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Contribution of the SAR-AD™ chromatographic separation to assess bitumen aging

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Introduction

For decades, there has been a lot of interest in bitumen aging. Study [1] reported that this process results chemically in a decrease of the aromatic fraction content and an increase of the asphaltenic fraction content. Their evolutions are often observed by separating bitumen according to the SARA chromatographic method [2] (Saturates, Aromatics, Resins and Asphaltenes). Recently, the Western Research Institute developed an High Performance Liquid Chromatography method called SAR-AD™ [3] (Saturates, Aromatics, Resins and Asphaltenes Determinator) which allows separating bitumen into 8 families with increasing polarities (versus 4 for SARA). The objective of this study is to present the different benefits of SAR-AD™ method in terms of aging mechanisms understanding.

Bitumen

3 bitumen selected for their different origins and chemical compositions.

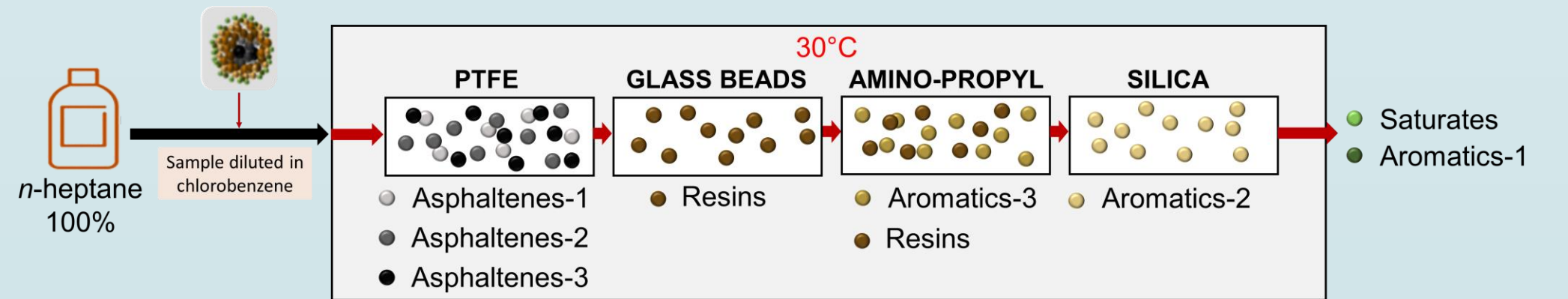|	Bitumen S	Bitumen G	Bitumen F
Penetration at 25°C (mm/10)	70	77	115
Softening Point (°C)	48.0	48.0	46.2
Kinematic Viscosity at 100°C (mm²/s)	4464	2056	507
Wax Content by DSC (w%)	0.7	5.2	8.8
Asphaltene Content (w%)	15.6	10.2	1.7

