www.revistageintec.net
ISSN: 2237-0722
revista GEINTEC
Gestão, Inovação e Tecnologias

Study on Recent Development of Graphene-based Nano Composites- A Review

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Abstract
Utilization of nano material become popular in recent years which enhances the overall behavior of the concrete and cement composites. Supplementary cementitious material, graphene based nano materials will reduce the consumption of cement and thus provides environmental sustainability benefits. Graphene based nano material will be the current focus of the research. This paper presents the review of dispersion of graphene oxide in different solvents, mechanical properties, hydration, durability, microstructure and thermal properties of the graphene reinforced cement composites.

Key-words: Nano Material, Cement, Graphene, Supplementary Cementitious Material, Solvents, Durability.

1. Introduction

India is the second largest cement producer in the world. Per capita consumption of cement will be 235 kgs in 2019. Production of cement will cause several environmental issues. Reducing cement consumption will reduce the carbon di oxide emission during production of cement. Due to the rapid development of construction industry consumption of amount of cement quantity will get increases, this will increase the production rate of cement. Increase in cement production will increase the high carbon di oxide emission and sustainability issues to the environment. Amount of

Emission of greenhouse gases will get reduced by reducing the amount of production of cement this can done by reducing the consumption of cement. Utilization of fly ash from the thermal power plants will reduces the substantial environmental risk [1]. Utilization of supplementary cementitious material, graphene based nano material will become alternative to reduce the consumption of cement.

Nano materials play a vital role in improvement of strength in cement modification. Engineered nanomaterials are classified into 0d nanoparticle, 1D nanofiber, and 2D nanosheet [2]. GO fabricated cement composite improves the Macro and micromechanical properties of the cement composite [3]. Flexural strength of the GO reinforced cement composite with a flyash dosage of 10% and 20% improves the strength by 32.9% and 31.9% with a optimum dosage of GO 0.03% by wt [4]. Properties and structure of GO based on the method of synthesis and degree of oxidation[5]. Improved properties of GO modified cement due to the formation of a 3D network structure [6]. Adhesion between the hydration products can be enhanced due to the addition of small quantity of carbon nanotubes with the cement and thus enhances the mechanical properties of the cement mortar [7]. Addition of small quantity of Silver nano particles in cementitious composites leads to higher thermal conductivity, ultrasonic pulse velocity, split tensile strength and reduces the lower electrical resistance, water absorption and porosity [8]. reduction in compressive strength was found due to the increase in size and high specific surface area of GO [9]. Rate of setting time gets reduced with the increasing of amount of GO with cement [10]. A compact and dense structure was found due to the incorporation of GO-based cement composites which enhances the hydrates networking with reduced porosity [11]. The addition of 0.05 wt% multiwalled nanotubes Chemically functionalized GO enhances the toughness of the cement matrix. The addition of a superplasticizer is important for the uniform dispersion of CNT and refinement of pore structure [12]. The formation of geopolymer gel and filling of nanopores reduces the porosity of the geopolymer [13]. Random distribution of GO particles in the cement matrix enhances the pore structure and decreases the sorptivity[14]. Multilayer graphene nanoparticle reduces the thermal stress and increases the hydration reaction within the cement matrix [15]. High energy conservation and durability behaviour was found with the incorporation of ultrafine PCMs composites [16]. Several researches have been made in the graphene-based cement composites. This review presents the recent development of cement-based composites and provides challenges of using graphene based nano materials. Effect of sonication energy in enhance of strength is not studied.

