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# Nanostructured Bitumen, Better Mechanical Performance and for Extreme Climates

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### **ABSTRACT**

Bitumen and aggregates are two elements without big affinity, they are mixed due to high temperature and high mechanical energy by asphalt plant mixers. This lack of affinity is always in the asphalt mix and the apparition of elements as water, or in a worse situation, water with salt in the Winter treatments, which make earlier appearance of pavements degradation.

In our determination to innovate, has develop a bitumen formula for situations where saline environments, used in Winter treatments, produced large damages in pavements. Alternative manufacturing process uses a few quantity of nanomaterials, chemical additives and special polymers in order to have a competitive product in the market. This bitumen fulfill with the performance grades binder demanded characteristics, moreover it presents a superior performance in the normalized water sensitivity test.

Normal adhesiveness in the bitumen-aggregate interaction used to be polar-polar character, depending on the aggregates surface charges has through -OH groups and bitumen acid fractions. This interaction is not strong enough how it is indicated at the first paragraph and only reached an improved adhesiveness with the use of aminic adhesiveness activants.

Using this technology has reached to improve this exponentially situation, achieving to bitumen-aggregate covalent bond, which is the strongest in chemistry. This union gives to the mix a great amelioration of properties due to obtaining the high performance interface in the asphalt mix, it acts like a key to improve the totality of the properties that are studied, beginning with adhesiveness, seeing better situations even in mechanical properties.

### **ABSTRAIT**

Le bitume et les agrégats sont deux éléments sans grande affinité, ils sont mélangés en raison de la température élevée et de l'énergie mécanique élevée par des mélangeurs d'usine d'asphalte. Ce manque d'affinité est toujours dans le mélange d'asphalte et l'apparition d'éléments comme l'eau, ou dans une situation pire, l'eau salée dans les traitements d'hiver, ce qui fait apparaître plus tôt la dégradation des chaussées.

Dans notre détermination à innover, nous avons développé une formule de bitume pour les situations où les environnements salins, utilisés dans les traitements d'hiver, produisaient des dommages importants dans les chaussées. Un procédé de fabrication alternatif utilise une quantité limitée de nanomatériaux, d'additifs chimiques et de polymères spéciaux afin d'avoir un produit compétitif sur le marché. Ce bitume remplit avec les caractéristiques

exigées de classeur de qualités exigées, de plus il présente une performance supérieure dans le test normalisé de sensibilité à l'eau.

Adhésivité normale dans l'interaction bitume-agrégat utilisé pour être polaire polaire, en fonction de la charge de surface des agrégats a travers les groupes -OH et les fractions d'acide de bitume. Cette interaction n'est pas assez forte comme elle est indiquée au premier paragraphe et n'atteint une adhésivité améliorée qu'avec l'utilisation d'activants d'adhésivité aminique.

L'utilisation de cette technologie a permis d'améliorer cette situation de façon exponentielle, atteignant la liaison covalente bitume-agrégat, qui est la plus forte en chimie. Cette union donne au mélange une grande amélioration des propriétés grâce à l'obtention de l'interface haute performance dans le mélange d'asphalte, il agit comme une clé pour améliorer la totalité des propriétés étudiées, en commençant par l'adhérence, en voyant des meilleures propriétés mécaniques .

## 1. EXPERIMENTAL TESTS AND RESULTS

Firstly, determination tests of water sensitivity of bituminous specimens (UNE EN 12697-12) were developed at several projects with the aim of corroborate the extreme climate bitumen better performance, being able to compare between indexes of retained strength reached with different types of mixes at various environments and weather conditions.

