

How Graphene Oxide Nanoparticles Can Revolutionize the Concrete Industry

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ABSTRACT: One of the most utilized materials for construction is concrete. Concrete is a fairly inexpensive, strong, and durable material to use for various construction. However, it has been shown that by adding Graphene Oxide Nanoparticles (GONPs) into the concrete mix, it is possible to improve its physical properties and durability. GONPs can be manufactured in various methods, Chemical Vapor Deposition (CVD), Exfoliation (Mechanical or Electrochemical), or Hummers' method. The dispersal of the NPs is also very important to the end physical properties of the dried concrete. Homogenous dispersal can be achieved by High Shear Mixing, Ultrasonication, Surfactant-Assisted Mixing, or Ball Milling. By adding 0.03% by weight of GO to the concrete mix, the compressive strength and the tensile strength both increased by 40% and the total porosity decreased by 13.5%. However, this led to a decrease in the workability by 34.6%. Additionally, this new form of concrete generates overall fewer carbon emissions. Although currently, this new form of concrete is significantly more expensive compared to conventional concrete, it has a longer lifetime and pays back in the long run.

Keywords: Concrete; Nanomaterials; Nanoparticles; Graphene; Graphene Oxide; Paper; Journal; Liaison Journal of Engineering; International University Liaison Indonesia;

1. INTRODUCTION

Concrete is considered one of the world's most used materials for various constructions. Its raw materials are readily available around the world and fairly cheap to produce and manufacture. Moreover, it is fairly durable and strong, which makes it suitable for large-scale construction from houses to skyscrapers and dams. Currently, approximately 30 billion tonnes of cement is used globally per year. Along with this, all of the manufacturing processes and transport of materials generate a significant carbon footprint. The cement industry accounts for about 8% of the total global CO₂ emissions and accounts for 2-3% of the energy consumption [Monteiro, et al., 2017].

Due to the prevalence of cementitious construction in the world, it is possible to see the various issues with using conventional concrete. The maintenance required for such construction

is rather extensive and often requires repairs and replacements, which can be rather costly [Gardner, et al., 2018]. Without routine maintenance, these faults can get out of hand and cause devastating damage. Additionally, the manufacture of concrete releases a significant amount of CO₂ emission and contributes to climate change, which is a current issue the world is trying to solve [Monteiro, et al., 2017].

By utilizing nanoparticles, it is possible to increase the compressive strength, increase its flexural strength, or even reduce internal microcracks [Aref, et al., 2017]. This would reduce the required amount of concrete for a given project and decrease the frequency of repairs and replacements required to be carried out in the future. Various types of nanoparticles have been analyzed and tested to see how they affect the mechanical strength of the concrete. These nanoparticles include inorganic oxides

such as TiO_2 and Al_2O_3 , and various carbon-based nanomaterials such as graphene oxide nanosheets and carbon nanotubes [Chhantyal, 2020].

1.1 BRIEF OVERVIEW

What are nanomaterials? In a nutshell, they are substances that have at least one dimension which is measured on the nanometer scale, between 1 and 100 nm. (1 nm = 10^{-9} m) Just for scale, the size of an atom is about 0.1 nm [ISO/TS80004-1:2015].

The field of nanotechnology was first postulated as a notion by Richard Feynman in 1959, it took the works of various scientists to develop this new field fully. K. Eric Drexler is one of the pioneers in this field and wrote the first book on nanotechnology, "Engines of Creation: The Coming Era of Nanotechnology", back in 1986 [Bayda, et al., 2020].

Experiments have shown that the nanoparticles have widely differing properties, both physical and chemical, compared to their bulk counterparts, this allowed scientists to use the same material for a wide array of applications. Additionally, due to the size of the nanoparticles, it has significantly more surface area compared to their bulk counterparts, and this is why nanoparticles are very attractive to the field of designing and manufacturing catalysts. This high surface-area-to-volume ratio allows for a much more efficient catalysis [Astruc, 2020].

