



## Experimental evaluation of the effect of Nano-Graphene on the durability of roller-compacted concrete pavements under freeze-thaw cycles

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**ABSTRACT:** Millions of dollars are spent every year on road pavements, which generally require rehabilitation every one to five years. Roller-compacted concrete (RCC) pavements are a good replacement for asphalt pavements due to their special properties, such as better durability and conformity with sustainable development principles. On the other hand, changes in the ambient temperature, which cause freeze-thaw cycles, can strongly affect the strength and durability of concrete pavements. Hence, a study of the impact of freeze-thaw cycles and additives on the properties of RCC seems necessary for developing suitable measures for controlling damage and durability loss in concrete. To this end, the present study investigated the influence of cement and nanographene content on the durability of pavements and the weight loss, dynamic modulus of elasticity, durability factor, and spalling of RCC. Nanographene with 0.05%, 0.07%, and 0.1% of cement weight was added to specimens prepared with cement grades of 200, 250, and 300. The specimens were subjected to freeze-thaw cycles after preparation and curing. According to the results, the addition of nanographene improved the properties of RCC under freeze-thaw cycles, reduced weight loss and spalling in the specimens with different cement contents, and enhanced their durability factor and modulus of elasticity. Although adding nanographene significantly contributed to better concrete properties, its effect was directly related to the concrete content. Finally, the optimal values of the cement and graphene content after 300 cycles were obtained to be 300 and 0.05% of cement weight, respectively.

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### 1- Introduction

The successful application of roller-compacted concrete (RCC) in large-scale construction projects, such as dam construction, and the high strength of this concrete at a low age sparked an increase in its use in road pavement and other engineering projects. On the one hand, RCC provides higher pavement strength than asphalt due to its higher rigidity. On the other hand, it entails significant technical and economic advantages due to the simplicity of pavement construction using road construction equipment and the lower consumption of earth materials. However, changes in ambient temperature on colder days lead to freeze-thaw cycles, which can adversely affect the strength of RCC. As a result, it is necessary to take measures to control damage along with strength and durability loss in this material.

Extensive research has so far been conducted on the addition of nanomaterial to RCC [1-6] and the application of various materials, including fibers, fly ash, slag, and deicer salts, in improving the behavior of RCC pavements.

Shafipour et al. (2021) studied the effect of polymer fibers on the durability of RCC. They concluded that adding fibers

increased its compressive strength but decreased its durability against freeze-thaw cycles. Hence, they reported that care should be taken when using fibers in RCC, especially in cold climates [7]. Hazaree et al. (2011) reported the results of experiments on RCC mixtures containing a wide range of cement content (100 kg/m<sup>3</sup> and 450 kg/m<sup>3</sup>). They evaluated the durability, water absorption, permeability, compressive strength, and resistance to freeze-thaw cycles of the mixtures. The results showed that high cement content and air bubbles influenced the strength and durability of the mixtures under freeze-thaw cycles [8].

In research on the impact of nanographene on the strength of concrete, Chen et al. (2019) reported that concrete containing nanographene had a finer pore structure than normal concrete [9]. Adding graphene reduced the performance of concrete while increasing the compressive strength of the specimens. The performance of concrete was evaluated with the workability, compressive strength, visual deterioration, and mass loss of concrete samples. It also increased concrete durability under freeze-thaw cycles. Nicula et al. (2020) examined the effect of blast furnace slag on the durability properties of road concrete, such as freeze-thaw resistance. According to the experimental results,

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**Table 1. Chemical composition of the cement used in this study (from GuilanSabz Co., Iran)**

Chemical Properties	CaO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	K <sub>2</sub> O	Na <sub>2</sub> O	SO <sub>3</sub>	Cl	L.O.I
%	64.32	21.8	5.36	3.64	1.7	0.82	0.03	1.6	0.03	0.7

