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DEVELOPMENT OF AN
ADVANCED SNOW-
MELTING GEOPOLYMER
MORTAR UTILIZING
GRAPHENE



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16. Abstract <p>Winter road maintenance often relies on salts to melt snow and ice, but repeated salt use can damage concrete infrastructure and increase long-term maintenance needs. This project examined whether fly ash-based geopolymer mortar containing graphene nanoplatelets could be developed as a lower-cement, electrically responsive material for future snow-melting applications. Graphene nanoplatelets can help reduce electrical resistivity, but they tend to clump together in water and cementitious materials. This can limit their effectiveness and may reduce mortar performance. Before mixture development, the commercial graphene nanoplatelets were characterized using transmission electron microscopy, scanning electron microscopy, Raman spectroscopy, and X-ray photoelectron spectroscopy to verify the quality and baseline characteristics of the supplied material. The study then screened graphene nanoplatelet suspensions prepared with three surfactants, sodium dodecyl sulfate, Triton X-100, and polyvinylpyrrolidone. The suspensions were compared using electrical conductivity, pH, and visual dispersion stability over time. Based on this preliminary screening, sodium dodecyl sulfate was selected as the surfactant for the geopolymer mortar mixtures because it provided the most practical balance of dispersion stability and measured suspension behavior. The selected sodium dodecyl sulfate and graphene nanoplatelet system was then incorporated into fly ash-based geopolymer mortar at different graphene nanoplatelet contents and surfactant-to-graphene nanoplatelet ratios. The mortar mixtures were evaluated for workability, compressive strength, ultrasonic pulse velocity, and bulk electrical resistivity. The results showed that adding more graphene nanoplatelets did not automatically improve performance. Lower graphene nanoplatelet contents, especially 0.05% and 0.1%, provided a better balance between maintaining hardened properties and reducing electrical resistivity. Higher graphene nanoplatelet contents often produced less favorable results, which may be explained by poorer dispersion, particle agglomeration, or disruption of the mortar matrix. These findings help narrow the mixture designs that should be considered in future electrical-heating and ice-melting tests.</p>					
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Development of an Advanced Snow-Melting Geopolymer Mortar Utilizing Graphene

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ABSTRACT

Winter road maintenance often relies on salts to melt snow and ice, but repeated salt use can damage concrete infrastructure and increase long-term maintenance needs. This project examined whether fly ash-based geopolymer mortar containing graphene nanoplatelets could be developed as a lower-cement, electrically responsive material for future snow-melting applications. Graphene nanoplatelets can help reduce electrical resistivity, but they tend to clump together in water and cementitious materials. This can limit their effectiveness and may reduce mortar performance. Before mixture development, the commercial graphene nanoplatelets were characterized using transmission electron microscopy, scanning electron microscopy, Raman spectroscopy, and X-ray photoelectron spectroscopy to verify the quality and baseline characteristics of the supplied material. The study then screened graphene nanoplatelet suspensions prepared with three surfactants, sodium dodecyl sulfate, Triton X-100, and polyvinylpyrrolidone. The suspensions were compared using electrical conductivity, pH, and visual dispersion stability over time. Based on this preliminary screening, sodium dodecyl sulfate was selected as the surfactant for the geopolymer mortar mixtures because it provided the most practical balance of dispersion stability and measured suspension behavior. The selected sodium dodecyl sulfate and graphene nanoplatelet system was then incorporated into fly ash-based geopolymer mortar at different graphene nanoplatelet contents and surfactant-to-graphene nanoplatelet ratios. The mortar mixtures were evaluated for workability, compressive strength, ultrasonic pulse velocity, and bulk electrical resistivity. The results showed that adding more graphene nanoplatelets did not automatically improve performance. Lower graphene nanoplatelet contents, especially 0.05% and 0.1%, provided a better balance between maintaining hardened properties and reducing electrical resistivity. Higher graphene nanoplatelet contents often produced less favorable results, which may be explained by poorer dispersion, particle agglomeration, or disruption of the mortar matrix. These findings help narrow the mixture designs that should be considered in future electrical-heating and ice-melting tests.

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LIST OF KEY TERMS

Alternating current, AC

An electric current that changes direction periodically. AC is relevant to future electrical-heating tests because self-heating performance depends on voltage, current, impedance, and power dissipation.

Analysis of variance, ANOVA

A statistical method used to test whether measured test results differ among factor levels. In this study, ANOVA was used to evaluate the effects of graphene nanoplatelet concentration, surfactant-to-GNP ratio, and their interaction.

Bulk electrical resistivity

A geometry-corrected measure of how strongly a material resists electrical current flow. Lower bulk electrical resistivity is generally consistent with higher electrical conductivity.

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Desirability analysis

A multi-response screening method that converts each test result into a normalized desirability value and combines the values into an overall ranking index.

Direct current, DC

An electric current that flows in one direction. DC is relevant as a comparison condition for future electrical-heating studies.

Graphene nanoplatelets, GNPs

Multilayer graphene-based particles used as conductive fillers in this study. GNPs can reduce electrical resistivity if they are sufficiently dispersed and form conductive pathways.

Log₁₀ bulk electrical resistivity

The base-10 logarithm of bulk electrical resistivity. This transformation was used because resistivity values can span a wider numerical range than strength or ultrasonic pulse velocity.

Multi-response desirability index

The overall desirability score calculated from individual desirability values for multiple test results. In this study, it was used as a screening metric rather than as proof of ice-melting performance.

Polyvinylpyrrolidone, PVP

A polymeric stabilizer evaluated during the preliminary graphene nanoplatelet suspension screening.

Principal component analysis, PCA

A multivariate analysis method used to summarize variation among mixtures when multiple test results are considered together.

Response surface regression model, RSM

A fitted regression model used to describe trends, curvature, and interaction patterns across the tested graphene nanoplatelet concentration and surfactant-to-GNP ratio range.

Root mean square error, RMSE

A model-fit statistic that describes the average magnitude of prediction error in the units of the modeled test result.

Sodium dodecyl sulfate, SDS

An anionic surfactant evaluated during the preliminary screening stage and selected for the geopolymer mortar phase.

Sodium hydroxide, NaOH

An alkaline activator component used to increase alkalinity and support geopolymer reaction.

Sodium silicate, Na₂SiO₃

A soluble silicate source used in the alkaline activator for fly ash-based geopolymer mortar.

Sodium oxide, Na₂O

A chemical constituent used in calculating the activator composition and silicate modulus.

Silicon dioxide, SiO₂

A chemical constituent used in calculating the silicate modulus of the alkaline activator.

Silicate modulus, SiO₂/Na₂O

The molar ratio of silicon dioxide to sodium oxide in the alkaline activator. This ratio influences geopolymer reaction and mixture behavior.

Saturated surface dry, SSD

A moisture condition in which the specimen surface is dry but internal pores contain moisture. SSD condition can affect electrical resistivity results.

Surfactant

A chemical additive used to improve the dispersion of graphene nanoplatelets by reducing particle agglomeration. Surfactants may also affect pH, workability, air content, pore structure, and hardened properties.

Surfactant-to-GNP mass ratio

The mass of surfactant divided by the mass of graphene nanoplatelets. This ratio was one of the main variables in the suspension and mortar phases.

Triton X-100, TX-100

A nonionic surfactant evaluated during the preliminary graphene nanoplatelet suspension screening.

Ultrasonic pulse velocity, UPV

A nondestructive test result based on the speed of an ultrasonic pulse through a specimen. In this study, UPV was used as an indirect indicator of internal continuity and relative material quality.

X-ray photoelectron spectroscopy, XPS

A surface-analysis method used to evaluate the chemical state and surface composition of materials. In this report, XPS was used to characterize graphene nanoplatelets.

Loss on ignition, LOI

The mass loss of a material after heating, commonly used as part of fly ash characterization.

EXECUTIVE SUMMARY

The original goal of this project was to develop an electrically conductive geopolymer material for potential snow-melting applications using graphene nanoplatelets and landfilled fly ash. The proposed work was broader than the work completed in this report. It included graphene nanoplatelet dispersion, landfilled fly ash activation, conductive geopolymer development, durability testing, and ice-melting performance evaluation. The completed report covers only part of that scope. The project funding was stopped after the principal investigator left Colorado State University and joined Boise State University, and the funds could not be transferred. As a result, the landfilled fly ash activation, durability testing, and direct ice-melting validation tasks were not completed.

The completed work focused on early-stage material screening. As an initial quality-verification task, the commercial graphene nanoplatelets were characterized using transmission electron microscopy, scanning electron microscopy, Raman spectroscopy, and X-ray photoelectron spectroscopy to verify their baseline morphology, graphitic structure, and surface chemistry before mixture preparation. The aqueous screening task then evaluated graphene nanoplatelet suspensions prepared using sodium dodecyl sulfate, Triton X-100, and polyvinylpyrrolidone. The suspensions were evaluated using electrical conductivity, pH, and visual stability over 12 days. This screening step was needed because graphene nanoplatelets can agglomerate, and poor dispersion can limit electrical performance or reduce mortar quality. Based on the preliminary screening, sodium dodecyl sulfate was selected for the mortar phase.

The mortar phase evaluated fly ash-based geopolymer mixtures containing different graphene nanoplatelet concentrations and sodium dodecyl sulfate-to-graphene nanoplatelet mass ratios. Surfactant-only controls were included to separate the effect of graphene nanoplatelet addition from the effect of surfactant dosage. The mixtures were tested for workability, ultrasonic pulse velocity, bulk electrical resistivity, and compressive strength. Statistical analysis was used to evaluate factor interactions, model fitted response trends, summarize mixture-level behavior using principal component analysis, and rank candidate mixtures using desirability analysis.

The results indicate that increasing graphene nanoplatelet content did not produce a consistent improvement in mortar performance. The measured behavior depended on the combination of graphene nanoplatelet concentration and surfactant-to-graphene nanoplatelet ratio. Lower graphene nanoplatelet contents, especially 0.05% and 0.1%, generally provided a more favorable balance between hardened-property retention and reduced electrical resistivity than higher graphene nanoplatelet contents. This suggests that dispersion quality and surfactant dosage may have been more influential than simply increasing conductive filler content.

The findings should be interpreted as screening results, not as proof of snow-melting performance. Bulk electrical resistivity can indicate whether a mixture is moving toward a useful electrical range, but it does not measure heating rate, power demand, temperature distribution, or ice-melting time. Direct electrical-heating and ice-melting tests remain necessary before the material can be considered a validated snow-melting geopolymer system.

1. INTRODUCTION AND BACKGROUND

1.1 Project Motivation

Concrete remains one of the most widely used construction materials, but its conventional binder system depends heavily on Portland cement clinker. Cement manufacturing is associated with high process and fuel-related carbon dioxide emissions, and global demand for concrete is expected to continue increasing as infrastructure systems expand and are renewed (World Economic Forum 2024). This creates a practical materials problem. Lower-clinker binders must be developed without treating sustainability as the only performance criterion. They must also provide workable mixtures, adequate hardened properties, and functions that are relevant to construction applications.

Fly ash-based geopolymer mortar is one candidate response to this problem because it uses an aluminosilicate-rich industrial by-product as the main precursor rather than as a partial replacement for Portland cement. Geopolymer binders are formed when reactive silica- and alumina-bearing phases dissolve in an alkaline solution and then reorganize into a hardened binding network (Davidovits 1991; Pacheco-Torgal et al. 2008). In low-calcium fly ash systems, the primary binding product is commonly described as sodium aluminosilicate hydrate, although the presence of calcium can lead to mixed reaction products and more complex gel assemblages (Singh and Singh 2019; Siyal et al. 2024). This distinction matters for the present project. The relevant material is not Portland cement concrete containing fly ash as a supplementary cementitious material. It is a mortar in which Class F fly ash is the primary reactive precursor activated by sodium hydroxide and sodium silicate.

The use of fly ash also connects the binder design to a broader materials reuse issue. Coal combustion products are generated in large quantities, and fly ash has been widely examined for beneficial use in construction materials (Heidrich and Feuerborn 2022; Yao et al. 2015). However, abundance alone does not establish suitability for a functional construction material. Fly ash source, oxide composition, glass content, fineness, activator formulation, and curing history can all influence geopolymer reaction products and hardened performance. For that reason, the sustainability argument must be tied to the actual performance of the fly ash-based geopolymer mortar produced in the study rather than to fly ash availability alone.

At the same time, cementitious materials are increasingly being designed for functions beyond load transfer and durability. Electrically conductive cementitious composites can support self-heating, deicing, self-sensing, and structural health monitoring when a sufficiently connected conductive network is formed inside the matrix (Han et al. 2017). For pavement and bridge deck applications, electrically conductive concrete has been studied as a self-heating material that can generate heat under an applied electrical field, which may reduce dependence on chemical deicers and repeated mechanical removal (Yehia and Tuan 2000). This application is relevant because snow and ice create safety and maintenance problems, while deicing chemicals can contribute to concrete deterioration and steel corrosion (Federal Highway Administration n.d.; Haselbach et al. 2021; Wang et al. 2024). The motivation for this report therefore extends beyond replacing cement. It is to examine whether a low-clinker geopolymer mortar can also provide electrical functionality for smart construction applications.

For ice-melting and self-heating applications, the objective is not to make the mortar fully conductive in the sense of a metallic conductor. The more appropriate objective is to reduce the electrical resistivity into a controllable range that allows heat generation under an applied electrical field without excessive current demand, localized overheating, or unstable electrical response. Under direct current or under alternating current conditions where the material response is predominantly resistive, the resistance of a prismatic cementitious element can be approximated as follows (Ling et al. 2016):

$$R = \rho \frac{L}{A} \quad (1)$$

where R is electrical resistance, ρ is bulk electrical resistivity, L is the current path length, and A is the cross-sectional area normal to current flow. For alternating current, the more general electrical response is described by impedance,

$$Z = R + jX \quad (2)$$

where Z is complex impedance, R is the resistive component, X is the reactive component, and j is the imaginary unit. The average real power converted to heat is:

$$P = V_{\text{rms}} I_{\text{rms}} \cos \phi \quad (3)$$

where V_{rms} and I_{rms} are the root-mean-square voltage and current, and ϕ is the phase angle between voltage and current. If the material behaves mainly as a resistor, $\cos \phi \approx 1$, and the relationship reduces to:

$$P \approx I_{\text{rms}}^2 R \approx \frac{V_{\text{rms}}^2}{R} \quad (4)$$

For a material subjected to an approximately uniform alternating electric field, the time-averaged volumetric heat generation can be expressed using resistivity as:

$$q''' \approx \frac{E_{\text{rms}}^2}{\rho_{\text{eff}}} \quad (5)$$

where ρ_{eff} is effective AC resistivity at the applied frequency. If the electrode spacing is L , then:

$$E_{\text{rms}} \approx \frac{V_{\text{rms}}}{L} \quad (6)$$

and therefore, Equation 5 can be written as:

$$q''' = \frac{V_{\text{rms}}^2}{\rho_{\text{eff}} L^2} \quad (7)$$

For the present material-screening stage, a reduction in resistivity is useful because it indicates increased electrical responsiveness relative to a more resistive matrix. However, resistivity alone cannot define the optimum for a snow-melting system. Final heating design would require an application-specific resistivity range established together with the applied voltage, electrode spacing, power demand, temperature response, and electrical stability. Conductive concrete overlays have been evaluated for bridge deck deicing and anti-icing because electrical resistance and impedance in the composite can generate heat when connected to a power source (Sassani et al. 2018; Tuan 2004; Tuan and Yehia 2004; Yehia and Tuan 1999, 2000). Field and slab studies reported that conductive concrete overlays could generate heat for snow and ice control, with reported average power levels on the order of several hundred watts per square meter (Tuan 2004; Yehia and Tuan 1999). Tuan also reported that AC and DC heating rates were similar, but AC power was preferred because it produced more uniform heating and reduced hot-spot formation (Tuan 2004). More recent reviews similarly identify self-heating conductive cementitious composites as candidate materials for deicing and electrical curing, but they also emphasize that performance depends on conductive filler type, dosage, dispersion, electrode configuration, moisture state, and electrical stability (Anis and Abdel-Raheem 2024; Oumer et al. 2024). This limitation is especially relevant for cementitious nanocomposites because measured conductivity and apparent percolation behavior can depend on moisture condition and testing frequency (Piana et al. 2023). Therefore, in the present study, bulk electrical resistivity is treated as a functional screening property rather than as direct proof of ice-melting performance. A reduction in resistivity is beneficial only if it occurs with acceptable workability, hardened performance, and stable electrical response. This test cannot independently confirm ice-melting capacity because direct AC heating and ice-melting experiments were not performed. It can only indicate whether the GNP-modified geopolymer mortar moved toward a resistivity range that may be suitable for future self-heating evaluation. Previous studies indicate that conductive fillers can reduce electrical resistivity in cementitious and geopolymer matrices when they form continuous or near-continuous conductive pathways. Carbon fibers, carbon nanotubes, graphite, graphene oxide, and related carbon-rich additives have been examined for this purpose (Gao et al. 2024; Górski et al. 2022; Krishna et al. 2023; Payakaniti et al. 2017). These studies support the basic mechanism, but they also show that electrical performance depends on filler type, dosage, dispersion state, and matrix chemistry. A conductive filler does not automatically produce a conductive composite. If the filler agglomerates, remains isolated, or disrupts the pore structure, the material may lose workability or strength without developing a stable conductive network.

Graphene nanoplatelets (GNPs) are relevant to this application because graphene-based materials have high intrinsic electrical conductivity, high aspect ratio, and the potential to form conductive networks at relatively low filler contents when dispersion is adequate (Cataldi et al. 2018; Geim and Novoselov 2007). However, intrinsic conductivity alone does not ensure a conductive cementitious or geopolymer composite. The measured electrical response depends on whether the conductive particles form continuous or near-continuous pathways within the hardened matrix. It is also affected by contact resistance, particle spacing, moisture condition, pore solution chemistry, and the stability of the filler network.

The same particle characteristics that make GNPs useful can also limit their effectiveness. GNPs have high specific surface area and strong interparticle attraction, mainly associated with van der Waals forces and hydrophobic surface behavior. These characteristics make them prone to

agglomeration in water, alkaline activator solutions, and cementitious matrices. Agglomeration can interrupt conductive pathway formation, reduce filler efficiency, and introduce weak regions or defects into the hardened matrix. Surfactants are commonly used to improve dispersion through noncovalent stabilization, but their effects are not limited to particle separation. They can also alter suspension pH, viscosity, air entrainment, workability, pore structure, and hardened properties (Hsieh et al. 2013; Lin et al. 2011; Qamar et al. 2024). Therefore, the central technical problem is not simply adding GNPs to the mortar. The more relevant problem is identifying a GNP-surfactant combination that reduces bulk electrical resistivity into a useful range without causing unacceptable losses in fresh or hardened mortar performance.