### 1.1. Navacerrada Mountain Ski Port, Madrid (Spain), Octobre 2016.

DRY SPECIMENS						
SPECIMEN	HEIGHT (mm)	SSD mass	LOAD (N)	TYPE OF BREAK traction (T) deformation (D) combined (C)	COMPRESSIVE STRENGTH OF DRY SPECIMENS (MPa)	INDEX OF RETAINED STRENGTH (%)
1	73,4	2.341,1	41.762	Traction (T)	3,567	
2	73,8	2.332,7	36.287	Traction (T)	3,085	
3	74,0	2.331,1	36.461	Traction (T)	3,089	
<b>AVERAGES</b>	73,717	2334,967	38170,000		3,247	
IMMERSED SPECIMENS						
SPECIMENS	HEIGHT (mm)	SSD mass	LOAD (N)	TYPE OF BREAK traction (T) deformation (D) combined (C)	COMPRESSIVE STRENGTH OF IMMERSSED SPECIMENS (MPa)	103,6
4	73,9	2340,1	39894	Traction (T)	3,387	
5	73,8	2344,4	38722	Traction (T)	3,289	
6	74,3	2325,2	40422	Traction (T)	3,413	
<b>AVERAGES</b>	73,967	2336,567	39679,333		3,363	

Table 1- determination tests of water sensitivity of bituminous specimens. Navacerrada. Madrid.

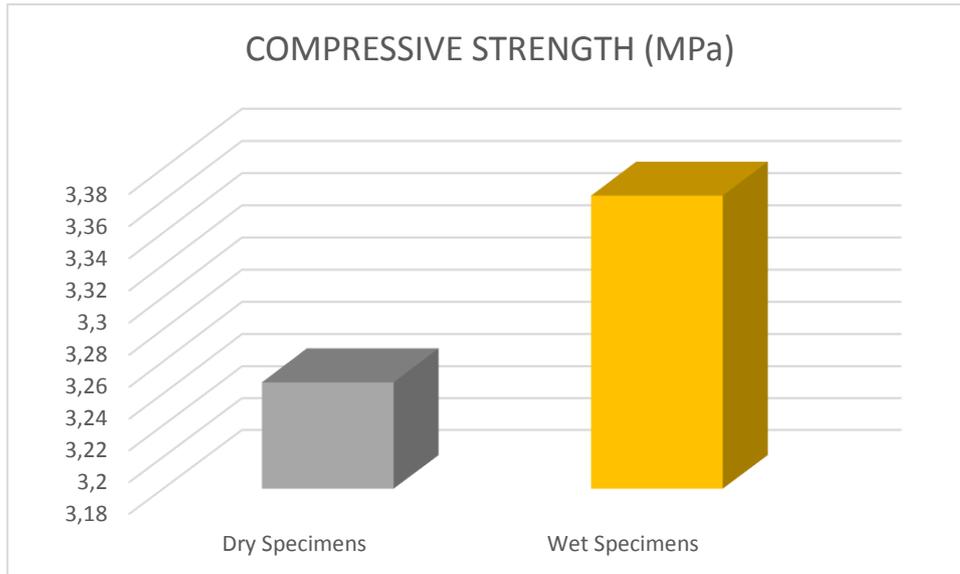


Figure 1- determination tests of water sensitivity of bituminous specimens, Navacerrada, Madrid.

This figure presents how the resistance of the immersed specimens are stronger than dry specimens, which helps increasing the finale index of retained strength.

#### 1.2. Standard Practice for Effect of Water on Bituminous-Coated Aggregate Using Boiling (ASTM D3625), December, 2016.

This practice covers a rapid procedure for visually observing the loss of adhesion in uncompact bituminous-coated aggregate mixtures due to the action of boiling water.

Furthermore, it is useful as an indicator of the relative susceptibility of bituminous-coated aggregate to water, but should not be used as a measure of field performance because such correlation has not been establish. If loss of adhesion due to water is indicated, testing by other procedures should be conducted to further evaluate mixture.

At our laboratory, aggregates were put under several saline resistance treatments, under the most exigent conditions according ASTM D3625 rules, with an extra condition, it was the add of 4% of NaCl. As has been noted, concrete asphalt manufactured with Nanostructured Bitumen has a 10 times higher resistance than the conventional ones.

