

# Examination of Adhesion between Aggregate and Bitumen in Asphalt Pavement

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This article is a summary based on the BSc -thesis at NTNU in Spring 2024. Further details can be found in the thesis

## INTRODUCTION

An asphalt pavement mixture is a relatively complex composite material when compared to its concrete counterpart. The standard composition of an asphalt pavement mixture consists primarily of bitumen/crude oil, rocks/aggregates, filler (pulverized limestone) and amine additives. The interactions between components in a mixture, alongside manufacturing parameters, will determine the physical and chemical properties of the overall mass, before being exposed to loading in field. It is thus detrimental to have an understanding of how the mixture will behave in the field of traffic.

A major property of asphalt is the local adhesion at the aggregate-bitumen interface and overall cohesion of the entire mixture. These properties

define the structural integrity of any asphalt mass. It is therefore crucial to develop an understanding and approach to measuring this property. Testing and analysis of aggregate-bitumen adhesion and mixture cohesion is comprehensive and will often require several theories from different fields of science.

The major topic of research in this thesis is how the aggregate surface contributes in the adhesion to bitumen polarized by cationic amine additives. Essentially a mapping of two asphalt aggregates, comparing their geochemical properties and how this affects adhesion to bitumen.

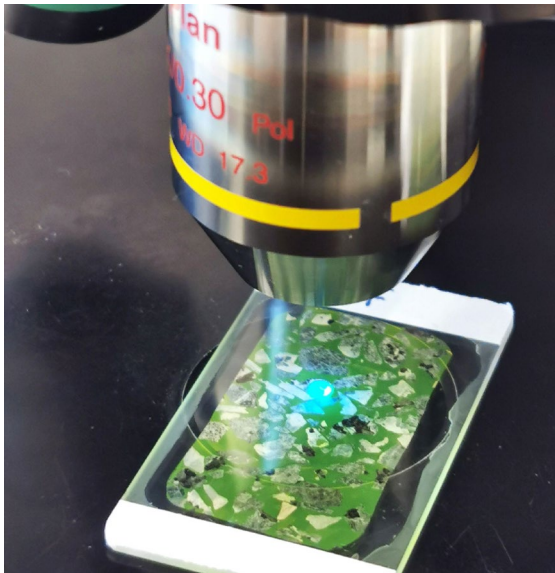
Properties such as net surface charge, mineralogy (XRD), micro structure (SEM), micro cracks (thin section) and water retention were characterized with various experiments.

The two aggregates tested were a gabbro and a feldspar-rich syenite. The binder was a 70/100 bitumen with cationic amine additives.

## METHOD

### Thin section microscopy

Examining micro-cracks utilizes a thin section cast in fluorescent epoxy along with a microscope equipped with fluorescent light (Nikon Eclipse E600 Spot Image). The method is to examine each rock in the thin section, pinpoint areas with micro-cracks and then quantify the overall severity of the sample.



*A thin section of a sample cast in fluorescent epoxy being examined in a microscope with fluorescent light*

### Polished surface SEM

In order to observe the samples in SEM they must be electrically conductive. This is achieved by gilding the samples in a plasma chamber with a nano-layer of gold and taping on copper tape to all sample surfaces.



*Photo of polished aggregate samples before and after SEM preparation.*

### Semi-quantitative phase analysis of minerals in aggregates with XRD

The aggregate sampling size should reflect the same fraction distribution used in the asphalt recipe, spanning from 0-11mm diameter. In a stone crusher the representative sample is crushed to an average size of 1mm. Lastly the powder is micronised to less than 10 microns using a micronizing mill.

In the XRD instrument (Bruker D8 Advance Serie 2 XRD) the samples undergo radiation for 69 minutes each. The angle of incidence is set between 3-80(2 $\theta$ ) at a 0.0116 (2 $\theta$ ) step and a 0.6 second interval. The filament used for radiation is cobalt (Co-K $\alpha$ 1) with a characteristic wavelength of 1.79Å.

