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Abstract. In this study, a dynamic shear measurement of bitumen using a rotative rheometer between two circular plates have been investigated. The experimental test was examined with pure-bitumen and bitumen-mixed with 10% and 20% of kaolin. In order to understand the behavior of the sample, the rheological measurements were conducted at different temperature 26°C, 50°C and 80°C. A study of the slip conditions was conducted to choose the appropriate plates to prevent the slip phenomena. The effect of the roughness of the faced disks on the viscosity and torque was studied. Furthermore, the evolution of the storage and loss modulus as a function of angular frequencies were examined. The experimental results reveal that pure-bitumen and bitumen-mixed with 10% of kaolin at both 26°C and 50°C presented viscoelastic behavior. However, bitumen-mixed with 20% of kaolin at 26°C becomes consistent and exhibits elastic solid behavior. At 80°C, the storage modulus of bitumen-mixed with 20% of kaolin was higher than the loss modulus, indicating an interaction and modification in macromolecular elements within the sample. Finally, the effect of kaolin addition is described and analyzed.

Keywords: dynamic shear, kaolin, Bitumen, complex modulus, complex viscosity

1 Introduction

Numerous researches have focused on studying the rheological behavior of non-Newtonian fluids between two circular disks. The first analytical description for the compressional rheometer was published by (Phan-Thien 1980). The earliest analysis of oscillatory flow to determine the complex viscosity and the complex modulus of viscoelastic fluid was established by the rheological tests of (Field et al. 1996; Phan-Thien et al. 2000). On the other hand, other researches devoted to the subject of complicated fluid focusing on the rheological characteristics of bitumen. (Farias et al. 2016) conducted a thermorheological study on bitumen, concentrating on the influence of nanocomposites and nanoclay on bitumen resistance and elasticity. They found an increase in complex modulus and a decrease in phase angle in the modified bitumen containing SBS-nanoclays indicating enhanced resistance and offering better protection against significant pavement distresses. They also demonstrated that the addition of CLO organoclay ameliorates the storage stability of polymer modified bitumen at high temperatures. Moreover, (Porto et al. 2019) studied the effect of polymer modified bitumen and their roles in improving road pavement performance and increasing resistance to crack propagation. In the same vein, many researchers (González et al. 2004; Peralta et al. 2013; Soenen et al. 2016; Yuliestyan et al. 2018; Shah and Mir 2020) have carried out studies on the impact of rheological and physical parameters at high temperature, including viscosity, penetration grade, and molecular-interaction, on the behavior of modified-bitumen. Recently, (Kraiem et al. 2022) investigated the squeezing test and the rheological measurements of bitumen and bitumen mixed with kaolin. Their study demonstrated that kaolin improves the rheological characteristics and identified the existence of yield stress using a squeeze-test instead of rheometry. However, the study of complex modulus on the rheological behavior of the sample was not addressed in their discussion. More recently, (Büchner et al. 2023) studied the asphalt binder relaxation at low temperatures using a dynamic shear rheometer. They examined the relaxation-behavior of bitumen to evaluate the impact of aging and rejuvenation on its resistance to cracking. Finally, as far as we know, researchers have conducted rheological measurements on filled-bitumen materials or asphalt, which are commonly used in road construction to enhance their durability and prevent cracking. The intention of this study is to analyze the rheological measurements of a different type of bitumen that is used in roofing and waterproofing applications at various temperatures. Besides, this study investigates the impact of incorporating a new filler (varying percentages of kaolin) into pure-bitumen. The present study is carried out as follows: The second section describes the rheological procedures. The dynamic shear tests are detailed in Section 3. Finally, Section 4 is devoted to the conclusion.

2 Rheological procedures

In this study, pure-bitumen and a blend of bitumen with kaolin at weight percentages of 10% and 20% were used. The rheological behavior of pure-bitumen and bitumen-mixed with kaolin was performed using the ARESG2 'rotational rheometer from TA instrument'. Tests were conducted using two parallel circular disks with a diameter of 10mm, while maintaining a regulated temperature of 26°C, 50°C and 80°C using the Advanced-Peltier system. Figure 1 shows the rheological test of the material between two parallel circular plates.



Fig. 1 Rheological test using ARESG2 with parallel-plate geometry

2.1 Effect of slip conditions

The roughness of the plates used in rheometric tests can induce varied flow conditions: flow with slip condition or adhesive flow. Rheometry measurements for both pure-bitumen and bitumen-mixed 10% and 20% of kaolin were conducted using a rough-surfaced plate to prevent the slip phenomena. In fact, the steady shear test of pure-bitumen at 26°C using a smooth-faced plates, demonstrates the presence of slip, which causes a decrease in torque and viscosity (fig.2(a)) compared to fig.2(b), where the test is conducted with rough plates. That is why rough disks were subsequently used in all experimental tests.



Fig. 2 steady shear test of pure-bitumen with smooth-faced plates (a) and with rough-faced plates (b) at 26°C

3 Dynamic shear tests

3.1 Storage and loss modulus measurements

In the present study, a dynamic shear measurement was examined for pure-bitumen and bitumen-mixed with 10% and 20% of kaolin at various temperature (26°C, 50°C and 80°C). For all the measurements, a low oscillation deformation was maintained below 0.5% within the linear viscoelasticity domain. These tests allowed for the determination of the loss and storage modulus. Figure 3 depicts the evolution of

the storage modulus (G') and the loss modulus (G'') of pure-bitumen as a function of angular frequency. At a temperature of 26° C, it was observed that pure-bitumen behaved as a viscoelastic fluid. Additionally, it was noted that G'' was higher than G' until reaching a critical angular frequency of 63 rad/s. The value of which G'=G'' allowed for the definition of a relaxation time for the sample. Thus, for pure-bitumen, the characteristic time was on the order of 0.016 s⁻¹ at a temperature of 26° C.