It could be observed in the following pictures that on the left side the aggregates have been totally discovered while on the right side aggregates present a first-rate coverage due to Nanostructured Bitumen.



Image 1- Boiling test, Madrid laboratory.

The following two data grids show the difference between Water sensitivity of bituminous specimens (UNE EN 12697-12) tests with and without Nanostructured Bitumen at Ribadesella, Asturias (Spain), January, 2017.

1.2.1. AC16Surf S 50/70 Mix without Nanostructured Bitumen:

DRY SPECIMENS						
SPECIMEN	HEIGHT (mm)	SSD mass	LOAD (N)	TYPE OF BREAK traction (T) deformation (D) combined (C)	COMPRESSIVE STRENGTH OF DRY SPECIMENS (MPa)	INDEX OF RETAINED STRENGTH (%)
1	68,1	2.397,6	30.665	Traction (T)	2,823	
2	68,6	2.399,6	33.318	Traction (T)	3,045	
3	68,3	2.412,9	30.265	Traction (T)	2,779	
<b>AVERAGES</b>	68,327	2403,367	31416,000		2,882	
IMMERSED SPECIMENS						
SPECIMEN	HEIGHT (mm)	SSD mass	LOAD (N)	TYPE OF BREAK traction (T) deformation (D) combined (C)	COMPRESSIVE STRENGTH OF IMMERSSED SPECIMENS (MPa)	96,9
4	68,6	2406,1	29785	combined (C)	2,722	
5	68,8	2407,6	32368	deformation (D)	2,952	
6	69,0	2410,6	29784	deformation (D)	2,706	
<b>AVERAGES</b>	68,783	2408,100	30645,667		2,793	

Table 2- determination tests of water sensitivity of bituminous specimens, Ribadesella, Asturias.

1.2.2. AC16Surf S 50/70 Mix with Nanostructured Bitumen:

DRY SPECIMENS						
SPECIMENS	HEIGHT (mm)	SSD mass	LOAD (N)	TYPE OF BREAK traction (T) deformation (D) combined (C)	COMPRESSIVE STRENGTH OF DRY SPECIMENS (MPa)	INDEX OF RETAINED STRENGTH (%)
1	68,0	2.407,0	36.558	Traction (T)	3,372	
2	68,0	2.400,9	33.653	Traction (T)	3,103	
3	67,9	2.412,3	33.717	Traction (T)	3,113	
<b>AVERAGES</b>	67,957	2406,733	34642,667		3,196	
IMMERSED SPECIMENS						
SPECIMEN	HEIGHT (mm)	SSD mass	LOAD(N)	TYPE OF BREAK traction (T) deformation (D) combined (C)	COMPRESSIVE STRENGTH OF IMMERSED SPECIMENS (MPa)	105,3
4	68,3	2408,7	39445	Traction (T)	3,620	
5	68,5	2405,5	35687	Traction (T)	3,265	
6	68,0	2405,0	34874	Traction (T)	3,215	
<b>AVERAGES</b>	68,280	2406,400	36668,667		3,367	

Table 3- determination tests of water sensitivity of bituminous specimens, Ribadesella, Asturias.