2. Dispersion of GO

Graphene and multi-walled carbon nanotubes were dispersed uniformly into cement paste by applying proper surfactant and ultrasonic treatment [17]. Depending on the amount of sonication energy and time the dispersion behavior of GO/CNT may get differs. Excessive sonication energy causes deterioration of dispersion [18]. Uniform dispersion of GO in water was achieved with the inclusion of Silica fume reduces the GO particle size from 1.8µm to 0.8µm [19]. Incorporation of DMF, NMP, THF, and Ethylene glycol based dispersing agent exhibits long-term dispersion stability [20]. incorporation of GO and graphene in water decreases the pH value of the graphene dispersed mix suspension simultaneously addition of reduced graphene oxide in water increases the pH value of the mix suspension [21]. Thin layer of silica coated surface on the GO surface exhibits a good dispersion stability by maintaining the silica to GO ratio as 1:1 [22]. Physical strength, oxygen-containing functional groups, dispersibility, chemical structure, size and interlayer distance of graphene oxide are different than reduced graphene oxide [23]. Uniform dispersion behaviour was obtained by using polycarboxylate based superplasticizer in GO based cement composites in alkaline environment [24]. Sika-PCE based superplasticizer shows good dispersion behaviour with 30% ultrasonic energy with a super plasticizer to GO ratio of 1:1[25]. Uniform dispersion of GO sheets in cementitious materials was achieved in strong alkaline based environment by using Sika-PCE based superplasticizer. Minimum 60 minutes sonication time is required to improve the deflocculation of GNP agglomerates and stabilization of GNPs in suspension for at least 1day [26]. Long term stability can be obtained by using solvents NMP, ethylene glycol and water[27]. The Graphene/Polyvinyl alcohol based aqueous solution directly replace the water during casting of cement. Uniform dispersion can be obtained at low cost with high production rate [28]. Polycarboxylate based superplasticizer shows good dispersion of graphene nanoplatelet in water [29]. Inclusion of 3% and 5% of silica fume in GO cement paste enhances the uniform dispersion of GO nanosheets. [30]. Surfactants used for the effective dispersion of GNP was CTAB, SDS, DMA, CO890 and SDBS. Among the above five surfactants SDBS surfactant provides greater dispersion of GNPs[31]. Sodium dodecyl benzene sulfonate (SDBS) based surfactant produces uniform dispersion of graphene sheets in water [32]. Uniform dispersion behavior was achieved by incorporating adequate quantity of silica fume in graphene. Silica fume improves the dispersion behavior and improves the interfacial strength between graphene and hydration products [33].

3. Effect of GO on Workability of Cement Paste

Workability of the cement paste gets reduced due the inclusion of GO. Cross linking of GO nanosheets by calcium cations will cause agglomeration and thus reduces the workability. The high water entrapment capacity of GO agglomerates reduces the free water [20]. Amount of adsorbed water will be increased with the increase in amount of GO with cementitious composite. friction between particle will get increased due to the increase in the amount of free water which causes a reduction in workability. workability of the cement composite reduces due to the higher viscosity and yield strength of the composite [21]. The inclusion of GO in cement composites increases the surface area of the cement composite. The presence of a higher rate of production of calcium silicate hydrates increases the surface area of the cement composite. The incorporation of GO reduces the workability of the cement paste [22].

High specific surface energy of the graphene will cause reduction in workability of the cement composite since Graphene oxide has large surface area and hydrophilic nature it consumes more water[34], [32], [23],[35],[36],[37]. Inter friction between the particles will cause higher amount of free water adsorption within the cement matrix by GO leads reduction in workability [36]. reduction in workability of the composite is proportional to the amount of graphene oxide present in the composite, reduced graphene comes under hydrophobic nature it does not consume more water from the composite [23]. Go had larger specific area than carbon nano tubes and contains more oxygen-containing functional groups with good hydrophilicity[38]. Due to good dispersion behaviour of coated silica on the GOS surface reduces the negative impact of agglomeration enhances the workability. GOS nanohybrids found to be effective in Nano reinforced OPC composites [22]. Fluidity of GO based cement composites enhanced due to the addition of fly ash. Since the fly ash has very fine particle size, less water requirement and ball effect plays a major role in enhancing fluidity of the GO based cement composites [30]. The rate of decrease in slump value is due to the increase in amount of GO content [39].

Due to the presence of oxygen functional groups and hydrophilic nature of GO will cause reduction in workability and final setting time of the cement composites. In case of rGO composites workability and final setting time increased due to the hydrophobic nature of rGO composite [40].