This problem is more specific in fly ash-based geopolymer mortar than in water-based suspensions or Portland cement systems. A surfactant that appears effective in distilled water may behave differently in a highly alkaline activator containing sodium hydroxide and sodium silicate. The ionic environment, dissolved silicate species, precursor particles, sand, and curing conditions can change GNP dispersion, surfactant adsorption, and conductive network formation in the hardened mortar. For that reason, the experimental program separated material quality verification, aqueous suspension screening, and mortar performance into distinct tasks. The GNP characterization task was used to verify the quality and baseline characteristics of the supplied material; it was not a separate performance objective. Suspension tests then provided an initial screening step, while mortar testing was required to determine whether the selected surfactant and GNP dosage produced a useful balance among workability, compressive strength, ultrasonic pulse velocity, and bulk electrical resistivity.

1.2 Research, Objectives, and Tasks

The original CTIPS proposal included a broader research program on conductive geopolymer materials for snow-melting applications. The proposed scope included graphene nanoplatelet dispersion, landfilled fly ash activation, conductive geopolymer development, durability testing, and direct ice-melting evaluation. However, the completed scope was narrowed after project funding was stopped when the principal investigator left Colorado State University and joined Boise State University, and the remaining funds could not be transferred. Therefore, this final report focuses on the completed portion of the project, which included quality verification of the supplied graphene nanoplatelets through material characterization, graphene nanoplatelet dispersion screening, and fly ash-based geopolymer mortar evaluation. Landfilled fly ash activation, durability testing, and direct ice-melting validation were not completed and are identified as future work.

The objective of the completed portion of this work was to evaluate graphene nanoplatelet-modified fly ash-based geopolymer mortar for potential smart construction applications. The study focused on identifying mixture conditions that could reduce bulk electrical resistivity while maintaining acceptable workability, ultrasonic pulse velocity, and compressive strength.

The first objective was to evaluate the dispersion behavior of graphene nanoplatelets in water using different surfactants. Sodium dodecyl sulfate, Triton X-100, and polyvinylpyrrolidone were examined at different graphene nanoplatelet concentrations and surfactant-to-GNP mass ratios. Electrical conductivity, pH, and visual agglomeration were used to select the surfactant system for the mortar phase.

The second objective was to incorporate the selected surfactant-GNP system into fly ash-based geopolymer mortar and evaluate the resulting fresh and hardened properties. The mortar mixtures were tested for workability, ultrasonic pulse velocity, bulk electrical resistivity, and compressive strength.

The third objective was to identify GNP concentration and surfactant-to-GNP mass ratio combinations that provided the best balance between electrical and hardened-property performance. Statistical analysis was used to evaluate whether the effects of GNP concentration and surfactant-to-GNP mass ratio acted independently or depended on their interaction.

The completed experimental program was organized into three tasks. Task 1 characterized the supplied GNPs to verify material quality and establish baseline morphological, structural, and surface-chemical characteristics. Task 2 screened aqueous GNP-surfactant suspensions. Task 3 evaluated geopolymer mortar mixtures prepared with the selected surfactant system. This reduced scope reflects the funding interruption and the inability to transfer the remaining funds after the institutional transition.

1.3 Report Overview

Chapter 2 describes the materials, mixture design, specimen preparation, and test methods used in the study. The chapter introduces the graphene nanoplatelets, surfactants, Class F fly ash, sand, sodium hydroxide, and sodium silicate solution. It first describes the characterization methods used to verify the quality and baseline characteristics of the supplied GNPs, then describes the preparation and screening of graphene nanoplatelet-surfactant suspensions used to select the surfactant for the mortar phase. The chapter also presents the geopolymer mortar mixture design and the procedures used to measure workability, compressive strength, ultrasonic pulse velocity, and bulk electrical resistivity.

Chapter 3 presents and discusses the experimental results. It begins with the GNP characterization results used to document the quality and baseline characteristics of the supplied material. The next part evaluates the electrical conductivity, pH, and visual stability of the graphene nanoplatelet-surfactant suspensions and provides the basis for selecting the surfactant used in the geopolymer mortar mixtures. The following part evaluates the fresh and hardened properties of the fly ash-based geopolymer mortars. The discussion focuses on how graphene nanoplatelet concentration and surfactant-to-GNP mass ratio affected workability, compressive strength, ultrasonic pulse velocity, and bulk electrical resistivity. The statistical analysis section then evaluates factor interactions, response surface trends, principal component patterns, and desirability-based mixture ranking.

Chapter 4 summarizes the main findings and identifies the mixture conditions that provided the most practical balance between electrical response and hardened mortar performance. It also discusses the limitations of the completed work and the additional testing needed before graphene nanoplatelet-modified fly ash-based geopolymer mortar can be evaluated for field-oriented smart construction applications.

2. METHODOLOGY

This chapter describes the materials, mixture proportions, specimen preparation procedures, and test methods used to develop electrically conductive fly ash-based geopolymer mortar containing graphene nanoplatelets. The experimental program was organized into three tasks. Task 1 characterized the supplied GNPs to verify material quality and establish baseline morphological, structural, and surface-chemical characteristics before mixture preparation. Task 2 screened graphene-surfactant suspensions in water to identify which surfactant provided the most suitable combination of dispersion stability, pH, and electrical conductivity. Task 3 used the selected surfactant in fly ash-based geopolymer mortar mixtures to evaluate fresh and hardened performance.

Task 2 was intentionally separated from the mortar phase because graphene nanoplatelets can agglomerate before they are introduced into the highly alkaline geopolymer system. In this screening task, three surfactants were evaluated at four graphene nanoplatelet concentrations and four surfactant-to-graphene mass ratios. This produced 48 aqueous suspensions. Electrical conductivity, pH, and visual evidence of agglomeration were used to compare the suspension systems. These measurements did not independently establish mortar performance, but they provided a controlled basis for selecting the surfactant used in Task 3.

Task 3 translated the selected dispersion system into mortar mixtures. Class F fly ash was activated with sodium hydroxide and sodium silicate, then combined with sand and graphene-surfactant suspension. The mortar mixtures were evaluated for workability, compressive strength, ultrasonic pulse velocity, and bulk electrical resistivity. This structure connects the material-selection stage to the performance stage and avoids treating suspension conductivity as a direct substitute for hardened mortar conductivity.

2.1 Materials

2.1.1 Fly Ash

In this study, Class F fly ash conforming to ASTM C618 (ASTM International 2025b) was used as the primary aluminosilicate precursor. Its chemical composition is presented in Table 1. The fly ash had a reported median particle size of 16.34 μm , specific surface area of 598 m^2/kg , and density of 2.39 g/cm^3 .

Table 1. Oxide composition and loss on ignition (LOI) of Class F fly ash (%mass)

SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	Na ₂ O	K ₂ O	LOI
54.24	18.16	10.42	7.37	1.46	1.30	1.24	2.71	1.13

2.1.2 Graphene Nanoplatelets

Commercial-grade graphene nanoplatelets were used as the conductive filler in this study. Supplier-reported properties were used for the initial material screening and were supplemented by independent characterization, as detailed in Section 3.1. The GNPs had a reported purity greater than 90 wt.%, a particle diameter of 0.5–0.7 μm , and fewer than 15 graphene layers.

2.1.3 Sodium Silicate

A commercial-grade sodium silicate solution was used as the soluble silicate component of the alkaline activator, which also provided a portion of the mixture water. The solution consisted of 28 wt.% SiO₂, 9 wt.% Na₂O, and 63 wt.% water. This yielded an SiO₂/Na₂O mass ratio of 3.11 and a molar ratio of approximately 3.21. The supplier-reported pH was 11.3, with a density of 1.39 g/cm³ at 20°C.

2.1.4 Sodium Hydroxide

Regent grade anhydrous sodium hydroxide was used to increase alkalinity and adjust the Na₂O equivalent of the activator. The reported purity was 99+%.

2.1.5 Surfactants

Three surfactants were evaluated in the preliminary screening stage to test their efficacy in reducing GNP agglomeration in water before the suspension was incorporated into mortar. Sodium dodecyl sulfate (SDS) was the anionic surfactant. Triton X-100 (TX-100) was the nonionic surfactant. Polyvinylpyrrolidone (PVP) was the polymeric stabilizer. Table 2 lists the physicochemical properties of these three surfactants.

Table 2. Physicochemical properties of surfactants used for GNP dispersion

Surfactant	pH	Density (g/cm ³)	Purity (%)
TX-100	9.7	1.07	90 to 100
PVP	5.0 to 8.0 at 10 g/L	1.69	99
SDS	9.1 at 10 g/L	0.370	98.40

2.1.6 Fine Aggregates

Washed multi-purpose sand was used as the fine aggregate in all mortar mixtures to maintain a consistent aggregate source across the experimental program. The sand had a bulk specific gravity of 2.63, an apparent specific gravity of 2.71, a water absorption of 1.75%, and a fineness modulus of 2.90. The particle size distribution of the sand is shown in Figure 1.

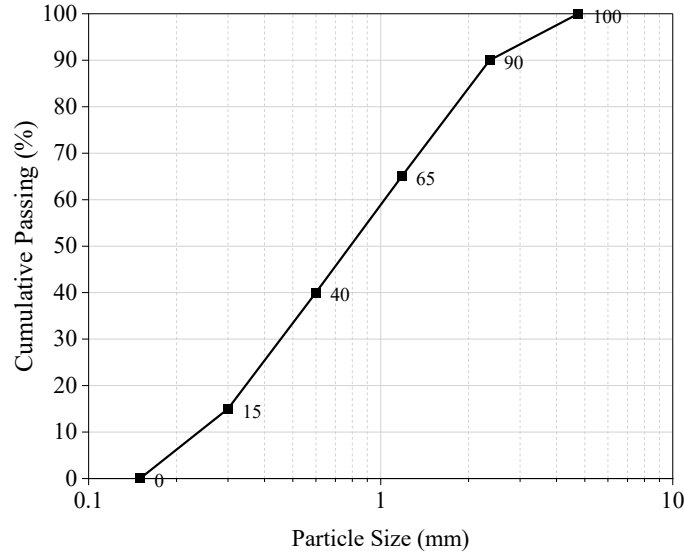


Figure 1. Gradation of multi-purpose sand.

2.2 Test Setup & Process

The experimental program was conducted in three tasks. Task 1 characterized the commercial GNPs to evaluate their platelet morphology, graphitic structure, defect-related features, and surface bonding states before mixture preparation. Task 2 evaluated the aqueous dispersion behavior of GNP-surfactant systems using 48 formulations prepared in distilled water. Suspension behavior was assessed through visual observations of sedimentation, clustering, and phase separation, supplemented by electrical conductivity and pH measurements. Based on this screening, one surfactant was selected for further evaluation. Task 3 incorporated the selected surfactant into fly ash-based geopolymer mortar and evaluated the effects of GNP dosage and surfactant-to-GNP mass ratio on workability, compressive strength, ultrasonic pulse velocity, and bulk electrical resistivity under two moisture conditions. Control mixtures containing the selected surfactant without GNPs were included to distinguish surfactant-related effects from those associated with GNP addition. Table 3 summarizes the three-task experimental program.

Table 3. Three-task experimental design used in the study

Tasks	Purpose	Main variables	Measured responses
Task 1	GNP quality verification	Morphological, structural, and surface-chemical	TEM, SEM, Raman spectroscopy, XPS
Task 2	Aqueous suspension stability screening	SDS, TX-100, PVP	Conductivity, pH, visual agglomeration
Task 3	Mortar performance testing	Selected surfactant from Task 2, GNP dosage, surfactant-to-GNP ratio	Flow, compressive strength, UPV, bulk electrical resistivity

2.2.1 Task 1: GNPs Characterization

The commercial GNPs were characterized before mixture preparation to verify that the supplied material exhibited morphological, structural, and surface-chemical characteristics consistent with graphene nanoplatelets and to establish a baseline description of the starting material. GNPs were characterized using transmission electron microscopy (TEM), scanning electron microscopy (SEM), Raman spectroscopy, and X-ray photoelectron spectroscopy (XPS). Electron microscopy was used to examine platelet morphology and lateral geometry, Raman spectroscopy evaluated graphitic structure and defect-related features, and XPS assessed near-surface elemental composition and carbon bonding states.

For TEM, approximately 5 mg of dry GNP powder was dispersed in ethanol and ultrasonicated for 10 minutes. A droplet of the ethanolic dispersion was deposited onto a carbon-coated copper grid and dried before imaging. TEM was performed using a Hitachi HT7700 microscope operated at 120.0 kV, and the representative micrograph was collected at 50,000 $\times$ magnification.

For SEM, dry GNP powder was mounted on conductive carbon tape attached to an aluminum stub. The powder was gently spread, and loosely attached particles were removed by tapping. No sputter coating was applied because GNPs are electrically conductive and imaging was conducted at low accelerating voltage. SEM was performed using a ZEISS Sigma 300 field-emission microscope equipped with an InLens detector. The representative image was collected at 1.00 kV, a working distance of 4.5 mm, and 5,000 $\times$ magnification.

Raman spectroscopy was performed using a WITec alpha300 R Raman microscope equipped with a 532 nm laser. Dry GNP powder was spread on a quartz slide and analyzed at room temperature using a 20 $\times$ objective, approximately 0.1 mW laser power, 0.5 s integration time, and 200 accumulations. The spectra were smoothed using the Savitzky-Golay method and baseline corrected.

XPS was performed using a Thermo Scientific ESCALAB 250Xi spectrometer equipped with a monochromatic Al K α source with a photon energy of 1,486.6 eV. GNP powder was pressed onto indium foil and analyzed under ultrahigh vacuum. Survey spectra were collected using a pass energy of 150 eV. High-resolution C 1s and O 1s spectra were collected using a pass energy of 20 eV. Binding energies were corrected using the C 1s reference peak at 284.8 to 285.0 eV. The C 1s spectrum was fitted after background subtraction using nonlinear least-squares curve fitting.

2.2.2 Task 2: Aqueous Suspension Stability Screening

In Task 2, we evaluated the suspension stability of GNP-surfactant systems in water. The screening matrix combined three dispersing agents, SDS, TX-100, and PVP, with GNP concentrations of 0.05, 0.10, 0.50, and 1.00 wt.% relative to the mass of water and surfactant-to-GNP mass ratios of 0.5:1, 1:1, 1.5:1, and 2:1. These levels were selected to span GNP concentrations and dispersant dosages representative of ranges investigated in previous GNP dispersion and cementitious-composite studies (Jiang et al. 2023; Nazari et al. 2019). The GNP concentrations included two lower levels, 0.05 and 0.10 wt.%, and two higher levels, 0.50 and 1.00 wt.%, with the highest concentration being 20 times the lowest. This range was selected to

examine how suspension behavior changed as GNP concentration increased. The surfactant-to-GNP ratios provided surfactant masses below, equal to, and above the GNP mass, allowing the influence of relative surfactant dosage to be compared across the selected GNP concentrations. These concentrations and ratios were used as discrete screening levels rather than as pre-determined optimum dosages. Combining the three dispersing agents, four GNP concentrations, and four mass ratios produced 48 aqueous suspensions.

The suspensions were compared using macroscopic stability, electrical conductivity, and pH as practical screening indicators for selecting a GNP-surfactant system for Task 3. Macroscopic observations were used to assess visible sedimentation, separation, and persistence of the suspended GNPs over time. Electrical conductivity provided a complementary measure of the ionic characteristics of the suspension, particularly for systems containing ionic surfactants, while pH was monitored to identify differences in the chemical conditions among the candidate systems. Tables 4 and 5 summarize the suspension screening design and surfactant mass corresponding to each surfactant-to-GNP ratio, respectively.

Table 4. Suspension screening design matrix

Design factor	Levels	Purpose
Surfactants	SDS, TX-100, PVP	Compare surfactant
GNP concentrations	0.05, 0.10, 0.50, 1.00 wt.% of water	Evaluate particle dosage
Surfactant-to-GNP mass ratios	0.5:1, 1:1, 1.5:1, 2:1	Evaluate stabilization dosage
Total suspensions	48	$3 \times 4 \times 4$ combinations

Table 5. Mass of surfactant used per 100 g water in the suspension screening design

GNP per 100 g water (g)	Surfactant mass in grams per each level of surfactant-to-GNP mass			
	0.5:1 ratio	1:1 ratio	1.5:1 ratio	2:1 ratio
0.05	0.025	0.050	0.075	0.100
0.10	0.050	0.100	0.150	0.200
0.50	0.250	0.500	0.750	1.000
1.00	0.500	1.000	1.500	2.000

Each suspension was prepared by first adding the required surfactant mass to distilled water in a 500 mL beaker. The solution was mixed for 5 minutes at 300 rpm using an OHAUS Guardian 5000 magnetic stirrer to distribute the surfactant before GNP addition. The required GNP mass was then added, and the suspension was mixed for 30 minutes at 300 rpm using a LabFish overhead stirrer. The suspension was subsequently ultrasonicated at 40 kHz for 1 hour while the temperature was maintained below 35°C.

These preparation conditions were selected based on preliminary trials conducted before the main experimental program. Among the conditions evaluated, the combination of mechanical mixing and ultrasonication provided the most satisfactory macroscopic suspension behavior. The same preparation procedure was therefore applied to all formulations to maintain a consistent basis for comparison. The procedure was not intended to establish an optimized sonication

condition because GNP dispersion can also depend on delivered ultrasonic energy, temperature history, particle concentration, and surfactant chemistry (Muthoosamy and Manickam 2017; Wang et al. 2016). Systematic optimization of these parameters was beyond the scope of this study.

Immediately after preparation, electrical conductivity and pH were measured using a SevenCompact pH/conductivity meter (Mettler-Toledo). These measurements were used as complementary indicators of the chemical and ionic characteristics of the GNP-surfactant suspensions rather than as direct measures of GNP dispersion.

Macroscopic suspension stability was evaluated visually. Preliminary UV-Vis measurements of the undiluted suspensions produced excessive absorbance and light scattering, preventing reliable quantitative comparison under the conditions tested. Dilution could have brought the measurements within the instrument range, but it would also have changed the absolute GNP and surfactant concentrations and could have altered adsorption and colloidal interactions, even though the surfactant-to-GNP mass ratio would remain unchanged. Because a validated dilution and UV-Vis measurement protocol was not established, visual assessment was used as a qualitative indicator of macroscopic suspension stability rather than as a quantitative measure of dispersed GNP concentration.

Following preparation, the suspensions were transferred to sealed vials and stored undisturbed for 12 days. At the end of the storage period, each suspension was examined for visible sedimentation, particle clusters, and phase separation.