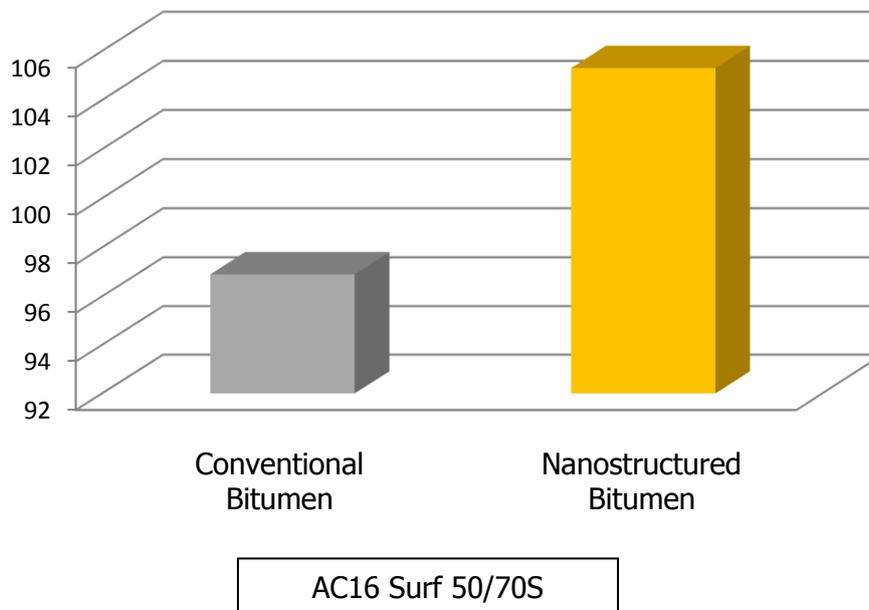


Figure 2- determination tests of water sensitivity of bituminous specimens, Ribadesella, Asturias.

On the left, at the RCTI (%) comparative figure, it is shown the increased percentage with Extreme Climate Bitumen.

The mix AC16 Surf 50/70S without Nanostructured Bitumen presents lower resistance at retained strength, obtaining a 8,4 points of difference, which means that resistance and duration of pavement versus adverse conditions are improved with Nanostructured Bitumen.

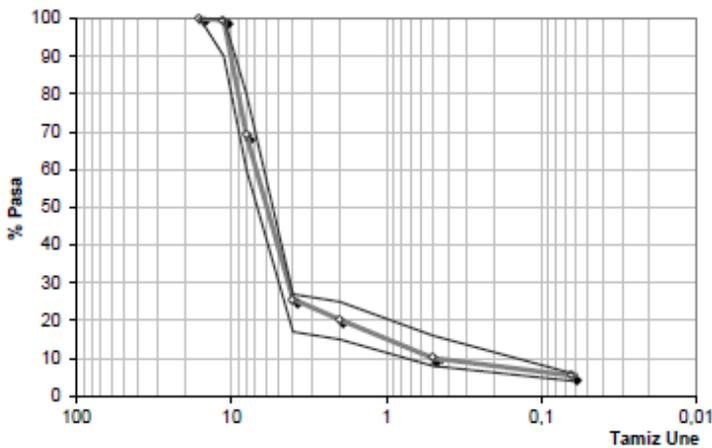
1.3. Assay with BBTM 11B PMB 45/80-65 at A2 highway, Zaragoza (Spain), June, 2017.

The new highway pavement was applied last June 28 in 1 kilometer of the Autovía A2 in the province of Zaragoza and is expected to be used generically for the actions in 2018.

The product has significantly improved mechanical properties compared to conventional bitumen, in addition to a high resistance at weather conditions, which translates into a special adaptation to the annual winter treatments that the concession has to carry out.

Moreover, bituminous tests were developed in order to corroborate the good performance of the new bitumen, as well as the water sensitivity test results below:

Determination of particle size distribution (UNE-EN 12697-2), this composition of a bituminous mixture in terms of binder content and aggregates grading is a significant quality parameter. The European Standard for bituminous mixtures contains some grading specifications Controlling the mixture grading is an important instrument for product quality management.



Sieve UNE (Tamiz)	% Pasa	Huso	
		BBTM	11B
16	100	100	100
11,2	100	90	100
8	65	60	80
4	23	17	27
2	19	15	25
0,5	12	8	16
0,063	5,2	4	6

Table 4- determination particle size distribution, A2 Highway, Zaragoza.

Figure 3- determination particle size distribution, A2 Highway, Zaragoza.

1.3.1. Bituminous mixtures – Test methods for hot mix asphalt – Soluble binder content (UNE-EN 12697-1)

% Binder content mix	5,03
% Binder content aggregates	5,29
Relation filler/binder	1,02

Table 5- determination soluble binder content, A2 Highway, Zaragoza.