4. Effect of GO on Hydration of Cement Paste

GO fabricated cement paste accelerates the hydration of cement paste due to presence of seeding effect of GO agglomerates which promotes nucleation sites because of their small particle size and large surface area [34]. incorporation of GO supports the formation of hydration crystals which enhances the compactness of the cement composites. on the first day of hydration small and irregular sphere shaped particles was found. Incomplete flower like hydration crystal was found by the presence of rod like crystal on the third day of hydration. incomplete flower with many petals was observed on the 7th day of hydration. A larger size flower-like shape found at 28 days. Denser hydration crystals are formed on 60days and 90days.Flower like hydration crystals forms dense and compact structure [41]. Inclusion of GO with 28.95% oxygen content modifies the microstructure of the hydration crystal and thus improves the toughness of the cement composites [30]. Addition of GO accelerate the hydration rate by nucleation effect and GO can greatly influence the hydration of C_3A rather than C_3S. Compared to conventional specimen GO based cement composites forms more denser microstructure and hydration crystal around the networking areas [11]. uniform and compact network structure was found due to the formation of refined crystal size due to the fabrication of GO in cement composites [42]. Numerous nucleation sites was found for the hydration products without reducing the growth rate of hydration products with a optimum dosage of 0.05% of EOGO based cement composite [43]. Compared to nano- SiO_2 based cement mortar has lower slump than nano-TiO_2 in cement mortar for the same dosage [44].

Incorporation of GO in geopolymer composite supports the hydration process. The compressive strength development in geopolymer composite reinforced with GO and rGO plays a vital role in hydration process [13].

The final setting time of GO and rGO based composites increased by an hour due to the acceleration of hydration process compared to the control specimen [45].

Incorporation of GNPs in cement composite improves the hydration reaction and supports the growth for more hydration products due to the ion exchange in earlier age and forms more compact microstructure. Enhanced dense microstructure improves the overall properties of GNPs cement composites [31].

Fig. 1 - SEM Images of GO with different Hydration Times (A) 1day (B) 3days (C) 7days (D) 28 days (E) 56 days (F) 90 days [41]

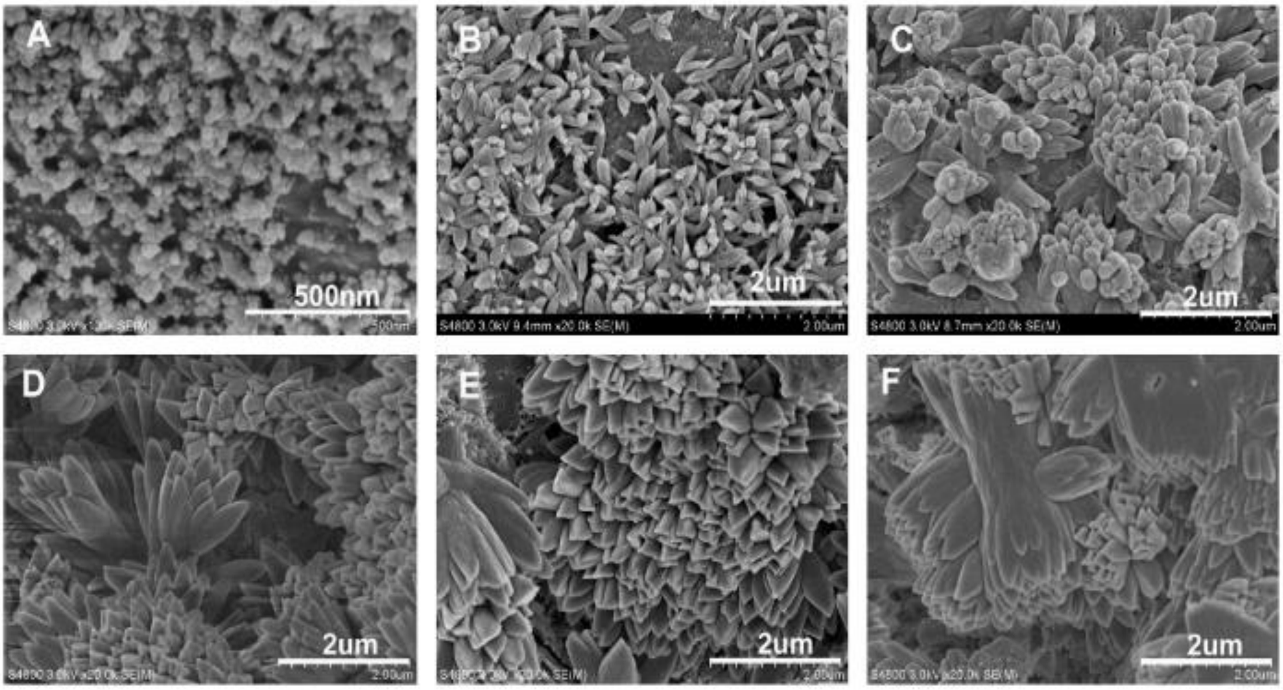
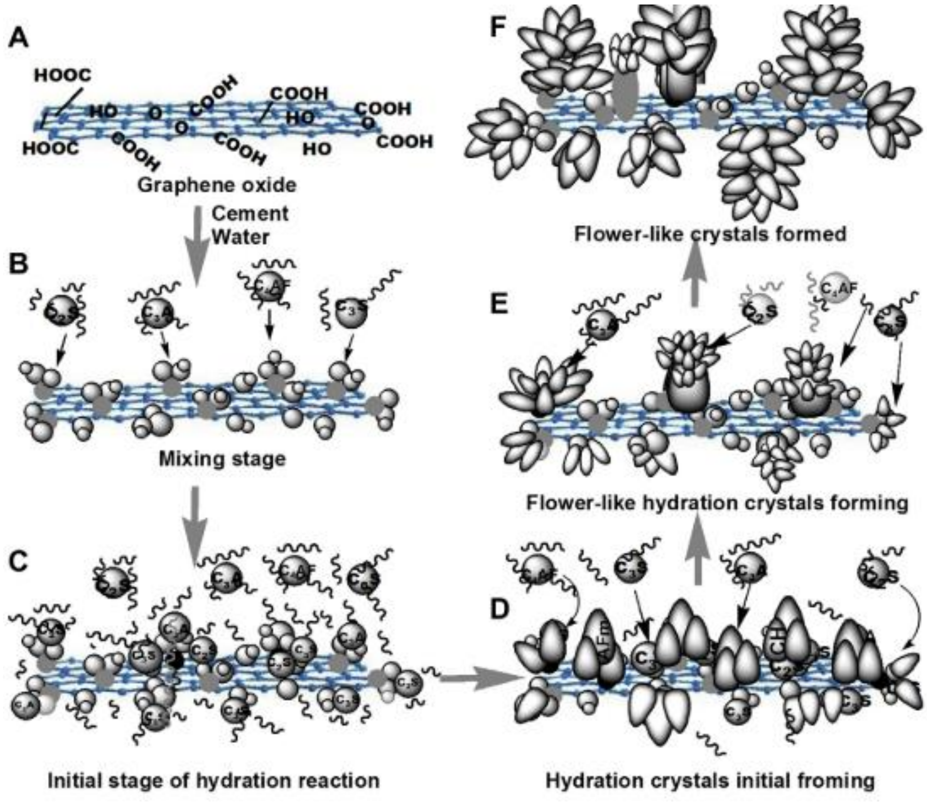