Pressure Aging Vessel

Simulation of binder aging after several years in service.
PAV conditions: 100°C, 21 bar during 24 and 48 hours.
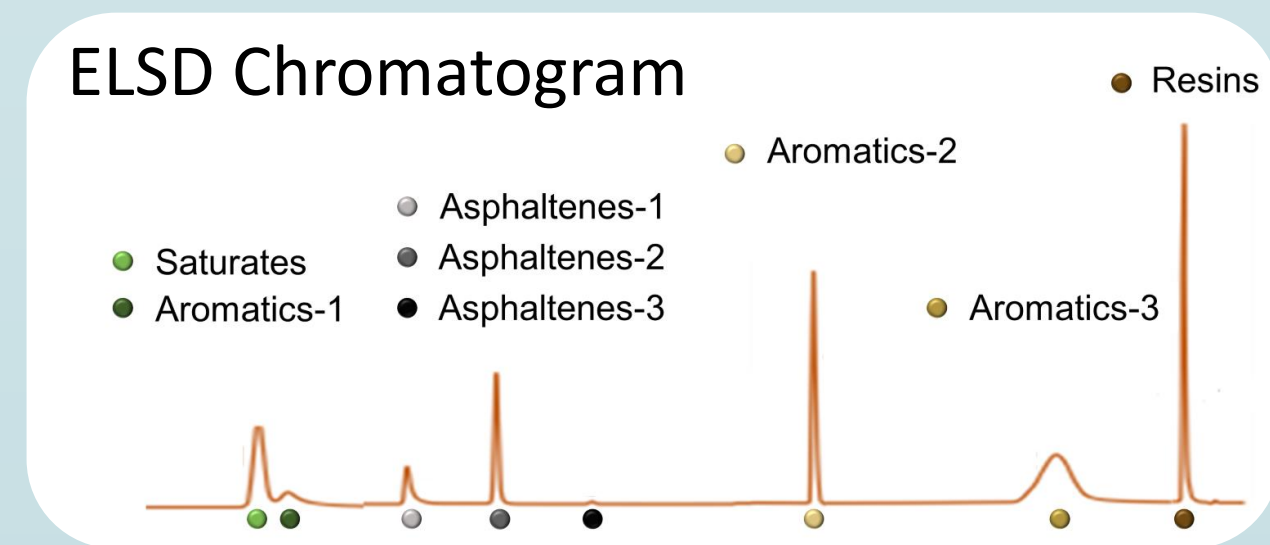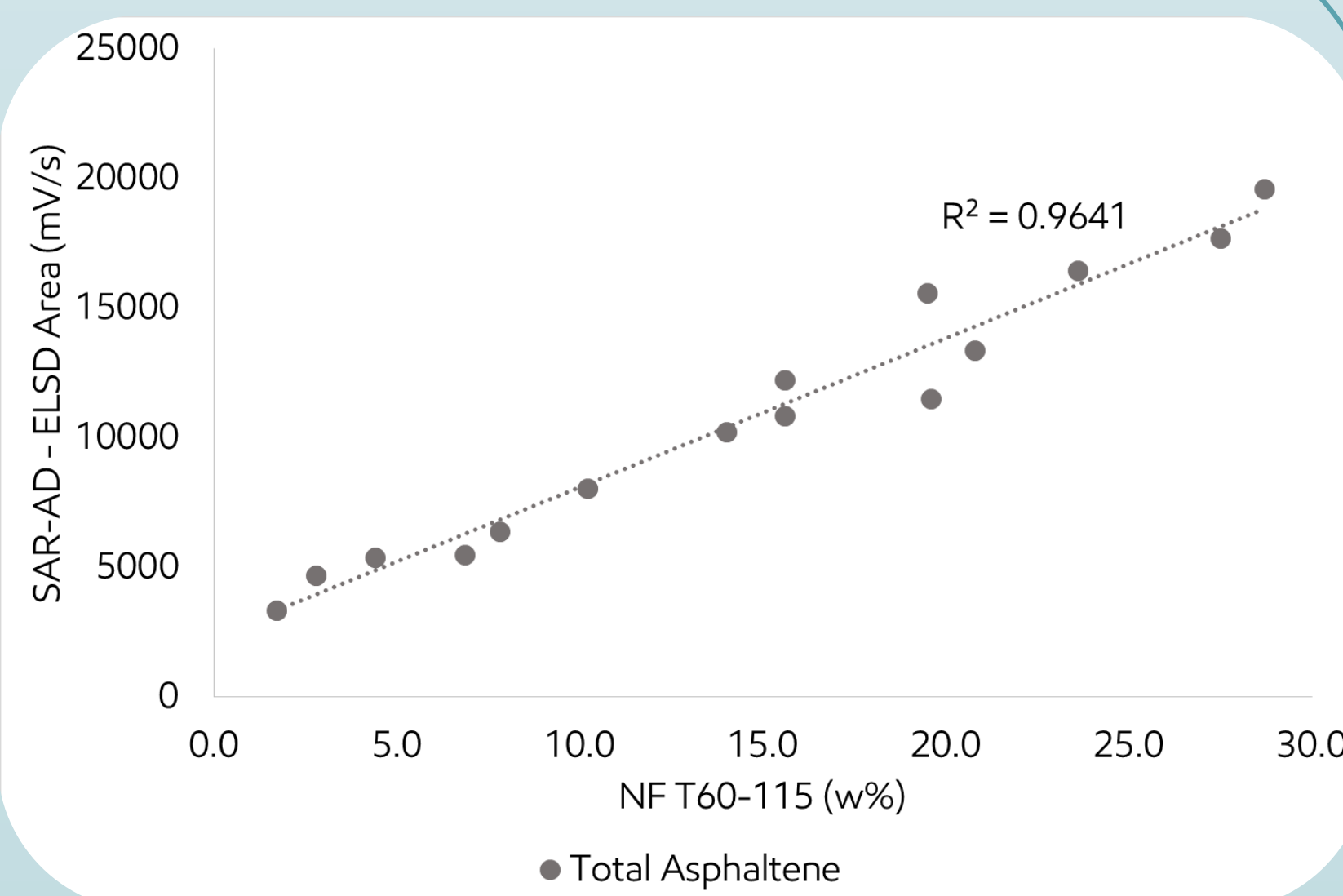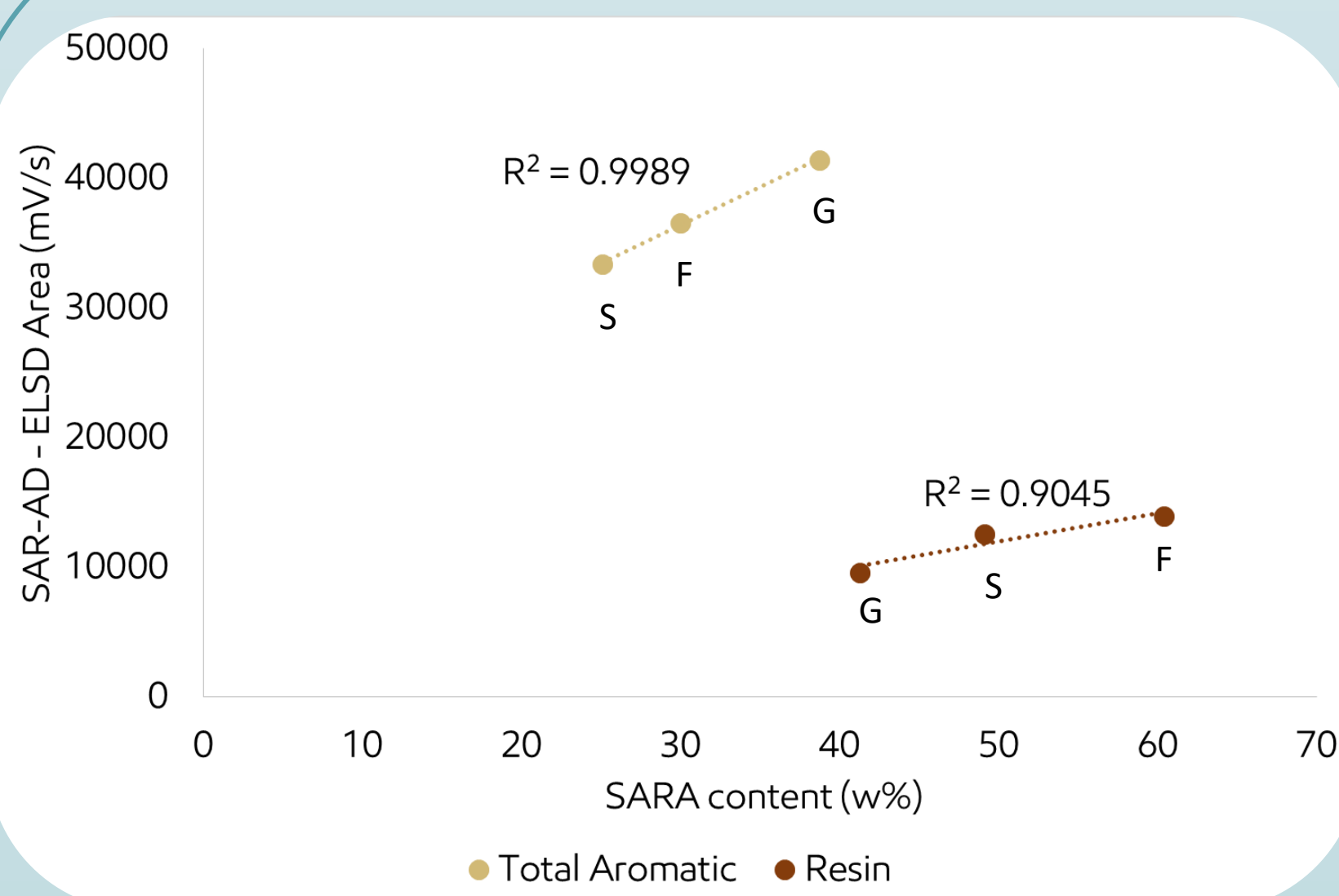
SAR-AD™
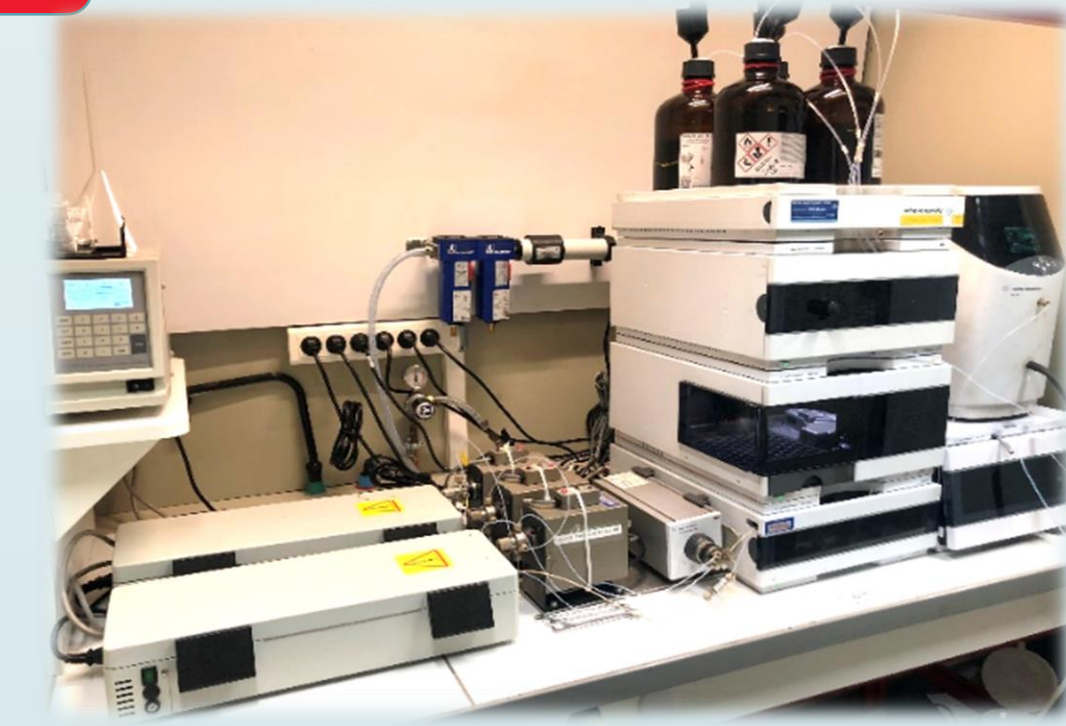
1. Separation of bitumen sample in 8 families as a result of the affinity between molecules and stationary phases / solvents.