### Semi-quantitative chemical analysis of aggregates with XRF

The same powder (10 micron) that came from the disc mill for XRD is also used for XRF. The XRF machine is programmed to follow strict patented systems of parameters according to sample preparation, in this case the system is called WROXI. The program analyzes the sample for main- and trace elements. When the XRF results are done the quantitative percentage values must be modified to

include the LOI-mass which was lost during pre-heating of the powder.



Photo of preheated powder and glass sample used for XRF.

## RESULTS

Both aggregates seem to exhibit the same amount of overall micro-crack occurrences in thin-section microscopy. The only visible trend is that micro-crack propagation seems to occur along grain boundaries of a "black mineral grain", most likely ferrite ( $Fe_2O_3$ ), in the feldspar (syenite) aggregate, see example 2,3,5 and 6. Ultimately the occurrence of micro-cracks is below moderate in both thin sections.

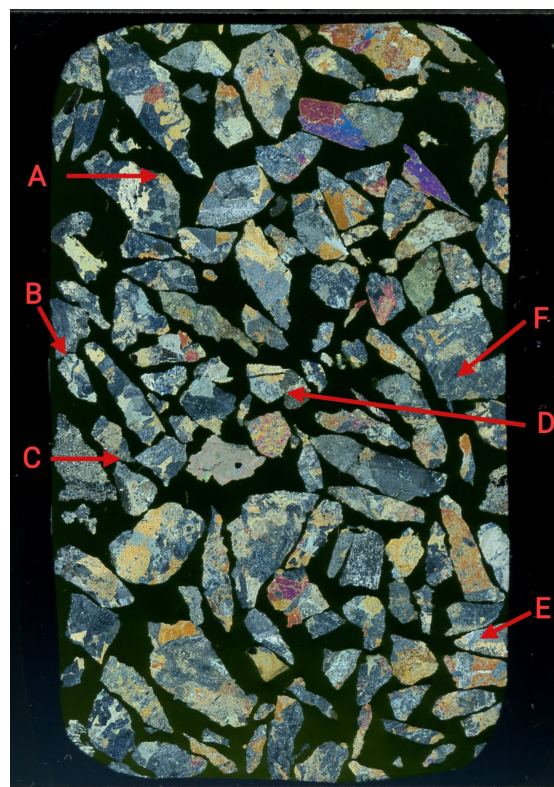
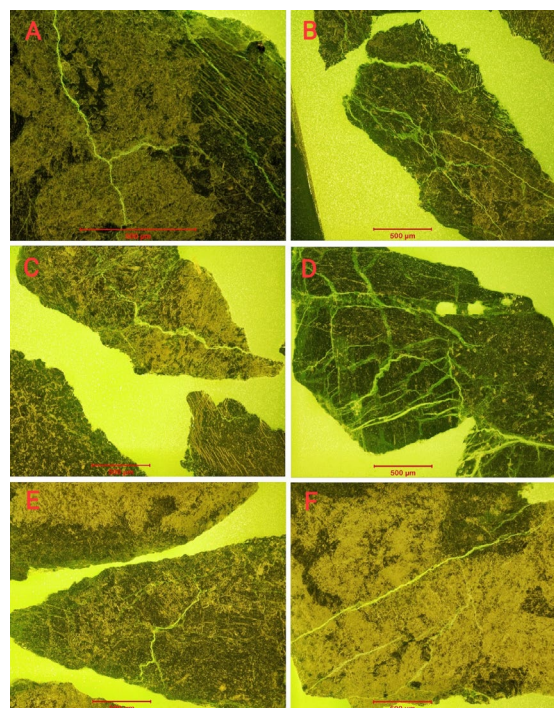


Photo-scan of gabbro thin section with overview of observed micro-cracks.