1.3.2. *Determination of void characteristics of bituminous specimens*

(UNE-EN 12697-8)

Max. density (kg/m <sup>3</sup> ) (UNE-EN 12697-5 C)	2.463
Density geo. 50 g (kg/m <sup>3</sup> ) (UNE-EN 12697-6 B)	2.089
(%) Air Voids (Vm)	15,2
(%) Air voids in the mineral aggregate (VMA)	25,3
(%) Fill voids with binder (VFB)	40,1

Table 6- determination of voids characteristics of bituminous specimens, A2 Highway, Zaragoza.

1.3.3. *Water sensitivity (15°C) (UNE-EN 12697-12)*

Average Dry Strength (kPa)	1.379,4
Average Wet Strength (kPa)	1.299,3
ITSR (%)	94,2

Table 7- determination of water sensitivity, A2 Highway, Zaragoza.

Once, explained all the figures about these tests at A2 highway, explaining all of the results, in order to give more test results, we seek to recap a little bit at the following results, only highlighting the final figures of ITS, dry and wet, besides the final index of tensile strength:

**CONVENTIONAL C-1**

PMB 45/80-65

ITSd =1305.7

ITSw= 1236.37

Relación ITSR (%)= 94.6

**PMB 45/80-65 NS-1**

ITSd =1641.6

ITSw= 1524.4

Relación ITSR (%)= 92.9

**CONVENTIONAL C-2**

PMB 45/80-65

ITSd =1101.8

ITSw= 1032.4

Relación ITSR (%)= 93.7

**PMB 45/80-65 NS-2**

ITSd =1550.7

ITSw= 1437.3

Relación ITSR (%)= 92.7

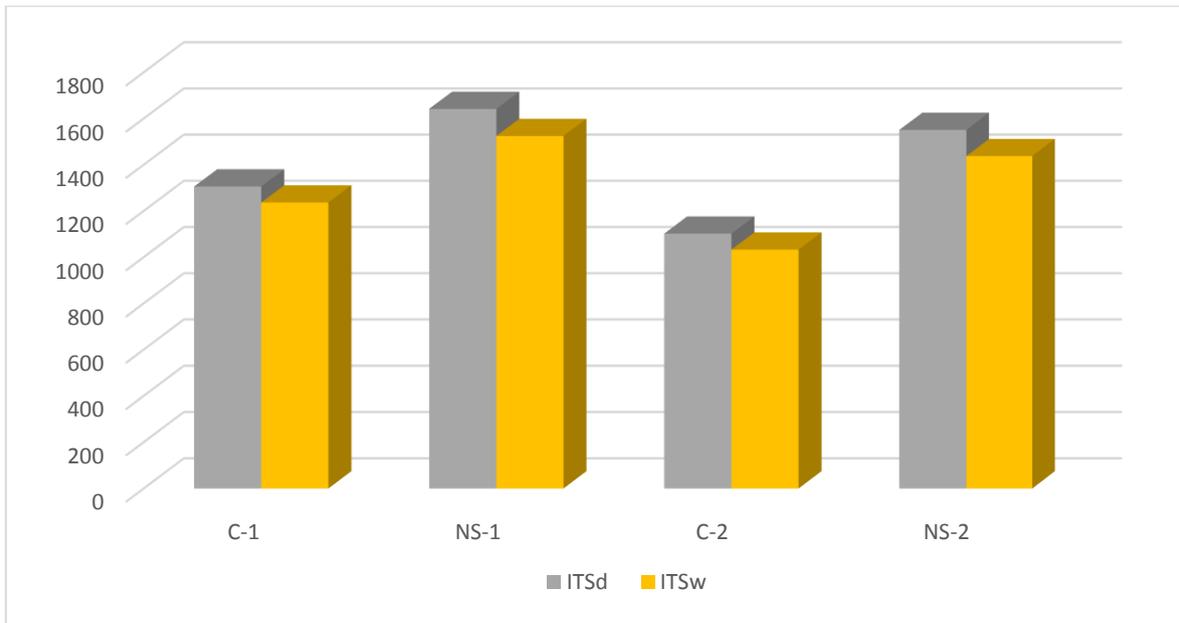


Figure 3- determination tests of water sensitivity of bituminous specimens, A2 Highway, Zaragoza.

