

# UNIVERSIDAD DE COSTARICA

# **Objectives:**

Quantify the adhesion between the binder source available in Costa Rica with different aggregate types typically used in construction,

Characterize the effect of different modifier contents on moisture damage susceptibility, Determine the effect of aging on adhesion for the analyzed materials

## **Materials:**

#### Pyrogenic Silica

- Synthetic, hydrophilic, amorphous silica, produced via flame hydrolysis.
- ✓ BET surface 175 225 m<sup>2</sup>/g
- ✓ Density at 20 °C approx. 2.2 g/cm<sup>2</sup>

#### Asphalt Binder

- Neat binder: PG 64-22
- + 0.5% nano SiO<sub>2</sub> (PG70-25)
- + 3% nano SiO<sub>2</sub> (PG76-25)
- + 6% nano SiO<sub>2</sub> (PG82-25)

# **Materials Characterization:**

#### Atomic Force Microscopy (AFM)

The surface texture of the bitumen obtained from AFM images is expected to be related to the tensile strength of the bitumen.

Bee-structures are related to natural species of the bitumen rather than the modifier.

> AFM topography (a) unmodified, (b) +0.5% SiO<sub>2</sub> (c) + 3% SiO<sub>2</sub> (d) + 6% SiO<sub>2</sub> bitumen

#### Differential Scanning Calorimetry Analysis (DSC)

- SiO<sub>2</sub> has crystal-like behavior and exhibits minor transitions it is more stable at the same temperature range of the bitumen.
- Addition of the modifier results in: Lower glass transition temperatures (Tg), lower melting temperatures (Tm) and higher content of fractional crystallization ( $\Delta$ H).
- Combination of some elements found in the bitumen with 4 point valency differential to silica helps in breaking up the network structure, thus reducing the Tg.

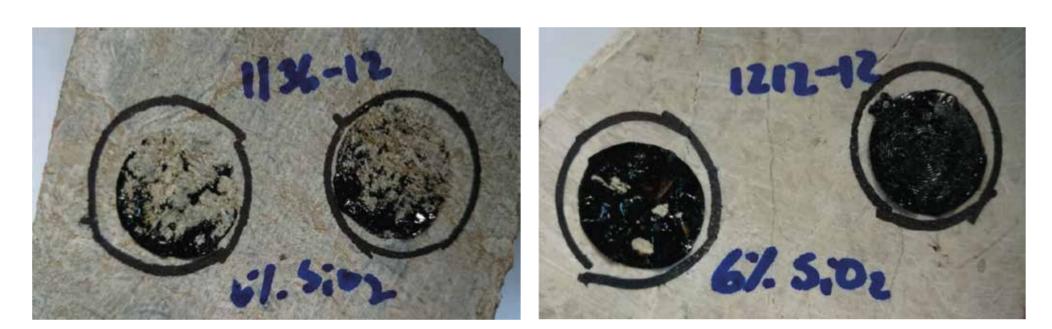
Bitumen	Tg Onset, °C	Tg, °C	Tm, °C	ΔH, J/g
PG64-22	-45.7	-27.5	24.4	7.8
+ 0.5% nano SiO <sub>2</sub>	-49.9	-33.9	24.0	8.7
+ 3% nano $SiO_2$	-43.3	-35.5	24.3	9.9
+ 6% nano SiO <sub>2</sub>	-40.3	-35.3	23.5	10.7