Observed occurrences of micro-cracks in gabbro thin section

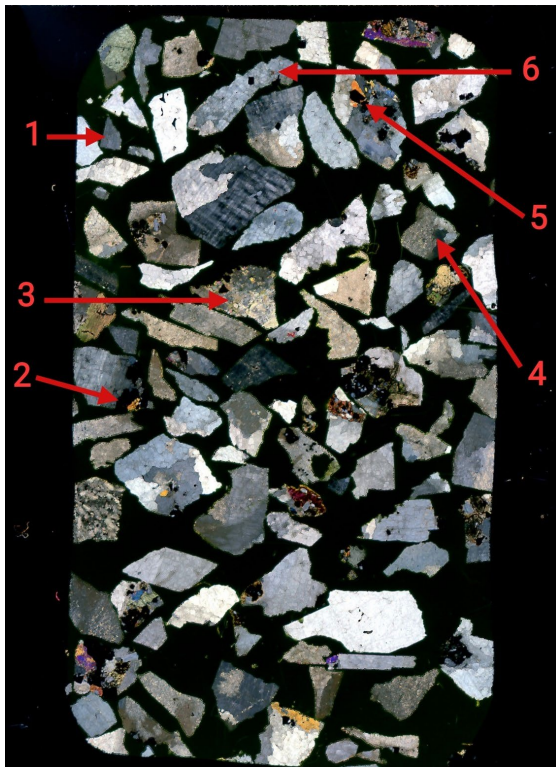
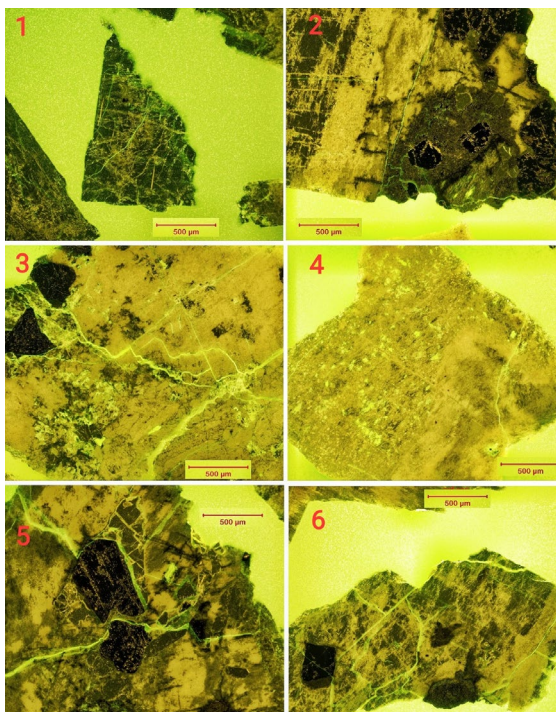


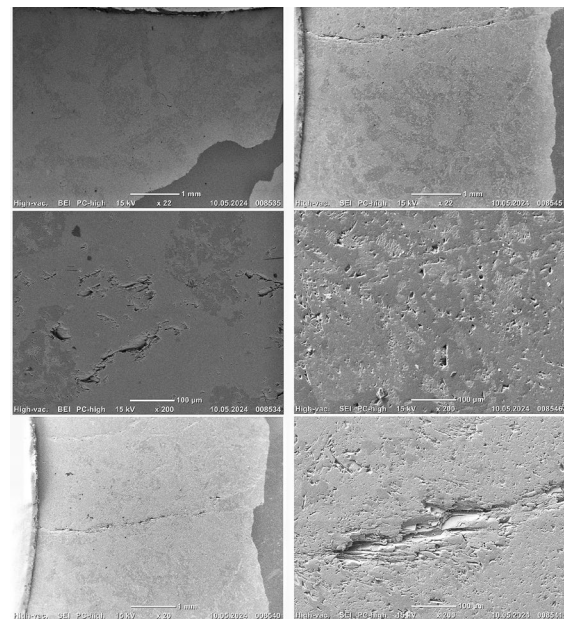
Photo-scan of feldspar aggregate thin section with overview of observed micro-cracks