Fig. 2 - Regulation Mechanism of GO Hydration Cement Crystal [41]



ISSN: 2237-0722
Vol. 11 No. 3 (2021)
Received: 19.04.2021 – Accepted: 10.05.2021

5. Effect of w/c Ratio

Morphology and hydration products of the cement based composites affected by the addition of GO. GO had large surface area this could promote the hydration of cement and nucleation effects. dense and compact structure will be formed by the presence of GO in the pores of the cement mortar. Due to the pore filling effect of GO in cement mortar will enables dense and compact structure of mortar and thus enhances the overall properties of the cement composites [42].

6. Pore Size Distribution

Pores present in cement paste could be filled with GO agglomerates which enhances the pore structure [20]. Presence of high ratio of gel pores in the cement will promotes the hydration process[22] . Capillary pore and gel pores can be reduced in GO-NS-based cement composites due to its nucleation and pozzolanic effect which offers superior mechanical properties and impermeability of cement composite [23]. cement composite reinforced with GO improves the pore structure due to the nucleation and filling effect of the structure. Refinement of pore structure will enhances the overall behavior of the structure [21].

GNP provides restraint against the generation and propagation of cracks which leads lower porosity and pore diameter[31]. GO fabricated with cement composites will increases the volume of gel pores in the composite. Addition of GO in cement composites increases the volume of gel pores in the composite [37]. GNP and GO reinforced cement composites refines the pore structure of the cement paste [34], [46]. incorporation of suitable amount silica fume with GO in cement composites will promotes the refinement of pore structure [33].