**Bitumen DSC properties** 

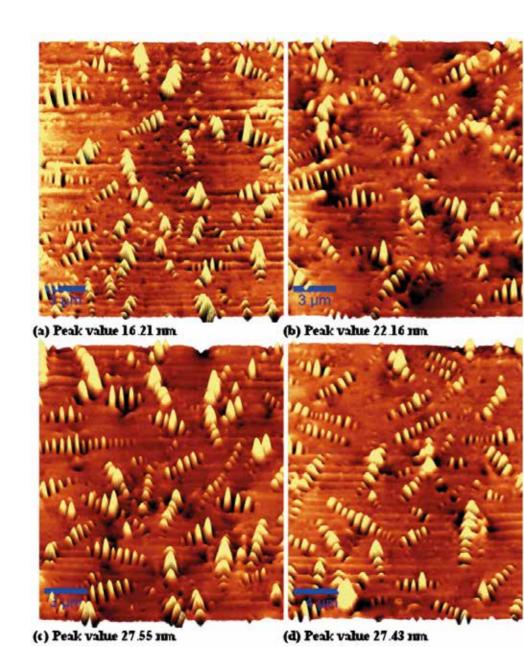
Aggregate

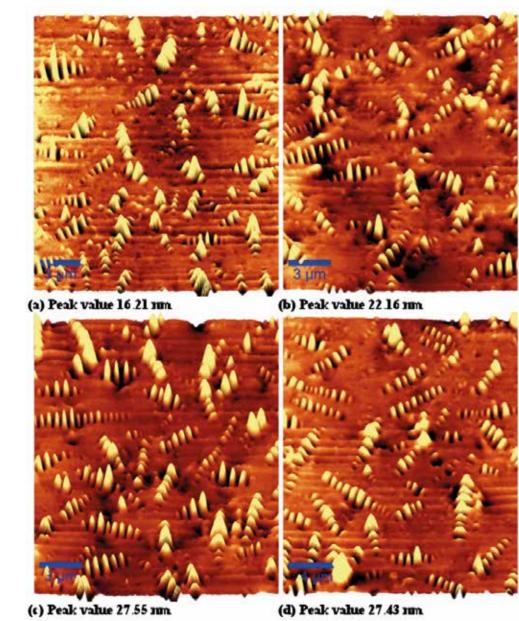


River Gravel 1 -Central Pacific







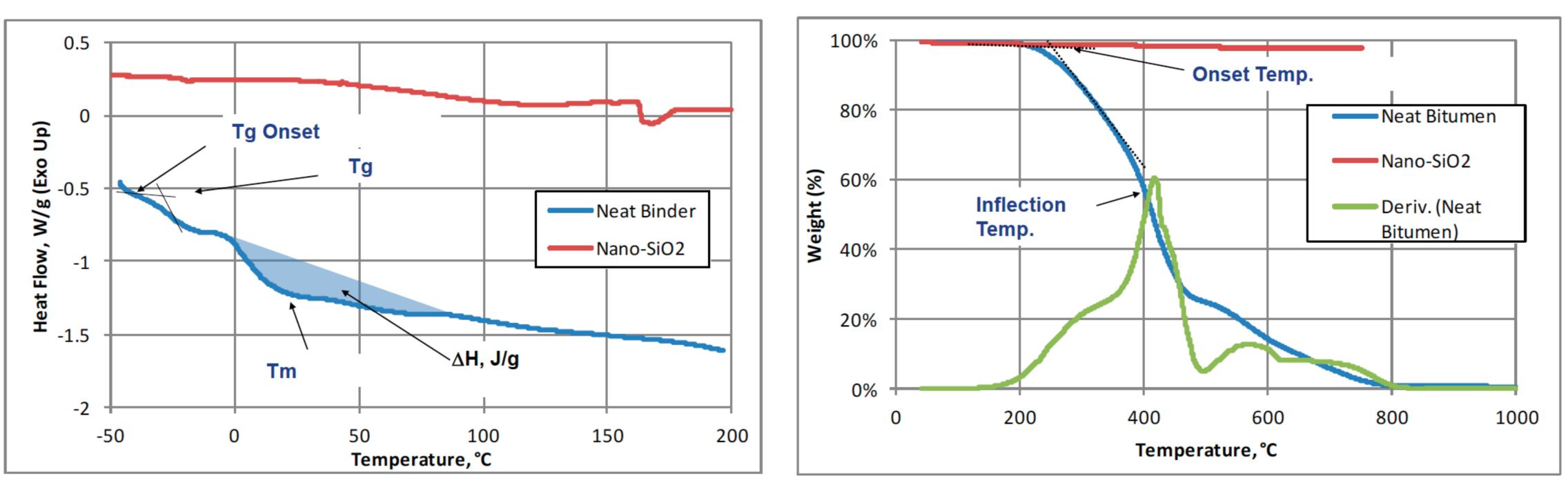


# Adhesion Performance of Nano-silica Modified Binder

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River Gravel 2 -Central Caribbean

