



# Mechanical and thermal properties of graphene modified asphalt binders

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## HIGHLIGHTS

- Graphene produces a more elastic response in asphalt binders.
- Elastomers provide a more effective recovery capacity than graphene.
- Graphene reduces the asphalt binders susceptibility at high temperature.
- Graphene increases the asphalt binders capacity to be heated and to produce heat transfer.

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## ABSTRACT

Graphene is considered to be a promising material for the development of the next generation of asphalt binders to be used in road engineering. Nonetheless, in spite of the potential advantages that graphene could add to bitumen, there are currently very few studies able to quantify its real contribution to the performance of existing binders. This research therefore aims to evaluate the mechanical and thermal properties of graphene modified binders. For this purpose, rheological and thermal tests were conducted on binders manufactured with different dosages of graphene flakes. The results indicate that the presence of graphene produces a more elastic response in asphalt binders. However, the resulting recovery capacity is not as effective as that provided by other asphalt modifiers such as elastomers. In addition, it has been shown that graphene can reduce the thermal susceptibility of these binders, increasing their capacity to be heated and to produce heat transfer.

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## 1. Introduction

Due to the demographic explosion, increased demands for mobility, and the development of global commerce, recent decades have seen a worldwide increase in the volume of traffic and axle loads supported by road pavements. At the same time, climate change is producing major temperature variations throughout the year and the occurrence of more severe weather events (such as storms and frosts). These circumstances have led to the need for developing new asphalt materials to enhance the mechanical response of road pavements in order to avoid their premature deterioration and to ensure a longer life span under these severe service conditions. Furthermore, considerable efforts have been made to adapt these new materials to the need for more efficient and multifunctional smart road infrastructures [1].

One of the most studied solutions for the development of these next generation road materials has been the use of nano-modifiers for improving the mechanical properties and smart application of asphalt materials [2–8]. In this respect, since its emergence in 2004 [9], graphene has opened up a number of new research lines in the field of construction engineering. One of the main uses of graphene in construction materials is as reinforcement for the improvement of the mechanical performance of matrixes such as polymers or other composites (which allows for enhancing the stiffness and strength of these materials) [10]. Another possibility is to use graphene as a controller of some of the functional properties of these materials such as electrical conductivity, gas barrier behavior, thermal conductivity, expansion, and stability. Several studies have demonstrated that graphene could enhance the electrical conductivity of cement mortars [11], as well as increase their stiffness, tensile strength, and durability (it has been shown that graphene can change the pore structures and hydration process of cementitious materials [12,13]). Similarly, the use of graphene or graphene oxide has been successfully applied as reinforcement

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for ceramic and metallic matrices [14,15]. Finally, the use of graphene-modified materials is also being studied to obtain self-sensing materials for smart constructions, as well as for the monitoring of infrastructures or the development of potential thermo-electric devices [16,17].

Due to its chemical composition and mechanical properties, graphene is regarded as a potential modifier that could help to achieve more durable and smart asphalt materials. Since graphene flakes are hydrophobic, they can react with the non-polar groups of asphalt binder to form stable nanocomposites that offer a continuous transition of stress from the asphalt binder to the graphene particle (some researchers have even demonstrated that it is possible to obtain graphene from asphalt [18,19]). In addition, the presence of graphene flakes could modify the thermal conductivity of these materials (as occurs in other construction materials such as cement [20]), and it has a lubricant effect on the material (which could improve its workability [21]). Nonetheless, given that its use as a modifier is still relatively recent, the study of graphene-modified asphalt materials is still rather limited [16], and there are relatively few investigations that analyze the real impact of graphene on the mechanical and thermal properties of asphalt binders.

Most of the studies carried out in this regard have focused on analyzing the rheological properties of the graphene-modified asphalts. For instance, Marasteanu et al. [21] evaluated the rheological properties of asphalt binders that had been modified with different types of graphene flakes (with a constant dosage of 6% of graphene over the total weight of the binder). The results indicated that, at lower temperatures ( $-24\text{ }^{\circ}\text{C}$ ), the use of graphene nano-flakes could produce an almost two-fold increase in the stiffness of the plain binder, along with an improvement in its strength and strain tolerance under these circumstances. With respect to the type of the graphene used, it was found that increasing the carbon content and reducing the surface area both help to increase bitumen stiffness. However, these authors demonstrated that the addition of graphene nano-flakes does not significantly affect the rheological properties of the binder (complex shear modulus and phase angle) at intermediate and high service temperatures (from  $4\text{ }^{\circ}\text{C}$  to  $70\text{ }^{\circ}\text{C}$ ). In relation to these outcomes, Habib et al. [22] – who studied the mechanical performance of graphene oxide (GO) modified binders – found that even by using a minimum amount of GO (0.1125 and 0.225% over the total weight of the binder), a slight increment in elastic modulus of the binder can be observed (this increment becomes larger at lower temperatures and higher frequencies, which is in accord with the findings of Marasteanu et al. [21]). Finally, the research study conducted by Brcic [23] also shows that increasing the graphene dosage (1%, 1.5%, and 2% over the total weight of the binder) produces a rise in the stiffness of both unaged and aged asphalt binders (with a 70/100 penetration grade). In addition, an increase in the elastic recovery of the binders was observed as a consequence of the addition of graphene (although this increment is much lower than that offered by polymer modifiers). This study also demonstrated that the fabrication process affects the final mechanical behavior of graphene-modified asphalt binders (graphene mix is not homogeneous).

