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EFFECT OF GRAPHENE OXIDE ON CHLORIDE ION PENETRATION IN STANDARD CANADIAN MIXES

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ABSTRACT

This study was conducted to evaluate the impact of a small percentage (0.03%) of graphene oxide (GO) on the properties of cement-based materials. The GO dispersion method (superplasticizers and sonication) and the use of binary binders were also variables of the study. To make this technology potentially accessible to concrete producers, commercially available GO was used for the trials. Through isothermal calorimetry, it was possible to characterize the hydration reaction of the GO-modified cementitious materials. The effect of GO is visible on the heat release rate curves, especially when dispersed by a superplasticizer and by a combination of superplasticizer and sonication. This effect is mainly noted in the amplitude of the calcium aluminate (C_3A) peak. The compressive strength tests were used to evaluate the mechanical strength of mortar at 1, 7, and 28 days. To evaluate the effect of GO from a practical application point of view, chloride ion penetration tests were carried out at 28, 56, and 91 days, on concretes based on the Ministère des Transports (MTQ) and Canadian Standard Association (CSA) V-S/C-1 mixes. The results showed that GO slightly reduces the penetration of chloride ions varying with the cure period and the type of concrete mix.

Keywords: *Graphene oxide, supplementary cementitious materials, calorimetry, RCPT, durability.*

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1. INTRODUCTION

The construction industry faces a significant challenge in reducing CO_2 emissions related to cement consumption. To address this, optimizing concrete mix designs and using efficient concrete grades can reduce material consumption and emissions [1]. Incorporating high range water admixtures (HRWAs) and supplementary cementitious materials (SCMs) into concrete mix designs empowers engineers and concrete producers to achieve concrete with exceptional properties and improved performance. By optimizing concrete mixtures with HRWAs and SCMs, cost-effective and sustainable solutions can be achieved while meeting specific performance criteria and environmental objectives [2].

The investigation of nanomodified cementitious composites, specifically using graphene oxide (GO), has shown promise in improving the mechanical properties and durability of cement-based materials. Numerous studies have demonstrated that the addition of a small percentage of GO to cement paste, mortars, and concrete can enhance compressive strength, flexural strength, tensile strength, and corrosion resistance [3-5]. The mechanism behind this improvement is believed to be the nucleation effect of GO on the growth of C-S-H crystals in the cement paste. Some researchers have compared this phenomenon to the well-known filler effect observed in supplementary cementitious materials [6-8].

GO-modified cementitious materials have also shown an impact on the pore structure of cement paste, reducing porosity and pore size, particularly in mixes with lower water-to-binder ratios [7][9]. Thermogravimetric analysis tests have further characterized the hydration of cement in the presence of GO, showing increased non-evaporable water content and calcium hydroxide content in GO-modified mixes [9][10]. The effect of GO on cement microstructure is not yet fully understood, and different studies have proposed varying explanations. However, the positive effects on properties of cement-based materials are generally acknowledged [11][12].

To facilitate the dispersion of GO nanoparticles, researchers have explored different methods, including the use of superplasticizers and sonication [3][12-14]. Superplasticizers act as a protective barrier around GO, preventing agglomeration, while sonication aids in dispersing GO in the cement matrix [13]. While GO has shown promise in enhancing cementitious materials' performance, some studies have indicated that it can negatively affect mortar performance, particularly due to poor workability in certain cases [15]. In terms of durability, few studies have been conducted on GO-modified concrete. Further research is needed to better understand the impact of GO on the durability of concrete structures, especially in terms of corrosion resistance [16].

A previous study showed the improved corrosion resistance of blended cement with slag and graphene oxide [17]. To continue the research on GO-modified cementitious materials, a multi-scale experimental program was conducted. Isothermal calorimetry, compressive strength tests, and chloride ion penetration tests were performed to evaluate the impact of GO on different properties and to investigate nanoparticle dispersion methods.

2. MATERIALS AND METHODS

2.1. Materials

In this study, various binders were employed for different stages of the research program, including general use Portland cement (GU), granulated ground blast furnace slag (GGBS), and silica fume (SF). The chemical analysis of these binders is detailed in Table 1. To introduce graphene oxide (GO) into the mixtures, a commercially available water dispersion of GO at a concentration of 0.4 wt% was utilized.

Table 1. Chemical analysis of binders.