2. Molecules are analyzed with an Evaporative Light Scattering Detector (ELSD).


SAR-AD™ vs SARA

For the three neat bitumen, the amount of aromatics and resins obtained by SARA separation correlate with SAR-AD™ ELSD areas.

For bitumen S, G and F before and after exposure to several aging durations, asphaltene content from NF T60-155 standard and asphaltene content from SAR-AD™ ELSD areas are correlated.
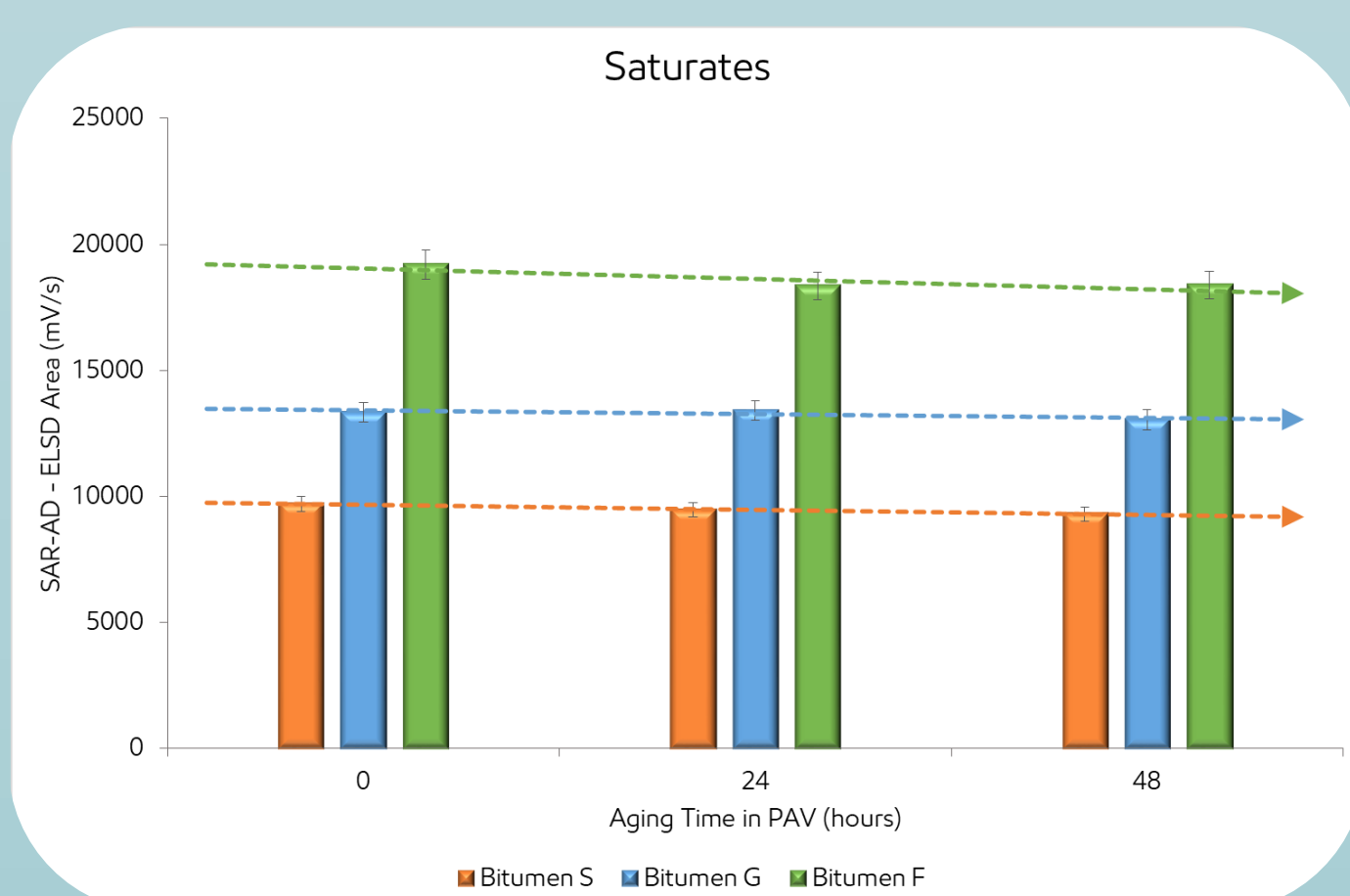
Bitumen chemistry