Limestone 2 - North Pacific



DSC analysis of the neat bitumen and modifier

#### Thermogravimetric analysis (TGA)

- The point of highest weight loss change rate is known as the inflection point. It indicates when Pyrolysis occurs. > Pyrolysis is a thermochemical decomposition of organic material at elevated temperatures in the absence of oxygen (or any halogen).
- This property is related to the chemical bond strength and structure stability of the material.
- Addition of the modifier produced higher inflections points

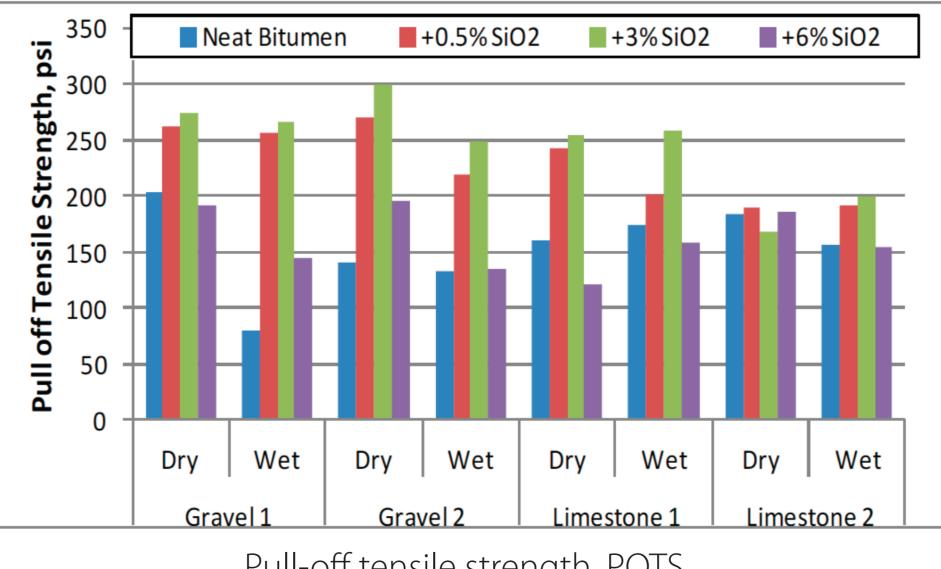
Bitumen TGA properties	Bitumen	% Loss @ 200 °C	Onset Temperature, °C	Inflection Point, °C	Residue, %
	PG64-22	0.92	255.3	416.3	0.67
	+0.5% nano SiO <sub>2</sub>	1.14	247.6	425.8	0.51
	+ 3% nano SiO <sub>2</sub>	1.17	302.8	433.4	9.67
	+ 6% nano SiO <sub>2</sub>	0.71	273.1	439.0	18.96

