Impact of water on asphalt mortars with aggregates of different mineralogy

L. Leon & S. Caro

Universidad de los Andes, Department of Civil and Environmental Engineering, Bogotá D.C., Colombia.

X. Sanchez

University of New Brunswick, Department of Civil Engineering. Fredericton, NB, Canada.

ABSTRACT: This work examines the influence of aggregate mineralogy and water exposure on the rheological behaviour of asphalt mortars. Three different mortars, fabricated with a single asphalt binder and three types of aggregates (quartzite, granodiorite, and serpentinite) with the same gradation, were tested after immersion in water for 0, 10, 60, and 160 days. Results show that aggregate mineralogy affects performance under moisture. Quartzite mortars hardened steadily due to binder oxidation and the inert nature of the aggregates. Granodiorite mortars initially stiffened initially but softened over time. Serpentinite mortars showed marked stiffness reductions after 60 days, indicating water-induced damage. In summary, aggregate mineralogy plays a critical role in the early and long-term moisture resistance of asphalt mortars.

1 INTRODUCTION

Moisture damage in asphalt mixtures is characterized by the progressive degradation of its mechanical properties due to the presence of water. This degradation results from loss of adhesion at the asphalt-aggregate interface and a change in the cohesive properties of the asphalt binder (Caro et al., 2007). Addressing moisture damage is crucial to developing durable and sustainable pavements.

Despite extensive research, the mechanisms behind moisture damage are not fully understood. Several theories, including boundary layer, mechanical, electrostatic, chemical and thermodynamic theories, have been proposed to explain these mechanisms (Hefer &Little, 2005; Bagampade et al., 2004). However, there is no comprehensive theory that fully explains the processes involved, so the causal mechanisms remain somewhat speculative (Bagampade et al., 2004).

There is consensus that the chemical composition and mineralogy of aggregates significantly influence the quality of adhesion between binder and aggregate under moisture conditions (Bagampadde et al., 2006; Cala & Caro, 2021; Hefer & Little, 2005; Rice, 1959). Studies have found strong correlations between silicon dioxide (SiO₂) content in the aggregates and the susceptibility to moisture damage, while ferric oxide (Fe₂O₃) and calcium oxide (CaO) promote more moisture-resistant adhesive bonds between aggregate and asphalt (Cala et al., 2019, 2021; Yoon and Tarrer, 1998).

While significant progress has been made in understanding the moisture degradation of the adhesion between binder and aggregates, less attention has been paid to how moisture affects the cohesive properties of asphalt binders, mastics or mortars. Since the durability of the asphalt materials depends on adhesion and cohesion conditions—and moisture affects both equally (Hicks et al., 2003)—, it becomes essential to explore the phenomena simultaneously.

This study aims to gain understanding of moisture damage by examining the effect of water on the mechanical response of an asphalt binder and asphalt mortars. For the mortars, the focus is on the role of aggregate mineralogy in such response. This work complements previous efforts on the change in the cohesive properties of mastics with different aggregates subjected to moisture conditions (Leon & Caro, 2023). Mortar samples were prepared using a single asphalt binder and fine aggregates with three different mineralogical compositions. The asphalt binder and mortar samples were submerged in water for different periods of time. The linear viscoelastic properties of the materials were quantified using dynamic shear rheometer (DSR) tests and Fourier transform infrared spectroscopy (FTIR) was performed on the binder samples to track the chemical changes induced by water exposure.

Studying the changes caused by water in the properties of asphalt binder and mortar separately, makes it possible to isolate the effect of water on the asphalt binder and on the asphalt-aggregate system. This approach provides a comprehensive assessment of

moisture-induced degradation processes affecting both binder cohesion and binder-aggregate adhesion.

2 MATERIALS AND METHODS

An asphalt binder with a penetration grade of 60–70 (mm/10) was selected along with three aggregates with different chemical compositions: serpentinite (SPT), quartzite (QTZ), and granodiorite (GRT).

The use of a single binder allows for the isolation of the effect of aggregate mineralogy. The selected aggregates cover a different lithological spectrum, ranging from a mafic rock (SPT) to a felsic rock (QTZ), and including an intermediate rock (GRT). Aggregates had low porosity and similar specific gravities which helps ensure fair comparisons in the mortar designs. Specific gravity tests performed following the ASTM D854-10 resulted in values of 2.73, 2.75, and 2.74 for SPT, QTZ, and GRT, respectively. Additionally, X-ray fluorescence (XRF) testing was conducted in accordance with ISO 12677-2011 to determine the chemical compositions of the rocks. The oxide composition of the rocks is listed in Table 1.