2.2.3 Geopolymer Mortar Mixture Design and Preparation

The mortar mixtures were prepared using the surfactant selected from the preliminary dispersion-screening stage. The fixed base formulation, as shown in Table 6, consisted of 300 g fly ash, 600 g sand, 124.5 g sodium silicate solution, 24.5 g dry NaOH, a nominal 25.6 g dispersion entry, and 10.4 g additional water. The sodium silicate solution-to-fly ash and sand-to-fly ash mass ratios were 0.415 and 2.00, respectively. The nominal design targets were a water-to-binder ratio of 0.30 and a combined activator $\text{SiO}_2/\text{Na}_2\text{O}$ molar ratio of 1.20, and these parameters were held constant across all mixtures. Total water content included the mixing water and the water contained in the sodium silicate solution, which contributed 78.435 g of water per batch, whereas the dry NaOH contributed no water. Holding the base formulation constant allowed the effects of GNP dosage and surfactant addition to be evaluated without simultaneously varying the fly ash or sand contents. For each mixture, the required GNP and surfactant masses were first dispersed in a portion of the mixing water using the same dispersion procedure applied during the preliminary screening stage. The resulting GNP-surfactant dispersion was then incorporated into the geopolymer mortar during mixing.

Table 6. Constant batch proportions used for main geopolymer mortar mixtures

Component	Mass per batch (g)
Fly ash	300.0
Sand	600.0
Sodium silicate solution	124.5
Sodium hydroxide flakes	24.5
Graphene-surfactant suspension	25.6
Extra water	10.4

A total of 20 mortar mixtures were prepared, consisting of 16 GNP-surfactant mixtures, designated M1 through M16 in Table 7, and four surfactant-only control mixtures, designated C1 through C4. The two experimental variables were the nominal GNP concentration and the surfactant-to-GNP mass ratio. GNP concentrations of 0.05, 0.10, 0.50, and 1.00 wt.% relative to the water used to prepare the dispersion were evaluated. Surfactant-to-GNP mass ratios of 0.5:1, 1:1, 1.5:1, and 2:1 were also evaluated. Throughout the results and figure legends, GNP concentration (%) refers to the mass of GNP per 100 g of dispersion water and should not be interpreted as the GNP content relative to fly ash. The corresponding GNP masses were approximately 0.0043, 0.0085, 0.0426, and 0.0852 wt.% of the 300 g fly ash, respectively.

The control mixtures C1, C2, C3, and C4 contained 0.006, 0.026, 0.192, and 0.511 g, respectively, of the surfactant selected during the aqueous suspension stability screening phase. These four dosages were selected to span the surfactant-content range represented in the GNP-containing mortar matrix. A separate SDS-only control at every unique surfactant dosage would have required a substantially larger control matrix and was not practical within the available experimental scope. Based on the listed rounded masses, the exact SDS-mass matches are C1-M1, C2-M6/M13, C3-M11, and C4-M16. Only these exact dosage-matched formulations are used for direct GNP-versus-control comparisons. The remaining GNP-containing mixtures do not have exact SDS-dosage-matched controls. Common superscript letters in Table 7 identify the exact matched formulations.

Table 7. Mixture proportions of geopolymer mortar mixes

ID	Fly ash (g)	Sand (g)	Na ₂ SiO ₃ (g)	NaOH (g)	Graphene suspension (g)	GNPs (g)	Surfactant (g)	Extra water (g)
M1 ^A	300	600	124.5	24.5	25.6	0.0128	0.006	10.4
M2	300	600	124.5	24.5	25.6	0.0256	0.013	10.4
M3	300	600	124.5	24.5	25.6	0.1278	0.064	10.4
M4	300	600	124.5	24.5	25.6	0.2556	0.128	10.4
M5	300	600	124.5	24.5	25.6	0.0128	0.013	10.4
M6 ^B	300	600	124.5	24.5	25.6	0.0256	0.026	10.4
M7	300	600	124.5	24.5	25.6	0.1278	0.128	10.4
M8	300	600	124.5	24.5	25.6	0.2556	0.256	10.4
M9	300	600	124.5	24.5	25.6	0.0128	0.019	10.4
M10	300	600	124.5	24.5	25.6	0.0256	0.038	10.4
M11 ^C	300	600	124.5	24.5	25.6	0.1278	0.192	10.4
M12	300	600	124.5	24.5	25.6	0.2556	0.383	10.4
M13 ^B	300	600	124.5	24.5	25.6	0.0128	0.026	10.4
M14	300	600	124.5	24.5	25.6	0.0256	0.051	10.4
M15	300	600	124.5	24.5	25.6	0.1278	0.256	10.4
M16 ^D	300	600	124.5	24.5	25.6	0.2556	0.511	10.4
C1 ^A	300	600	124.5	24.5	25.6	0	0.006	10.4
C2 ^B	300	600	124.5	24.5	25.6	0	0.026	10.4
C3 ^C	300	600	124.5	24.5	25.6	0	0.192	10.4
C4 ^D	300	600	124.5	24.5	25.6	0	0.511	10.4

Note. The water present in the composition of sodium silicate and sodium hydroxide was taken into consideration when calculating the total water content. C1-C4 are representative SDS-only controls selected to span the SDS dosage range in the GNP-containing mixtures. Exact SDS-mass matches are C1-M1, C2-M6/M13, C3-M11, and C4-M16, as identified by common superscript letters. The remaining GNP-containing mixtures do not have exact dosage-matched controls.

To prepare the geopolymer mortar, Class F fly ash was first placed in a 4.7 L mechanical mixer bowl. The alkaline activator was then added, followed by the GNP-surfactant dispersion and additional water. The GNP-surfactant dispersion consisted of the prescribed GNP and surfactant masses added to water and prepared using the same mixing and ultrasonication procedure employed during the aqueous suspension screening phase. The mortar mixing sequence was adapted from ASTM C305 (ASTM International 2020b). The constituents were mixed for 30 seconds at low speed, after which the full mass of sand was added gradually over the next 30 seconds while mixing continued at low speed. The mixer was then operated at medium speed for 30 seconds, followed by a 90-second rest period. During the first 15 seconds of the rest period, mortar adhering to the sides of the bowl was scraped down. Mixing was then resumed for an additional 60 seconds at medium speed.

The fresh mortar was cast into 50 mm × 50 mm × 50 mm cube molds in two layers, consistent with the specimen geometry specified in ASTM C109 (ASTM International 2024). Each layer was consolidated for 10 seconds using a vibrating table. Three replicate cubes were prepared for

each mixture. The same three cubes were used sequentially for UPV, as-cured bulk electrical resistivity, 24-hour water-immersed bulk electrical resistivity, and finally compressive strength. The specimens were initially cured at 49°C for 24 hours and then stored at room temperature for an additional 27 days. UPV and the as-cured resistivity measurement were performed at the end of this 28-day curing period.

2.2.4 Fresh and Hardened Property Testing of Geopolymer Mortar

2.2.4.1 Workability

Workability was evaluated using a flow table in accordance with ASTM C1437 (ASTM International 2020a). Fresh mortar was placed in the flow mold in two layers, with each layer tamped 20 times, and the surface was then leveled with a straightedge. The mold was lifted one minute after mixing ended, and the flow table was dropped 25 times within 15 seconds. The spread diameter was measured using a digital caliper. Flow was reported as the percentage increase in the average final spread diameter relative to the original base diameter of the flow mold. The resulting flow percentage was treated as an empirical measure of mortar workability and was not interpreted as an independent measure of yield stress, plastic viscosity, or thixotropy.

2.2.4.2 Ultrasonic pulse velocity

Ultrasonic pulse velocity (UPV) was used as a nondestructive indicator of internal uniformity and relative changes in the geopolymer mortar matrix (Malhotra and Carino 2004). UPV measurements were performed in accordance with ASTM C597 (ASTM International 2022) on 50 mm × 50 mm × 50 mm geopolymer mortar cubes. Measurements were conducted using a Pundit Ultrasonic Pulse Velocity Tester (Proceq AG, Switzerland) equipped with 54 kHz longitudinal transducers in a direct through-transmission configuration, as shown in Figure 2. Acoustic coupling gel was applied between the transducers and specimen surfaces to improve contact. The instrument had a transit-time resolution of 0.1 μs and automatic gain optimization.

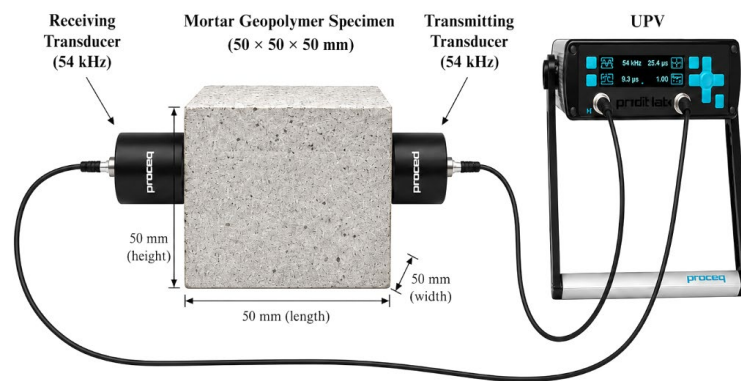


Figure 2. UPV testing setup used in this study.

Before testing, the propagation path length of each cube was measured using a digital caliper. The transducers were positioned on opposite faces of the specimen, and UPV was determined from the measured propagation path length and recorded transit time. UPV was measured first on

the same three replicate cubes that were subsequently used for both resistivity conditions and compressive-strength testing, and the mean UPV was reported for each mixture.

2.2.4.3 Bulk electrical resistivity

Bulk electrical resistivity was measured to evaluate the electrical response of GNP-modified geopolymer mortar. Measurements were performed on 50 mm cube specimens using a uniaxial plate-electrode configuration adapted from ASTM C1876 (ASTM International 2025a). The procedure was treated as an adapted bulk-resistivity method because cube specimens were used rather than the cylindrical specimens specified in the standard. A Resipod resistivity meter (Proceq AG, Switzerland) equipped with the bulk-resistivity attachment was used for testing. The instrument operated at 40 Hz with an applied voltage of approximately 10 V and a nominal probe spacing of 38 mm.

Measurements were performed under two moisture-conditioning states using the same three cubes. The first resistivity measurement was made in the as-cured laboratory condition at the end of the 28-day curing period described in Section 2.2.3. The specimens had been heat-cured for 24 hours and then stored at room temperature for 27 days, with no additional drying treatment before testing. After the as-cured resistivity measurement, the same cubes were immersed in water for 24 hours. At the end of that 24-hour period, corresponding to 28 days of curing plus one day of water immersion, visible surface water was removed immediately before the second resistivity measurement. This condition is referred to as the 24-hour water-immersed, surface-dried condition. Water immersion was expected to reduce resistivity by increasing pore-liquid continuity, although this was not measured directly. The 24-hour immersion period also does not establish complete pore saturation. Accordingly, comparisons between the two conditions represent responses to different moisture-conditioning states rather than a comparison between fully dry and fully saturated specimens.

The test configuration, as shown in Figure 3, consisted of two stainless-steel plate electrodes positioned on opposite specimen faces. Saturated sponge inserts were placed between the electrodes and specimen surfaces to improve electrical contact. Before testing, the sponge inserts were saturated with a calcium hydroxide solution prepared by adding 1 g of calcium hydroxide to 1 L of distilled water.

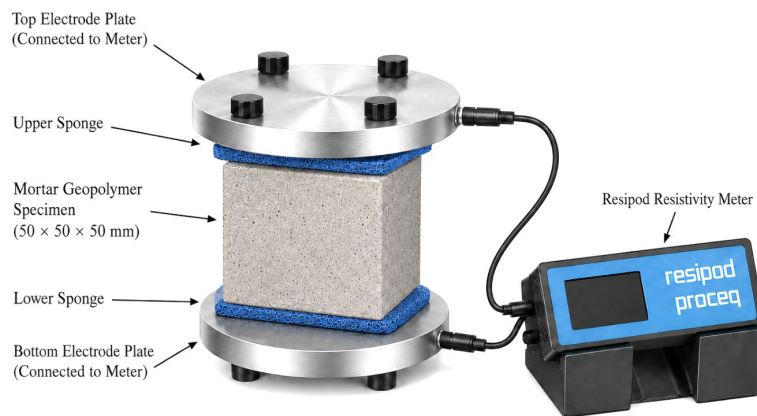


Figure 3. Bulk electrical resistivity testing setup.

Because the sponge inserts also contributed to the measured electrical response, three instrument readings were obtained for each test condition. First, the full assembly was measured with the specimen positioned between the two sponge inserts. The resistivity of the upper and lower sponge inserts were then measured separately. The two sponge readings were subtracted from the full-assembly reading to obtain the corrected specimen response. The corrected value was then converted to specimen bulk resistivity using Eq. 8.

$$\rho_{bulk} = \frac{(R_{specimen+sponges} - R_{bottom\ sponge} - R_{top\ sponge})}{2\pi a} \times \frac{A}{L} \quad (8)$$

Here, ρ_{bulk} denotes the specimen bulk resistivity in $k\Omega \cdot cm$, and $R_{assembly}$ is the measured resistivity of the specimen-sponge assembly ($k\Omega \cdot cm$). $R_{bottom\ sponge}$ and $R_{top\ sponge}$ denote the instrument display resistivity values in $k\Omega \cdot cm$ for the top and bottom sponges, a is probe spacing, which in this case was 3.8 cm, A is the average cross-sectional area (cm^2), and L is the specimen length (cm). Values calculated in $k\Omega \cdot cm$ were converted to $\Omega \cdot m$ by multiplying by 10.

2.2.4.4 Compressive strength

Compressive strength was measured last, after the same cubes had completed UPV testing, the 28-day as-cured resistivity measurement, 24-hour water immersion, and the second resistivity measurement. The strength test was conducted in accordance with ASTM C109 using 50 mm cube specimens (ASTM International 2024). Before testing, loose sand grains were removed from the cube faces that would contact the loading platens. The cubes were then tested using a Test Mark Industries compression testing machine with a capacity of 1,112 kN. Three replicate cubes were tested for each mortar mixture, and the average compressive strength was reported. The reported strength, therefore, corresponds to specimens cured for 28 days and then water-immersed for 24 hours before strength testing, rather than to an as-cured 28-day strength condition.

2.2.5 Statistical Analysis

2.2.5.1 Data preprocessing and standardization

Data processing was performed in R before analysis of variance (ANOVA), response surface modeling, and desirability analysis. Specimen-level observations were retained for compressive strength, UPV, and bulk electrical resistivity. Compressive strength and UPV were analyzed in their original units. For bulk electrical resistivity, measurements obtained under the as-cured condition and expressed in $\Omega \cdot m$ were $\log_{10}$ -transformed before statistical modeling. The transformation was used to reduce scale-dependent variance and improve the distribution and homogeneity of model residuals. Its appropriateness was assessed using residual-versus-fitted, normal Q-Q, and scale-location plots, together with the Shapiro-Wilk test for residual normality and Levene's test for homogeneity of variance. Resistivity is therefore reported as $\log_{10}(\rho_{bulk})$ in the statistical models, while the measured values in $\Omega \cdot m$ are also presented to retain physical interpretability.

2.2.5.2 Analysis of variance

For the 16 GNP-containing mortar mixtures, two-way ANOVA was performed separately for compressive strength, ultrasonic pulse velocity UPV, and log-transformed bulk electrical

resistivity to evaluate the effects of GNP concentration, surfactant-to-GNP mass ratio, and their interaction. These three terms were treated as fixed effects. Surfactant-only controls were excluded because the surfactant-to-GNP ratio is not physically defined in the absence of GNPs. When the interaction term was statistically supported, the corresponding main effects were not interpreted independently. Instead, Tukey-adjusted simple-effect comparisons were used to compare GNP concentrations within each surfactant-to-GNP ratio and surfactant-to-GNP ratios within each GNP concentration. This approach allowed the specific formulation combinations contributing to the interaction to be identified.

2.2.5.3 Response surface regression

Response surface regression was used to describe fitted trends in compressive strength, ultrasonic pulse velocity, and log-transformed bulk electrical resistivity across the tested GNP concentration and surfactant-to-GNP ratio space. Before regression, both factors were centered and scaled so that the linear, interaction, and quadratic terms were estimated on comparable numerical scales. The second-order model is given in Equation 9. The fitted response surfaces were used to evaluate overall trends, curvature, and interaction patterns across the tested formulation space and to complement the categorical comparisons obtained from ANOVA. The models were used as descriptive representations of the experimental response surface and were not intended to establish physical mechanisms or to extrapolate beyond the investigated factor ranges. The model used in this study is given as:

$$Y = \beta_0 + \beta_1 G_c + \beta_2 R_c + \beta_{12} G_c R_c + \beta_{11} G_c^2 + \beta_{22} R_c^2 + \varepsilon \quad (9)$$

where Y is the response variable, G_c is the coded GNP concentration, R_c is the coded surfactant-to-GNP mass ratio, β_0 is the intercept, β_1 and β_2 are the linear coefficients, β_{12} is the interaction coefficient, β_{11} and β_{22} are the quadratic coefficients, and ε is the residual error. The surfactant-only controls were not included in this ratio-based model because the surfactant-to-GNP ratio is not physically defined when GNPs are absent.

2.2.5.4 Principal component analysis

Principal component analysis (PCA) was performed using standardized mixture-level mean values. Mixture ID, GNP concentration, surfactant-to-GNP ratio, and control designation were not included as PCA input variables. They were used only to label and interpret the PCA score plots.

2.2.5.5 Desirability analysis

A desirability-function approach was used to rank the mortar mixtures by combining multiple test responses with different units and optimization directions into a single dimensionless measure. Each response was converted to an individual desirability value, d_i , ranging from 0 to 1, where 0 represents the least favorable response and 1 represents the most favorable response according to the specified criterion. The individual desirability values were then combined to obtain an overall desirability for each mixture, allowing the mixtures to be compared based on their combined performance rather than on any single test result. The approach follows the multi-response desirability framework introduced by Harrington (1965) and subsequently formalized for simultaneous response optimization by Derringer and Suich (1980). Additional details on the desirability-function framework are provided by Costa and Lourenço (2016).

