The internal structure of bitumen : original way using Atomic Force Microscopy and ultramicrotomic section

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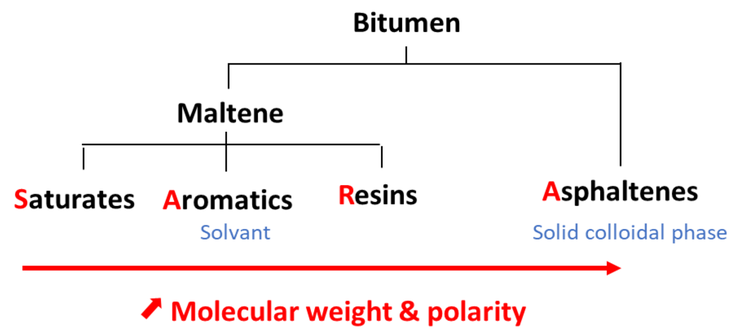
Context

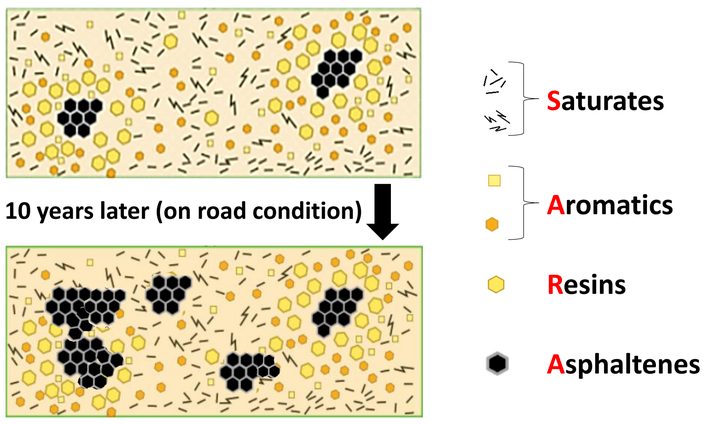
Bitumen is a viscoelastic material: it behaves like a fluid at high temperatures and like a solid at low temperatures. It is made of a complex mixture of hydrocarbons, divided into four fractions classified by their molecular weight and polarity.

Over time, under road conditions, bitumen undergoes oxidative aging. Its rheological properties change, making it harder and more brittle, which eventually leads to road cracking.

To address this issue, the NANOroad project aims to link the micro- and nanostructures of bitumen with its macroscopic mechanical properties in order to improve recyclability and resistance to aging—contributing to the sustainable development of road materials.

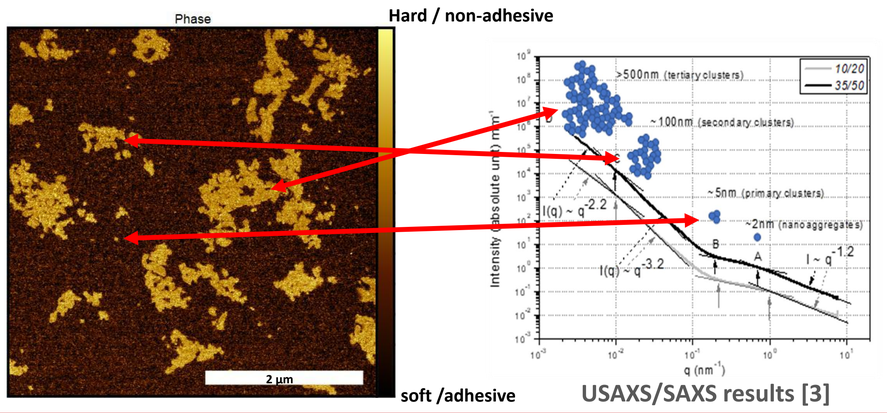


Evolution of colloidal/micellar of bulk structure bitumen



Modified from [2]

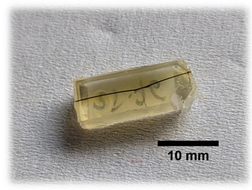
Consistent with indirect observation results from SAXS