Oxide (%)	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O_{eq}	S	SO_3
GGBFS	36.3	10.3	0.9	43.4	6.6	0.5	0.9	0.2
SF	96.5	0.5	0.45	0.4	0.4	0.11	-	0.08
GU	20.4	4.4	2.5	63	1.7	0.53	-	3.7

For the concrete mixes, two types of coarse aggregates were employed: 5-20 mm limestone aggregates adhered to the CSA specifications for particle size distribution. Natural sand served as the fine aggregates, and its particle size distribution and fineness modulus complied with Canadian standards. To achieve the desired slump and air content in the concrete mixes, two types of polycarboxylate superplasticizer and air entraining admix were employed.

2.2. Sample preparation and testing

2.2.1. GO sonication

Sonication was employed for some mixes using GO-water solutions. The 0.04% graphene oxide dispersion was added to the required amount of water for each paste and mortar mix. The GO dispersion in a beaker was placed inside a protective box, and the instrument probe was submerged into the solution. Sonication induced cavitation, causing agglomerates to separate from the particles, resulting in a more uniform dispersion of the nanoparticles. A Qsonica brand and model Q700 device, programmed at an amplitude of 30 for 15 minutes, was used for nanoparticle dispersion. Mixes with sonicated GO are indicated with the "+" symbol.

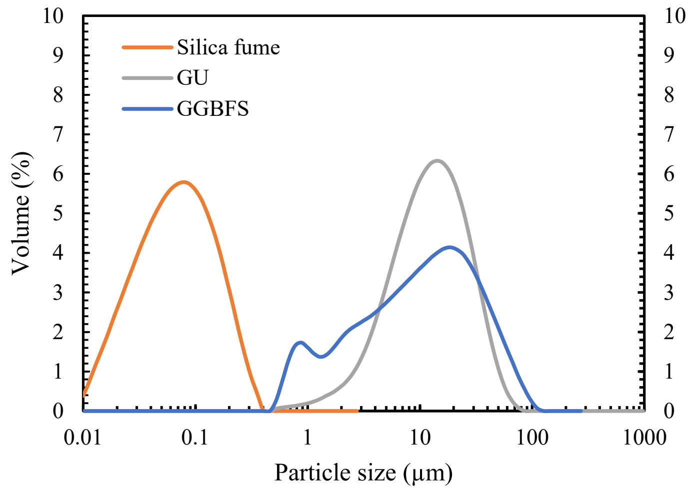

Figure 1. : Particle size distribution of binders.

2.2.2. Isothermal calorimetry

Isothermal calorimetry tests were conducted following the ASTM C1702 standard [18], which is the "Standard Test Method for Measurement of Heat of Hydration of Hydraulic Cementitious Materials Using Isothermal Conduction Calorimetry." The objective of this standard is to determine the heat of hydration of a cement paste by comparing the results obtained for different binders with an inert reference sample. The tests were carried out for a duration of 7 days at a constant temperature of 23°C. The tested pastes were mixed manually in the ampoule using a glass rod. Table 2 presents the samples used for the isothermal calorimetry tests, providing a detailed overview of the different combinations of mixtures analyzed during the experimental program.

Table 2. Materials proportions for isothermal calorimetry mixes.

# Mix	GU (g)	GGBFS (g)	Water (ml)	W/B	GO (%)	SP	Sonication
1	2.7332	0.8382	1.4286	0.40	-	-	No
2	2.7332	0.8382	1.1607	0.40	0.03	-	No
3	2.7332	0.8382	1.4286	0.40	-	1	No
4	2.7332	0.8382	1.4286	0.40	-	-	Yes
5	2.7332	0.8382	1.1607	0.40	0.03	1	No
6	2.7332	0.8382	1.1607	0.40	0.03	1	Yes

2.2.3. Concrete tests

In this part of the study, the only dispersion method used for graphene oxide (GO) was superplasticizer. However, it is important to note that the volume of GO solution to be dispersed for concrete manufacturing was much larger compared to pastes and mortars (359 ml for mortars versus 7 liters for concrete). Due to the significant volume, sonication with the available equipment in this research was not feasible.

Concrete is often required to meet specific standards and performance criteria to ensure its safety and durability as a construction material. For this research, the concrete mix designs were based on Quebec Ministry of Transportation type V-S, as well as CSA A23.1 type C-1 mixes. Table 3 presents the target characteristics of these concrete mixes. The mixes adhering to the requirements of type V-S/C-1 concretes were prepared using the 5-20 mm limestone aggregate. The binder mass used was 390 kg/m^3, and the water/binder ratio was set at 0.4. Admixture dosages were adjusted during batching to achieve the desired slump and air content. In this category of mixes, different types of binders were utilized.