As well as, it is possible to conclude that the ITS, dry and wet, results are always better with Nanostructured Bitumen, obtaining higher figures. Means the proper election of Nanostructured Bitumen due to its improved mechanical properties and its fantastic resistance.

#### 1.4. Ribadesella road pavement section with a monitored compaction

On January 20th 2017 another road experiment was carried out, a field experiment due to it was realized a compaction test in the north of Spain, Ribadesella, (Asturias).

This test consisted in compact the pavement but with a particularity, it was done with a special compaction machine and its software, which measures the density while compaction. Therefore, it can be known the number of passes needed to reach the optimum compaction grade of roads, besides the money and time saving due to performance a properly compaction work.

This time mainly monitoring the evolution of compaction with the Nanostructured Bitumen, not only faster compaction was obtained but also the final compaction was 7% higher, in terms of density.

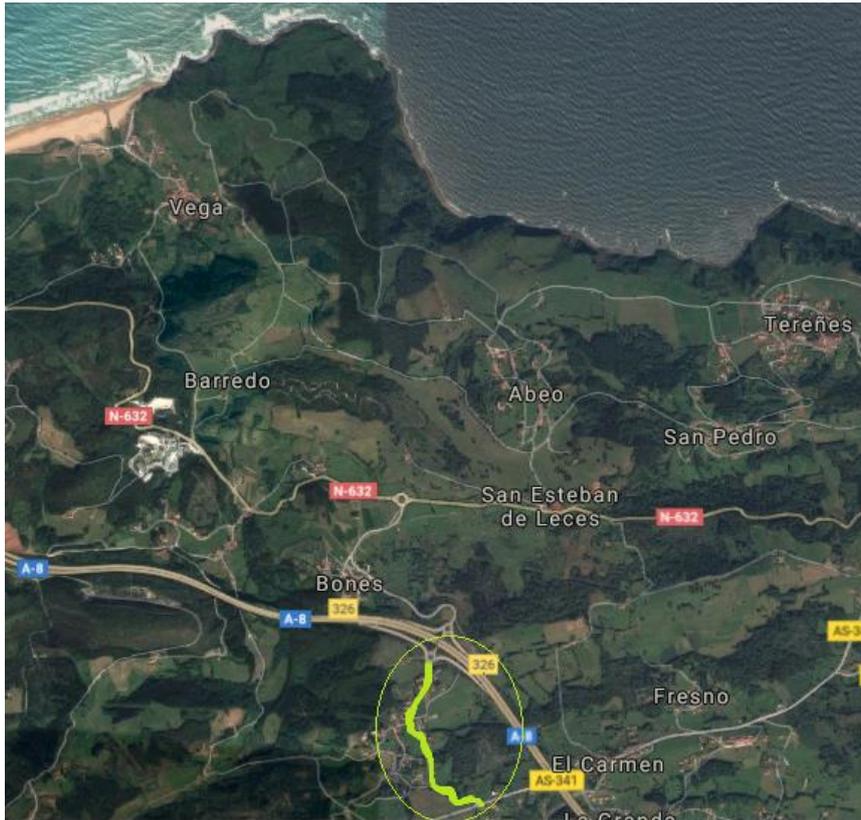
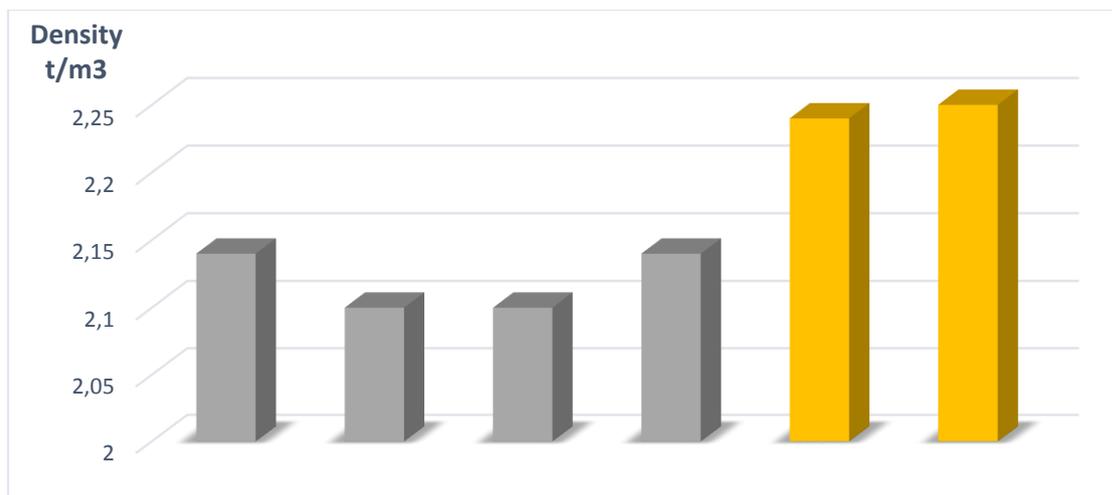