**Table 2. Properties of the NGO used in this study (from Nanotech Innovation United, Iran)**

Parameters	Thickness	Product Purity	Number of Layer	Surface Area	Bulk Density	Electrical Conductivity	Lateral dimension
Approximate Values	3-6 nm	99%	8-10	>120m <sup>2</sup> /g	0.42 g/cc	Insulator	5-10 $\mu$ m

the durability of concrete with 15% slag powder and 60% artificial aggregate increased under 300 freeze-thaw cycles. Moreover, the results for the mixture with 15% slag powder and 20% artificial aggregate were considerably better than those for mixtures containing regular materials [10]. Zhang et al. (2021) studied the influence of graphene nanoplatelets on the carbonation depth of concrete under varying weather conditions. The authors reported that the carbonation depth of graphene concrete was 15%-30% of that of normal concrete and that this difference decreased under humidity levels above 78%. Based on the overall results, nanographene contributed to the anti-carbonation performance under varying weather conditions [11].

Since the consumption of RCC is progressing and its use in cold and humid areas is associated with problems, therefore, research should be done on how it behaves in different conditions. A literature review shows a lack of comprehensive research on the effect of nanographene on the strength and durability of RCC pavements under freeze-thaw cycles. Therefore, the present work studied the extent and manner of the influence of nanographene on the mechanical and durability properties of pavements made of RCC subjected to freeze-thaw cycles.

## 2- Materials and methods

### 2- 1- Materials

In this research, average aggregate gradation was used according to PCA (2010) [12-14] for better mixing of graphene nanoparticles (NGO) with RCC. Figure 1 displays the gradation of the aggregate used in this research.

The specimens were prepared using Portland cement (Type-2) procured from GuilanSabz Co., Iran. Table 1 shows

the chemical composition of this type of cement. In addition, the water used in this study was supplied from tap water. Also, the Graphene-oxide nanomaterial was procured from Nanotech Innovation United with the specifications shown in Table 2.

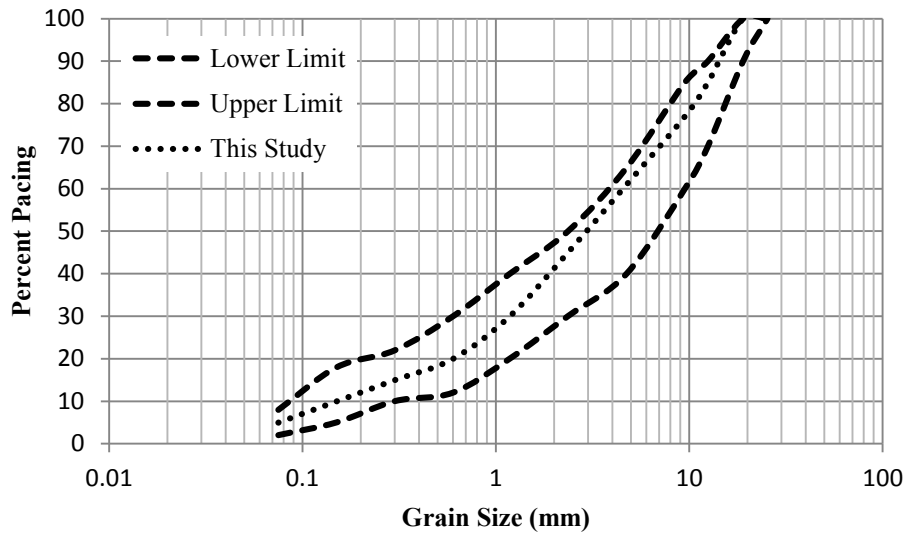
### 2- 2- Methods

In this research, 12 specimens were prepared and tested, namely 9 specimens with 3 different NGO contents and 3 different cement contents along with 3 control specimens. Cubic specimens with dimensions of 400×75×100 mm were prepared according to the PCA (2010) and the ASTM C666 [15-18] to examine the durability of their surface under freeze-thaw cycles. Specimens with cement contents of 200, 250, and 300 and NGO contents of 0.05%, 0.075%, and 0.1% of the cement weight were prepared. The water-to-cement ratio recommended for this concrete is 0.4-0.42 based on its preparation method. The designations and mix designs of all the specimens are shown in Table 3.