7. Mechanical Properties of GO

Mechanical properties of the cement based composite will get enhanced with incorporation of GO. Optimal dosage of 0.03% of addition of GO increased the flexural strength about 78.6% and the corresponding increase in compressive strength will be 38.9% compared to conventional specimen. incorporation of GO with nanosheet supports crystal growth which provides the dense and compact structure [41]. Incorporation of rGO with a optimal dosage of 0.6% shows slight improvement in early strength and there is no significant improvement in strength at 28days [47]. Incorporation of GO and carbon fibre reinforced cement composites show greater improvement in flexural strength than compressive strength [48]. Flower like hydration crystals and uniform dispersion of all cement particles improves the flexural strength and polyhedral hydration crystals are responsible for enhancement of compressive strength. toughness of the cement composites enhanced due to the modification of microstructure and hydration crystals and thus reduces the brittleness of the cement composites [30]. Incorporation of high amount of GO content reduces the strength due to the GO has large specific energy and high specific surface area, this results capability of absorbing more water and leads to higher water demand [42]. Uniformly dispersed GNPs exhibits enhanced mechanical properties of cement based composites [31]. An increase of compressive strength and flexural strength is found about 33% and 58% with addition of 0.05% of GO [37]. Incorporation of GO in fly ash based geopolymer composite enhances the strength and durability of geopolymer composites. incorporation of small quantity of GO improves the compressive strength about 41.4 MPa with a GO dosage of 0.02% [1]. Incorporation of GO in concrete enhances the compressive strength (21-55%) and flexural strength (16-38%) [39]. Optimum content 0.03% by wt enhances the compressive strength of the cement paste [34]. Mechanical properties of the composites enhanced with the addition of 0.05% of EOGO in cement composite [43]. mechanical behaviour of CNTs improved due to the attachment CF surface. Adhesion between CF-CNTs in cement matrix will forms interlocking between CF-CNTS which plays major role in enhancing the mechanical properties of the composites [49]. Incorporation of GO in geopolymer composite improves the flexural strength by 25% with a optimum dosage of 0.3% by wt[13]. hydration crystals are formed due to the active participation of functional groups with chemical bonding. formation of hydration crystals bridge the gap between the composite matrix and enhances the toughness of the matrix. increase in compressive strength was found in rGO composites compared to GO composites. flexural strength of the GO composite was higher compared to the rGO composite. improved hydration products was found in addition of rGO composites due to the presence of physical strength and low content functional groups [40]. Smaller quantity of addition of Nano-Graphite reinforced composite enhances the mechanical properties of cement composite [50]. Incorporation of rGO with a small quantity enhances the compressive strength with an optimum dosage of 0.1% [51]. addition of suitable quantity of silica fume with graphene improves the overall properties of the cement composites [33].

The addition of GO in cement enhances the mechanical properties of the cement-based composites. The improvement in strength with the optimum dosage of GO is given in table1.

Table 1 - Mechanical Properties of GO Reinforced Cement Paste

Matrix	Dosage of Graphene (wt%)	w/c ratio	ultrasonication	Admixture	Improvement of mechanical properties	Ref
Cement paste	0.03	0.37	-	Polycarboxylate based superplasticizer	38.9% compressive strength 78.6% tensile strength 60.7% flexural strength	[24]
Cement paste	0.04	0.3	500 W, 5min	-	59.31 Mpa to 81.56 Mpa compressive strength	[20]
Cement paste	0.033	0.4	-	-	131.6% split tensile strength 95.7% compressive strength	[14]
Cement paste (hybrid GO/CNT)	0.02 wt % GO 0.04 wt% CNTs	0.4	300 W, 30 min	Polycarboxylate based superplasticizer	23.9% compressive strength 16.7% flexural strength	[25]
Cement paste (GO-NS)	0.03 wt%	0.44	-	-	133% compressive strength	[23]
Cement paste	0.05	0.5	-	-	15-33% compressive strength 41-59% flexural strength	[22]
Cement paste	0.04	0.4	150 W, 5 min	-	67% tensile strength	[13]
Cement paste (pristine graphene)	0.07	0.48	30 min	MasterGlenium SKY8100	34.3% compressive strength 26.9% tensile strength	[26]
Cement paste	0.04	0.4	10min	-	37% compressive strength 14.2% flexural strength	[10]
Cement paste	-	0.4	15 min	PC	53.2% young’s modulus 78.8% flexural strength 62.3% fracture energy	[17]
Cement paste	0.04	0.4	5min	-	83% flexural strength	[27]
Cement paste	0.03	0.27	-	PC	40% flexural strength	[28]
Cement paste	0.05	0.5	-	-	90.9% compressive strength 181.2% flexural strength	[29]
Cement paste	0.003	0.44	-	-	9.4-31% compressive strength 8.1-36.7% flexural strength	[30]