In this study, compressive strength and UPV were treated as criteria to be maximized. Log-transformed bulk electrical resistivity was treated as a criterion to be minimized only as a directional screening preference for identifying formulations with reduced resistivity within the tested set. This screening choice should not be interpreted as an application-level objective of minimizing resistivity without bound. A final snow-melting design would require an application-specific resistivity range established from direct electrical-heating tests together with voltage, electrode spacing, power demand, temperature response, and electrical stability. For a criterion to be maximized, the individual desirability was calculated using the following expression:

$$d_i = \begin{cases} 0, & y_i \leq L_i \\ \left(\frac{y_i - L_i}{T_i - L_i} \right)^{s_i}, & L_i < y_i < T_i \\ 1, & y_i \geq T_i \end{cases} \quad (10)$$

where y_i is the measured test result, L_i is the lower unacceptable limit, T_i is the target value, and s_i is a shape parameter. For a criterion to be minimized, the individual desirability was calculated as:

$$d_i = \begin{cases} 1, & y_i \leq T_i \\ \left(\frac{U_i - y_i}{U_i - T_i} \right)^{t_i}, & T_i < y_i < U_i \\ 0, & y_i \geq U_i \end{cases} \quad (11)$$

where T_i is the desired low value, U_i is the upper unacceptable limit, and t_i is a shape parameter. Equal weighting was used for all criteria. The overall desirability index was then calculated using the geometric mean of the individual desirability values:

$$D = (d_1 d_2 d_3)^{1/3} \quad (12)$$

where d_1 , d_2 , and d_3 represent the individual desirability values for compressive strength, UPV, and log-transformed bulk electrical resistivity, respectively. The geometric mean penalizes mixtures that perform poorly in any one criterion. Therefore, if any individual desirability value is zero, the overall desirability value also becomes zero. The desirability index was used only as a screening metric and was not treated as evidence of electrical-heating or ice-melting performance.

For application-level material selection, the desirability procedure should be implemented with explicit feasibility limits rather than relying only on scaling within the observed experimental range. A candidate formulation should first satisfy a minimum concrete mechanical-performance requirement and fall within an acceptable electrical-resistivity range established from direct heating and power-demand measurements; formulations outside either acceptable range would then receive zero application-level desirability. Numerical application limits were not imposed in

the present analysis because this study used mortar rather than pavement concrete and did not establish a heating-validated resistivity window. Applying pavement-concrete strength limits directly to 50 mm mortar cubes or imposing an unvalidated resistivity threshold would not be technically supported. Accordingly, the present desirability analysis is a comparative screening tool, while future concrete and heating tests are needed to define the condition limits for application-level optimization.

3.1 GNPs Characterization Results

The TEM image in Figure 4 shows a thin, translucent platelet with visible folding, wrinkling, and overlapping regions. Variations in image contrast are consistent with local differences in thickness resulting from platelet overlap or folding. The observed platelet extends across a micrometer-scale field of view, indicating a relatively large lateral dimension. These features confirm the thin, sheet-like morphology and high lateral-to-thickness aspect ratio of the GNPs. Such morphology provides a large potential contact area between platelets but can also promote folding and restacking when the particles come into close contact.



The SEM image in Figure 5 shows plate-like features with irregular edges and micrometer-scale lateral dimensions. Several apparent features exceed 10 μm in width; however, these may represent overlapping or agglomerated platelets rather than individual GNPs. Local stacking and association among platelets are also evident in the dry powder. This platelet association indicates a tendency for the as-received GNPs to form larger assemblies, supporting the use of surfactant-assisted mixing and ultrasonication during dispersion preparation.

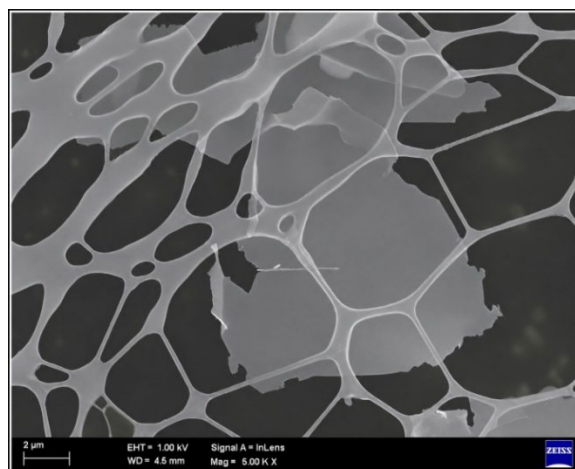


Figure 5. SEM image of graphene nanoplatelets.

The Raman spectrum in Figure 6 shows D, G, and 2D bands at approximately 1,350, 1,580, and 2,720 cm^{-1} , respectively, consistent with predominantly graphitic sp^2 -bonded carbon. The dominant G band is associated with in-plane vibrations of sp^2 -bonded carbon, whereas the much smaller D band indicates a relatively low defect-related response. Because the D band can arise from edge defects, finite platelet dimensions, and basal-plane disorder, it should not be attributed solely to processing-induced damage. The measured I_{2D}/I_G ratio of 0.83 is consistent with few-layer to multilayer graphene nanoplatelets rather than monolayer graphene and agrees with the folded and stacked platelet morphology observed by TEM and SEM. However, Raman spectroscopy alone cannot determine the exact number of graphene layers because the 2D-band shape and intensity can also be influenced by defects, strain, doping, and measurement conditions.

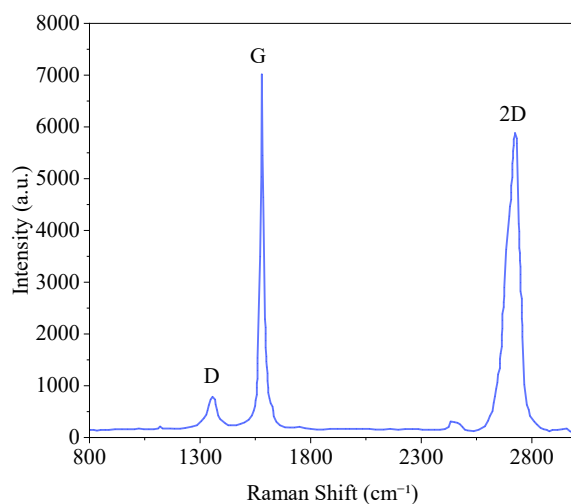


Figure 6. Raman spectrum of graphene nanoplatelets.

The XPS results, including the O 1s spectrum shown in Figure 7, indicate that the GNP surface was predominantly carbonaceous. The survey spectrum yielded a surface composition of 92.84 at.% C and 7.16 at.% O. Deconvolution of the C 1s spectrum assigned 97.75% of the fitted

carbon signal to C=C bonding and 2.25% to C–O bonding, indicating that the carbon signal was dominated by graphitic sp^2 -bonded carbon. Deconvolution of the O 1s spectrum assigned 14.29% of the fitted oxygen signal to C–O species and 85.71% to a component attributed to adsorbed surface H_2O . When the oxygen contribution assigned to surface H_2O is excluded, the normalized non-water surface composition corresponds to approximately 98.9 at.% carbon. These results are consistent with a predominantly graphitic GNP surface containing a relatively small contribution from oxygen-containing surface species. The adjusted carbon value should be interpreted as a fitting-based estimate of the non-water surface composition rather than as an independently measured bulk purity.

Although XPS detected oxygen-containing surface species, the presence of oxygen alone does not classify the material as graphene oxide. The measured survey composition corresponds to an O/C atomic ratio of approximately 0.077. ISO/TS 80004-13:2024 describes graphene oxide as graphene with extensive oxidative modification of the basal plane and notes a typical O/C atomic ratio of approximately 0.5 (ISO 2024). In combination with the predominantly graphitic C=C contribution in the C 1s spectrum and the Raman response, the measured composition is more consistent with commercial graphene nanoplatelets containing a relatively small amount of oxygen-containing surface species than with graphene oxide. The term pristine graphene is not used for the material in this study because the supplied product consists of multilayer nanoplatelets and contains measurable surface oxygen and structural defects.

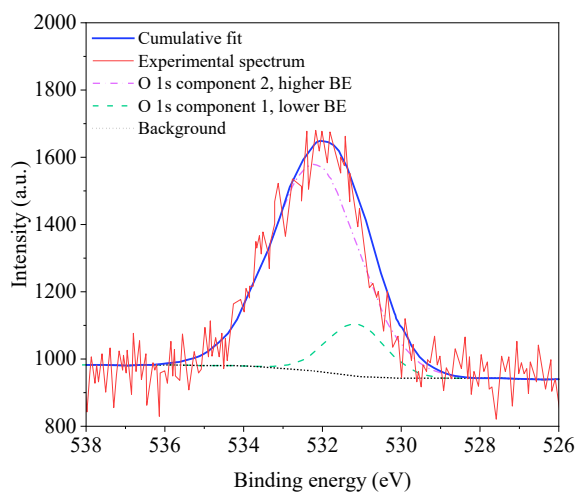


Figure 7. X-ray photoelectron spectroscopy (XPS) O 1s spectrum of graphene nanoplatelets.

The characterization results confirm that the commercial GNPs used in this study exhibited the expected platelet-like morphology and predominantly graphitic structure. These characteristics are directly relevant to the scope of the present study. The high-aspect-ratio platelet morphology and the observed tendency for stacking provide a physical basis for the dispersion screening conducted before mortar preparation; this is because platelet association can promote agglomeration in aqueous suspensions. The predominantly graphitic structure also supports the selection of GNPs as a conductive filler for evaluating changes in bulk electrical resistivity, although conductivity of the GNP powder itself was not measured. Accordingly, the characterization establishes the nature of the starting GNP material and supports the

experimental design, but it does not establish the degree of GNP dispersion, interfacial bonding, or formation of conductive pathways within the hardened geopolymer mortar.

3.2 Evaluation of Aqueous Stability of GNP Dispersions

Task 2 focused on the aqueous stability of GNP-surfactant dispersions before their incorporation into geopolymer mortar. The purpose of this phase was to assess whether the selected dispersion variables produced suspensions that remained sufficiently uniform for subsequent mortar preparation and to identify obvious tendencies toward settling, clustering, or phase separation. Electrical conductivity, pH, and post-storage visual observations were considered complementary screening indicators, with the visual assessment providing qualitative information on macroscopic suspension stability. These measurements were not intended to quantify nanoscale GNP dispersion or predict behavior under the highly alkaline conditions of the geopolymer system.

3.2.1 Visual Screening of Aqueous Suspension Stability

Visual observation was used as a screening step in Task 2 to assess the macroscopic stability of the aqueous GNP-surfactant dispersions before considering conductivity and pH. Figures 8 and 9, Figures 10 and 11, and Figures 12 and 13 show the post-storage appearance of the GNP dispersions prepared with SDS, TX-100, and PVP, respectively. Each formulation represents a single batch preparation; therefore, the images were used for comparative screening rather than for evaluation of batch-to-batch variability.

Figures 8 and 9 show the visual dispersion behavior of GNP aqueous dispersions prepared with SDS after 12 days of storage. The vial images in Figure 8 indicate that SDS generally limited visible sedimentation and large-scale GNP separation across most GNP concentrations and SDS-to-GNP ratios. The suspensions became darker as the GNP concentration increased from 0.05% to 1.0%, which is expected because higher GNP loading increases opacity. Therefore, darker color should not be interpreted by itself as better dispersion. The more relevant visual indicators are the absence of a distinct clear upper layer, visible bottom sediment, and large GNP deposits.

At SDS-to-GNP ratios of 0.5, 1.0, and 1.5, the vial images show relatively uniform suspensions, particularly at 0.1%, 0.5%, and 1.0% GNP. The 0.05% GNP dispersions appear lighter in all panels, which is mainly attributed to the lower GNP content. No obvious sediment layer is visible in these vials. This suggests that SDS provided effective macroscopic stabilization over this range of ratios. However, the response at the 2.0 ratio is less uniform. In Figure 8d, the 0.05% GNP vial shows a lighter upper region and a darker lower region, suggesting partial concentration gradient or settling. The 1.0% GNP vial also appears lighter and more brown-gray than the corresponding 1.0% GNP samples at lower SDS-to-GNP ratios. These observations indicate that increasing the SDS-to-GNP ratio to 2.0 did not consistently improve visual stability.

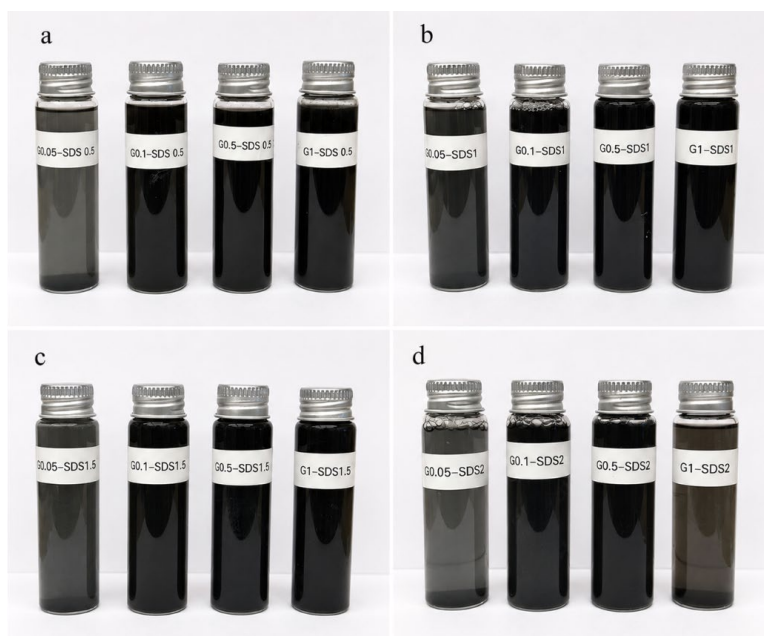


Figure 8. Visual dispersion stability of GNP aqueous dispersions prepared with sodium dodecyl sulfate at different SDS-to-GNP ratios after 12 days: (a) 0.5, (b) 1.0, (c) 1.5, and (d) 2.0. Within each panel, the GNP concentration increases from left to right as 0.05%, 0.1%, 0.5%, and 1.0%, respectively.

The beaker images in Figure 9 support this interpretation. After pouring the dispersions into beakers placed on white paper, the 0.5%, and 1.0% GNP samples generally appear dark and relatively uniform across most SDS-to-GNP ratios. The 0.05% GNP samples remain light because of the lower solids content, but they do not show severe localized deposits in the central field. Some local dark regions are visible in selected samples, such as G0.1-SDS2, but these features are limited compared with the stronger clustering observed in the PVP system. The circular boundary corresponds to the beaker wall and should not be interpreted as sedimentation or particle accumulation.

The combined vial and beaker observations suggest that SDS produced good macroscopic dispersion stability, with the most consistent visual performance occurring at SDS-to-GNP ratios of 0.5 to 1.5. The 2.0 ratio did not show a clear visual advantage and showed signs of nonuniformity in selected vials. This result is consistent with the idea that an intermediate SDS dosage can be sufficient to stabilize GNP particles, while excess SDS may not further improve dispersion. Possible explanations include saturation of available GNP surfaces, free SDS remaining in solution, or micelle formation at higher surfactant concentration. However, the images cannot independently distinguish these mechanisms.

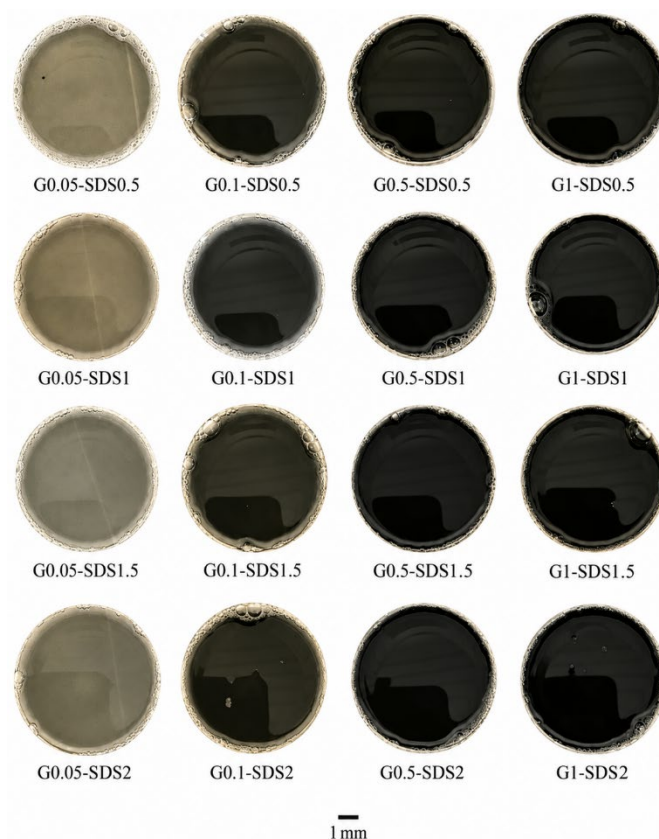


Figure 9. Images of GNP aqueous dispersions prepared with SDS at different SDS-to-GNP ratios and GNP concentrations after pouring into beakers placed on white paper. Rows correspond to SDS-to-GNP ratios of 0.5, 1.0, 1.5, and 2.0, respectively. Columns correspond to GNP concentrations of 0.05%, 0.1%, 0.5%, and 1.0%, respectively.

Figures 10 and 11 indicate that TX-100 provided strong macroscopic stabilization of the GNP aqueous dispersions after 12 days. In the vial images in Figure 10, the suspensions remained visually dark and uniform across all tested TX-100-to-GNP ratios and GNP concentrations, with no clear upper transparent layer, distinct bottom sediment, or obvious large agglomerated deposits. The 0.05% GNP mixtures appear lighter than the higher-concentration mixtures, but this is expected from the lower solids content and should not be interpreted as weaker stability by itself. Overall, the vial images suggest that TX-100 limited visible settling effectively over the full dosage range examined.

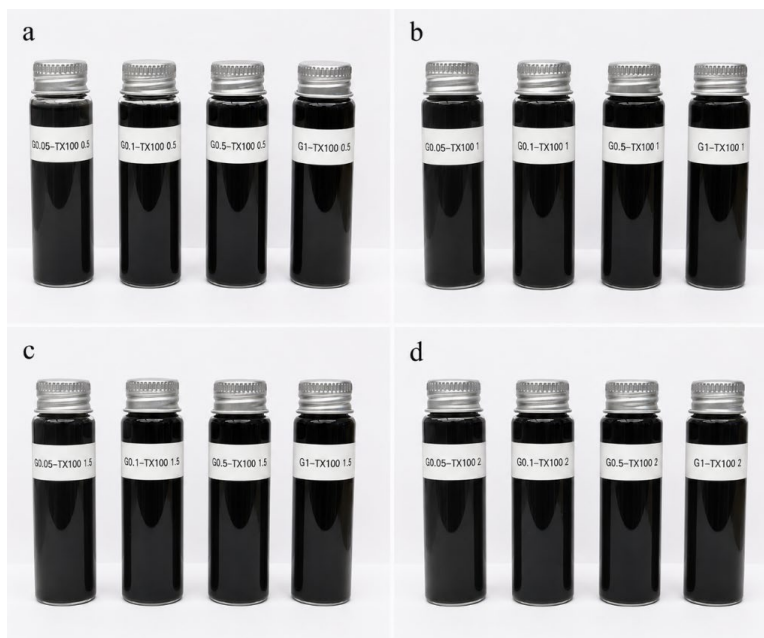


Figure 10. Visual dispersion stability of GNP aqueous dispersions prepared with TX-100 at different TX-100-to-GNP ratios after 12 days: (a) 0.5, (b) 1.0, (c) 1.5, and (d) 2.0. Within each panel, the GNP concentration increases from left to right as 0.05%, 0.1%, 0.5%, and 1.0%, respectively.