Bitumen molecules polarity increase [4]
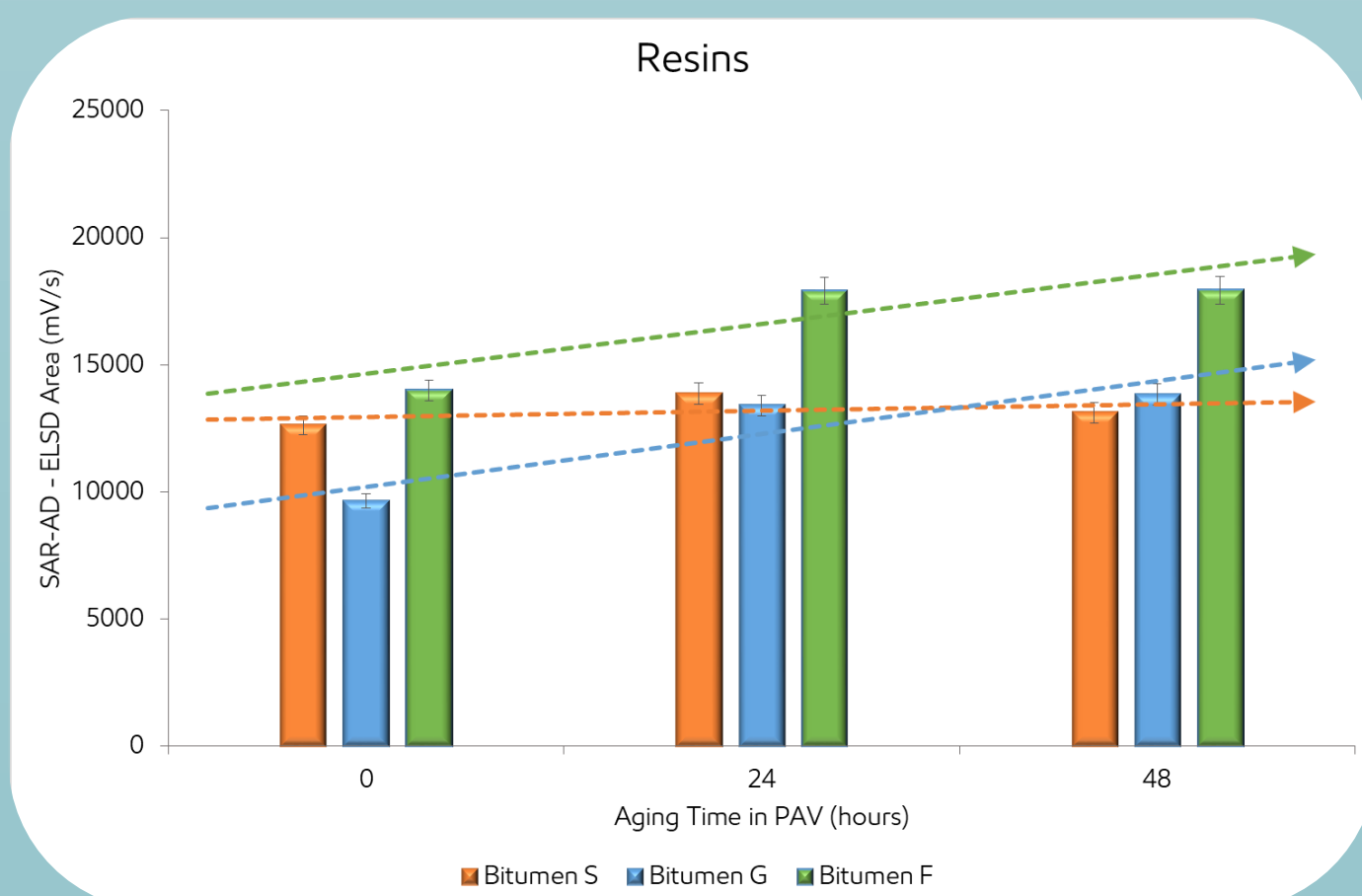

Saturates · Aromatics · Resins · Asphaltenes
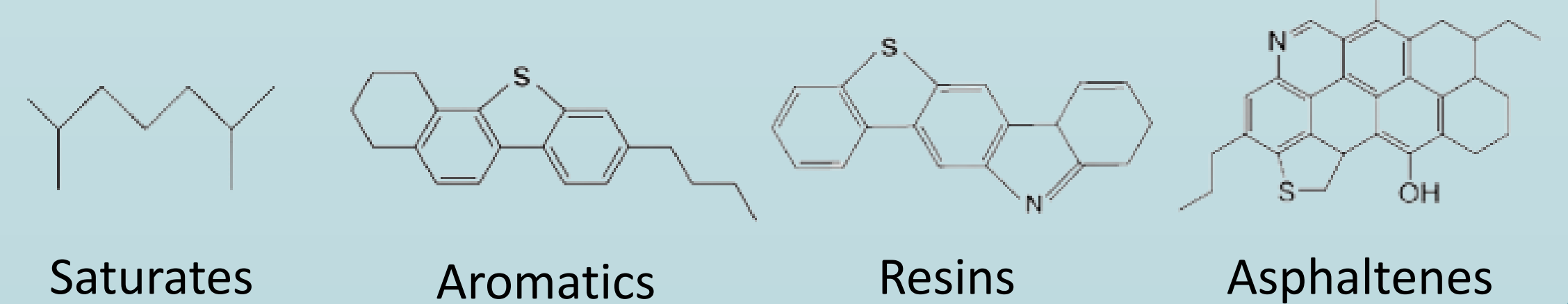
Evolution of SAR-AD™ families after PAV aging