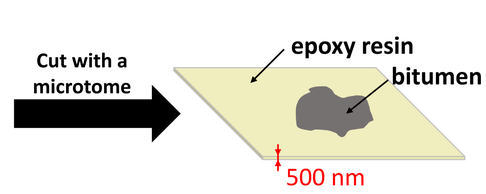


USAXS/SAXS results [3]

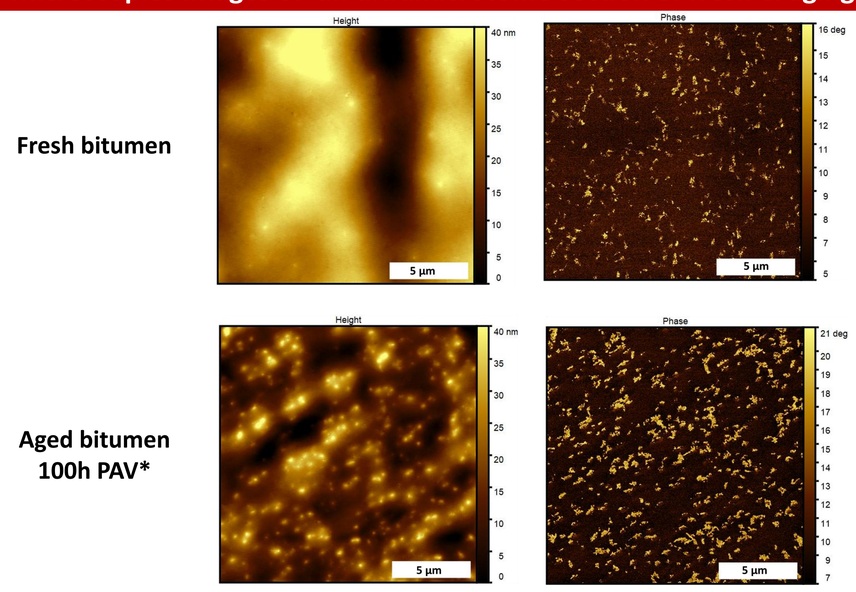
Ultramicrotomic section

To investigate the internal structure of bitumen, we prepare an artificial surface using a microtome, which slices an ultrathin section (approximately 500 nm) from epoxy resin blocks in which a bitumen stick has been embedded.



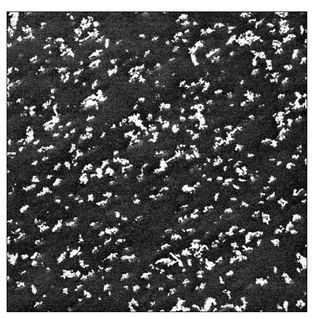


Atomic Force Microscopies images of an ultramicrotome section of fresh & aging bitumen

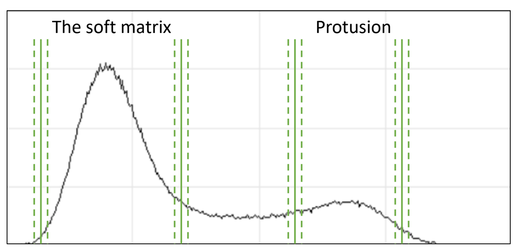


Contrast phase images obtained in tapping mode provide information about the internal structure of bitumen and reveal an adhesive and soft matrix with harder protrusions on the surface. As bitumen ages, these protrusions become larger and appear more aggregated. This observation is consistent with the aggregation of asphaltenes in agreement with SAXS and mechanical data.