Table 1. Chemical composition of the aggregates studied (% by weight total).

	Aggregate		
	QTZ	GRT	SPT
Oxide	% Weight total		
SiO ₂	98.18	65.75	40.21
Al_2O_3	1.09	15.26	7.81
Fe_2O_3	0.09	4.09	10.24
MgO	0.01	2.45	23.45
CaO	0.06	3.77	6.35
TiO_2	0.07	2.68	0.42
P_2O_5	0.01	0.14	0.04
SO_3	0.00	0.01	0.23
Volatiles lost after igniting	0.46	1.63	9.29
Other basic oxides	0.07	4.14	1.96

2.1 Design and fabrication of mortar samples

A representative mortar of the fine fraction of a dense-graded hot dense asphalt mix with maximum nominal aggregate size of 19 mm was designed and prepared. The mortar includes the binder and the material passing through the #16 sieve (1.18 mm). The asphalt binder content of the mortars was 9.75%, a value derived from estimating the amount of binder present in the fine fraction of the dense mix. The target air void content for the compacted specimens was 10%.

The maximum theoretical specific gravity (G_{mm}) of each type of mortar, computed according to the AASHTO T 209–12 standard, was 2.38 for SPT, 2.41 for QTZ, and 2.43 for GRT.

Cylindrical specimens measuring 50 mm in height and 13 mm in diameter were fabricated individually using a metallic mould. The asphalt binder and aggregates were preheated and mixed at 160 °C, while the

steel mould was preheated to 140°C. The loose mortars were subjected to short-term aging for two hours in an oven at a compaction temperature of 140°C to simulate the natural aging during the production and placement of asphalt mixtures.

The loose mortar was then poured into the preheated mould and compacted in a press at a pressure of 0.80 MPa for 45 minutes at room temperature –approximately 20°C– to achieve the desired density and void content, using the procedure proposed by Caro et al. (2014). Finally, metal holders were placed at the ends of specimens to facilitate their installation in the rheometer.

2.2 Moisture conditioning

Asphalt binder and mortar samples were submerged in distilled water at room temperature, avoiding the incorporation of high temperatures or pressures since they could act as catalysts for more complex chemical reactions that would hinder the effect of water in the materials. In the case of the asphalt binder, 3 g of binder were poured in multiple silicone moulds with a cavity size of 43.2 mm (L) × 25.2 mm (W) × 2.8 mm (H) and the moulds were submerged in water. In the case of the mortars, the specimens were directly submerged in water. The selected conditioning times were 0, 10, 60 and 160 days. Each specimen was uniquely assigned to a specific conditioning time and remained continuously under water throughout its designated period.

2.3 Fourier Transform Infrared Spectroscopy characterization of the asphalt binder

To determine the changes in the chemical composition of the asphalt binder samples, FTIR testing using an Attenuated Total Reflectance (ART-FTIR) cell was performed at each conditioning period. FTIR indices were calculated by integrating the absorbance over defined wavenumber ranges for oxidation-related functional groups (e.g., carbonyls and sulfoxides).

2.4 Frequency and temperature sweep tests

Four replicates were tested at each conditioning period (i.e. 0, 10, 60, and 160 days in water) to determine the linear viscoelastic properties of asphalt binder and mortars. The oscillatory tests consisted of frequency and temperature sweep procedures under a controlled percentage angular strain of 0.01 for the asphalt binder and 0.00075 for the mortar. These strain values were selected after verifying that the materials were in the linear viscoelastic range based on oscillatory shear deformation sweep tests.

The temperature and frequency sweep tests were performed in a temperature range between 25°C and 65°C with steps of 10°C and frequencies between 1

and 30Hz with steps of 1 Hz using an TA AR2000ex rheometer with 8 mm parallel plate geometry for the asphalt binder and with a solid geometry for the mortar specimens.

3 RESULTS AND DISCUSSION

3.1 *Linear viscoelastic and chemical changes of the asphalt binder with water conditioning time*

The master curves of the average dynamic shear modulus ($|G^*|$) of the asphalt binder at a reference temperature of 25°C in the different water conditioning periods are presented in Figure 1.