### 2- 3- Preparation of specimens

RCC beam specimens with a size of 100×75×400 mm were used for the freeze-thaw cycle tests based on the ASTM C666 standard. In order to prepare the specimens, first, the materials were weighed to the required amounts and were then mixed for RCC placement.

Mixing simple RCC materials were mixed in a dry state inside the concrete mixer for 1 minute. To mix the materials of designs, NGO solution was added to the RCC at the final stage with 140 revolutions per minute for 5 to 7 minutes. The prepared RCC was then compacted using a vibrating table. For curing the specimens, the molds were left in a



**Fig. 1. Gradation of the aggregate used in this study**

**Table 3. Designations and mix designs of the specimens**

Quantities	Mixtures											
	R1	R1G1	R1G2	R1G3	R2	R2G1	R2G2	R2G3	R3	R3G1	R3G2	R3G3
Cement (C) (kg/m <sup>3</sup> )	300	300	300	300	250	250	250	250	200	200	200	200
Water (W)	120	120	120	120	100	100	100	80	80	80	80	80
W/C	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4	0.4
NGO	—	0.15	0.225	0.3	—	0.125	0.19	0.25	—	0.1	0.15	0.2
NGO/C (%)	—	0.05	0.075	0.1	—	0.05	0.075	0.1	—	0.05	0.075	0.1
Sand (kg/m <sup>3</sup> )	1410	1410	1410	1410	1430	1430	1430	1430	1460	1460	1460	1460
Coarse aggregate (kg/m <sup>3</sup> )	570	570	570	570	620	620	620	620	660	660	660	660

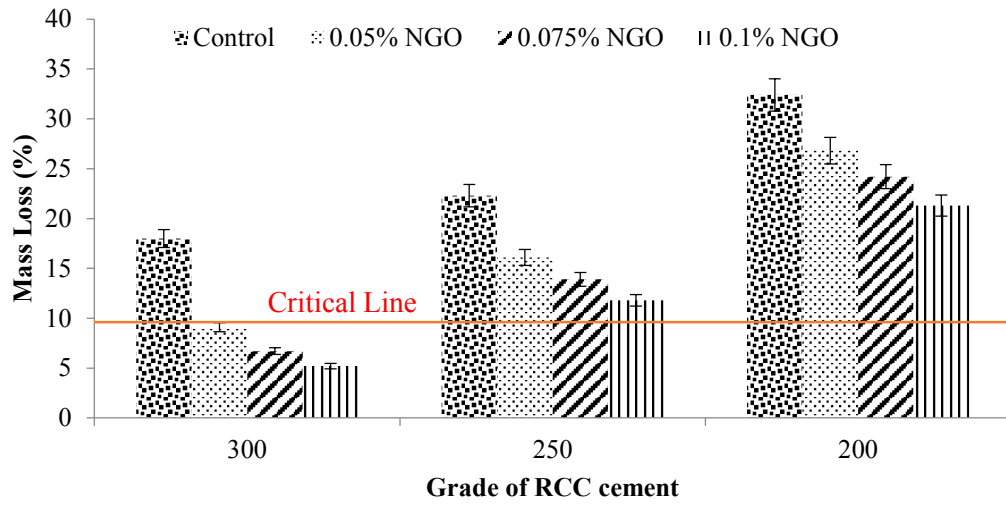
humid location at a temperature of 23±2°C for 24 hours. The specimens were then removed from the molds and placed in the curing basin. The specimens were removed from the basin after 14 days, left to rest for 48 hours at 23±2°C, and placed in the freeze-thaw device to examine their surface behavior after freeze-thaw cycles.

**2- 4- Test procedures**

The specimens were placed in standard steel molds after mold removal and 14 days of curing. These steel molds were then put in the freeze-thaw device and filled with tap water up to 0.5 cm above the RCC surface. The device settings were

then configured according to ASTM C666 and the device was turned on. Based on this standard, the device brings the specimen to a temperature of -18°C and then to 4°C over a period of 5 hours. In order to evaluate the weight loss of the specimens, they were removed from the device and weighed every 36 cycles. According to the ASTM C666 standard, a weight loss of 10% of the initial weight of the specimens after 300 cycles is acceptable.