Mask for numerical analysis with Gwyddion software

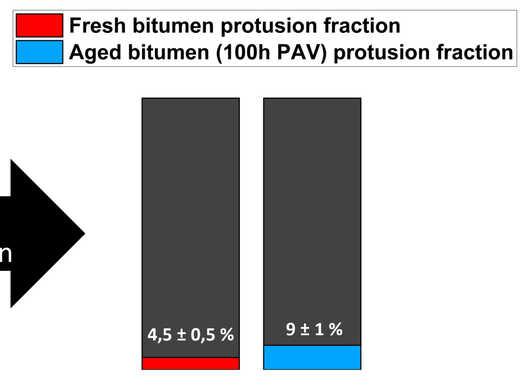


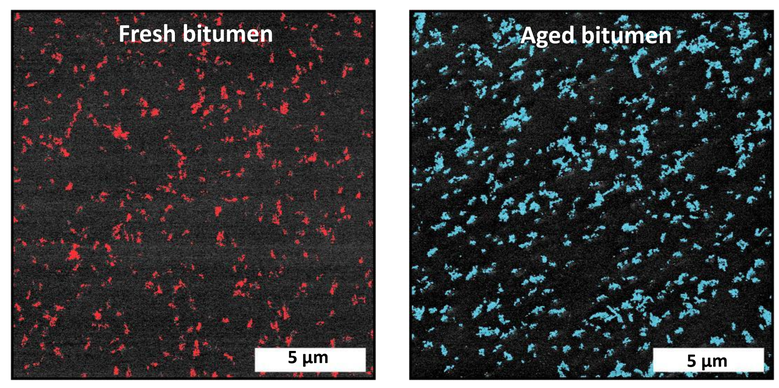
Histogramm of the contrast phase image:



Otsu's automatic thresholding method separates the values into two classes by minimizing the intra-class variance.

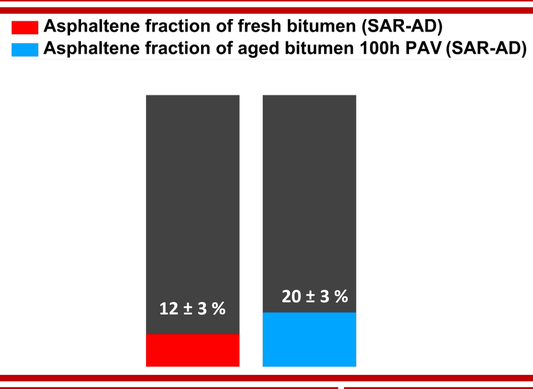
Statistical determination





Chemical analysis of bitumen's fractions : SAR-AD

Saturates, aromatics, and resins—Asphaltene Determinator (SAR-AD) is a separation technique developed by the Western Research Institute. It combines precipitation with chromatographic analysis to fractionate petroleum, heavy oil, bitumen, or asphalt into distinct solubility fractions.[4]



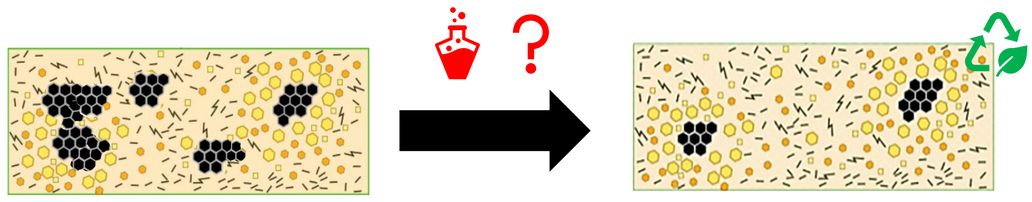
	FRESH BITUMEN	AGED BITUMEN [100H PAV]
SATURATES (± 1%)	10	10
AROMATICS (± 1%)	65	55
RESINS (± 1%)	15	17
ASPHALTENES (± 3%)	12	20

Conclusion

- AFM outcomes consistent with chemical results obtained by SAR-AD on fraction volume proportions
- AFM outcomes consistent with the theoretical model of bitumen internal structure and indirect observations (SAXS/USAXS)
- Direct observation of the internal structure of bitumen ⟹ Understanding the mechanism of aging

Outlook

What Is the Effect of Recycling Agents on Bitumen?



Recycling agents may restore bitumen's rheological properties to their original state during pavement application. Our AFM approach will allow us to assess their impact on bitumen's internal structure and improve understanding of the aging mechanism.

References

[1] PAV : Pressure Aging Vessel process - To simulate the long-term aging of bitumen, samples are subjected to ~100–110 °C and 2.1 MPa air pressure, reproducing in the laboratory the effects of several years of oxidation.
[2] Bitume.info 2005, p7
[3] F. Lahjiri & al.- *Energy & Fuels* 2023 *37* (12), 8444-8455
[4] J. Adams & al. – *Energy & Fuels* 2015, 29, 2774–2784
Project from ANR Nanoroad

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