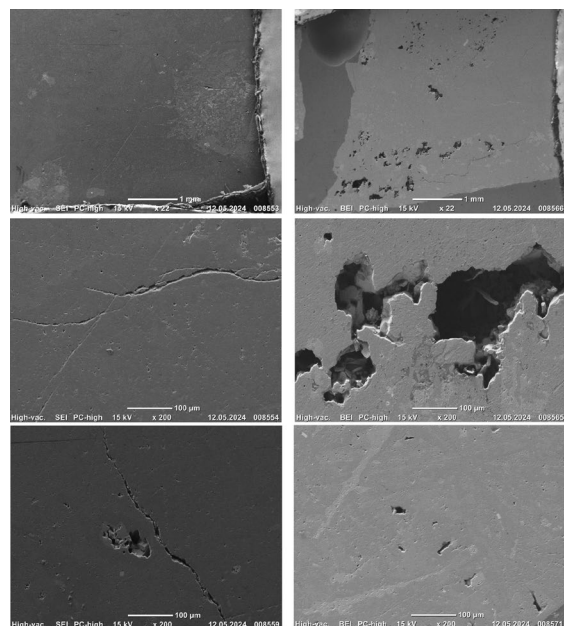
Observed occurrences of micro-cracks in feldspar aggregate thin section. A trend of micro-crack propagation appears along grain boundaries of magnetite (dark mineral) in 2,3,5 and 6.

and 6.

Examination of polished cross-section for the two aggregates in SEM shows observed higher occurrence of micro-cracks and pores in the feldspar aggregate compared to the gabbro aggregate. The polished cross-section of the gabbro exhibits a seemingly rougher texture than the polishes cross-section of the feldspar (syenite).



SEM photos of micro-structure on polished cross-section of gabbro aggregate.



SEM photos of micro-structure on polished cross-section of feldspar (syenite) aggregate

The quantification of mineral structures with XRD of both aggregates can be interpreted with the x-ray diffractograms.

Mineral	Gabbro	Syenite
Hornblende	40-43%	2-3%
Clinozoisite	25-26%	5%
Albite	19-20%	60-64%
Chlorite	9%	-
Microcline	2-3%	16-19%
Magnetite	-	4-5%
Muscovite	-	2-3%

XRD quantification of mineral structures in both gabbro and syenite aggregate.

Results of the most reoccurring compounds from chemical quantification using XRF are shown in the following table. The silica content shows that the gabbro is a basic rock and the syenite is an intermediate rock. Syenite has a higher amount of alkali metals (potassium and sodium), while gabbro has a higher amount of alkaline earth metals (magnesium and calcium). Also, the syenite has a higher "loss of ignition" amount than the gabbro.

Compounds	Gabbro	Syenite
SiO <sub>2</sub>	48.4%	56.2%
Al <sub>2</sub> O <sub>3</sub>	14.9%	19.5%
CaO	11.8%	4.76%
Fe <sub>2</sub> O <sub>3</sub>	9.68%	5.29%
Na <sub>2</sub> O	2.79%	5.86%
MgO	8.24%	1.22%
K <sub>2</sub> O	0.06%	3.59%
LOI	0.91%	2.55%

XRF chemical quantification of both aggregates.

In summary the findings align with mineral theory of why silica- and alkali content along with refractive index of syenite contribute to the reduced adhesion in the feldspar mixture. Examining micro-structure found that the syenite

exhibited more micro-cracks and porosity than the gabbro.

## CONCLUSION

In conclusion, the aggregates display different water retention. The syenite (feldspar) aggregate exhibits a higher water retention value and requires longer drying time than the gabbro aggregate. By examining polished cross-sections of the aggregates in SEM, it was found that the syenite aggregate displayed higher occurrences of micro-cracks and pores than the gabbro aggregate.

Syenite falls below the experimental threshold, set at 1.6, relating the mineral refractive index to the susceptibility to stripping. This finding aligns with the results found in the experimental testing of stripping with ITSR, resulting in the feldspar (syenite) mixture being more inflicted by stripping than the gabbro mixture.

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