The images in Figure 11 support this interpretation. After pouring the dispersions into beakers placed on white paper, most mixtures still showed relatively uniform dark fields, especially at GNP concentrations of 0.5% and 1.0%. The lower-concentration mixtures, particularly 0.05% GNP, appeared lighter because of reduced opacity, but they did not show the pronounced localized clustering observed in the PVP system. Although minor brightness differences are visible among some beaker images, these variations appear to follow GNP concentration more than obvious instability or severe agglomeration. Therefore, the second set of images generally confirms the good visual uniformity indicated by the vial-scale observations.



Figure 11. Images of GNP dispersions prepared with TX-100 at different TX-100-to-GNP ratios and GNP concentrations. Rows correspond to TX-100-to-GNP ratios of 0.5, 1.0, 1.5, and 2.0, respectively. Columns correspond to GNP concentrations of 0.05%, 0.1%, 0.5%, and 1.0%, respectively.

Figures 10 and 11 suggest that the effect of TX-100-to-GNP ratio was limited. Increasing the ratio from 0.5 to 2.0 did not produce a clear monotonic change in appearance in either the vial images or the beaker images. All four ratios yielded dispersions with broadly similar macroscopic stability. This suggests that TX-100 was effective across the tested dosage range and that increasing surfactant content beyond the lower ratios did not produce a clear visual improvement. These figures therefore do not identify a sharply optimal TX-100-to-GNP ratio based on macroscopic appearance alone.

Figures 12 and 13 show that the apparent stability of the PVP-based GNP dispersions depends strongly on the observation method. In the sealed vials, most suspensions appear dark and relatively uniform after 12 days, especially at GNP concentrations of 0.1%, 0.5%, and 1.0%. The 0.05% GNP suspension at a PVP-to-GNP ratio of 0.5 appears lighter than the other mixtures, which is expected because of its lower GNP content. Based only on the vial images, PVP appears to limit gross sedimentation for several mixtures. However, this interpretation is incomplete because the vial images do not reveal the internal distribution of GNPs after the suspension is disturbed or poured.

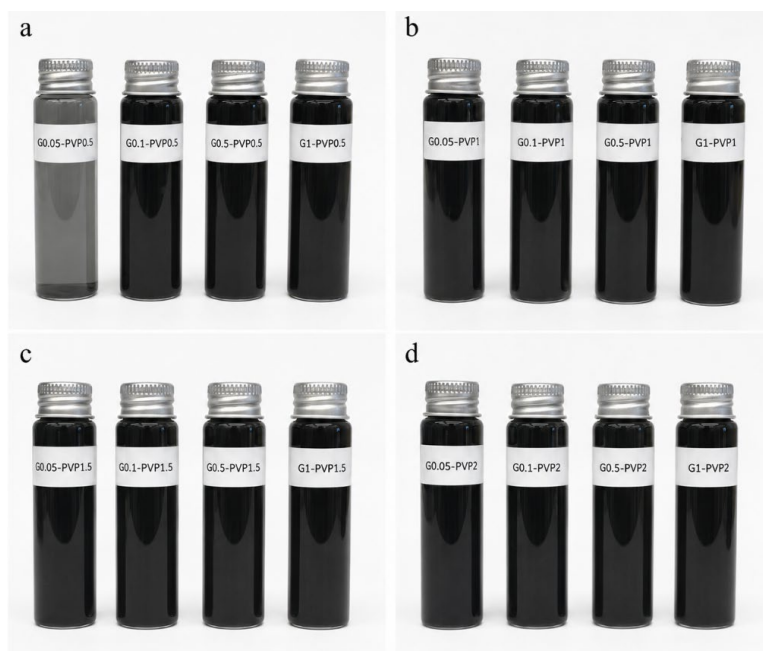


Figure 12. Visual dispersion stability of GNP aqueous dispersions prepared with PVP at different PVP-to-GNP ratios after 12 days: (a) 0.5, (b) 1.0, (c) 1.5, and (d) 2.0. Within each panel, the GNP concentration increases from left to right as 0.05%, 0.1%, 0.5%, and 1.0%, respectively.

The beaker images in Figure 13 show a less uniform dispersion state. Visible dark clusters and nonuniform particle distribution are present in several PVP mixtures. These features are apparent in the 0.05% GNP mixtures across multiple PVP-to-GNP ratios and are also visible in selected higher-concentration mixtures, especially G1-PVP0.5, G1-PVP1, G1-PVP1.5, and G0.5-PVP2. The 2.0 PVP-to-GNP ratio does not remove these clusters. In fact, G0.05-PVP2, G0.1-PVP2, and G0.5-PVP2 show visible localized deposits or uneven dark regions. This indicates that increasing PVP dosage did not produce a monotonic improvement in visual dispersion stability.

The comparison between Figures 12 and 13 is the key result. The vial images suggest acceptable macroscopic stability, but the beaker images reveal residual agglomeration that is not obvious in the sealed-vial view. This means that PVP reduced visible bulk settling in several suspensions but did not consistently suppress localized GNP clustering. Therefore, describing PVP as preventing agglomeration would be too strong. A more defensible interpretation is that PVP provided partial macroscopic stabilization, while visible agglomerates remained under the selected dispersion procedure.

The observed behavior is consistent with the expected role of PVP as a nonionic polymeric dispersant. PVP can stabilize graphitic particles through adsorption and steric hindrance, but its effectiveness depends on polymer concentration, GNP surface area, mixing energy, and the degree of initial particle separation. If platelet stacks are not sufficiently separated during dispersion, PVP may not fully penetrate or stabilize all particle surfaces. Excess polymer may also fail to improve stability if surface coverage is already reached or if local polymer-rich regions promote bridging or nonuniform particle distribution. The present images cannot

distinguish among these mechanisms, but they indicate that PVP stabilization was not sufficient to eliminate visible agglomeration.

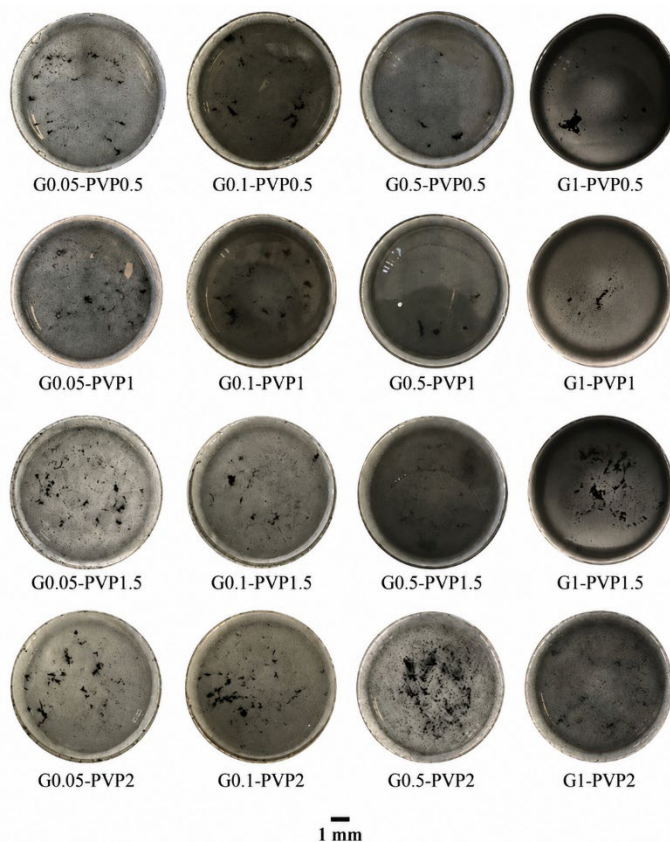


Figure 13. Images of GNP dispersions prepared with PVP at different PVP-to-GNP ratios and GNP concentrations. Rows correspond to PVP-to-GNP ratios of 0.5, 1.0, 1.5, and 2.0, respectively. Columns correspond to GNP concentrations of 0.05%, 0.1%, 0.5%, and 1.0%, respectively.

The visual screening therefore narrowed the candidate dispersing agents from three to two, with SDS and TX-100 showing more favorable macroscopic suspension behavior than PVP. Because visual observation alone does not distinguish the physicochemical differences between the remaining SDS and TX-100 formulations, the subsequent conductivity and pH results were used as complementary screening criteria to support the final surfactant selection.

3.2.2 Electrical Conductivity of Graphene-Surfactant Suspensions

Figure 14 presents the electrical conductivity of the aqueous GNP dispersions prepared with SDS, TX-100, and PVP at different GNP concentrations and surfactant-to-GNP mass ratios. Each formulation was prepared as a single batch; therefore, the reported values represent the measured response of the screened formulations. Following the visual screening, conductivity was used as a complementary physicochemical parameter to further compare the candidate dispersion systems.

The results in Figure 14 show that the largest conductivity differences were associated with dispersant type. SDS produced substantially higher conductivity than TX-100 and PVP across the tested conditions. For SDS, conductivity increased from approximately 115 to 300 $\mu\text{S}/\text{cm}$ at 0.05% GNP, 180 to 485 $\mu\text{S}/\text{cm}$ at 0.10% GNP, and 560 to 1,430 $\mu\text{S}/\text{cm}$ at 0.50% GNP as the surfactant-to-GNP ratio increased from 0.5 to 2.0. At 1.00% GNP, conductivity increased from approximately 860 $\mu\text{S}/\text{cm}$ at a ratio of 0.5 to 1,930 $\mu\text{S}/\text{cm}$ at a ratio of 1.5, followed by a decrease to approximately 1,660 $\mu\text{S}/\text{cm}$ at a ratio of 2.0. This response shows that the electrical characteristics of the SDS formulations generally changed with formulation composition, although the increase was not proportional at the highest GNP concentration.

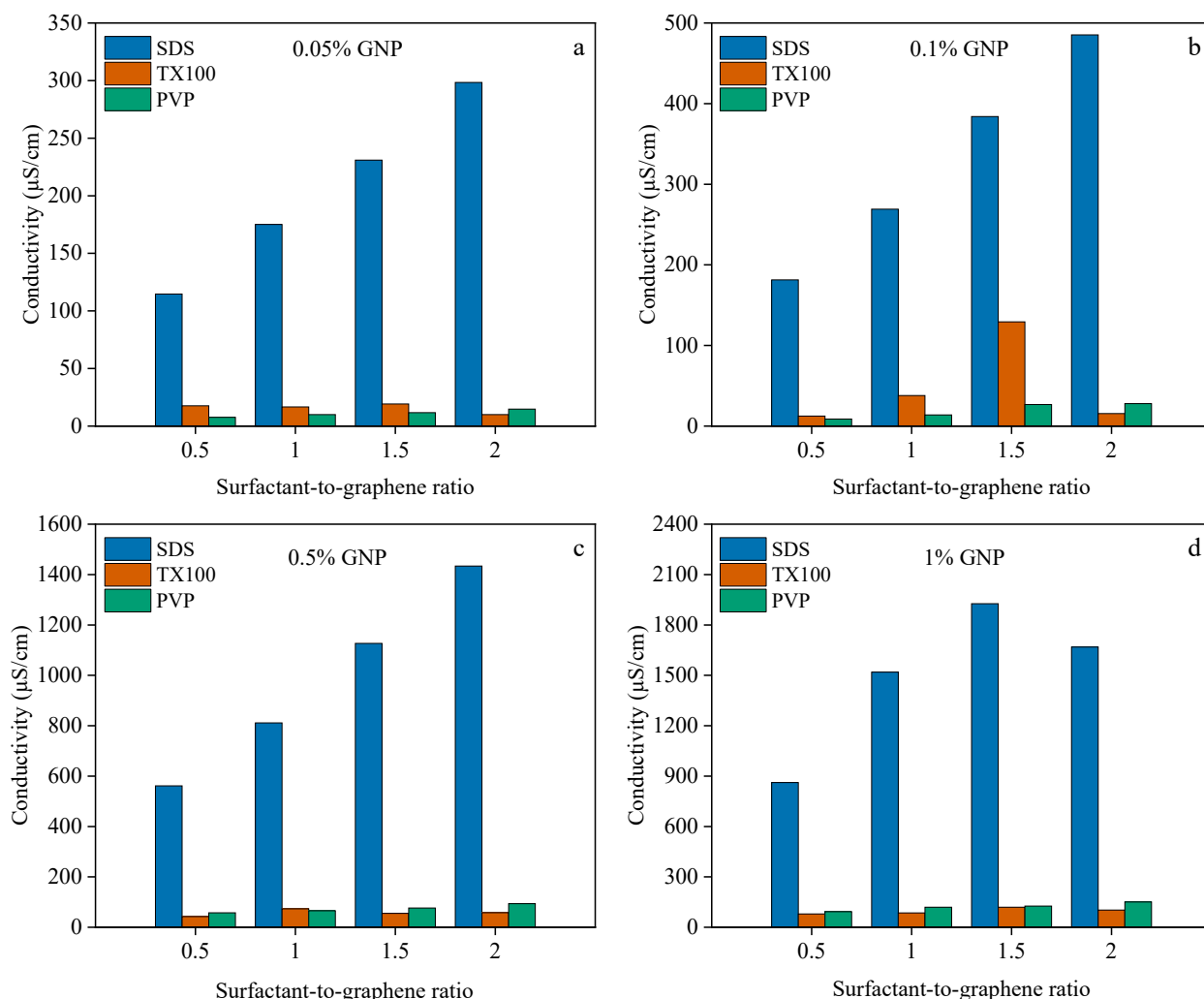


Figure 14. Electrical conductivity of GNP suspensions prepared with SDS, TX-100, and PVP at different GNP concentrations and surfactant-to-GNP mass ratios.

TX-100 and PVP exhibited much lower conductivity, generally ranging from approximately 10 to 130 $\mu\text{S}/\text{cm}$ and 8 to 150 $\mu\text{S}/\text{cm}$, respectively. The higher conductivity of the SDS formulations is consistent with the ionic nature of SDS, whereas TX-100 and PVP are nonionic dispersing agents. Conductivity of aqueous SDS solutions is known to vary with surfactant concentration and can also be influenced by micellization and counterion association near the critical micelle

concentration (Shah et al. 2000). Accordingly, the conductivity response was interpreted as a characteristic of the complete aqueous formulation rather than as a direct measure of GNP dispersion quality.

At a fixed surfactant-to-GNP ratio, increasing GNP concentration also increased the absolute surfactant content. The trends in Figure 14 therefore reflect the combined composition of each formulation. Although conductive GNP networks can influence electrical behavior in solid composites as filler concentration increases (Al-Saleh and Al-Sharman 2022; Fang et al. 2020; Mutlay and Tudoran 2014), the aqueous conductivity measurements in this study were not intended to establish particle-to-particle contact, tunneling, or percolation among suspended GNPs. Within the screening framework, this formulation-level response was useful because the objective was to compare the actual GNP-surfactant systems subsequently considered for mortar preparation rather than to isolate the electrical contribution of each constituent. In combination with the visual observations, the conductivity results provided an additional basis for distinguishing the physicochemical behavior of the candidate dispersion systems.

3.2.3 pH of GNP-Surfactant Suspensions

Figure 15 presents the pH of the same aqueous formulations and provides a complementary measure of their acid-base environment. The results show that SDS dispersions remained mostly near neutral to mildly alkaline, ranging from approximately pH 7.0 to 8.9. TX-100 exhibited a wider range, from approximately pH 4.4 to 9.4, with higher values occurring mainly under selected low-GNP conditions and lower values at higher GNP concentrations. PVP produced the lowest pH values overall, ranging from approximately 4.1 to 7.0. At 0.50% and 1.00% GNP, SDS consistently exhibited higher pH values than TX-100 and PVP.

Within the screening design, pH was used to complement the visual and conductivity results by characterizing the chemical environment associated with each formulation. The narrower near-neutral to mildly alkaline range observed for SDS indicates a more consistent aqueous acid-base environment across the tested formulation space, whereas TX-100 showed greater variation with composition. These differences were interpreted at the formulation level because the measured pH reflects the combined influence of the dispersing agent, GNP content, and solution composition.

The strongly alkaline environment of the geopolymer activator is substantially different from that of the aqueous screening solutions. Accordingly, the pH measurements were used as a comparative screening parameter rather than as a representation of the final geopolymer chemistry. Considering the absence of visible macroscopic agglomeration, the comparatively high conductivity associated with the ionic character of SDS, and the predominantly near-neutral to mildly alkaline aqueous environment, SDS was selected as the dispersing agent for the subsequent geopolymer mortar phase. The selection was therefore based on the combined screening response rather than on any single parameter as a direct measure of GNP dispersion quality.

