gandhi RA, et al. 2021. Graphene/graphene nanoplatelets reinforced polyamide nanocomposites: a review. *High Perform Polym* 33(9): 981-997. https://doi.org/10.1177/09540083211011216
4. Krystek M, Pakulski D, Patroniak V, Górski M, Szojda L, et al. 2019. High-performance graphene-based cementitious composites. *Adv Sci* 6(9): 1801195. https://doi.org/10.1002/advs.201801195
5. Shuang D, Baomin W. 2019. Study on dispersion of graphene nanoplates and rheological properties, early hydration of cement composites.

*Mater Res Express* 6(9): 095086. https://doi.org/10.1088/2053-1591/ab31ee

6. Liu J, Fu J, Yang Y, Gu C. 2019. Study on dispersion, mechanical and microstructure properties of cement paste incorporating graphene sheets. *Constr Build Mater* 199: 1-11. https://doi.org/10.1016/j.conbuildmat.2018.12.006
7. Tong T, Fan Z, Liu Q, Wang S, Tan S, et al. 2016. Investigation of the effects of graphene and graphene oxide nanoplatelets on the micro-and macro-properties of cementitious materials. *Constr Build Mater* 106: 102-114. https://doi.org/10.1016/j.conbuildmat.2015.12.092
8. Qi X, Zhang S, Wang T, Guo S, Ren R. 2021. Effect of high-dispersible graphene on the strength and durability of cement mortars. *Materials* 14(4): 915. https://doi.org/10.3390/ma14040915
9. Wang B, Zhao R. 2018. Effect of graphene nano-sheets on the chloride penetration and microstructure of the cement based composite. *Constr Build Mater* 161: 715-722. https://doi.org/10.1016/j.conbuildmat.2017.12.094
10. Shamsaei E, de Souza FB, Yao X, Benhelal E, Akbari A, et al. 2018. Graphene-based nanosheets for stronger and more durable concrete: a review. *Constr Build Mater* 183: 642-660. https://doi.org/10.1016/j.conbuildmat.2018.06.201
11. Camacho MD, Galao O, Baeza FJ, Zornoza E, Garcés P. 2014. Mechanical properties and durability of CNT cement composites. *Materials* 7(3): 1640-1651. https://doi.org/10.3390/ma7031640
12. Kang D, Seo KS, Lee H, Chung W. 2017. Experimental study on mechanical strength of GO-cement composites. *Constr Build Mater* 131: 303-308. https://doi.org/10.1016/j.conbuildmat.2016.11.083
13. Alsadey S. 2015. Effect of superplasticizer on fresh and hardened properties of concrete. *J Agric Sci Eng* 1(2): 70-74.
14. Khan M, Ali M. 2018. Effect of super plasticizer on the properties of medium strength concrete prepared with coconut fiber. *Constr Build Mater* 182: 703-715. https://doi.org/10.1016/j.conbuildmat.2018.06.150
15. Alkhateb H, Al-Ostaz A, Cheng AH, Li X. 2013. Materials genome for graphene-cement nanocomposites. *J Nanomech Micromech* 3(3): 67-77. https://doi.org/10.1061/(ASCE)NM.2153-5477.0000055
16. Shah SA, Tantray MA. 2022. Graphene nanoengineered concrete. *J Pharm Negat Results* 31: 1046-1052.