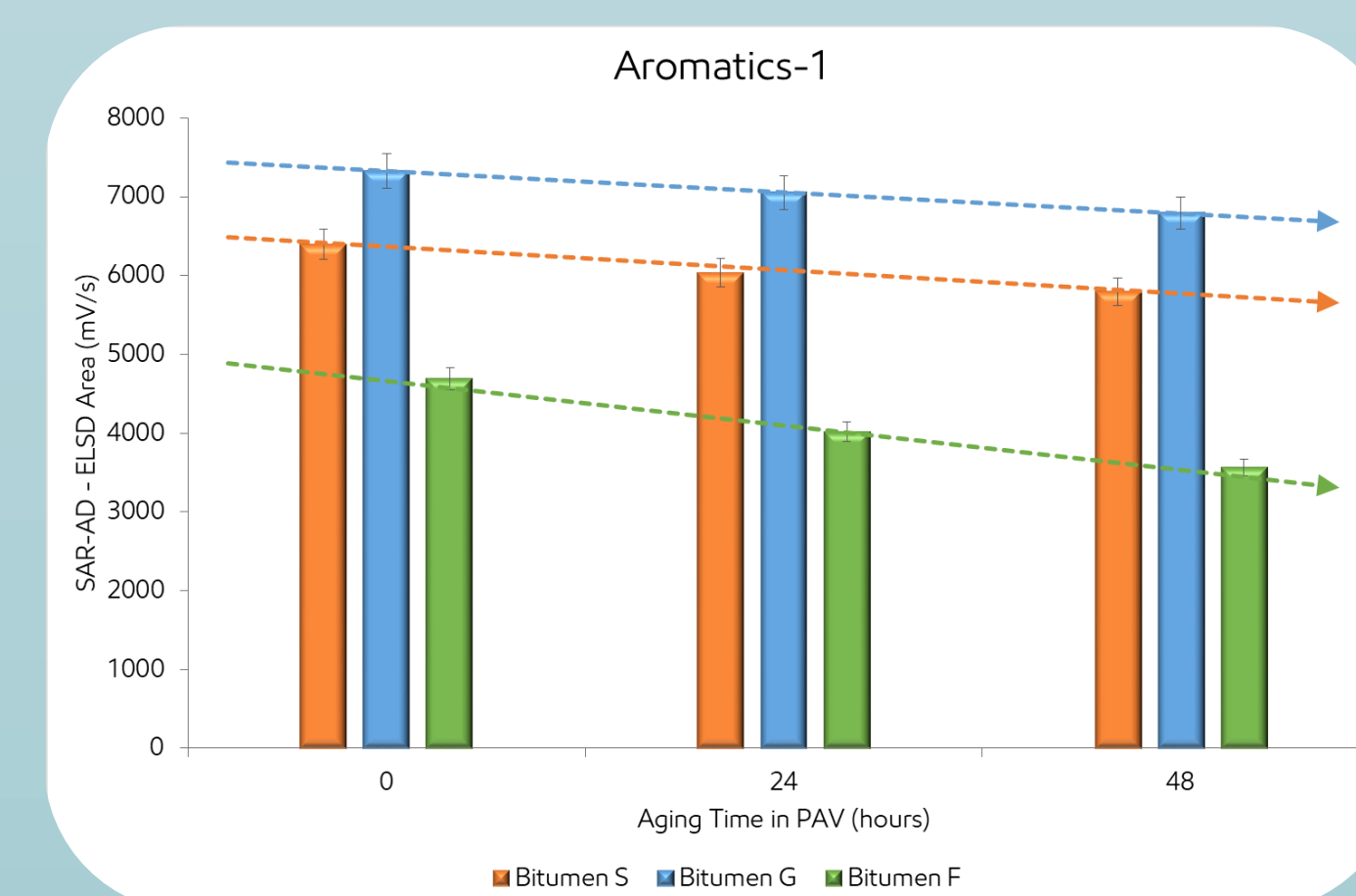❖ SAR-AD separates saturates and resins as single entities similarly to traditional SARA.

❖ SAR-AD™ enables further segregation of aromatics and asphaltenes into three sub-families versus SARA.

- Almost no aging effect on saturates whatever tested bitumen.

- All aromatic contents decreased during aging for all three bitumen studied.
- Aromatics content decrease is most significant for the most polar aromatics fraction (Aromatics-3>2>1).