8. Electrical Resistivity

Inclusion of greater amount of graphene with adequate amount of silica fume improves the electrical conductivity of the composites [33]. Giatec device was used to measure the resistivity of GO solutions. Li et al proposed that incorporation of GO reinforced cement composites found to be electrically more insulative. Flexural strength of the cement composite enhanced with the addition of GO in cement composites [27]. The electrical resistivity of the GO-based composite higher compared to the conventional specimen. An increase in electrical resistivity was found during the dissolution stage which increases the nucleation effect of GO [10].

9. Resistance to Chloride Penetration

Addition of GNP in concrete refines the microstructure and tortuosity of concrete and enhances the resistance of concrete to chloride ion and water penetration. incorporation of GNP in cement composites enhances the durability and reduces the water permeability, chloride diffusion coefficient and chloride migration coefficient [46].

10. Water Absorption

GO reinforced concrete shows reduction in surface absorption after the allowing sufficient curing period. due to the filling effect of GO in cement composites GO act as filling material fill the voids and reduces the absorption of water. Depending on the amount of GO content the porosity of the reinforced mixes will reduced since the nucleation formation of cement hydrates accelerate with respect to GO content in the cement matrix [39]. Inclusion of rGO in cement composite enhances the dense structure of the composites their by improves the water absorption capacity of the composite [51].

11. Microstructural Analysis

Scanning Electron Microscopy (SEM)

Morphological characteristics can be analysed with the help of SEM analysis. Graphene sheet consists of wrinkles and crumbled texture on the surface [28]. GO had a layered structure with a thin, folded sheet connected to shape a disordered solid [29].

XRD analysis shows that EOGO act as a nano seeding material in cement pore solution to promote CSH and other hydration products. SEM analysis also agrees with XRD data. In SEM analysis also EOGOs are found in the presence of CSH [43]. XRD analysis reveals that addition of GO sheet lowers the orientation index calcium hydroxide crystals. SEM analysis confirms that incorporation of GO sheets produce high degree of dispersion and promote the hydration and restraints the extension process of cracks [32] .Stronger degree of connectivity with the matrix was found in the multi-scale structure of CF-CNTs [49]. Compared to rGO composites GO composite consist higher content of $Ca(OH)_2$ and C-S-H. Amorphous C-S-H gel and a combination of crystalline hydrated products such as $Ca(OH)_2$ are filled in the pores of GO composites. Random pore filling was found in rGO composites and common hydration products mostly ettringite with partial portlandite and other hydration products. [40]. compared to conventional cement paste GO based cement composite found to be more electrically insulative [52].

12. Conclusion

In this research review a substantial research effort has been focused on the compressive, tensile strength and flexural strength of cement modified by inclusion of GO.

From the extensive review the following conclusions can be drawn

GO reinforced cement composites alters the fresh properties of the cement paste. incorporation of GO with cement reduces slump, initial and final setting time due to the large surface area. The large surface area of the GO plays the major role in controlling the workability, hydration mechanism, strength enhancement and durability behavior.

GO fabricated cement composite improves the hardened properties of the cement composite. flexural strength and tensile strength of the GO modified cement composites enhanced greatly compared to compressive strength.

Refined pore structure enhances the dense and compact structure and thus improves the mechanical properties of the composite.

Smaller particle size and the large surface area promotes the nucleation sites due to the seeding effect of GO agglomerates which in turn accelerates the hydration of cement paste. Flower like hydration crystals was found with the addition of GO which forms the more compact and dense structure.

GO modified cement composites results higher electrical resistivity than the conventional specimens. Increased electrical resistivity accelerates the nucleation sites.

The main barrier in harnessing maximum benefits from it is the agglomeration and difficulty in the proper dispersion of graphene in cement. Ultrasonication is used for the dispersion of graphene in water. Dispersion potential and the amount of energy and time required to achieve uniform dispersion to be further developed and investigated to enhance the overall behaviour of cement-based composites.

Cost of fabrication of graphene solution is quite high due to the complex procedure and extensive time for processing. Fabrication of graphene solution at lower cost for its production has to be further developed and investigated.

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