In terms of thermal properties, some studies have demonstrated that graphene could improve the high-temperature properties of asphalt binders, as their glass transition temperature increases as the graphene content increases [24]. In addition, the latter study has shown that the thermal expansion coefficients are larger in the case of the graphene-modified asphalt. Nonetheless, it should be noted that the conclusions drawn on from this research were based mainly on molecular simulations rather than laboratory observations. Other studies related to thermal properties have suggested that the use of graphene can produce energy from paved roads [25]. Taking advantage of the large heat capacity

of asphalt materials, a simple method for harvesting this free energy is to embed pipes beneath the road surface [26]. If a fluid such as water is pumped through the pipes it will absorb the heat stored in the pavement and transport it to a point where the thermal energy can be converted into electricity or used to heat a secondary source. In this respect, the addition of small quantities of graphene to the asphalt could enhance the thermal conductivity of the pavement, transforming it into an effective and efficient medium to transfer the stored heat to the piping system. This research, however, is still in progress and there are still no quantitative results or information with respect to whether the heat transfer is still possible in spite of the fact that the flakes are not in contact with each other.

Based on this review, it appears that the positive effect of graphene on asphalt binders has been demonstrated at low temperatures, whilst little conclusive information has been provided with regard to its effects at real temperatures that are expected to occur during the service life of road pavements. Similarly, relatively few quantitative results are available regarding the effect of graphene on the thermal conductivity of the binder, which is one of the most promising features that this modifier can provide. In addition, more information is needed about the optimal characteristics and dosage of the added graphene for bitumen modification. Thus, in order to address this lack of information, the Laboratory of Graphene and 2D materials and the Laboratory of Construction Engineering of the University of Granada (Spain) are working on a joint project whose main objective is to determine the optimal type and dosage of graphene to be used for the production of high performance and multifunctional asphalt binders. The purpose of the present paper is to present those results that focus on the evaluation of graphene-modified asphalt binders, paying special attention to the dosage of graphene used and its effect on both the rheology and thermal properties of the binder.

## 2. Materials and methods

### 2.1. Materials

The graphene materials used for this study were processed using a thermal reduction of graphene oxide in  $\text{H}_2$  ambient. It should be noted that the final properties of graphene-modified materials are highly dependent on the type of graphene used as well as the process by which it is obtained [27]. For this purpose, various tests were carried out in order to establish the optimal graphene flakes, which should have the lowest impurity content possible (the major carbon content), a low aspect ratio L/W (relationship between the length and width of the flake, which should have values of around 1; as the aspect ratio increases, the potential for clustering is increased during mixing), the highest specific surface (which is essential for ensuring a good degree of dispersion in the binder, which in turn guarantees homogeneous thermal and mechanical properties in the material produced [28,29]), good tensile strength, and a competitive production price. The final process used for the production of the graphene is described in the following paragraphs.

The initial material was graphite oxide (GO) flakes, which were prepared from commercial graphite powder using a modified version of the Hummers' method [30]. This method makes use of Hummers' reagents with additional amounts of  $\text{NaNO}_3$  and  $\text{KMnO}_4$ . Typically, concentrated  $\text{H}_2\text{SO}_4$  (115 mL) is added to a mixture of graphite (5 g) and  $\text{NaNO}_3$  (2.5 g), and in our procedure the mixture was cooled down to  $0\text{ }^{\circ}\text{C}$  by means of an ice bath.  $\text{KMnO}_4$  (15 g) was added slowly at small doses to keep the reaction temperature below  $20\text{ }^{\circ}\text{C}$ . The solution was heated to  $35\text{ }^{\circ}\text{C}$  and stirred for 3 h, at which point 3%  $\text{H}_2\text{O}_2$  (1.5 L) was slowly added, giving rise to a pronounced exothermal effect to  $98\text{ }^{\circ}\text{C}$ . The reaction mixture was stirred for 30 min and, finally, the mixture was filtered. The remaining solid material was then washed with de-ionized water and filtered again, this process being repeated until the pH was neutral [30]. Following Kaniyoor et al. [31,32], exfoliation and reduction of GO was carried out as follows: The graphene oxide sample was taken in a boat and placed inside a tubular quartz furnace. The furnace is sealed at both ends with end couplings having provisions allowing for the release of gas. The furnace was flushed with Ar for 15 min, followed by  $\text{H}_2$  for 5 min at room temperature. The temperature was then raised to  $200\text{ }^{\circ}\text{C}$  in the presence of  $\text{H}_2$ . The flow of  $\text{H}_2$  was continued for another 30 min. The furnace was allowed to cool naturally to room temperature. Fig. 1 shows photographs of the obtained rGO (reduced Graphene Oxide), as well as an image of the optical microscopy, where the

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