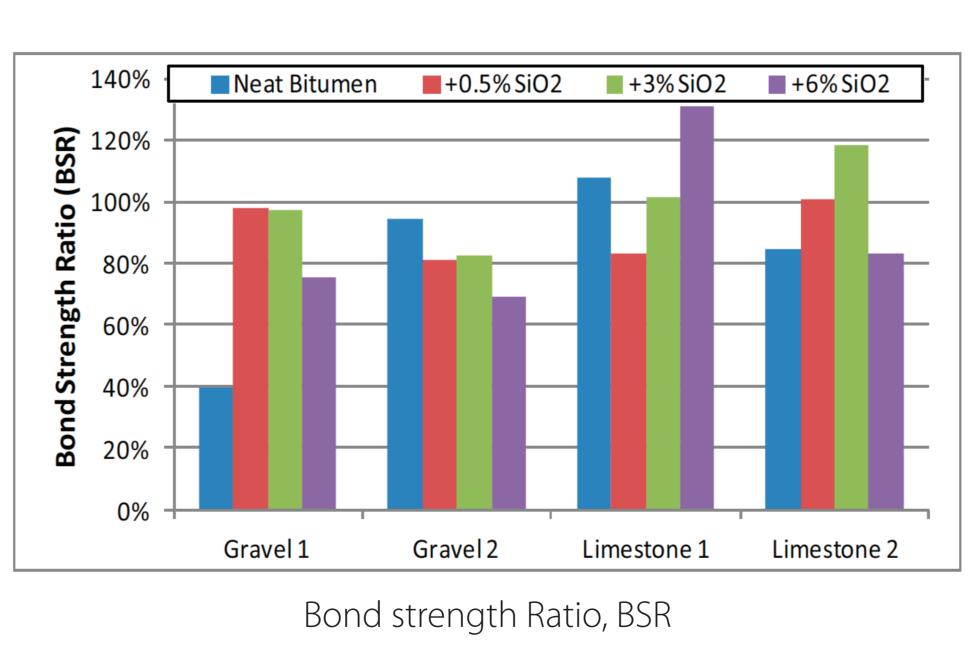
# Effect of Modifier Content, Aging and Aggregate Source on Adhesion

Bitumen-aggregate adhesion was characterized based on the Bitumen Bond Strength (BBS) test. The test was performed following AASHTO TP-91 using the P.A.T.T.I. equipment.

The bitumen-aggregate system is subjected to a constant load rate of 100 psi/s (690 kPa/s) and is analyzed after 24 hours of conditioning at room temperature (Dry) and 48 hours conditioning in a water bath at 40 °C (Wet).