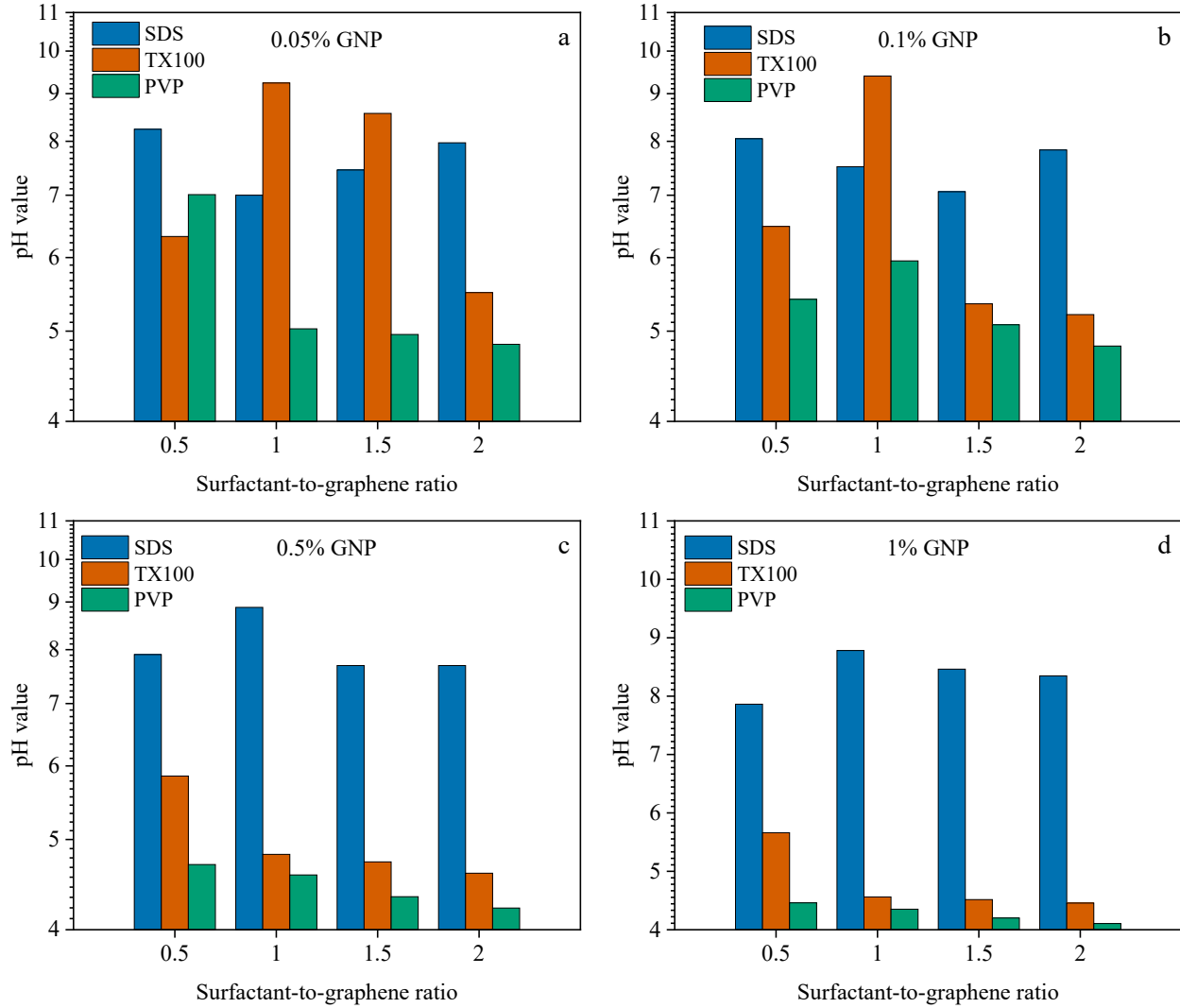


Figure 15. pH of GNP suspensions prepared with SDS, TX-100, and PVP at different GNP concentrations and surfactant-to-GNP mass ratios.

3.3 Performance Evaluation of SDS-GNP Geopolymer Mortar

3.3.1 Workability

Figure 16 presents the flow-table results for the SDS-GNP geopolymer mortar formulations. Workability varied with both GNP concentration and SDS dosage. At an SDS-to-GNP ratio of 0.5, flow remained near 129% for the 0.05% and 0.10% GNP mixtures, then decreased to 123.5% at 0.50% GNP and 104.4% at 1.00% GNP. Similar reductions were observed at the higher ratios. From 0.05% to 1.00% GNP, flow decreased by approximately 19% at a ratio of 0.5, 30% at a ratio of 1.0, and 24% at a ratio of 1.5. At a ratio of 2.0, the highest flow occurred at 0.10% GNP, at approximately 123.1%, followed by a decrease to 94.6% and 78.7% at 0.50% and 1.00% GNP, respectively. These trends show that higher GNP contents generally reduced workability, particularly at 0.50% and 1.00% GNP.

The SDS-only controls also showed a clear change with surfactant dosage. Flow was approximately 117.6% and 117.9% at the two lower SDS levels, then decreased to 99.0% and 73.1% at the two higher levels. This corresponds to an overall reduction of approximately 38% from the lowest to the highest SDS dosage, showing that SDS content itself contributed to the workability response.

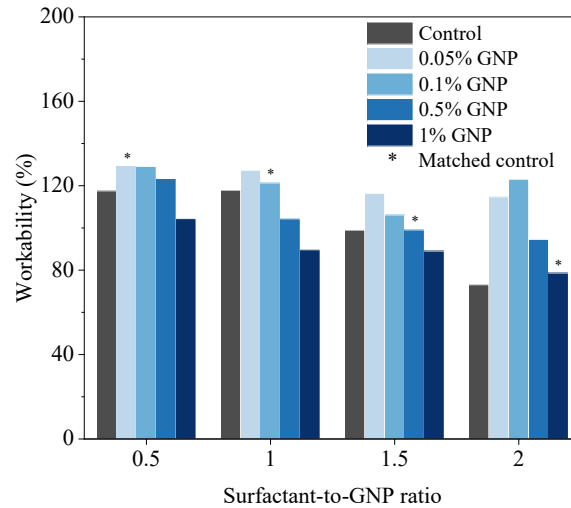


Figure 16. Flow table results of geopolymer mortar mixtures made with different GNP concentrations and SDS-to-GNP mass ratios. Asterisks indicate GNP-containing mixtures with SDS content matched to the corresponding control.

The asterisks in Figure 16 identify GNP-containing mixtures that had the same SDS content as the corresponding SDS-only control. These matched comparisons provide a clearer indication of the change associated with GNP addition. At a ratio of 0.5, the matched 0.05% GNP mixture increased flow from 117.6% to 129.5%, corresponding to an increase of approximately 10.2%. At a ratio of 1.0, the matched 0.10% GNP mixture increased flow from 117.9% to 121.3%, an increase of about 2.9%. At a ratio of 1.5, the matched 0.50% GNP mixture produced essentially the same flow as the control, with values of 99.1% and 99.0%, respectively. At a ratio of 2.0, the matched 1.00% GNP mixture increased flow from 73.1% to 78.7%, an increase of approximately 7.7%. Thus, when SDS content was held constant, GNP addition produced either a modest increase or little change in flow rather than a consistent reduction.

The overall workability response reflects the combined influence of SDS dosage and GNP concentration. The reduction in flow at higher surfactant levels was already evident in the SDS-only controls, while increasing GNP concentration within a given formulation generally reduced flow at the higher GNP contents. At lower GNP contents, SDS-assisted particle separation and improved packing may help maintain flow, as reflected by the similar or slightly higher workability observed in the surfactant-matched comparisons. As GNP content increases, however, the high specific surface area and platelet geometry of the particles can increase liquid demand and strengthen particle-particle interactions, leading to greater resistance to flow. This interpretation is consistent with previous studies showing that the fresh behavior of graphene-modified cementitious materials depends strongly on nanoparticle dosage and dispersion

condition, and that surfactant-carbon nanomaterial systems can modify interparticle interactions rather than simply increase flow (Ge et al. 2021; Mendoza Reales et al. 2018; Zhou et al. 2022).

3.3.2 Ultrasonic Velocity

The 28-day UPV results in Figure 17 complement the compressive-strength results by providing a comparative indicator of internal continuity and stiffness across the SDS-GNP geopolymer mortar formulations. Under the same testing conditions, higher UPV was interpreted as indicating greater internal continuity and stiffness, whereas lower UPV was associated with greater internal heterogeneity or discontinuity. The response was formulation-dependent and broadly consistent with the strength trends. Lower GNP concentrations, particularly 0.05% and 0.10%, generally produced the highest UPV values. The maximum value, approximately 3.0 km/s, occurred at 0.05% GNP and an SDS-to-GNP ratio of 1.5. A comparable value of approximately 2.95 km/s was obtained at 0.10% GNP and a ratio of 0.5. In contrast, the 1.00% GNP formulations ranged from approximately 2.08 to 2.50 km/s and were among the lowest across most SDS-to-GNP ratios.

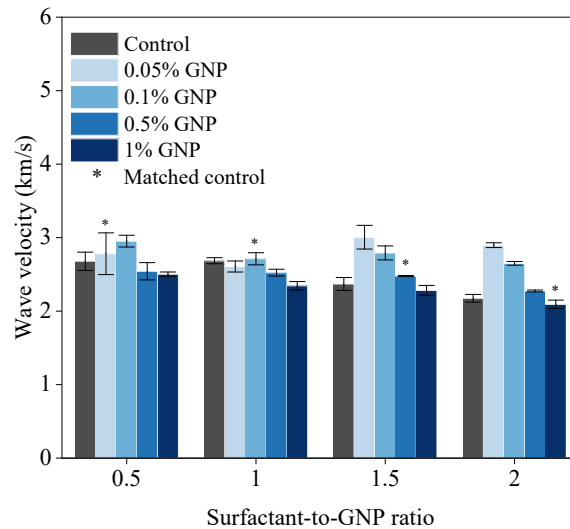


Figure 17. UPV results of 28 d geopolymer mortar specimens made with different GNP concentrations and surfactant-to-GNP mass ratios. Error bars represent 95% confidence intervals for the mean ($n = 3$). Asterisks indicate GNP-containing mixtures with SDS content matched to the corresponding control.

Comparing the SDS-only controls with their matched GNP-containing counterparts shows that GNP addition does not produce a uniform effect on internal continuity. The SDS-only controls isolate the influence of surfactant dosage from the contribution of GNP. For the two lower SDS levels, the UPV remained stable between 2.6 and 2.7 km/s before dropping to approximately 2.35 and 2.2 km/s at higher dosages. Overall, the control UPV decreased by roughly 17% from the lowest to the highest SDS level. Although the response was not strictly monotonic, this downward trend suggests that higher SDS concentrations reduce internal continuity at the upper end of the tested dosage range. At the lowest matched condition, adding 0.05% GNP increased the UPV by about 6% from approximately 2.6 to 2.8 km/s. The 0.10% GNP addition produced essentially no change, keeping both values near 2.7 km/s. The third matched condition of 0.50%

GNP yielded another 6% increase, raising the UPV from 2.35 to 2.50 km/s. Conversely, the highest concentration of 1.00% GNP reduced the UPV by about 5% from 2.2 to 2.1 km/s. These findings confirm that low and intermediate GNP contents provide modest increases relative to their SDS-matched controls, whereas the highest GNP content directly reduces the UPV.

The 95% confidence intervals indicate that specimen-level variability differed among formulations, although most intervals were relatively narrow. Because each mean was based on three replicate cubes, the intervals are sensitive to differences among individual specimens and were considered when interpreting the mean values. The generally higher UPV at lower GNP concentrations is consistent with greater internal continuity, while the lower values at higher GNP contents suggest greater internal heterogeneity. Differences in particle packing, pore structure, entrapped air, interfacial contact, and matrix stiffness may contribute to this behavior. Similar dosage- and dispersion-dependent effects of graphene-family nanomaterials have been reported in cementitious and geopolymeric systems (Ge et al. 2021; Saafi et al. 2015; Zhou et al. 2022). At suitable dosages, improved particle distribution may contribute to a more continuous matrix, whereas higher particle contents can increase platelet association and local heterogeneity. The UPV results therefore provide complementary evidence that the internal condition of the geopolymer mortar depended on the GNP-SDS formulation and that increasing GNP content beyond an appropriate range did not provide further improvement.

3.3.3 Bulk Electrical Resistivity

Electrical resistivity was evaluated under two moisture-conditioning states to determine how the GNP-SDS formulations responded under different levels of pore-liquid availability. Figure 18 presents the as-cured results measured at the end of the 28-day curing period. Figure 19 presents the results from the same cubes after an additional 24 hours of water immersion followed by surface drying, corresponding to 28 days of curing plus one day of immersion. The as-cured specimens generally exhibited resistivity values of approximately 60 to 180 $\Omega\cdot\text{m}$, whereas the 24 hour water-immersed specimens were substantially lower, generally ranging from approximately 5 to 29 $\Omega\cdot\text{m}$. This reduction after water immersion is consistent with greater ionic conduction through the pore solution and shows that moisture condition had a strong influence on the measured electrical response.

The as-cured results in Figure 18 show a nonmonotonic response to GNP concentration. The highest mean resistivity, approximately 180 $\Omega\cdot\text{m}$, occurred at 0.50% GNP and an SDS-to-GNP ratio of 0.5. Other relatively high values occurred at 0.05% and 0.10% GNP across several ratio groups. The surfactant-matched comparisons provide a clearer indication of the response associated with GNP addition. At a ratio of 0.5, adding 0.05% GNP increased resistivity from approximately 66 to 74 $\Omega\cdot\text{m}$. At a ratio of 1.0, the matched 0.10% GNP formulation increased resistivity from approximately 72 to 110 $\Omega\cdot\text{m}$. At a ratio of 1.5, the matched 0.50% GNP formulation increased resistivity from approximately 66 to 91 $\Omega\cdot\text{m}$. At a ratio of 2.0, the matched 1.00% GNP formulation increased resistivity from approximately 59 to 84 $\Omega\cdot\text{m}$. Thus, GNP addition did not reduce as-cured resistivity within the matched formulations. The nonmonotonic response instead suggests that bulk resistivity was governed by competing contributions from pore-liquid conduction, GNP distribution and contact, platelet association, pore structure, and electrode contact rather than by a single continuous GNP pathway.

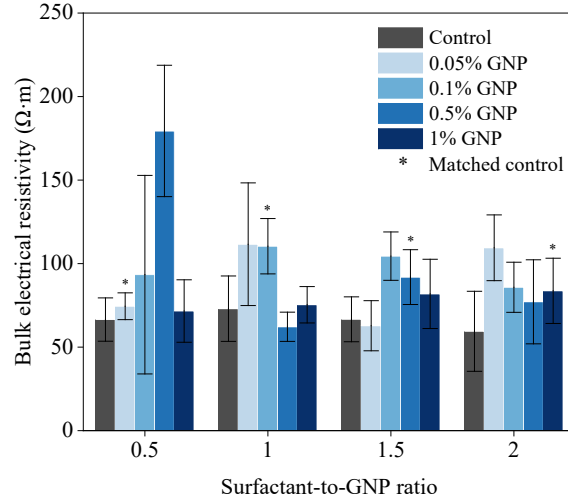


Figure 18. Bulk electrical resistivity results of 28 d as-cured geopolymer mortar specimens made with different GNP concentrations and surfactant-to-GNP mass ratios ($n = 3$). Asterisks indicate GNP-containing mixtures with SDS content matched to the corresponding control.

The 24-hour water-immersed results in Figure 19 show a different absolute resistivity range but a similarly formulation-dependent response. Resistivity of the SDS-only controls increased across the control series from approximately $5 \Omega \cdot \text{m}$ at the lowest SDS level to approximately $26 \Omega \cdot \text{m}$ at the highest. Within the surfactant-matched comparisons, the 0.05% GNP formulation at a ratio of 0.5 increased resistivity from approximately 5 to $11 \Omega \cdot \text{m}$, while the matched 0.10% GNP formulation at a ratio of 1.0 increased it from approximately 9 to $29 \Omega \cdot \text{m}$. At a ratio of 1.5, the matched 0.50% GNP formulation increased resistivity from approximately 18 to $26 \Omega \cdot \text{m}$. At a ratio of 2.0, the matched 1.00% GNP formulation produced a resistivity close to that of its control, at approximately $26 \Omega \cdot \text{m}$. The lower overall values after immersion are consistent with increased pore-liquid continuity, while the variation among formulations indicates that moisture condition and mixture composition acted together to control the measured response. The 24-hour immersion condition was used to provide a consistent moisture-conditioning state and was not intended to establish complete saturation.

The results show that a consistent reduction in resistivity was not observed with increasing GNP concentration under either moisture condition. This is important because a monotonic reduction might be expected if bulk electrical transport were controlled primarily by a continuous GNP network. The present results instead indicate that ionic transport through the pore solution remained an important contributor and that GNP incorporation may simultaneously influence particle connectivity, pore structure, and ionic transport pathways. Under the as-cured condition, residual pore liquid remains part of the measured electrical response, while the immersed condition increases the contribution of pore-liquid conduction. The two conditioning states therefore provide complementary information on how the formulations respond under different moisture conditions rather than isolating purely electronic conduction through the GNPs.

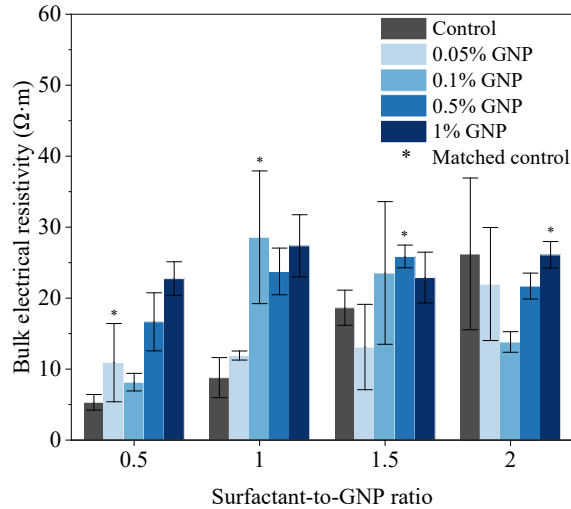


Figure 19. Bulk electrical resistivity results for geopolymer mortar specimens after 28 d curing followed by 24 h water immersion and surface drying. Specimens were made with different GNP concentrations and surfactant-to-GNP mass ratios. Error bars represent 95% confidence intervals for the mean ($n = 3$). Asterisks indicate GNP-containing mixtures with SDS content matched to the corresponding control.

The comparatively wide 95% confidence intervals observed in Figure 19 for selected formulations also indicate greater specimen-level variability in resistivity than in UPV. This behavior is consistent with the sensitivity of electrical measurements to local moisture distribution, pore connectivity, GNP distribution, and conductive-path continuity (Azarsa and Gupta 2017; Mu et al. 2021). Within the scope of this study, the resistivity measurements establish that the GNP-SDS geopolymer mortars exhibit formulation- and moisture-dependent electrical behavior. They do not, however, show that increasing GNP concentration alone consistently improves electrical performance. Rather, the results provide the electrical basis for identifying promising formulations for subsequent application-specific evaluation, such as sensing, Joule heating, or electromagnetic response, where those functions would need to be measured directly.

For the present screening stage, lower resistivity was treated as evidence of increased electrical responsiveness, but resistivity alone cannot identify the optimum mixture for ice-melting. In Joule heating, lower resistance increases current flow and heat generation at a fixed applied voltage, while very low resistance can increase current demand and reduce controllability. Conversely, very high resistance can limit heat generation under practical voltage limits. Therefore, the resistivity results in this study are used to identify mixtures that move toward greater electrical responsiveness, while the final target for a snow-melting system must be an application-specific operating range established through direct heating and power-demand measurements.