Nanomaterials can be used to enhance the properties of concrete in various ways. The incorporation of nanomaterials into concrete can lead to improvements in strength, durability, and other performance characteristics. Some common types of nanomaterials used in concrete enhancement include Nano-Titanium Dioxide (nano- TiO_2), Carbon Nanotubes (CNTs), and Graphene Oxide (GO). Graphene oxide is often considered an excellent nanomaterial for enhancing concrete due to its unique properties, which can impart several beneficial characteristics to the concrete matrix, such as preventing fracture by increasing its various

physical properties. Additionally, the long-term benefit of using graphene oxide-enhanced concrete is reducing the contribution of CO_2 emissions, because less amount of concrete is required for the construction and routine maintenance [Monteiro, et al., 2017], [Walunjkar, et al., 2023].

The exceptional characteristics of two-dimensional graphene oxide (GO) are its substantial surface area and the existence of diverse oxygen functional groups. Making it not only suitable for applications in electronic industries, as highlighted by [Tiwari, et al., 2018]. but also, in construction industries as the reinforcement additive in cement [Chuah, et al., 2014].

2. GRAPHENE NANOMATERIAL SYNTHESIS

Graphene is an arrangement of carbon atoms in the form of a single sheet of honeycomb lattice structure and has remarkable mechanical, electrical, and thermal properties. It is the basic unit for various graphitic materials of any other dimension. It is a 2D building material for carbon materials of all other dimensions. If it is wrapped up tightly onto itself, it forms a 0D buckyball. Rolling the sheet into a cylinder forms 1D nanotubes, and if the layers are stacked up together, they form 3D graphite.

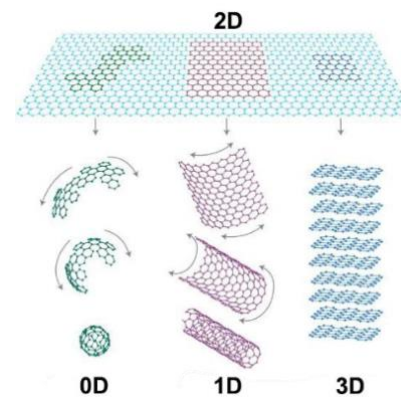


Figure 1. Representation of the various molecular structures of graphene-based NPs [Liu, et al., 2018]

Graphene nanoparticles (GN) have been investigated as a potential additive for cement

and concrete to improve certain of its qualities, such as high mechanical strength, electrical conductivity, and thermal conductivity. Because of this, it is a material that shows promise for a variety of uses [Alateah (2023)]. Cement characteristics can be improved with a variety of graphene-based compounds. The selection of graphene oxide (GO), reduced graphene oxide (rGO), graphene nanoplatelets (GNPs), and graphene nanoparticles (GNPs) is contingent upon the particular demands of the cement or concrete application [Shamsaei, et al., 2018]. Strong acids and other oxidizing chemicals are used to oxidize 3D graphite, putting oxygen-based functional groups into the layers in order to create GO [Liu and others, 2018].

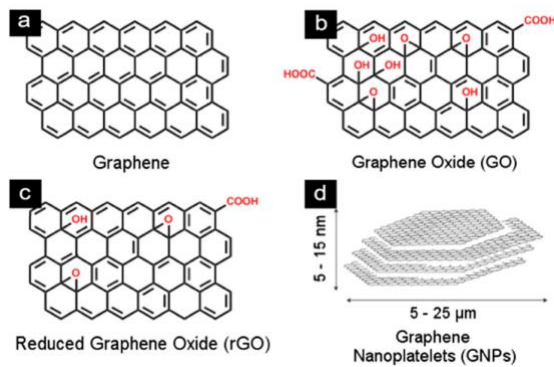


Figure 2. Various structures of GNs [Sahmsaei, et al., 2018]

The synthesis of graphene-enhanced cement involves incorporating graphene-based materials into the cement matrix during the mixing process. However, unlocking the full potential of graphene in cement-based materials requires addressing the considerable challenge of achieving uniform dispersion of nanosheets without compromising their quality. In recent times, various techniques have been employed for graphene synthesis. This process involves extracting graphene based on considerations of purity and the specific desired outcome.