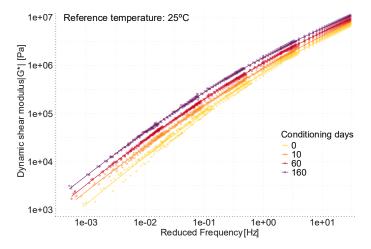


Figure 1. Master curves of $|G^*|$ of the asphalt binder at 25°C

The results show a significant increase in stiffness with increasing conditioning time. Specifically, compared to the unconditioned samples, the average modulus increased by 16.7% after 10 days, 39.8% after 60 days, and reached 69.1% after 160 days of water immersion. Statistical testing at a significance level of 0.05 confirmed that these changes were significant, particularly for the conditioning periods of 60 and 160 days (p-value < 0.01 and p-vale < 0.001, respectively).

Chemical analysis using the ART-FTIR results revealed significant changes in the functional groups of the asphalt binder after conditioning in water for 0, 10, 60, and 160 days, as observed in Figure 2.

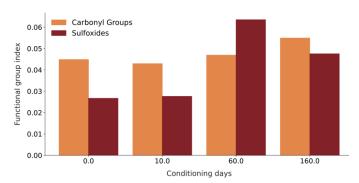


Figure 2. Functional group indices of the asphalt binder.

Despite the fact that water immersion limits oxygen availability and environmental temperatures

could restrict the required energy for oxidation to form additional polar fraction, ART-FTIR results show a progressive increase in carbonyls and sulfoxides groups. The observed average growth rates were approximately 0.241% per day for carbonyl groups and 0.776% per day for sulfoxides. Since measurements were taken only at 0, 10, 60, and 160 days, these values represent average growing rates rather than linear trends. Carbonyls exhibited the highest correlation with the dynamic shear modulus (r=0.94) and sulfoxides (r=0.64), indicating that oxidation and the formation of polar compounds are the primary factors responsible for the observed hardening.

3.2 Linear viscoelastic changes of the asphalt mortars

Figure 3 illustrates the evolution of the mean modulus values ($|G^*|$) for the three mortars (QTZ, SPT, and GRT) subjected to water conditioning. Error bars represent the 95% confidence interval of the mean.

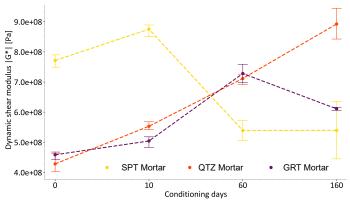


Figure 3. Variation of $|G^*|$ as a function of conditioning time in QTZ, SPT, and GRT mortars.

For the mortar with QTZ, $|G^*|$ increased consistently with water conditioning. After 10 days, there was an increase of 34.9%, followed by further increases of 22.7% and 26.4% at 60 and 160 days, respectively. This stiffening progress may be mainly due to the oxidative aging of the asphalt binder.

The results of $|G^*|$ for the mortar with GRT show an initial increase of 8.7% during the initial 10 days. The stiffening became more pronounced after 60 days, with an increase of 40.4% compared to the initial value. However, a reduction of 14.5% was reported between the 160- and 60-days period, although $|G^*|$ remained higher than in the dry state. This suggests that while short-term water exposure stiffened the GRT mortar due to asphalt binder oxidation, prolonged immersion could start degrading the adhesive properties between the binder and the aggregates.

In the case of the mortar with SPT, $|G^*|$ increased by 17.1% after 10 conditioning days. However, a significant reduction of 40.9% was observed at 60 days. At 160 days, there was a slight recovery of 2.2%, but the value remained below the unconditioned modulus. Similar to the GRT mortar, prolonged water

immersion negatively impacted the mechanical integrity of the material, likely due to adhesive degradation.

The effect of water conditioning was statistically significant (*p*-value<0.001) in all cases, indicating that immersion time substantially influences the mechanical properties of the mortars. Similarly, the aggregate type exhibited a highly significant effect (*p*-value<0.001), confirming that the mineralogical composition of the aggregate impacts the properties of the mortar. The significant interaction between conditioning time and aggregate type (*p*-value<0.001) suggests that the impact of water conditioning varies with the type of rock.