In addition, visual inspections were carried out to measure the apparent change in the specimens according to ASTM C672. This standard offers a rating system of 0-5 for the visual inspection of concrete surfaces under freeze-thaw



**Fig. 2. Percentage of weight loss in the specimens with and without NGO**

cycles based on spalling. Based on this rating system, a rating of 0 represents no spalling, while a rating of 5 represents strong spalling where aggregates are exposed throughout the surface.

Ultrasonic wave velocity testing with a frequency of 54 kHz and a measurement accuracy of 0.1  $\mu$ s was used to determine the dynamic modulus of elasticity of the specimens. Based on the ASTM C666 standard, the dynamic modulus of elasticity is calculated as follows:

$$P_c = (n_1^2 / n^2) \times 100 \quad (1)$$

where:

$P_c$ : is the relative dynamic modulus of elasticity in %,

$n$ : denotes the transverse frequency in the zeroth freeze-thaw cycle, and

$n_1$ : represents the transverse frequency after the  $c^{\text{th}}$  freeze-thaw cycle.

Moreover, according to ASTM C666, the durability factor of the specimens can be calculated as follows:

$$DF = PN/M \quad (2)$$

where:

DF: is the durability factor of the specimen,

P: is the dynamic modulus of elasticity in % at N cycles, and

N: is the number of cycles at which P reaches the threshold for ending the test or the number of cycles at which the test must end, whichever is smaller. Also,

M: is the number of cycles at which the test is terminated.