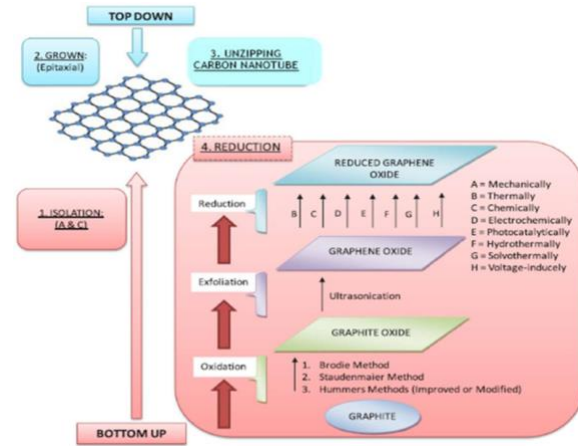


Figure 3. Graphene, GO, and rGO synthesis strategies [Deepa, et al., 2022]

There are two approaches to synthesizing nanomaterials, "top-down" and "bottom-up". In the top-down method, synthesis starts from a bulk material, broken down into smaller particles and eventually reaching the nanoscale. In the bottom-up method, the synthesis starts from a solution and then is aggregated together to form the nanoparticles [Astruc, 2020].

Initially, a straightforward graphite taping technique is employed in the production of single-layer graphene. Subsequently, mechanical and chemical exfoliation, along with chemical vapor deposition (CVD), have been introduced. Presently, various methods, including reduction in graphene oxide (GO), micro-mechanical exfoliation, liquid-phase exfoliation (LPE), and epitaxial growth techniques, are utilized for graphene manufacturing. Among these approaches, heat treatment stands out as a particularly environmentally friendly method [Deepa, et al., 2022].

2.1 CHEMICAL VAPOUR DEPOSITION (CVD)

One "bottom-up" method to synthesize high-quality graphene is chemical vapor deposition (CVD). This technique can be applied to conduct the synthesis on a large scale. [Mbayachi, et al., 2021]. In CVD, monolayers or few layers of pure graphene or GO are produced from graphite powder. This is achieved by solution-based chemical oxidation [Deepa, et al.,

2022]. Different substrates are employed in CVD for graphene film generation. These substrates could be composed of Nickel (Ni), Copper (Cu), Iron (Fe), or Stainless steel. Common carbon sources include Methane (CH_4) and acetylene (C_2H_2). The carbon source is activated using either thermal CVD or plasma-enhanced CVD (PECVD) [Mbayachi, et al., 2021].

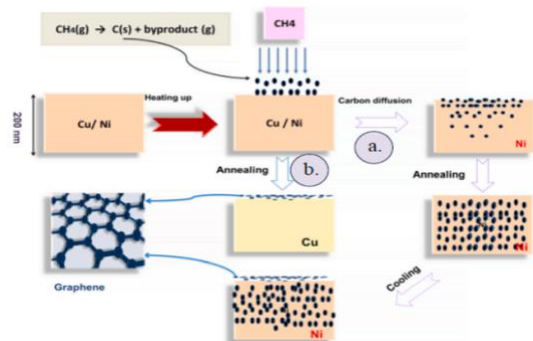


Figure 4. A diagram illustrating the process of CVD Graphene formation on a nickel substrate. (a) The graphene is synthesized via dissolution and precipitation, and (b) direct deposition mechanism [Mbayachi, et al., 2021]

Hydrogen and argon gases are used as carrier gases in CVD to remove unwanted oxides from the surface of the metal catalyst. Traditional CVD-based graphene development requires two primary steps and has used transition metal substrates such as Cu and Ni. The gas precursor is first decomposed through heating at a high temperature to make carbon, followed by using the segregated carbon on the metal catalyst surface to create the graphene carbon structure. To illustrate, the synthesis of graphene using polycrystalline Ni includes annealing Ni in an H_2 environment at a temperature of 900–1000 °C, with a grain size. After that, an H_2/CH_4 gas mixture is introduced to the substrate, with CH_4 acting as the carbon source. The decomposition of hydrocarbons induces the dissolution of carbon atoms in the Ni film, leading to the creation of a solid solution. Due to Ni's high solubility at elevated temperatures, the Ni-C precipitate is created as the solid solution cools down in an argon gas environment, leading to the etching of graphene (Fig.4). Despite Ni

being a generally suitable substrate for graphene production, the size and percentage of the graphene monolayer can be affected by the quality of the Ni film.[Mbayachi, et al., 2021].