3.3. Additional considerations on QTZ mortars

It is noteworthy that despite being associated with aggregates that typically show higher moisture susceptibility due to weaker asphalt-aggregate adhesion (Cala et al., 2021), QTZ mortars did not display signs of adhesive damage within the studied conditioning periods. This result contrasts with expectations and previous findings on more basic and intermediate rock types.

Several factors may contribute to this apparent contradiction. For instance, differences in the filler particle size distribution (passing sieve # 200) were not fully controlled in this study and could have led to a denser internal structure in the QTZ mortars, slowing down water infiltration and delaying the onset of adhesion loss. Additionally, the high silica content in quartzite may foster stronger initial interactions with the increasingly polar asphalt species formed during oxidation, temporarily reinforcing the asphalt-aggregate bonds. While these hypotheses provide plausible explanations, further research—incorporating more controlled filler gradations, advanced pore structure characterizations, and detailed chemical analyses of the asphalt-aggregate interface—is needed to fully understand the interplay of microstructural and chemical factors delaying moisture-induced damage in QTZ mortars.

4 CONCLUSIONS AND RECOMMENDATIONS

This study evaluated the effect of water conditioning on the dynamic shear modulus of one control asphalt binder and asphalt mortars fabricated with three aggregates with different mineralogy. The materials were subjected to four moisture conditioning periods of 0, 10, 60 and 160 days of water immersion.

Results show that aggregate mineralogy plays a key role in the moisture susceptibility of asphalt mortars. The mechanical response of mortars under water exposure is governed by a complex interaction between the chemical changes of the asphalt binder and the intrinsic properties of the aggregates.

Also, the impact of moisture on the mechanical properties of the asphalt mortars does not follow a clear trend with water immersion. While the serpentinite mortars exhibited rapid mechanical degradation, granodiorite mortars initially stiffened before softening over time, and quartzite mortars remained structurally stable throughout time.

These results suggest that moisture effects may be delayed based on the mortar's microstructure, showing the importance of evaluating not only the chemical composition of the aggregates but also the particle size distribution and other relevant properties.

5 REFERENCES

- Bagampadde, U., Isacsson, U. & Kiggundu, B.M. (2004). Classical and contemporary aspects of stripping in bituminous mixes. Road Materials and Pavement Design 5(1): 7-33.
- Bagampadde, U., Isacsson, U. & Kiggundu, B.M. (2006). Impact of bitumen and aggregate composition on stripping in bituminous mixtures. Materials and Structures 39(287): 303-315
- Cala, A., Caro, S., Lleras, M., & Rojas-Agramonte, Y. (2019). Impact of the chemical composition of aggregates on the adhesion quality and durability of asphalt-aggregate systems. Construction and Building Materials, 216, 661–672.
- Cala, A. & Caro, S. (2021). Predictive quantitative model for assessing the asphalt-aggregate adhesion quality based on aggregate chemistry. Road Materials and Pavement Design.
- Caro, S., Masad, E., Bhasin, A., & Little, D. N. (2008). Moisture susceptibility of asphalt mixtures, Part 1: Mechanisms. International Journal of Pavement Engineering, 9(2), 81–98.
- Caro, S., Sánchez, D. & Caicedo, B. (2014). Methodology to characterise non-standard asphalt materials using DMA testing: application to natural asphalt mixtures. International Journal of Pavement Engineering 16(1): 1-10.
- Hefer, A. & Little, D. (2005). Adhesion in bitumen-aggregate systems and quantification of the effects of water on the adhesive bond. Research Report ICAR-505-1.
- Hicks et al. (2003). Moisture sensitivity of asphalt pavements: a national seminar. San Diego, California. Transportation Research Board The National Academies Press: Washington, D.C.
- ISO 2011. ISO 12677:2011: Chemical analysis of refractory products by X-ray fluorescence (XRF) Fused cast-bead method. Geneva: ISO.
- León, L. & Caro, S. (2023). Influence of water on the rheological properties of asphalt mastics fabricated with fillers having different mineralogical compositions. In Airfield and Highway Pavements 2023: 139-149.
- Rice, J. (1959). Relationship of aggregate characteristics to the effect of water on bituminous paving mixtures. Transportation Research Record: Journal of the Transportation Research Board 59: 1-10.
- Yoon, H. H., and A. R. (1988). Tarrer. Effect of Aggregate Properties on Stripping. In Transportation. Research Record: Journal of the Transportation Research Board, 1171: pp. 37-43.