- Different aging effect on resins due to different bitumen chemistry.
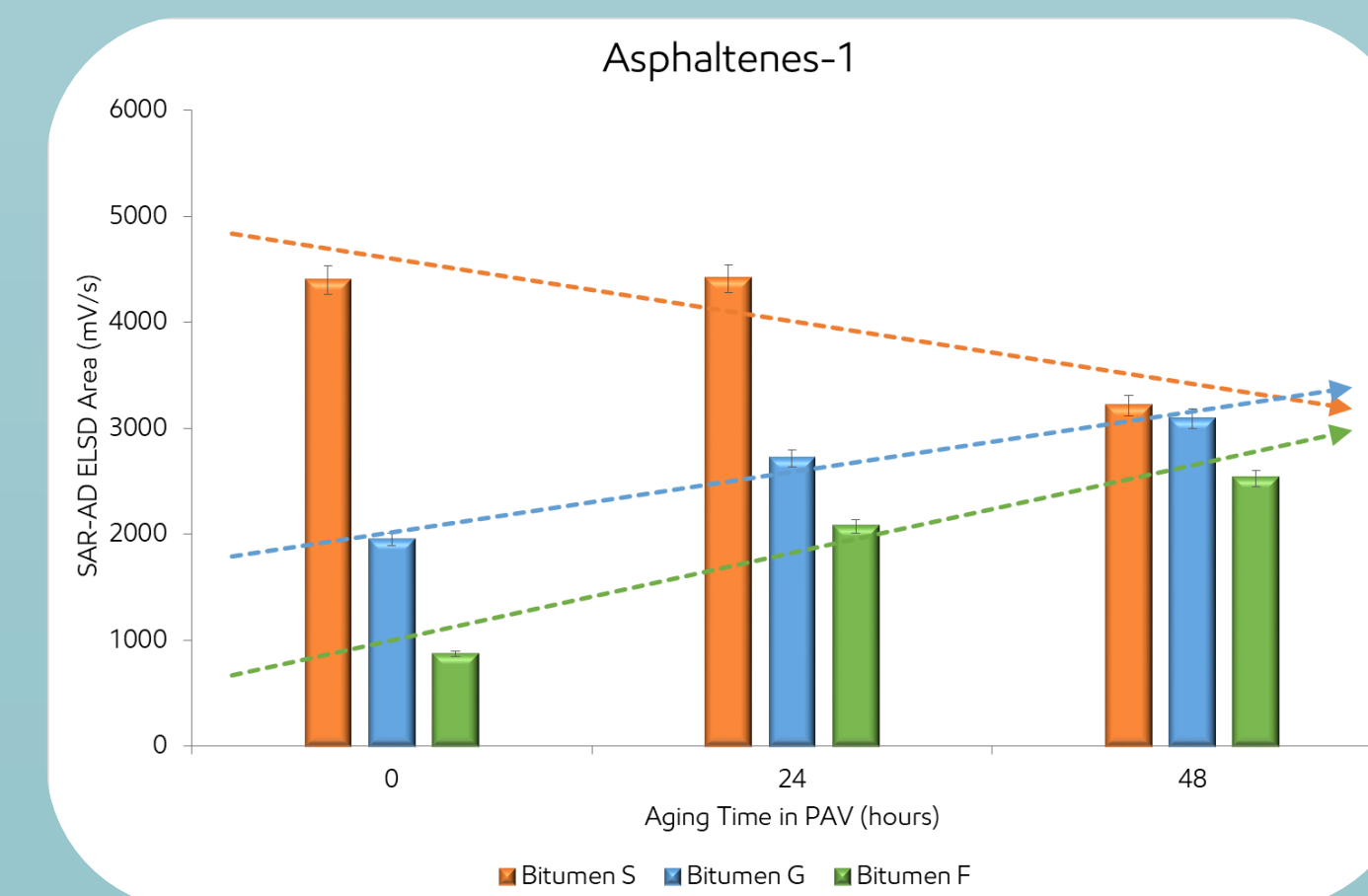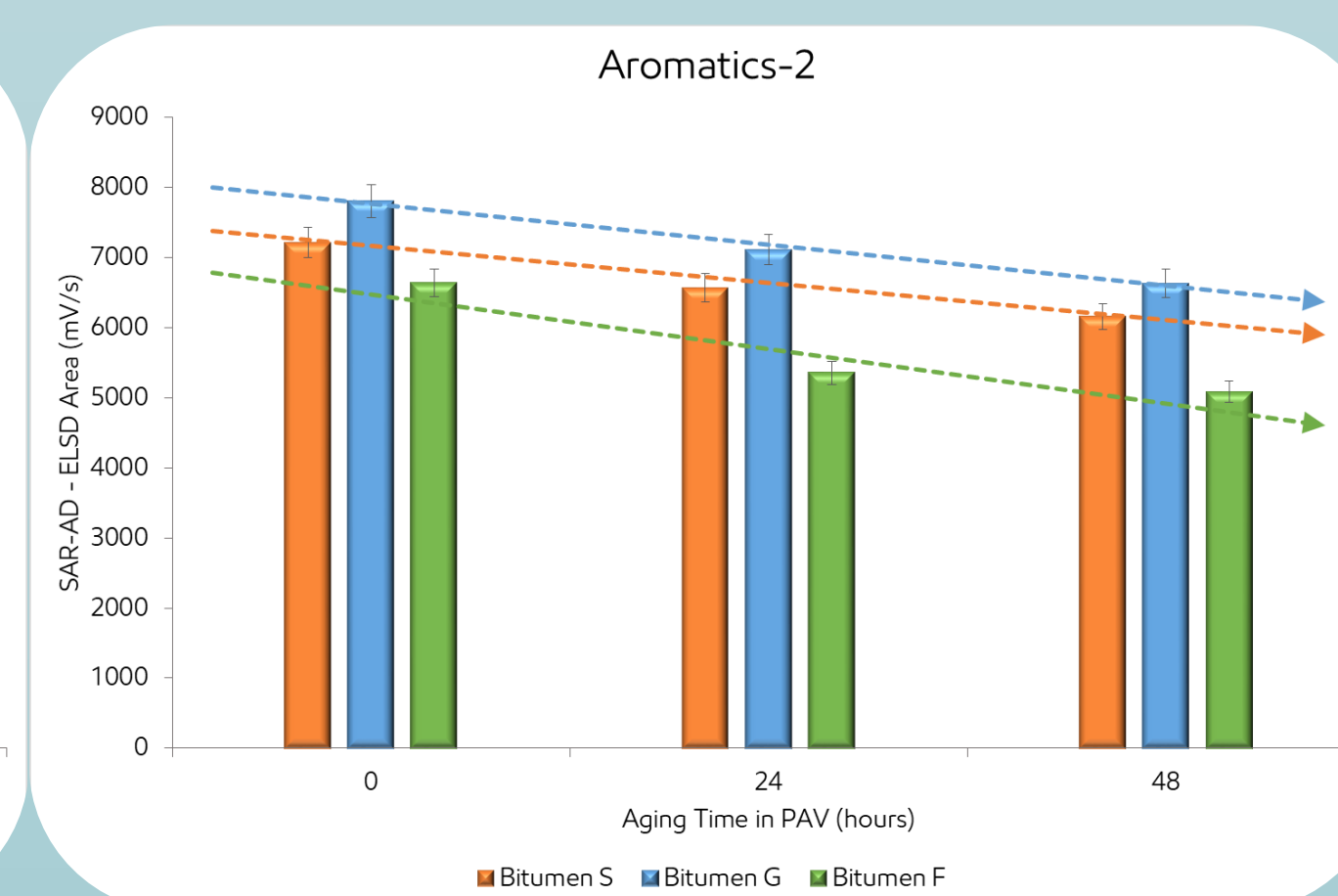
- Asphaltenes-2 has the strongest correlation with SARA/NF T60-115 while asphaltenes-1 and -3 exhibit different behaviors due to different bitumen chemical composition.
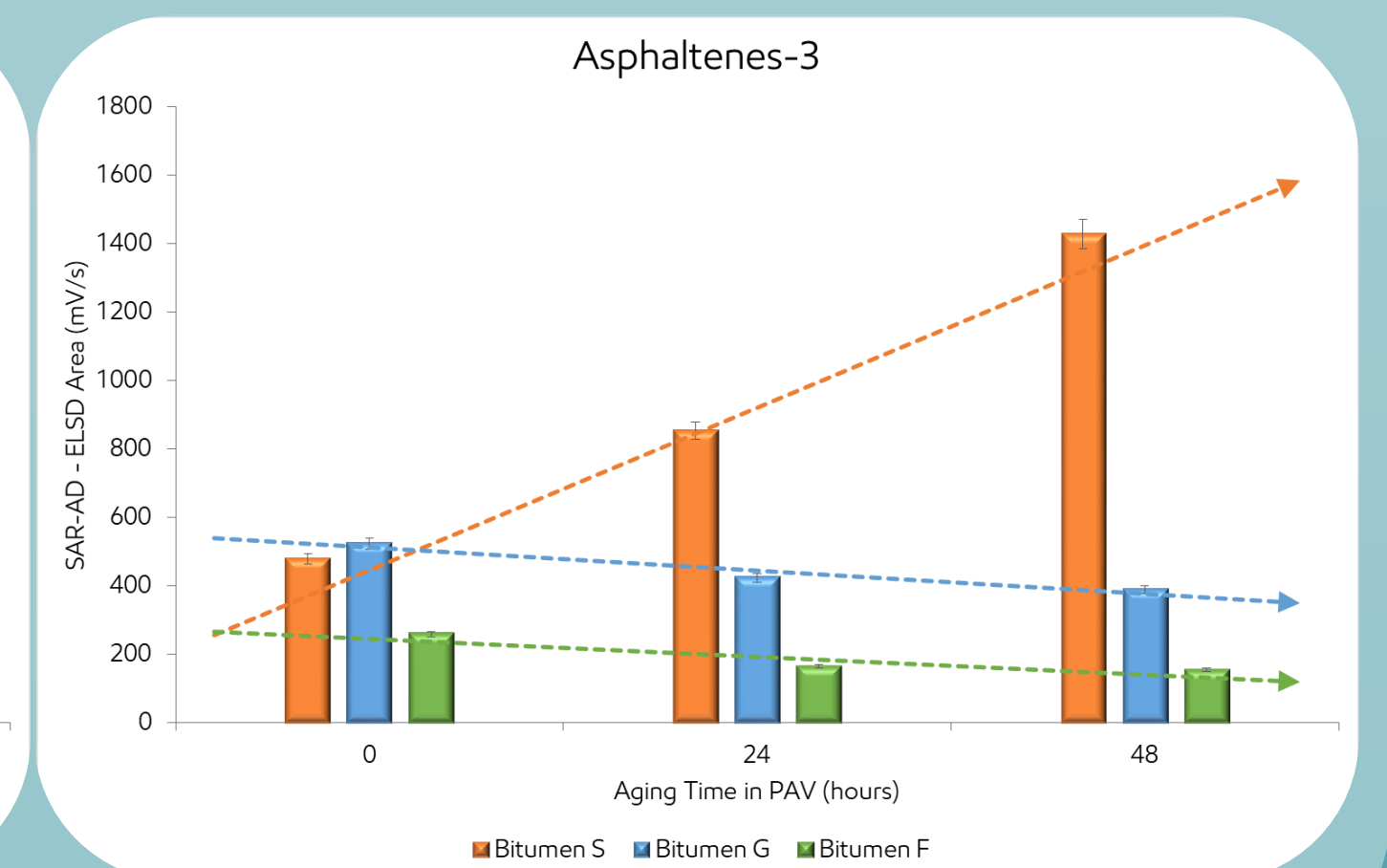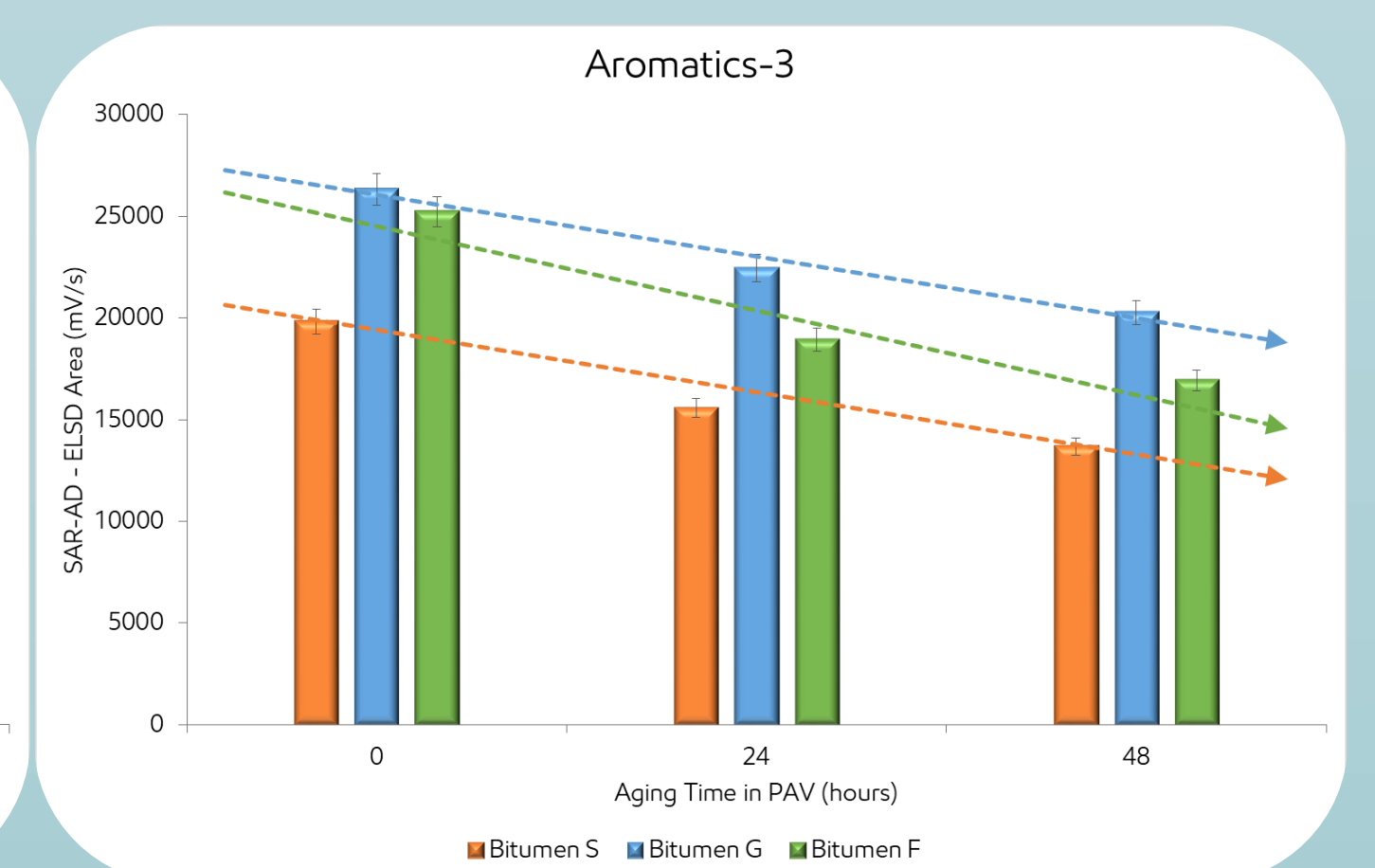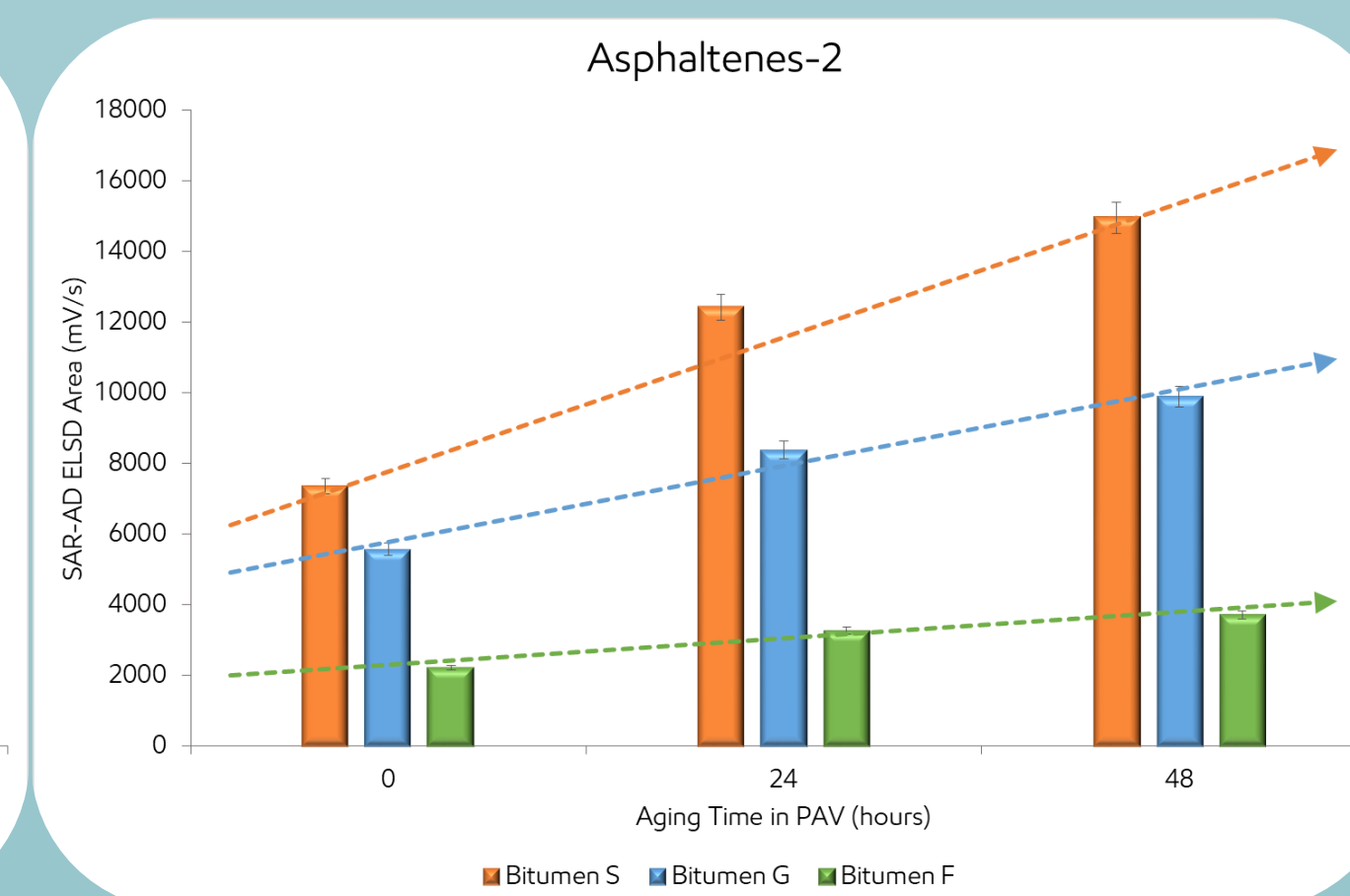
Benefits & drawbacks SAR-AD™ vs SARA separation

➕
- Separation into 8 families instead of 4 allowing finer analysis of aromatic and asphaltene molecules
- Time saving (90 min vs 1.5 day / sample) / Automated analysis
- SAR-AD™ requires smaller sample amount

➖
- Non-preparative method / not possible to analyze SAR-AD™ physical properties (rheology…)

[1] J. Mirwald, S. Werkovits, I. Camargo, D. Maschauer, B. Hofko, and H. Grothe, "Understanding bitumen ageing by investigation of its polarity fractions," Constr. Build. Mater., vol. 250, p. 118809, Jul. 2020
[2] C. Yang et al., "Investigation of physicochemical and rheological properties of SARA components separated from bitumen," Constr. Build. Mater., vol. 235, p. 117437, Feb. 2020
[3] J. Rovani, Western Research Institute, "Automated SAR-AD Using an Agilent 1260 HPLC System with Agilent 1260 ELSD – Western Research Institute Standard Operating Procedure and Supporting Information", April 2017
[4] J. Wang, T. Wang, X. Hou, and F. Xiao, "Modelling of rheological and chemical properties of asphalt binder considering SARA fraction," Fuel, vol. 238, pp. 320–330, Feb. 2019