Within this framework, the resistivity results suggest that GNP-modified geopolymer has potential as a controlled resistive material for ice-melting applications, but its effectiveness cannot be confirmed from resistivity measurements alone. The mixtures did not show evidence of an excessively conductive, fully percolated network. This is favorable for controlled resistive

heating because a fully connected conductive network could reduce resistance too much, increase current demand, and reduce controllability. Instead, the response was moderate and mixture-specific, indicating that selected GNP-modified mixtures may provide enough electrical transport for heat generation while avoiding unstable current demand. However, bulk electrical resistivity remains an indirect screening metric. It does not measure surface temperature rise, heating rate, heat distribution, power demand, ice-melting time, or performance stability under wet and freezing conditions. Electrical resistivity in cementitious materials is also affected by pore solution conductivity, saturation state, pore connectivity, and transport path tortuosity, so it cannot isolate GNP-related conduction by itself (Sengul 2014; Whittington et al. 1981).

Therefore, a direct ice-melting experiment is needed to determine whether the measured resistivity values translate into functional deicing performance. The original study plan included an ice-melting experiment as the next validation step, but this task was removed after the discontinuation of project funding. As a result, the project ended without the functional test needed to confirm whether the GNP-modified geopolymer could generate sufficient, uniform, and stable heat under realistic wet or ice-covered conditions. Such a test should apply a controlled voltage or current to the specimens while monitoring current draw, surface temperature, heating uniformity, and the time required to melt ice. Until that validation is completed, the results should be interpreted as evidence of potential suitability for controlled resistive heating, not as confirmation of ice-melting effectiveness.

3.3.4 Compressive Strength

Figure 20 presents the compressive strength measured after the same cubes had completed the 28-day curing period, UPV testing, as-cured resistivity testing, 24-hour water immersion, and the second resistivity measurement. Mean strength ranged from approximately 5.5 to 16.8 MPa and varied with both GNP concentration and SDS-to-GNP ratio. The highest mean strength was approximately 16.8 MPa at 0.10% GNP and an SDS-to-GNP ratio of 1.5. A second high value of approximately 15.6 MPa was obtained at 0.05% GNP and a ratio of 2.0. In contrast, the 1.00% GNP formulations generally exhibited lower strength across the tested ratios. The results therefore indicate that increasing GNP concentration did not produce a continuous improvement in compressive strength.

The SDS-only controls show that their strength decreased from approximately 14.6 MPa at the lowest SDS content to 12.8, 9.5, and 7.0 MPa as SDS dosage increased. This represents an overall reduction of about 52%, indicating that increasing SDS content alone was associated with a progressive reduction in compressive strength. The contribution associated with GNP can then be examined by comparing GNP-containing mixtures with controls having the same SDS content and no GNP. At the lowest matched condition, adding 0.05% GNP reduced strength by about 28%, from 14.6 to 10.5 MPa. At the next two matched conditions, 0.10% and 0.50% GNP increased strength by approximately 13%, from 12.8 to 14.4 MPa and from 9.4 to 10.6 MPa, respectively. At the highest matched condition, 1.00% GNP reduced strength by about 21%, from 7.0 to 5.5 MPa. These comparisons show that SDS and GNP affected strength differently. Increasing SDS dosage was associated with a general downward trend in the control mixtures, while the response associated with GNP depended on dosage. Moderate GNP contents produced modest strength gains relative to their SDS-matched controls, whereas the lowest and highest matched GNP contents did not improve strength.

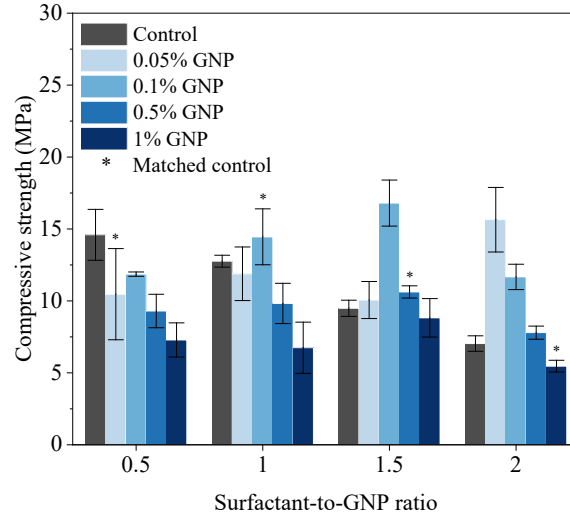


Figure 20. Compressive strength results of geopolymers mortar specimens tested after 28 d curing followed by 24 h water immersion and resistivity testing. Specimens were made with different GNP concentrations and surfactant-to-GNP mass ratios. Error bars represent 95% confidence intervals for the mean ($n = 3$). Asterisks indicate GNP-containing mixtures with SDS content matched to the corresponding control.

The 95% confidence intervals in Figure 20 show that specimen-level variability also differed among formulations. Most mixtures exhibited relatively narrow intervals, while selected low-GNP formulations showed wider intervals. Because each mean was based on three replicate cubes, the confidence intervals are sensitive to moderate differences among individual specimens. Local variation in GNP distribution, particle association, entrapped air, and consolidation may contribute to this variability. The mean strength values were therefore interpreted together with their associated uncertainty.

The overall response is consistent with a balance between beneficial particle effects at moderate GNP contents and increasing particle interaction at higher contents. At suitable dosages, improved particle separation may promote packing, nucleation, and local crack-bridging effects. As GNP content increases, greater platelet association may introduce local discontinuities and reduce effective load transfer. The influence of SDS on fresh-state behavior and subsequent matrix formation also contributes to the hardened response. These trends are consistent with previous studies showing that the mechanical contribution of graphene-family additives depends strongly on dosage and dispersion condition (Ranjbar et al. 2015; Saafi et al. 2015; Zhou et al. 2022), while surfactant-carbon nanomaterial systems can alter fresh behavior in ways that affect hardened properties (Mendoza Reales et al. 2018). The lower strengths observed at higher GNP concentrations also coincided with the lower flow values in Figure 16 and the generally lower UPV values in Figure 17, indicating a consistent formulation-level response across the fresh and hardened measurements.

Published graphene-modified geopolymer systems show a wide range of strength responses. Abiodun et al. (2023) reported a 28-day compressive strength of 63.5 MPa at 0.07 wt.% pristine graphene relative to precursor. Ranjbar et al. (2015) reported a 1.44-fold increase in compressive strength at 1 wt.% GNP, while Sajjad et al. (2021) reported an approximately 10% increase at

1% graphene in a slag/fly ash mortar. These values provide useful context but are not direct benchmarks because graphene type, dosage basis, precursor chemistry, activator composition, curing regime, and reference formulations differ among studies. Within the scope of the present work, the measured strength range of 5.5 to 16.8 MPa establishes the mechanical response of the selected SDS-GNP geopolymer formulations and provides the mechanical context for the subsequent evaluation of their electrical behavior.

The measured mortar strengths should not be interpreted as representative of geopolymer concrete intended for pavement construction. The experimental mixtures contained no coarse aggregate and were tested as 50 mm mortar cubes, whereas pavement concrete acceptance is based on concrete-scale test methods and project-specific requirements. FHWA guidance notes that typical minimum values for concrete pavement are about 27.6 MPa (4,000 psi) in compression, although the specified value depends on the pavement design (Fick et al. 2012). The highest mortar mean in the present study, 16.8 MPa, is below that contextual value, but a direct pass/fail comparison is not valid because the material scale, aggregate system, specimen geometry, and test method differ. The present results therefore establish only the mechanical response of the mortar screening matrix. Future work should introduce coarse aggregate, re-optimize the mixture after scale-up, and verify compressive strength, flexural strength, electrical resistivity, and other pavement-relevant properties using concrete-scale test methods and the applicable agency requirements.

3.4 Statistical Analysis of Geopolymer Mortar Results

3.4.1 Factor Effects and Interaction Structure

Two-way ANOVA was used to evaluate the effects of GNP concentration, SDS-to-GNP ratio (and their interaction) on compressive strength, UPV, and log-transformed bulk electrical resistivity. The surfactant-only controls were excluded because the SDS-to-GNP ratio is not physically defined when GNP is absent. The ANOVA results are summarized in Table 8.

A statistically significant GNP concentration $\times$ SDS-to-GNP ratio interaction was observed for all three responses ($p < 0.001$). Therefore, the effects of GNP concentration and SDS-to-GNP ratio were interpreted in combination rather than as independent effects. Tukey-adjusted simple-effect comparisons were subsequently used to identify differences among GNP concentrations within each SDS-to-GNP ratio.

For compressive strength, GNP concentration had the largest partial effect size among the fitted model terms, $F(3, 32) = 55.60$, $p < 0.001$, $\eta_p^2 = 0.839$. The interaction was also strongly supported, $F(9, 32) = 7.18$, $p < 0.001$, $\eta_p^2 = 0.669$, while SDS-to-GNP ratio had a smaller partial effect size, $F(3, 32) = 4.21$, $p = 0.013$, $\eta_p^2 = 0.283$. Tukey comparisons showed that at a ratio of 0.5, the 1.00% GNP mixture had lower strength than the 0.05% and 0.10% mixtures. At a ratio of 1.0, the 0.10% mixture had higher strength than the 0.50% and 1.00% mixtures, while the 1.00% mixture was also lower than the 0.05% and 0.50% mixtures. At a ratio of 1.5, the 0.10% mixture had higher strength than the 0.05%, 0.50%, and 1.00% mixtures ($p < 0.001$ for all three comparisons). At a ratio of 2.0, strength decreased from the 0.05% mixture to the higher GNP levels, with the 0.05% mixture exceeding the 0.10%, 0.50%, and 1.00% mixtures.

These results confirm that the influence of GNP concentration depended strongly on the SDS-to-GNP ratio.

For UPV, GNP concentration again had the largest partial effect size, $F(3, 32) = 94.228, p < 0.001, \eta_p^2 = 0.898$. SDS-to-GNP ratio, $F(3, 32) = 13.77, p < 0.001, \eta_p^2 = 0.563$, and the interaction, $F(9, 32) = 6.64, p < 0.001, \eta_p^2 = 0.651$, were also statistically significant. Tukey comparisons generally showed higher UPV at the lower GNP concentrations. At a ratio of 0.5, both 0.05% and 0.10% GNP produced higher UPV than 0.50% and 1.00% GNP. At a ratio of 1.0, both 0.05% and 0.10% GNP produced higher UPV than 1.00% GNP. At a ratio of 1.5, the 0.05% mixture exceeded the 0.10%, 0.50%, and 1.00% mixtures, while the 0.10% mixture also exceeded the 0.50% and 1.00% mixtures. A similar pattern occurred at a ratio of 2.0, where 0.05% GNP exceeded all higher concentrations and 0.10% GNP exceeded both 0.50% and 1.00% GNP. These results support the observed tendency toward higher UPV at lower GNP contents, although UPV alone cannot distinguish among effects associated with GNP distribution, entrapped air, and matrix heterogeneity.

For log-transformed bulk electrical resistivity, GNP concentration, $F(3, 32) = 18.004, p < 0.001, \eta_p^2 = 0.628$, SDS-to-GNP ratio, $F(3, 32) = 11.85, p < 0.001, \eta_p^2 = 0.526$, and their interaction, $F(9, 32) = 5.09, p < 0.001, \eta_p^2 = 0.589$, were all statistically significant. The Tukey results showed a less systematic pattern than for strength and UPV. At a ratio of 0.5, the 0.10% mixture had lower log-transformed resistivity than the 0.50% and 1.00% mixtures, while the 0.05% mixture was also lower than the 1.00% mixture. At a ratio of 1.0, the 0.05% mixture had lower resistivity than the 0.10%, 0.50%, and 1.00% mixtures. The same pattern occurred at a ratio of 1.5, where the 0.05% mixture was lower than all three higher GNP concentrations. At a ratio of 2.0, only the difference between 0.10% and 1.00% GNP was statistically significant. The absence of a consistent concentration-dependent decrease across the four ratios agrees with the resistivity trends discussed previously and indicates that the electrical response depended on the specific GNP-SDS formulation rather than on GNP concentration alone.

The statistical results confirm that the performance of the geopolymer mortar was governed by the specific combination of GNP concentration and SDS-to-GNP ratio rather than by either factor alone. Moderate GNP contents generally provided the most favorable mechanical and ultrasonic responses, whereas the electrical response remained formulation-dependent and did not show a consistent concentration-driven improvement.

Table 8. Two-way ANOVA results for the effects of GNP concentration, surfactant-to-GNP ratio, and their interaction on compressive strength, ultrasonic pulse velocity, and log-transformed bulk electrical resistivity of geopolymer mortar

Test result	Term*	F-statistic	p-value	Partial η^2
Compressive strength	A	F(3, 32) = 55.60	<0.001	0.839
Compressive strength	B	F(3, 32) = 4.21	0.013	0.283
Compressive strength	A $\times$ B	F(9, 32) = 7.18	<0.001	0.669
UPV	A	F(3, 32) = 94.23	<0.001	0.898
UPV	B	F(3, 32) = 13.77	<0.001	0.563
UPV	A $\times$ B	F(9, 32) = 6.64	<0.001	0.651
$\log_{10}(\rho)$	A	F(3, 32) = 18.00	<0.001	0.628
$\log_{10}(\rho)$	B	F(3, 32) = 11.85	<0.001	0.526
$\log_{10}(\rho)$	A $\times$ B	F(9, 32) = 5.09	<0.001	0.589

*A represents GNP concentration, B represents surfactant-to-GNP ratio, and A $\times$ B represents their interaction. Partial η^2 is reported as the effect-size measure for each model term.

3.4.2 Response Surface Regression Model

Response surface regression was used to summarize the overall variation in compressive strength, UPV, and log-transformed bulk resistivities across the tested GNP concentration and SDS-to-GNP ratio ranges. The fitted models were intended to describe broad direction, curvature, and combined factor behavior rather than reproduce every difference among the 16 individual formulations. The estimated coefficients are reported in Table 9.

Table 9. Response surface regression coefficients for strength, UPV, and log-transformed resistivity

Response	β_0	β_1	β_2	β_{11}	β_{22}	β_{12}
Strength	10.951***	-2.624***	0.237	0.361	-0.776†	-0.587†
UPV	2.514***	-0.259***	-0.063**	0.082**	-0.004	-0.063**
$\log_{10}(\rho)$	1.380***	0.125***	0.059**	-0.05	-0.067*	-0.033†

Note. Values are regression coefficients, with coefficient standard errors shown in parentheses. The fitted second-order model was $\hat{y} = \beta_0 + \beta_1 G_c + \beta_2 R_c + \beta_{12} G_c R_c + \beta_{11} G_c^2 + \beta_{22} R_c^2 + \varepsilon$. G_c is the coded GNP concentration, R_c is the coded surfactant-to-GNP mass ratio, β_0 is the intercept, β_1 and β_2 are the linear coefficients, β_{12} is the interaction coefficient, β_{11} and β_{22} are the quadratic coefficients, and ε is the residual error. ***p < 0.001, **p < 0.01, *p < 0.05, †0.05 ≤ p < 0.10.

The overall model fit statistics are summarized in Table 10. All three response surface models were statistically significant, with model p -values less than 0.001. However, the explained variance differed among responses. UPV had the strongest model fit, with $R^2 = 0.791$ and adjusted $R^2 = 0.766$. Strength showed moderate explanatory capacity, with $R^2 = 0.622$ and adjusted $R^2 = 0.577$. Log-transformed resistivity had the weakest fit, with $R^2 = 0.528$ and adjusted $R^2 = 0.472$. The RMSE values indicate the typical residual error in the units of each modeled response. Strength had an RMSE of 1.996, UPV had an RMSE of 0.120, and $\log_{10}(\rho)$ had an RMSE of 0.125. AIC and BIC are reported for completeness, but they should not be used

to compare strength, UPV, and resistivity directly because the responses have different scales and error structures. AIC and BIC are only meaningful for comparing alternative model forms fitted to the same response.

Table 10. Model fit statistics for the response surface regression models

Response	R ²	Adjusted R ²	RMSE	Model p-value	AIC	BIC
Compressive strength	0.622	0.577	1.996	<0.001	216.55	229.65
UPV	0.791	0.766	0.12	<0.001	-53.13	-40.03
log ₁₀ (ρ)	0.528	0.472	0.125	<0.001	-49.7	-36.6

For compressive strength, the linear coded-GNP coefficient was negative ($b_1 = -2.624$, $p < 0.001$), indicating an overall decrease in fitted strength as GNP concentration increased. The linear SDS-to-GNP ratio term was not statistically significant. The ratio-squared and GNP concentration $\times$ SDS-to-GNP ratio terms had $0.05 \leq p < 0.10$ and therefore were not statistically significant at $\alpha = 0.05$. The model produced an R^2 of 0.622 and an RMSE of 1.996 MPa, indicating that the quadratic surface captured the general response but left substantial formulation-level variation unexplained.

As shown in Figure 21, the fitted surface places the highest compressive strengths in the lower-GNP region and shows a general decrease in strength toward 1.00% GNP. The surface also indicates some dependence on SDS-to-GNP ratio, particularly at the lower GNP concentrations, although this effect is less pronounced than the overall GNP concentration trend. This agrees with the experimental results, where selected low and intermediate GNP formulations produced higher strengths, while the 1.00% GNP mixtures generally exhibited lower values.

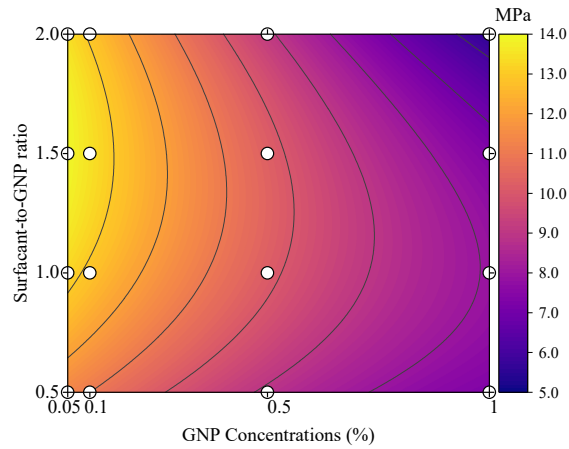


Figure 21. Response surface heat map for fitted compressive strength as a function of GNP concentration and surfactant-to-GNP ratio.

The response-surface model should be interpreted together with the factorial ANOVA. The categorical ANOVA identified a statistically significant GNP concentration $\times$ SDS-to-GNP ratio interaction, whereas the single bilinear interaction term in the quadratic regression was not significant at $\alpha = 0.05$. These findings are not contradictory because the two models represent interaction differently. The factorial ANOVA allows the effect of GNP concentration to vary

separately at each tested SDS-to-GNP ratio, while the quadratic model approximates the 16 formulations using a smooth continuous surface. The response surface therefore provides a useful visualization of the overall trend, while the ANOVA and Tukey comparisons provide stronger evidence of the formulation-specific differences.