2.2 EXFOLIATION

Graphene exfoliation refers to the process of separating individual layers of graphene from a bulk material, typically graphite. GO could be produced in large quantities and at a lower cost compared to carbon nanotubes (CNTs) by chemically oxidizing graphite. The GO can be customized to blend well with other elements that have higher Young's modulus, hardness, and flexibility. In the process of producing graphene by chemical exfoliation, the solvent's surface energy remains relatively close to that of graphene [Deepa, et al., 2022].

2.3 MICROMECHANICAL EXFOLIATION

The mechanical exfoliation process, often called the peel-off or Scotch tape method, was the first way Novoselov and Geim used to produce graphene. This technique involves mechanically separating graphene layers using sticky tape (Fig. 5). With this method, graphene sticks to the tape in layers after it is peeled, but after repeated peeling, it breaks into several individual graphene flakes. [Mbayachi, et al., 2021].



Figure 5. The technique for synthesizing graphene using the "Scotch tape" method was initially documented in 2004 by Novoselov and Geim [Mbayachi, 2021]

Recent efforts have been directed toward enhancing the sustainability and scalability of mechanical exfoliation by enhancing the

efficiency and dimensions of exfoliated flakes. An illustrative example involves the substitution of conventional adhesive tape with dicing tape, commonly referred to as UV release tape. This replacement simplifies the tape's residue-free removal after transferring graphene [Alateah, 2023].

2.4 ELECTROCHEMICAL EXFOLIATION

The electrochemical exfoliation of graphite has emerged as a straightforward yet highly efficient approach for the large-scale synthesis of graphene in recent years. This process employs different graphite forms, such as foils, plates, rods, and powders, as electrodes in either aqueous or non-aqueous electrolytes. By applying an electric current, the electrodes undergo expansion. Depending on the applied power, two types of electrodes are utilized: Cathodic (Negative) and Anodic (Positive) [Mbayachi, et al., 2021].

2.5 HUMMERS' METHOD

Using Hummer's approach, a flake containing potassium persulfate, phosphorus pentoxide, and graphite powder was heated at 80°C for 4.5 hours in order to create graphene oxide (GO). After undergoing pre-treatment, the graphite powder was cleaned with deionized water and then dried in a vacuum oven. Sulfuric acid was added to the pre-treated components, and the mixture was then agitated at 35°C for 12 hours. Afterward, deionized water was added. Centrifugation was performed on the resultant mixture for one hour at 7500 rpm. Methanol/ether was used for several washes to neutralize the supernatant, and the solution was centrifuged in consecutive cycles. The gathered sediments underwent vacuum drying at room temperature for a duration of 12 hours. Cyclodextrin/graphene was later produced from GO and graphite powder using a modified Hummers' process [Deepa, et al., 2022].

3. METHODS OF DISPERSING GRAPHENE IN CONCRETE MIXTURE

The production of graphene-enhanced cement involves the dispersion of graphene nanoparticles within the cement matrix.

Achieving a uniform distribution of graphene in the cement is crucial for enhancing the material's mechanical and electrical properties. Mixing and agitation methods play a key role in ensuring effective dispersion. According to research, the introduction of a small amount of graphene nanosheets (GNS) or graphene oxide (GO) can substantially enhance both the mechanical strength and transport properties of cement-based materials. Several researches have shown different mechanical mixing/agitation methods and techniques have been devised [Alateah, 2023] to ensure the uniform dispersion of graphene within the cementitious matrix, fully leveraging its nanoscale properties.

3.1 HIGH SHEAR MIXING

One common and useful technique for dispersing graphene in concrete matrices is high shear mixing. In order to guarantee a consistent distribution of graphene agglomerates throughout the concrete mixture, it is necessary to break them down. This process applies strong shear and impact pressures using equipment such as rotor-stator mixers, homogenizers, and ball mills to overcome the van der Waals interactions between layers of graphene. This method creates strong turbulence that disperses graphene evenly throughout the matrix and efficiently separates the layers. Utilizing high mixing rates is typically necessary to achieve good dispersion. [Alateah, 2023]