Initially, reference mixes were created, consisting of concrete made with general use (GU) cement and GUb-30S blended cement containing 30% of slag in mass. To assess the combination of graphene oxide with these two types of binders, the same mixes were then produced by adding 0.03% of GO based on the total binder mass. Lastly, a reference mix meeting MTQ requirements was prepared using a GUb-S/SF binder type with 30% slag and 5% silica fume in mass. This mix will serve as a reference to evaluate the performance of graphene oxide concrete compared to concrete typically used for civil engineering structures.

The admixture dosage was adjusted differently for each concrete mix to achieve the desired fresh state characteristics. These mix designs used the same types of binders mentioned earlier, resulting in the creation of 3 formulations inspired by a type V-S/C-1 concrete. The concrete mixes were prepared following the guidelines of CSA Standard A23.2-2C. The GO dispersion was mixed with water and superplasticizer (SP) and added to the mixer during the mixing process. In addition to the mixing, fresh concrete was subjected to slump tests, air content tests, and density determination. Each of the different mixes yielded a total of 22 concrete cylinders. The manufacture of these 100 mm diameter and 200 mm long cylinders followed the CSA A23.2-3C test method for making and curing concrete compression and flexural test specimens. The specimens were then demolded after 23 hours ± 1 hour and placed in a 100% humidity chamber.

Table 3. Type V-S, and C-1 concrete requirements.

Concrete characteristics based on 3101 and CSA standards	Requirements	
	MTQ	CSA
	V-S	C-1
Compressive strength (MPa)	35 at 28 d	35 at 28 d
Minimal binder content kg/m^3	365/340	-
Binder type	GUb-S/SF, GUb-SF	-
W/B	0.40	< 0.4
Coarse aggregate (mm)	5 - 20	-
Air content (%)	6 - 9	5 - 8
Slump (mm)	90 - 150	-
Chloride ion penetration (coulombs)	1500	1500

2.2.4. Rapid chloride penetration testing

Rapid chloride penetration testing (RCPT) was performed according to the ASTM 1202-19 [19] Standard Test Method for Electrical Indication of Concrete's Ability to Resist Chloride Ion Penetration. For each mix, three 50 mm samples were sawed from the concrete cylinders and subjected to RCPT at 28, 56, and 91 days. A Germann Instrument RCPT testing device was utilized to test the specimens. The initial current measurement and subsequent measurements every 30 minutes were recorded for 6 hours. The values were plotted on a current (amperes) versus time (seconds) graph, and the integral of the area under the curve represented the load in Coulomb (ampere-seconds). RCPT results provide a qualitative indication of the concrete's resistance to chloride ion penetration, with smaller Coulomb values indicating better resistance.

3. RESULTS AND DISCUSSION

3.1. Isothermal calorimetry

The results of the isothermal calorimetry tests were presented figure 2 and table 4 respectively for the heat flow and cumulative heat of hydration. To improve resolution, the x-axis of the heat flow graphs was limited to 60 hours. For mixes with a binary binder consisting of 30% slag, similar observations were made compared to the general use Portland cement (GU) mixes. The addition of superplasticizer caused a rightward shift of the maximum peak, and the amplification of the C_3A peak was observed for mixes with both superplasticizer (SP) and graphene oxide (GO). Moreover, the second peak's amplitude increased with lower water-to-binder (W/B) ratios. Additionally, for samples with SP, those with GO+ showed a moderately higher peak compared to mixes with SP alone. However, there was no visible evidence of significant synergy or interaction between GO and slag that notably affected the curves' shapes. Table 4 provided information on the mixtures with the highest heat of hydration at 160 hours, which was found to be those with superplasticizer GUb-S 0.4 SP-GO.