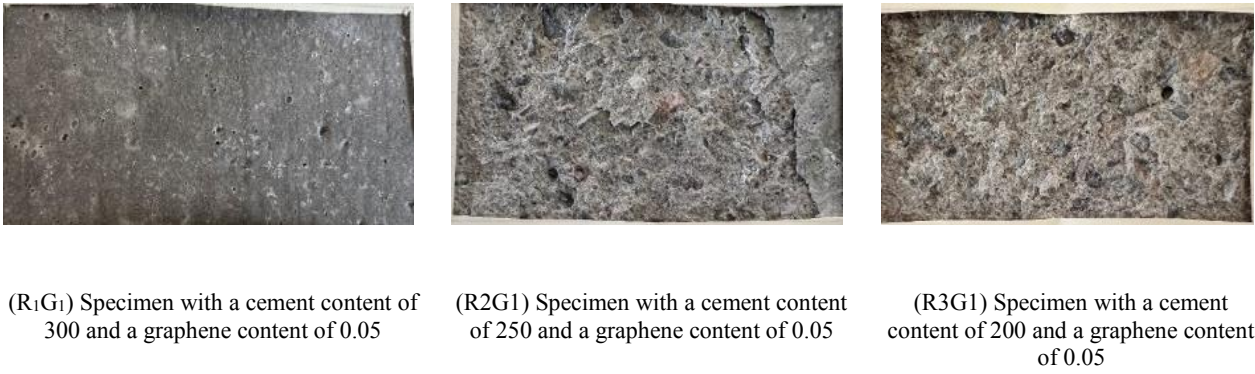
### 3- Results and discussion

#### 3- 1- Specimen weight loss after 300 freeze-thaw cycles

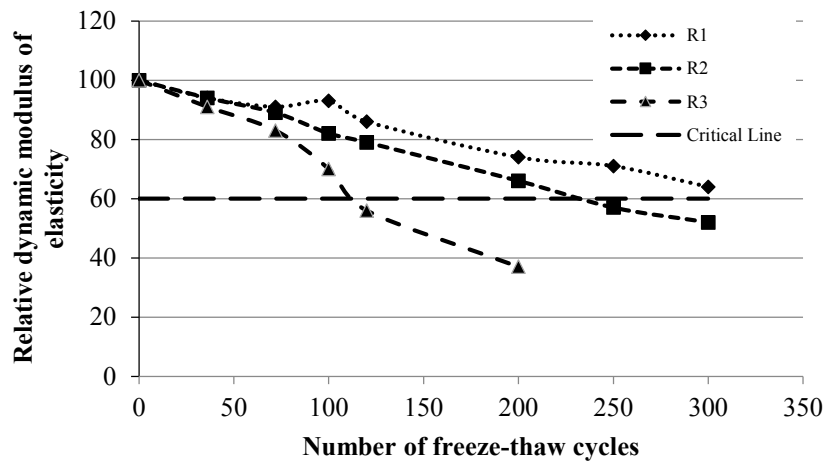
A study of the data showed that adding specific amounts of NGO to the RCC mixture improved the properties of RCC and its resistance to freeze-thaw cycles. According to the results obtained from the addition of nanographene oxide, the weight of samples with a cement content of 300 was reduced by 70%, with a cement content of 250, 40%, and with a cement content of 200, 35%. The reason for the mass loss in the RCC was due to the damage caused by the freeze-thaw cycles. However, it must be noted that an increase in the cement content simultaneously with the addition of NGO can lead to the best results. As seen in Figure 2, the specimens with a cement content of 300 exhibited acceptable weight loss based on the standard with the addition of NGO. However, this weight loss was beyond the limit allowed by the standard in the specimens with lower cement content even though the addition of NGO improved their performance under freeze-thaw cycles. The obtained results are in good agreement with the findings of various articles [8, 9].

#### 3- 2- Visual inspection of the specimens

The visual inspection of the three specimens with identical graphene and different cement contents after 100 cycles, performed according to ASTM C672, is displayed in Figure 3. The results indicated that using nanographene increased the strength at the surface layer of the RCC by decreasing the permeability of this layer. The highest increase in surface layer strength corresponded to the specimen with the highest cement content. In addition, deeper spalling and a higher weight loss were observed in the control specimens.



**Fig. 3. Visual inspection of specimens with different cement and identical graphene contents**



**Fig. 4. Changes in the dynamic modulus of elasticity of the control specimens**

Hence, it may be concluded that adding nanographene to the RCC mixture together with good cement content improves the spalling resistance of concrete under freeze-thaw cycles.

### 3- 3- Dynamic modulus of elasticity

Changes in the dynamic modulus of elasticity of the control specimens with different cement contents after 0-300 freeze-thaw cycles are displayed in Figure 4.

The resistance of mixes increased with the increase in the cement content. Moreover, Figure 5 depicts variations in the dynamic modulus of elasticity in the specimens with cement contents of 200, 250, and 300 after the addition of 0.05%, 0.07%, and 0.1% of cement weight of graphene and after 0-300 freeze-thaw cycles. According to Figure 5, it can be seen that the samples with low cement grade will last up to 100 cycles, and after adding NGO, this amount will increase significantly [8]. Adding NGO makes RCC mixes more cohesive with a

significant increase in density. The results obtained from the graphs indicated that adding nanographene to the RCC mixture significantly lowered the reduction in the dynamic modulus of elasticity of the specimens and contributed to the durability of RCC under freeze-thaw cycles. Furthermore, the results showed that an increase in the cement content reinforced the positive influence of adding nanographene. Based on the results obtained from the experiments, it was found that by adding the cement and NGO composition, a better effect was created on the samples. This effect is less by adding each of the above ingredients alone.

### 3- 4- Durability factor

Based on the results presented in Table 4, the durability factor was obtained to be 93% in mix designs with a cement content of 300 and a nanographene content of 0.1% of cement weight. In addition, this factor was determined to be above