3.2 ULTRASONICATION

Ultrasonication plays a crucial role in disassembling agglomerates and enhancing the interfacial interaction between graphene sheets and cement particles. Because of its high specific surface area, graphene sheets form significant aggregates in water before undergoing ultrasonic treatment. When sonicated, the vibrations generated by ultrasonic waves can surpass the van der Waals interactions among the graphene sheets, resulting in their dispersion [Liu, et al., 2019]. Additionally, the separation of monolayer graphene from graphene stacks is caused by the cavitation effect created during the ultrasonic process, which opposes the van der Waals forces between graphene sheets. Because of this,

thinner graphene sheets show more molecules absorbing on their surfaces, which enhances graphene's stability and dispersibility in aqueous solution. [Lin & Du, 2020]

3.3 SURFACTANT-ASSISTED MIXING

Introduce surfactants to the mixture to improve the wettability of graphene oxide (GO), reducing agglomeration and enhancing dispersion. Four surfactants show promise for dispersing graphene oxide (GO): polycarboxylate, air entrainer, and Gum Arabic. The addition of GO significantly improves flexural strength, especially when using polycarboxylate and Gum Arabic surfactants. These surface-active agents play a crucial role in preserving the dispersion of GO nanosheets in the alkaline environment of cement, effectively reinforcing the cement paste [Chuah, et al.].

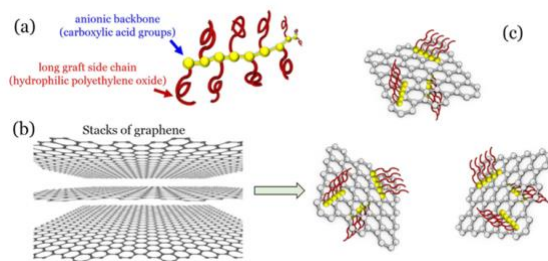


Figure 6. Micro-view of (a) polycarboxylate molecule, (b) flocculation of graphene sheets, (c) homogeneous dispersed graphene [Lin & Du, 2020]

Fig. 6 shows the dispersion mechanism of graphene nanosheets (GNS) in water facilitated by polycarboxylate (PC). The PC molecule's backbone binds to the surface of graphene sheets, while the PC molecule's side chain, featuring negative charges, creates electrostatic repulsion forces, leading to the dispersion of graphene particles and preventing their agglomeration. [Lin & Du, 2020] Nevertheless, an excessive amount of remaining surfactant can have adverse effects on both cement hydration and pore structure if not eliminated before the mixing process. [Alateah, 2023]

3.4 BALL MILLING

The dispersion of graphene oxide (GO) in a cement matrix was enhanced through the utilization of ball milling in a planetary ball mill.

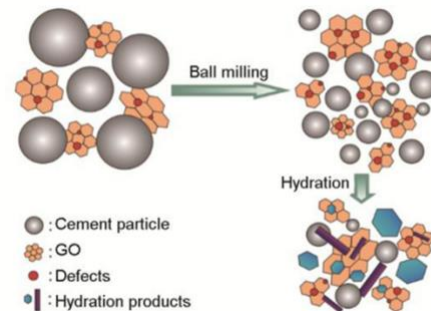


Figure 7. Schematic illustration of Graphene Oxide (GO) reinforced cement paste [Jing, et al., 2019]

A possible strengthening mechanism of graphene oxide (GO) in cement paste is shown in Fig. 7. As depicted, ball milling leads to a reduction of cement particle size, leading to an enhanced specific surface area. Ball milling creates more friction and collisions between cement particles and graphene oxide (GO), which improves the interactions between the two. This modification considerably enhances the dispersion efficiency of GO, resulting in its dispersion state after grinding and obtaining a uniform distribution inside the matrix. Furthermore, intrinsic flaws are introduced into the GO by mechanical grinding. [Jing and others, 2019] The mechanical characteristics of the cement paste are improved by the combined effects of flaws on GO and homogenous dispersion of GO. [Alateah (2023)]

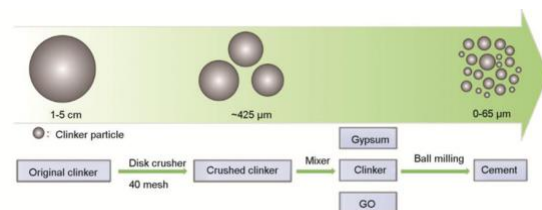


Figure 8. Schematic diagram of the preparation of cement.