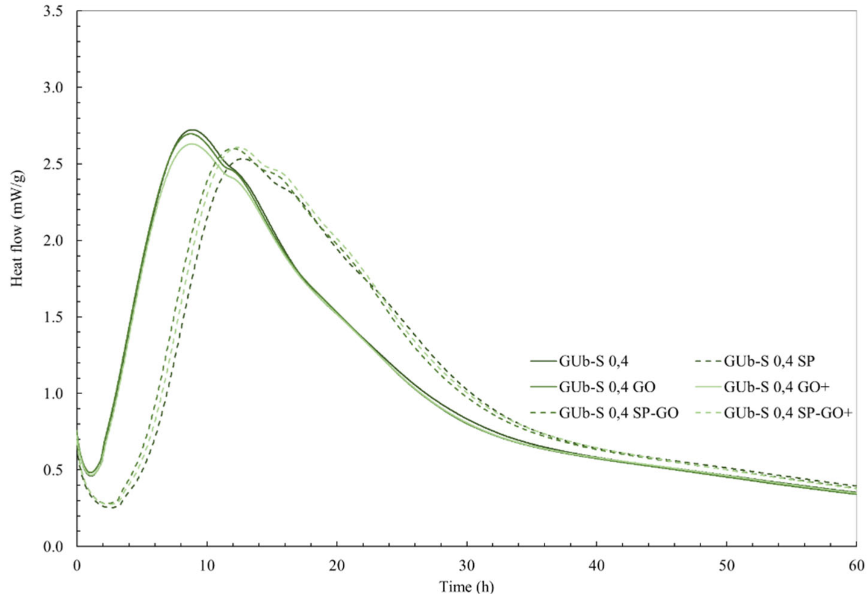

Figure 2. : Heat flow vs. time for GUb-S mixes.

The calorimetry tests investigated the impact of graphene oxide (GO) and its dispersion method on cement paste with general use Portland cement (GU) and GU blended cement made with 30% ground granulated blast furnace slag (GGBFS). As observed before, the type of binder and the use of superplasticizer had a more significant effect on the shape of the heat release rate and cumulative heat of hydration curves. The influence of GO was more subtle, necessitating the separation of curves based on binder type.

The addition of GO slightly increased the height of the heat rate peak, particularly for mixes with superplasticizer (SP). However, this observation was not consistent for mixes without SP. The most notable effect of GO was on the shape of the calcium aluminate hydration peak, which was amplified by its addition. This phenomenon was evident in both binary binder pastes and GU cement pastes and was more prominent when GO was dispersed with superplasticizer. This phenomenon can be attributed to the interaction between the functional groups of GO and Ca^{2+}, leading to a reduction in the concentration of Ca^{2+} in the porous solution and facilitating the dissolution of the C_3A (calcium aluminate) component [20]. This occurrence might be linked to the nanofiller effect of GO, similar to the effect observed in studies using quartz and limestone powder as fillers in cementitious materials. The nanofiller effect of GO could accelerate and amplify the hydration of cement components, leading to an acceleration of the C_3A peak [21].

Table 4. Cumulative heat at 160 hours for GUb-S mixes.

Mix	Cumulative heat at 160 hours (J/g)
GUb-S 0.4	287.9
GUb-S 0.4 GO	285.5
GUb-S 0.4 SP	287.9
GUb-S 0.4 GO+	285.3
GUb-S 0.4 SP-GO	292.5
GUb-S 0.4 SP-GO+	291.9

3.2. Concrete

Table 5 presents the fresh concrete properties and 28-day compressive strength for each mix, and these tests are conducted to verify compliance with the Quebec MTQ Standard 3102. These tests are essential for assessing whether the mixes meet the specified requirements for concrete used in civil engineering constructions, as per the standards. In this paper, the focus is primarily on discussing and analyzing the RCPT results.

With the exception of Type V GUb-S/SF mix, where the air content exceeds the standard limit, all other mixes meet the requirements for slump, air content, and compressive strength. However, it was determined that reworking the batches of non-compliant concretes was unnecessary due to the minimal impact of entrained air on the resistance to chloride ion penetration.

Table 5. Fresh and hardened characteristics of concrete mixes.

Properties		Air content	Slump	Density	Compressive strength (28d)
Mixes		%	mm	kg/m^3	MPa
TYPE V-S	**GU 0.4**	10	115	2263	37.2
	GUb-30S 0.4	9.0	115	2280	38.5
	GUb-30S+GO 0.4	7.5	100	2343	45.0
	GUb-S/SF 0.4	10.0	120	2255	38.3

3.3. Rapid Chloride Penetration Test (RCPT)

Figure 3 illustrates the results of the chloride ion penetration tests conducted at 28, 56, and 91 days for mixes based on V-S concrete with a water-to-binder (W/B) ratio of 0.4. The graph includes two reference lines: the red line represents the 1000-coulomb limit required by the

MTQ standard at 56 days, and the blue line represents the 1500-coulomb limit required by the CSA standard at 91 days. The error bars on the graph represent the confidence interval calculated from a 95% confidence index.