#### Evaluation of the modifier content on the aggregate/binder interaction, Unaged





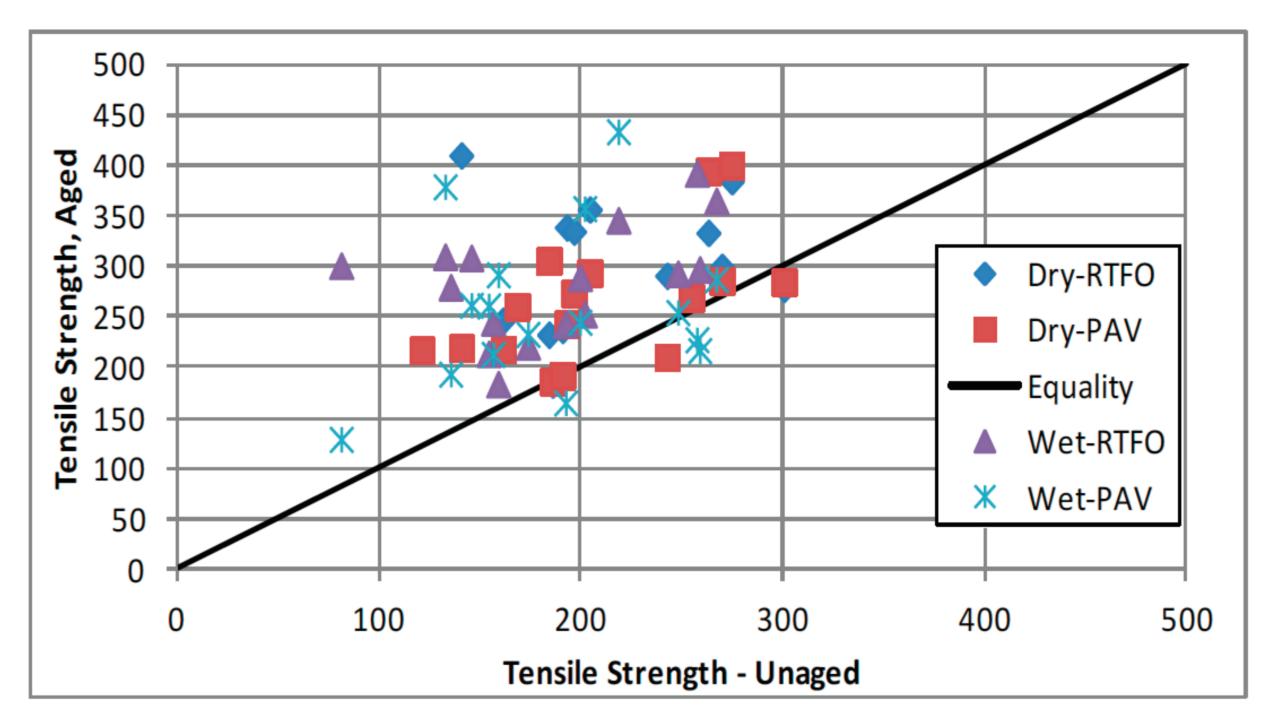
Pull-off tensile strength, POTS

- As the additive content is increased, an increase in adhesion is also obtained.
- The phenomenon occurs up to a critical modifier concentration after which adhesivity begins to drop (around 3%).
- The use of modification improves resistance to moisture damage (shown by the bond strength ratio)

TGA analysis of the neat bitumen and modifier



### Effect of Aging



Unaged vs Aged tensile strength

#### Effect of Aggregate Source

- Effect of Aggregate source observed only on tensile strength in Dry condition (ANOVA).
- be statistically explained by any of the analyzed treatments.
- Variability of the response Bond Strength Ratio (BSR) cannot Interactions were not statistically significant.
- Tensile strengths for the dry and wet conditions were not statistically different between RTFO and PAV aging stages but greater than the unaged stage.
- River gravel aggregate showed higher POTS than the limestone.
- Addition of the modifier content exhibit statistically different tensile strengths for the dry and wet conditions between two groups: 0.5% and 3.0% nano-SiO<sub>2</sub> vs. 6.0% and unmodified with greater values for the first group.

### **Conclusions:**

- ✓ Superior bitumen performance and higher thermal stability produced higher bond and tensile strength (from TGA and DSC analyses).
- ✓ Significant differences in strength of adhesion between the different aggregate-bitumen combinations were obtained. A minimum Bond Strength Ratio of 70% is recommended.
- ✓ An optimal adhesion/cohesion performance can be obtained for the studied modifier to maximize the adhesivity, and consequently moisture damage resistance at a concentration near 3%.
- RTFO aged bitumens showed a considerable increment in strength of adhesion which indicates that the
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The POTS results for aged binders indicate a 50% increase in POTS when RTFO aging was performed and 35% for PAV aging for both Dry and Wet conditions.

Analysis of variance, $\alpha = 5\%$				
Response	Pull-off tensile strength - Dry			
Source	DF	F-statistic	P-value	Result
Aging	2	15.59	<<0.05	Significant
Agg. Type	3	11.68	<<0.05	Significant
Mod. Cont.	3	4.27	0.011	Significant
Error	39			
Response		Pull-off tens	le strength	-Wet
Source	DF	F-statistic	P-value	Result
Aging	2	12.88	<<0.05	Significant
Agg. Type	3	1.94	0.139	
Mod. Cont.	3	3.92	0.015	Significant
Error	39			
Response			BSR	
Source	DF	F-statistic	P-value	Result
Aging	2	0.91	0.412	
Agg. Type	3	2.05	0.123	
Mod. Cont.	3	0.63	0.601	
Error	39			

#### Tukey-test results, $\alpha = 5\%$

Treatment	Condition	Mean Tensi	Croupipa*	
	Condition	Dry	Wet	Grouping*
Aging	RTFO	292.4	284.1	А
	PAV	266.1	259.6	Α
	Original	209.2	186.1	В
Mod. Cont.	3% SiO <sub>2</sub>	283.8	274.2	A
	0.5% SiO <sub>2</sub>	267.6	268.5	Α
	Original	247.8	215.8	В
	6% SiO <sub>2</sub>	224.3	214.6	В
Agg. Type	River Gravel 1	306.8		Α
	River Gravel 2	274.6	ΝΙΛ	Α
	Limestone 1	226.5	NA	В
	Limestone 2	215.7		В

\* Means that do not share a letter are significantly different

 $\checkmark$  Higher particle interlock (AFM images texture) was accomplished by modification with nano-silica.