Fig. 8 shows a schematic illustration of the preparation process of the cement. The initial cement clinker, with a particle size of 1-5 cm, underwent primary crushing using a disk crusher and subsequently traversed 40 mesh screens. Achieving a consistent dispersion of clinker particles involved uniformly blending the resultant clinker (with a size less than 425 μm) in a mixer for 24 hours. To obtain the ball-milled cement (0-65 μm in size), the resulting clinker, gypsum, and GO powder are ground at 300 r/min for 3.5 h inside a planetary ball mill [Jing, et al., 2019].

4. ADVANTAGES AND DISADVANTAGES

One of the key factors that are affected by the addition of GO is the workability of the concrete mix. The workability of a mortar mix shows how the material is mixed, transported, placed, and finished with ease and homogeneity. This factor also affects the finished hardened properties of the concrete as it determines how the components are suspended within the concrete mix and how the mix is placed properly. [N, Ajay & S, Girish, 2015]

The addition of GO has been shown to decrease the workability of the mortar mix. Free water present within the mix is absorbed by the GO-NPs due to its hydrophilic property, this leads to a decrease in lubrication within the mix and an increase in the overall viscosity of the mix. [T. Naresh Kumar, et al., 2021] When 0.03% by weight of GO was added, the diameter of the mini-slump was 34.6% lower than the control. [Gong, Kai, et al., 2015] A method to deal with this issue is with the aid of surfactants, or in this case, superplasticizers. These agents will decrease the viscosity and disperse the NPs throughout the mixture more evenly. However, using these agents is not without problems, as some superplasticizers can lead to foaming and decrease the mechanical strength of the final hardened concrete. [Birenboim, et al., 2019]

Another large disadvantage of using GO-concrete is the cost. Due to the high cost of the GONPs, the GO-concrete mix would cost 5-10 more than the conventional mix. [Monteiro, et

al., 2017] But at the same time, the amount of concrete mix required for a given project would be significantly less, as the GO-concrete could endure and withstand more stress. Additionally, the long-term benefit arises when it comes to the frequency of maintenance and replacement, as the GO-concrete is much more durable. When all of these factors are taken into account, the cost difference between the use of GO-concrete over conventional concrete is not as substantial as expected.

One advantage of using GO-concrete over conventional concrete is the reduced amount of carbon footprint. Since less concrete is required for a particular construction, and fewer repairs and replacements are required over the lifetime of the construction, fewer carbon emissions are being produced overall.

Another advantage is the improvement of cement properties. In contrast to traditional Portland cement mixtures, incorporating graphene into concrete has been observed to enhance its strength, hardness, and overall durability significantly [Walunjkar, et al., 2023]

This happens because when graphene oxide (GO) is introduced into the cement matrix, graphene oxide (GO) serves as both a template and a bridging agent during the formation of cement hydration crystals. This dual role facilitates and regulates the process of cement hydration, leading to a denser microstructure in the cement-hydrated crystals and an enhancement in the macroscopic properties of the overall cement matrix.

The summarized enhancement in the macroscopic properties results is detailed in Table 1. Enhanced mechanical properties such as compressive strength and flexural strength are important in construction as these properties make structures more robust and better able to withstand external forces, leading to longer-lasting constructions and more resistant to cracking and bending. Additionally, the freeze-thaw durability of concrete, a critical parameter in structural integrity, is positively influenced. The incorporation of graphene oxide results in

reduced mass loss after several freeze-thaw cycles compared to non-modified concrete.

Table 1. Summary of the different graphene-oxide weight percentages in cement and its changes in properties