By increasing the temperature to 50° C, a predominance of the viscous behavior was observed. At this temperature, it was not possible to reach the intersection point of the G' and G'' curves because the machine is limited to a certain maximum frequency. Consequently, it can be predicted that this intersection may be found at high frequencies. However, at high temperatures (80°C), it was difficult to conduct the oscillation measurements for pure-bitumen due to the existence of a free surface, which may affect the measurement of oscillation torque, strain rate, etc., at the plate edges.



Fig. 3 Storage modulus and loss modulus as a function of angular frequency of pure-bitumen

Figure 4 shows the evolution of the storage modulus (G') and the loss modulus (G') of bitumen-mixed with 10% of kaolin as a function of angular frequency. At a temperature of 26° C, it was observed that bitumen-mixed with 10% of kaolin

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exhibited a viscoelastic fluid behavior. Besides, it was noticed that G'' was higher than G' until reaching a critical angular frequency of 158.5 rad/s. The relaxation time of bitumen-mixed with 10% of kaolin was approximately 0.006s⁻¹, which is smaller than that of pure-bitumen. This indicates that the addition of kaolin facilitated the transition from the solid elastic behavior.

By increasing the temperature to 50°C and 80°C, it was noticed that G'' remained higher than G'. At these temperatures, bitumen-mixed with 10% of kaolin behaved as a viscous fluid behavior.



Fig. 4 Storage modulus and loss modulus as a function of angular frequency of bitumen-mixed with 10% of kaolin

On the other hand, previously published results (Kraiem et al. 2022) have investigated that bitumen-mixed with 20% of kaolin exhibited high consistency and pointed out the difficulty of achieving the steady-shear measurement at 26°C.

Figure 5 illustrates the effect of temperature on the storage modulus and loss modulus for bitumen-mixed with 20% of kaolin. At a temperature of 26°C, G' remained higher than G''. Thus, the sample was considered to be more solid than fluid. This finding confirms the result of (Kraiem et al. 2022).

By increasing the temperature to 50°C, G' was higher than G'', indicating the viscous behavior of the bitumen-mixed with 20% of kaolin. However, at a temperature of 80°C, it was observed that G' was higher than G'' and a plateau in G' began to appear at low frequencies. It is important to note that the appearance of a plateau region has frequently been associated to the development of interaction among the macromolecular constituents of the modified-bitumen which enhance the elastic

response of the fluid. This result is in good agreement with the experimental results of (García-Morales et al. 2006). For this reason, it can be assumed that at high frequencies, the storage and loss modulus got closer until G'' become higher than G' and it was possible that at a temperature of 80°C, the sample may undergo a destruction of its microstructure within the material.



Fig. 5 Storage modulus and loss modulus as a function of angular frequency of bitumen-mixed with 20% of kaolin

3.2 Influence of kaolin percentage addition on rheological behavior

The impact of kaolin addition on the complex viscosity at different temperatures is represented in Fig. 6. As can be seen, the complex viscosity increases with the decrease in temperature for all the samples, both pure-bitumen and bitumen-mixed with kaolin. Therefore, as the percentage of kaolin addition increased (20% compared to 10%), the complex viscosity also increased. This can be explained by the structural modifications caused by the inclusion of kaolin particles, which make the sample more consistent. Furthermore, decreasing the oscillation strain rate expands the gap between the complex viscosity curves of pure-bitumen and bitumen-mixed

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with kaolin. This could be attributed to the significant cohesion property of kaolin on the one hand, and to the frictional force inside the material layer on the other (Karatas et al. 2020). Nevertheless, at a higher strain rate, the complex viscosity profiles of pure-bitumen and bitumen-mixed with kaolin became closer. This indicates that the effect of the bitumen matrix has a predominant effect and that the rheological flow is not influenced by the presence of kaolin particles. On the other hand, as observed, the complex viscosity curve of bitumen-mixed with 20% of kaolin at 80°C increases significantly, which may reveal modification to the structure inside the sample, as previously demonstrated by the complex modulus.



Fig. 6 Complex viscosity of pure-bitumen and bitumen-mixed with kaolin at 26°C, 50°C, and 80°C

4 Conclusion

The current study allowed us to experimentally analyze the dynamic shear measurements of pure-bitumen and bitumen-mixed with 10% and 20% of kaolin between

two circular disks at different temperatures. The influence of the slip conditions was investigated. On the other hand, the storage and the loss modulus were evaluated to characterize the behavior of the sample. The experimental tests showed that the loss modulus was higher than the storage modulus for pure bitumen and bitumen-mixed with 10% of kaolin at all applied temperature. However, at 26°C, bitumen-mixed with 20% of kaolin behaved more like a solid than a fluid. For the same sample, at 80°C, it was noticed that there could be modifications in the macromolecular elements within the sample due to the existence of a plateau in G' at low frequencies, confirming previous research. Moreover, an increase in temperature, decreases the viscosity, while an increase of the percentage of kaolin addition enhances the rheological properties.

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