For UPV, the response surface model showed a stronger fit than the compressive-strength model. The linear coefficients for GNP concentration and SDS-to-GNP ratio were both negative and statistically significant, with $b_1 = -0.259$ ($p < 0.001$) and $b_2 = -0.063$ ($p < 0.01$), respectively. The interaction term was also negative and statistically significant, with $b_{12} = -0.063$ ($p < 0.01$), indicating that the effect of GNP concentration depended on the SDS-to-GNP ratio. The positive quadratic GNP term, $b_{11} = 0.082$ ($p < 0.01$), indicates curvature along the GNP concentration axis. Overall, the fitted response was characterized by lower UPV toward higher GNP concentrations, particularly when combined with higher SDS-to-GNP ratios. The model had an R^2 of 0.791 and an RMSE of 0.120 km/s, providing the strongest fit among the three response-surface models.

The UPV response surface in Figure 22 places the highest fitted values in the low-GNP region, particularly at intermediate SDS-to-GNP ratios, and lower values toward the high-GNP and high-ratio region. This broad trend agrees with the factorial ANOVA and Tukey comparisons, which showed that lower GNP concentrations generally produced higher UPV than the 0.50% and 1.00% formulations. The response is consistent with differences in internal continuity among the mixtures. Higher GNP contents may promote platelet association, entrapped air, or other matrix discontinuities that reduce ultrasonic wave transmission. However, UPV alone cannot distinguish among effects associated with GNP distribution, air content, pore structure, matrix stiffness, moisture condition, or transducer contact.

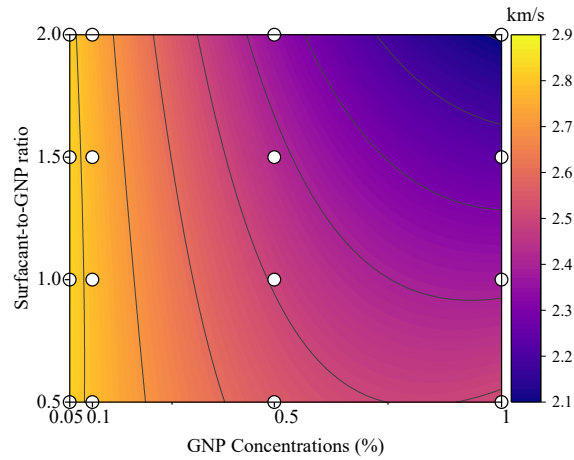


Figure 22. Response surface heat map for fitted UPV as a function of GNP concentration and surfactant-to-GNP ratio.

For log-transformed resistivity, the linear coefficients for GNP concentration and SDS-to-GNP ratio were positive and statistically significant, with $b_1 = 0.125$ ($p < 0.001$) and $b_2 = 0.059$ ($p < 0.01$), respectively. However, these coefficients represent the fitted slopes at the coded origin and should not be interpreted as evidence of monotonic increases across the full

experimental range. The negative SDS-to-GNP quadratic term was statistically significant ($b_{22} = -0.067$, $p < 0.05$), while the GNP quadratic term was not statistically significant. The interaction term showed weak statistical support ($b_{12} = -0.033$, $0.05 \leq p < 0.10$). Examination of the fitted surface showed that log-resistivity generally increased with GNP concentration over much of the tested range, but the response flattened and, at higher SDS-to-GNP ratios, decreased near the upper GNP range. The model had an R^2 of 0.528 and an RMSE of 0.125, representing the lowest explanatory capacity among the three response-surface models.

Figure 23 shows the fitted log-resistivity response within the tested design space. The broad increase toward higher GNP concentrations is therefore not uniform across all SDS-to-GNP ratios. This behavior agrees with the factorial ANOVA and Tukey comparisons, which showed that resistivity depended on the specific combination of GNP concentration and SDS-to-GNP ratio rather than on GNP concentration alone. The fitted response does not support a simple assumption that increasing GNP necessarily decreases bulk resistivity. It also cannot distinguish changes in GNP particle connectivity from changes in pore-liquid conduction or pore structure and does not establish the presence or absence of an electrically percolated GNP network.

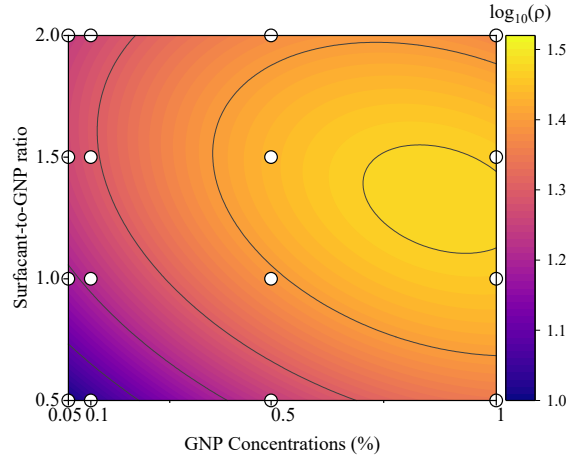


Figure 23. Response surface heat map for fitted log-transformed bulk electrical resistivity as a function of GNP concentration and surfactant-to-GNP ratio.

3.4.3 Principal Component Analysis of Mixture-Level Test Results

Principal component analysis was used to examine how the mixtures were distributed when compressive strength, UPV, and log-transformed bulk electrical resistivity were considered together. The analysis was performed using mixture-level mean values rather than individual replicate values. This avoids treating replicate specimens as independent formulation-level observations. The variables were centered and scaled before PCA because the three test results have different units and numerical ranges.

Since three measured test results were included in the PCA, the analysis produced three principal components. Each component is an orthogonal linear combination of compressive strength, UPV, and log-transformed bulk electrical resistivity. The components are ordered by the amount of variance they explain. Therefore, PC1 captures the largest share of variation in the combined

test-result profile, PC2 captures the largest remaining variation after PC1, and PC3 captures the remaining variation after PC1 and PC2.

Table 11 presents the PCA loadings for the three measured test results. The loadings indicate how strongly each test result contributes to each principal component and whether it contributes in the positive or negative direction. Larger absolute loading values indicate a stronger contribution to that component. Therefore, the loading table is needed before interpreting the PCA biplot because PC1 and PC2 do not directly represent single measured properties.

PC1 explained 70.6% of the total variance. Compressive strength and UPV had positive loadings on PC1, while log-transformed bulk electrical resistivity had a negative loading. This means that mixtures with higher PC1 scores generally had higher strength, higher UPV, and lower resistivity. Therefore, PC1 can be interpreted as a favorable combined test-result direction for the intended application. This interpretation is only valid because the loading signs align with the desired performance pattern.

PC2 explained 21.7% of the total variance. The largest loading on PC2 was associated with log-transformed bulk electrical resistivity, while compressive strength also contributed and UPV contributed less. Therefore, PC2 mainly separated mixtures according to electrical behavior, with a secondary contribution from strength. A higher PC2 score should not be interpreted as automatically favorable because it is associated with higher log-transformed resistivity. PC3 explained only 7.7% of the total variance and was not used as a primary interpretation axis.

Table 11. Principal component analysis loadings for mixture-level strength, ultrasonic pulse velocity, and log10 bulk electrical resistivity

Variable	PC1 loading	PC2 loading	PC3 loading
Compressive strength	0.588	0.528	0.613
UPV	0.633	0.172	-0.755
log10 bulk electrical resistivity	-0.504	0.831	-0.234

Figure 24 shows the PCA biplot based on PC1 and PC2, which together explained 92.3% of the total variance. The plot provides a visual summary of how the mixtures relate to one another based on the combined test-result profile. Mixtures located toward the positive PC1 direction are more consistent with the desired balance of higher strength, higher UPV, and lower resistivity. Mixtures located toward the negative PC1 direction show a less favorable combined profile. The distribution of points also indicates that increasing GNP concentration did not produce a uniform shift toward the favorable PC1 direction.

The PCA results are consistent with the surfactant-matched comparisons, ANOVA interaction results, and response surface models. The mixture behavior cannot be interpreted using GNP concentration alone. Instead, the combined test-result profile depended on the specific GNP concentration and surfactant-to-GNP ratio combination.

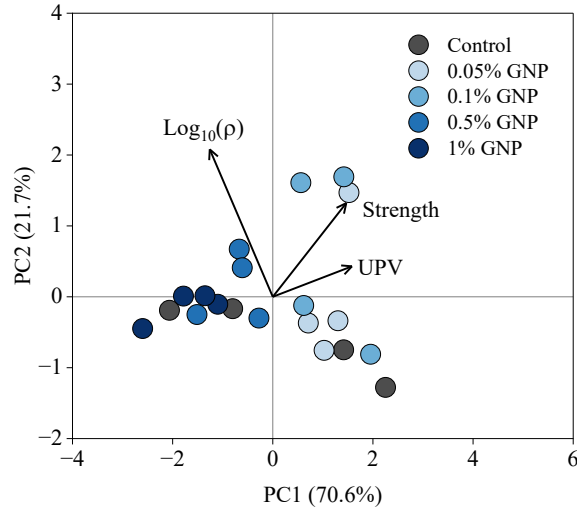


Figure 24. PCA biplot of mixture-level test results. Points represent mixtures plotted using PC1 and PC2 scores. Arrows represent variable loadings for compressive strength, UPV, and log-transformed bulk electrical resistivity. Colors indicate GNP concentration.

3.4.4 Mixture Screening Using Desirability Analysis

Figure 25 presents the desirability ranking of the GNP-containing mixtures based on compressive strength, UPV, and log-transformed bulk electrical resistivity. Compressive strength and UPV were assigned maximize objectives. Log-transformed resistivity was assigned a minimize direction only for comparative screening within the tested formulations, because the present stage sought mixtures that reduced resistivity while retaining mechanical performance. Each response was linearly scaled between the minimum and maximum values observed across the complete set of tested formulations, including the surfactant-only controls. The three individual desirability values were then combined using an equally weighted geometric mean. This ranking does not define the optimum resistivity for a heating system; an application-specific target range requires direct electrical-heating and power-demand testing.

Among the GNP-containing mixtures shown in Figure 25, M2 had the highest overall desirability, approximately 0.73. M2 contained 0.10% GNP relative to dispersion water and an SDS-to-GNP ratio of 0.5. The next highest-ranked mixtures were M1 and M9, both containing 0.05% GNP, followed by M5, M14, and M13. The ranking therefore generally favored the lower GNP concentrations, while no single SDS-to-GNP ratio was consistently preferred across the highest-ranked mixtures.

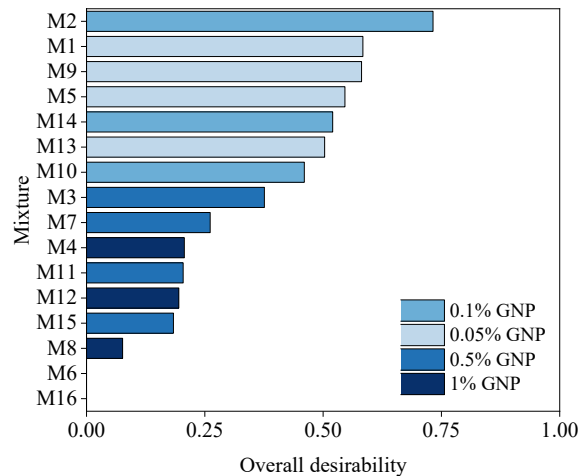


Figure 25. Desirability ranking of GNP-containing geopolymer mortar mixtures.

The desirability values should be interpreted as a comparative screening metric rather than as evidence of a unique optimum. The ranking depends on the selected responses, the specified maximize or minimize directions, the observed scaling limits, and the equal weighting assigned to the three criteria. A different application objective or weighting scheme could therefore produce a different ranking. In addition, the ranking compares the measured performance of complete formulations and does not isolate the individual contributions of GNP and SDS.

The zero overall desirability values for M6 and M16 do not indicate zero measured performance. M6 reached the least favorable end of the selected resistivity scale, while M16 reached the least favorable end of the strength and UPV scales. Because the overall desirability was calculated as the geometric mean of the three individual desirabilities, any individual desirability of zero produces an overall value of zero. This approach prevents a strong result in one response from fully compensating for a poor result in another.

The desirability ranking should be treated as a screening result, not as proof of a mechanistic or application-level optimum. It depends on the selected responses, target directions, scaling limits, and equal weighting. In particular, the minimize direction assigned to resistivity was used only to identify formulations with lower resistivity within the tested set and does not imply that the lowest achievable resistivity is the desired snow-melting design target. A different application target or weighting scheme could change the ranking. Therefore, the ranking is most defensible as a decision-support tool for selecting candidate formulations for follow-up electrical-heating tests. In future application-level use, formulations that do not satisfy validated minimum mechanical requirements or the heating-validated resistivity operating range should receive zero desirability before the remaining candidates are ranked.

Within the tested factor range and selected desirability criteria, the screening results favor selected mixtures containing 0.05–0.10% GNP relative to dispersion water, corresponding to approximately 0.0042–0.0085 wt.% relative to fly ash. M2 was the highest-ranked GNP-containing formulation under the selected criteria. However, this ranking should not be interpreted as a universal material optimum because it is conditional on the selected performance objectives and does not include uncertainty in the desirability calculation or independent batch validation.

4. CONCLUSIONS AND RECOMMENDATIONS

This study evaluated graphene nanoplatelet-modified fly ash-based geopolymer mortar as an early-stage candidate material for electrically functional construction applications. The completed work supports several conclusions:

- First, suspension behavior was strongly affected by surfactant type, graphene nanoplatelet concentration, and surfactant-to-graphene nanoplatelet mass ratio. The water-based suspension screening was useful for selecting a surfactant system before mortar production, but it cannot independently predict hardened mortar performance. Suspension conductivity and visual stability should therefore be treated as preliminary indicators, not as substitutes for mortar-scale testing.
- Second, sodium dodecyl sulfate was selected for the mortar phase based on the combined suspension screening results. This selection was reasonable for the completed experimental program, but it should not be interpreted as proof that sodium dodecyl sulfate is universally optimal for geopolymer systems. Surfactant performance can change in highly alkaline activators and in the presence of fly ash, sand, dissolved silicate species, and curing effects.
- Third, graphene nanoplatelet-modified geopolymer mortar did not show a simple monotonic improvement with increasing graphene nanoplatelet concentration. Higher graphene nanoplatelet contents did not consistently improve bulk electrical resistivity, ultrasonic pulse velocity, or compressive strength. This indicates that conductive filler dosage alone is not an adequate design variable.
- Fourth, the surfactant-matched comparisons showed that selected low-graphene nanoplatelet mixtures performed more favorably relative to their corresponding surfactant-only controls than the highest graphene nanoplatelet mixtures. This suggests that the beneficial range of graphene nanoplatelet addition is limited under the tested dispersion and mortar conditions.
- Fifth, the analysis of variance showed that the interaction between graphene nanoplatelet concentration and surfactant-to-graphene nanoplatelet ratio was statistically supported for compressive strength, ultrasonic pulse velocity, and log-transformed bulk electrical resistivity. This means that the effect of graphene nanoplatelet concentration depended on the surfactant-to-graphene nanoplatelet ratio. Therefore, the main effects should not be interpreted independently.
- Sixth, principal component analysis showed that mixture performance should be interpreted as a combined test-result profile rather than as a single-property outcome. Mixtures with favorable combined behavior were associated with higher strength, higher ultrasonic pulse velocity, and lower log-transformed resistivity. The highest graphene nanoplatelet concentration did not consistently move mixtures toward this favorable combined-performance direction.
- Seventh, desirability analysis provided a practical screening tool for ranking mixtures based on simultaneous strength, ultrasonic pulse velocity, and resistivity criteria. The desirability ranking should not be interpreted as direct proof of ice-melting performance. It identifies candidate formulations for follow-up testing.

- Finally, the original project scope was not fully completed because the principal investigator moved from Colorado State University to Boise State University and the project funds could not be transferred. The completed work represents a partial implementation of the original project. It provides useful material-screening evidence, but it does not complete the proposed landfilled fly ash activation, durability evaluation, or direct snow-melting validation.

While the completed study establishes an initial basis for identifying promising GNP-modified geopolymer mortar formulations, it represents only part of the research program originally proposed. The completed work should therefore be interpreted as the material-screening stage of a broader research program. The originally planned landfilled fly ash activation, durability evaluation, and direct ice-melting validation were not completed, and translation from the present mortar system to concrete was also not evaluated.

Moving forward, the first next step should be to translate the most promising mortar formulations to concrete. Coarse aggregate should be introduced and the paste volume, GNP content, SDS dosage, water content, air content, and workability should be re-optimized. Trends observed in mortar should not be assumed to transfer directly to concrete because coarse aggregate changes the volume and continuity of the conductive paste, the interfacial transition zone, mechanical response, electrical transport paths, and thermal behavior. Concrete-scale testing should therefore include workability, air content, compressive and flexural strength, bulk electrical resistivity, and other pavement-relevant properties. This stage would determine whether the lower-GNP formulations identified in the present screening remain favorable after scale-up.

Concrete mixtures that satisfy the required mechanical criteria should then undergo direct electrical-heating and ice-melting testing. These experiments should control electrode configuration, applied voltage or current, specimen geometry, moisture condition, initial temperature, and ice thickness. Current draw, power consumption, heating rate, surface-temperature distribution, heating uniformity, and ice-melting time should be measured. These measurements would determine whether the resistivity values identified during screening translate into functional snow-melting performance and would establish the acceptable resistivity operating range and performance limits needed for a constrained, application-level desirability analysis.

Functional durability should then be evaluated by repeating mechanical, electrical, and heating measurements after transportation-related deterioration exposures. Freeze-thaw cycling, deicing-salt or chloride exposure, abrasion, wet-dry cycling, shrinkage, and repeated electrical-heating cycles should be considered. Measuring strength, resistivity, heating uniformity, and ice-melting efficiency before and after exposure would indicate whether deterioration disrupts the conductive network or reduces functional performance. This step is necessary because initial resistivity and strength alone cannot establish long-term pavement suitability.

Finally, the originally proposed landfilled fly ash activation should be revisited to determine whether reclaimed material can reproduce the mechanical and electrical behavior observed with the Class F fly ash used here. Mechanical activation and source variability should be evaluated together with the same mortar-to-concrete translation and functional tests. Additional dispersion and microstructural measurements—including zeta potential, ultraviolet-visible spectroscopy, microscopy, pore-structure analysis, or X-ray computed tomography—would help distinguish whether future performance changes are associated with GNP dispersion, surfactant dosage, entrained air, pore structure, matrix disruption, or precursor variability.

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