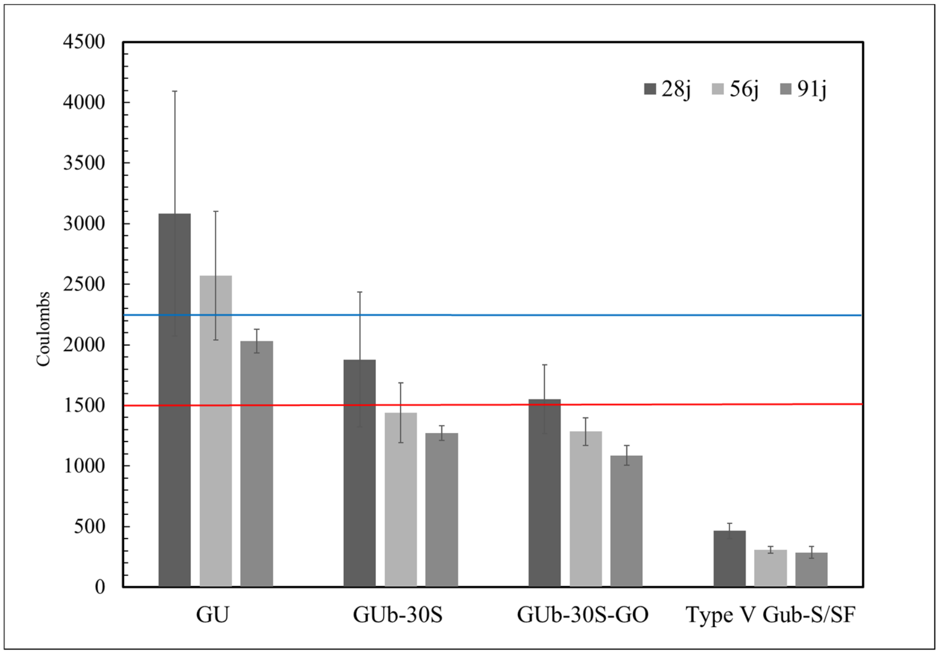

Figure 3. : RCPT results for type V-S/C-1 mixes (the red line shows the standard required and the mixes below the line are acceptable).

The analysis of the chloride ion penetration test results reveals two significant observations for all concrete mixes. Firstly, the coulomb value consistently decreases with the age of the concrete, indicating an improved resistance to chloride penetration over time, from early age to 91 days. Secondly, the addition of supplementary cementitious materials (SCMs) to the concrete mixes significantly reduces the coulomb value, indicating enhanced chloride ion resistance. When blended with 30% slag, the penetration of chloride ions is significantly reduced. On average, it decreases by 38.8% compared to the GU mix. Although the GU-GO mix does not improve the performance of the concrete, the addition of GO to the blended binder slightly reduces the chloride ion penetration, with an average decrease of 7.4% compared to the GUb-S mix and 47.7% compared to the GU 0.4 mix. On the other hand, the typical V-S mix with silica fume shows significantly superior performance to the other four mixes, with an average decrease of 86% compared to the GU mix. This mix also meets the MTQ requirements with a value of 307 coulombs at 56 days. Additionally, for the 1500-coulomb limit of the CSA standard, GUb-30S and GUb-30S-GO concretes meet the requirements. The addition of SCMs, such as slag and silica fume, significantly improves the chloride ion resistance of concrete, while the addition of GO to GU cement concrete does not show notable improvement in this property. The use of blended binders appears to be more effective in enhancing the durability of concrete with respect to chloride penetration compared to the sole use of GU cement.

4. CONCLUSION

In conclusion, this study aimed to explore the potential of graphene oxide (GO) in cement-based materials, both from a fundamental and practical perspective. The multiscale analysis of the effectiveness of graphene oxide (GO) in GU and compound binder cementitious matrices yielded mixed results. Calorimetry tests showed that superplasticizer-dispersed GO affected the heat flow peaks and intensity of the C_3A peaks, suggesting a possible nucleation effect. However, compressive strength tests on mortar did not yield expected results, showing a decrease in strength without superplasticizer, and only marginal improvement with properly dispersed GO.

However, the addition of 0.03% GO was insufficient to significantly enhance concrete durability, failing to meet the 1000-coulomb limit specified by the Quebec Ministry of Transport standard. Several reasons may explain these results. GO being a relatively new material lacks standardized quality assurance procedures, leading to varied conclusions in different studies. The quantity of GO used (0.03%) might have been too low to exhibit substantial effects, while higher dosages may have practical limitations and increased costs. Incompatibility with some superplasticizers and contamination issues in the presence of cement or lime may also affect GO dispersion and stability.

While there is a significant interest in carbon-based nanomaterials, including GO, for various industries, the study highlighted the need for further research and standardization in the field of GO-modified cementitious materials. Reproducibility of results and larger-scale studies are necessary to fully explore the potential benefits of GO in concrete and cement applications.

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