GO in Cement	Properties	Percentage	Reference
0.03% (Water to cement ratio of 0.5)	Compressive strength	Increased by 40%	[Gong, et al., 2015]
	Tensile strength	Increased by 40%	
	Total Porosity	Decreased by 13.5%	
0.05% (Water to cement ratio of 0.5 in Ultra-High Strength Concrete)	Compressive strength	Increased by 15.5%	[Lu, L., et al., 2017]
	Flexural strength	Increased by 21.1%	
0.1% (Water to cement ratio of 0.4)	Compressive strength	Increased by 55.8%	[Du, Hongjian, 2019]
0.03% (Water to cement ratio of 0.2)	Compressive strength	Increased by 21.37%	[Wang, et al., 2019]
	Tensile strength	Increased by 53.77%	
	Flexural strength	Increased by 39.62%	
0.03% (water to cement ratio of 0.36 and GO with oxygen content of 29.75%)	Compressive strength	Increased by 38.9%	[Lv, Shenghua, et al., 2013]
	Tensile strength	Increased by 78.6%	
	Flexural Strength	Increased by 60.7%	
0.05% (Water to cement ratio of 0.5)	Compressive strength	Increased by 15.0% - 33.0%	[Pan, Zhu, et al., 2015]
	Elastic modulus	Increased by ~6.5%	
	Flexural Strength	Increased by 41.0% - 59.0%	
3.00% (Water to cement ratio of 0.6)	Elastic modulus	Increased by 200% - 500%	[Horszczaruk, et al., 2015]
0.05% (Water to cement ratio of 0.3)	Chloride ion permeability	Decreased by 12%	[Chu, Hongyan, et al., 2020]
	Mass loss rate (after 300 cycles of freeze-thaw)	0.44%	
0.06% (Water to cement ratio of 0.28 in Ultra-High-Performance Concrete)	Chloride ion permeability	Decreased by 10.70%	[Yu, et al., 2020]
	Mass loss rate (after 300 cycles of freeze-thaw)	0.13%	
	Relative dynamic modulus of elasticity (after 300 cycles of freeze-thaw)	98.51%	
0.06% (water to cement ratio of 0.448)	Mass loss rate (after 540 cycles of freeze-thaw)	0.25%	[Mohammed, et al., 2016]
	Total water adsorption	6.4%	

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	Elastic modulus	Increased by ~6.5%	
	Flexural Strength	Increased by 41.0% - 59.0%	
0.3% (water to cement ratio of 0.45)	Total volume of abrasion	Decreased by 70%	[Damith, et al., 2022]

Table is summarized from [Li, H., et al. 2023] and [Maria Acheing Akulu, et al., 2023]

The research findings on the incorporation of graphene oxide (GO) in cement reveal a diverse range of effects on various properties, highlighting its potential for enhancing the performance of concrete.

The findings indicate that varying concentrations of GO, ranging from 0.03% to 3.00%, result in distinct improvements in concrete properties. Notably, increases in compressive strength, tensile strength, and reductions in total porosity are observed as the percentage of GO in concrete mixtures is adjusted.

The second aspect examined in the data is the water-to-cement ratio, an essential parameter in concrete mix design. The study explores

different ratios, such as 0.2, 0.3, 0.4, 0.5, and 0.6, revealing varying effects on concrete performance. Lower water-to-cement ratios, particularly 0.2, show positive impacts on compressive and flexural strengths. Conversely, higher ratios, like 0.6, demonstrate significant increases in elastic modulus.

Across various concentrations and water-to-cement ratios, a consistent pattern of increased compressive strength, tensile strength, and flexural strength emerges. These improvements signify a promising development for creating higher-performance concrete with superior structural integrity and resistance to applied forces.

The influence of GO on durability-related properties is another distinguishing factor. For instance, at a water-to-cement ratio of 0.3, Chu et al. (2020) observed a 12% reduction in chloride ion permeability, indicating enhanced resistance to corrosion.

In summary, the table highlights the intricate relationship between graphene oxide concentration, water-to-cement ratio, and diverse concrete properties. Tailoring these factors allows for the optimization of concrete mixtures, paving the way for enhanced mechanical strength, durability, and overall performance in various applications.

5. CONCLUSION AND FUTURE PROSPECTS

In this paper, various methods are discussed on how to synthesize graphene oxide nanoparticles. Also, the techniques on how to disperse the nanoparticles throughout the concrete mix. Although adding the GO to the concrete mix decreases its workability, it increases the physical properties of the hardened concrete, such as its compressive strength, tensile strength, and flexural strength.

Currently, research is still being done to determine the optimal mixture of graphene oxide and the proper additives to ensure that the desired physical properties of the hardened concrete are achieved. As well as the techniques to ensure the mixture is well agitated and to achieve homogeneity throughout the cement mix.

While the cost of graphene oxide is still relatively expensive, as more techniques are being developed to manufacture these NPs faster and more efficiently, the cost of these materials would eventually decrease and allow it to become a more economically viable resource for large-scale constructions.

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