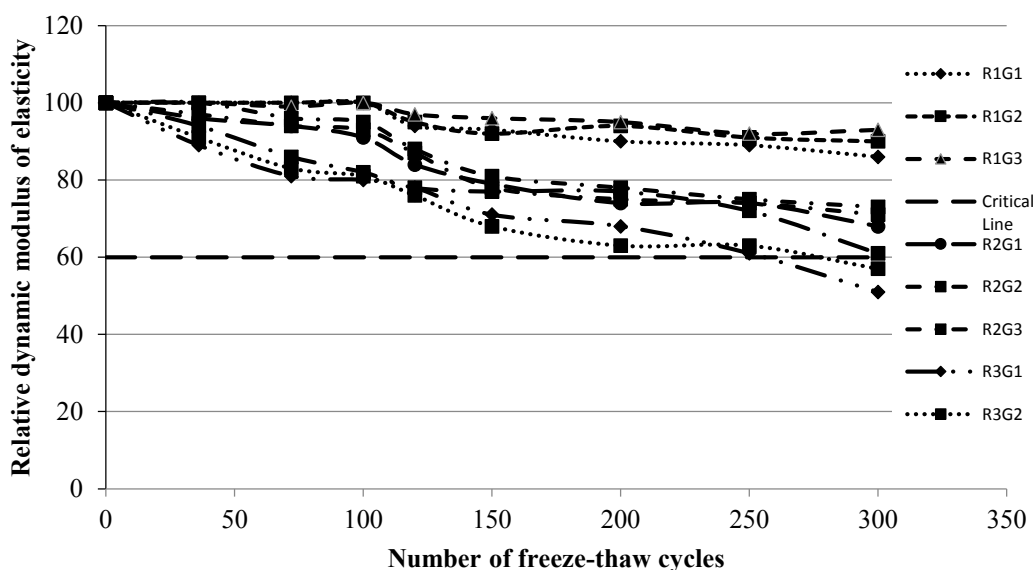


Fig. 5. Changes in the dynamic modulus of elasticity of specimens after the addition of nanographene

Table 4. The durability factor of the specimens

Mixture ID	R1	R1G1	R1G2	R1G3	R2	R2G1	R2G2	R2G3	R3	R3G1	R3G2	R3G3
Number of cycles	300	300	300	300	Failed	300	300	300	Failed	Failed	Failed	Failed
Durability factor (%)	21	86	90	93	Failed	26	32	43	Failed	Failed	Failed	Failed

50% in specimens with cement contents of 0.05% and 0.07% of cement weight. On the other hand, the control specimen with a cement content of 250 was destroyed before reaching the 300th cycle, and the durability factor was found to be below 50% in the rest of the specimens with a cement content of 250 and with added nanographene. In addition, the specimens with a cement content of 200 were also destroyed before the 300th cycle.

#### 4- Conclusion

This research aimed to study the effect of NGO on the durability of RCC subjected to freeze-thaw cycles. The following results were obtained:

1. The weight loss of the control specimens decreased with an increase in their cement content. However, this decrease was not enough, Because the weight loss of the specimen with a cement content of 300 exceeded the standard limit. The results after the addition of NGO indicated that adding

a specific amount of graphene to the RCC mixture enhanced the properties of RCC and its strength under freeze-thaw cycles. Although adding graphene to specimens with a low cement content considerably decreased weight reduction, the weight loss still exceeded the standard limit. Therefore, one may conclude that adding a specific amount of nanographene while using a specific cement content can remarkably improve the properties of RCC under freeze-thaw cycles.

2. A visual inspection of three specimens with identical graphene contents but different cement contents after 100 cycles showed that the specimen with a cement content of 300 and a graphene content of 0.05% of cement weight achieved a rating of 0-1, indicating good performance under freeze-thaw cycles.

3. Based on the elastic modulus graphs, the addition of nanographene to the RCC mixture led to a significant improvement in the durability of RCC subjected to freeze-thaw cycles. The graphs also showed that an increase in

the cement content reinforced the positive effect of adding nanographene.

4. According to the durability factor results, the control specimen with a cement content of 250 was destroyed before reaching the 300th cycle. Also, the durability factor for the rest of the specimens with 250 cement content was under 50% with added nanographene. In addition, the specimens with a cement content of 200 were also destroyed before the 300th cycle. In the specimens with a cement content of 300, adding nanographene greatly contributed to the performance and durability of the specimens under freeze-thaw cycles.

Thus, it can be concluded that the addition of nanographene has a positive influence on the performance of RCC. Based on the results, the optimal amount of nanographene can be considered to be 0.05% of cement weight. However, it must be noted that one cannot use RCC with low cement contents in pavements since there is a direct relationship between the use of appropriate cement content and the addition of nanographene. Although adding nanographene to concrete led to better RCC properties, the prepared RCC is acceptable only when the cement content is within an acceptable range.

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