Image 2- Ribadesella road site, Asturias.



Grey: Conventional / Yellow: Nanostructured Bitumen

Figure 4- determination tests of compaction density, Ribadesella, Asturias.

As is shown above, it has been taken six compaction samples, four of them from conventional bitumen mixes (in grey) and the other two with Nanostructured Bitumen (in

yellow). In essence, these samples present the results, reaching higher density with Extreme Nanostructured Bitumen.



Image 3- Ribadesella work site, Asturias.

Additionally to all of these tests, this Nanostructured Bitumen has a patent process in course due to the successful results reached at several fields, as mechanical properties, focus on extreme weather conditions, resistance at negative agents...

#### 1.5. Resistance of Compacted Hot Mix Asphalt (HMA) to Moisture-Induced Damage – AASHTO T 283-07 (2011)

The test was developed on August, 2017, to Projects in Canada.

This method covers preparation of specimens and the measurement of the change of diametral tensile strength resulting from the effects of water saturation and accelerated water conditioning with a freeze-thaw cycle, of compacted HMA. The results may be used to predict long-term stripping susceptibility of the HMA and evaluate liquid anti-stripping additives that are added to the asphalt binder or pulverulent solids, such as hydrated lime or Portland cement, which are added to the mineral aggregate.

The summary of the method is to test specimens for each set of mix conditions, such as those prepared with untreated asphalt binder, asphalt binder treated with anti-stripping agent, or aggregate treated with lime, are prepared. Each set of specimens is divided into subsets. One subset is tested in dry conditions for indirect-tensile strength. The other subset is subjected to vacuum saturation and a freeze cycle, followed by a warm-water soaking cycle, before being tested for indirect-tensile strength.

Numerical indices of retained indirect-tensile strength properties are calculated from the test data obtained by the two subsets: dry and conditioned.

Regarding the method way of procedure, the next data grids show the figures of sampling and the data obtained in the tests of determination the resistance of compacted hot mix asphalt:

<b>Moisture Sensitivity Data</b>							AASHTO T-283
Mix: Ditecpesa/AME SP12.5FC1 D12 Cat D				exp. Global Number: 276720-2			
Sample Remarks: Lab. Crushed Stone with 0.5% Pave Bond Lite				Date sampled: July 31, 2017.			
Sample		1	2	3	4	5	6
Diameter,mm	D	150	150	150	150	150	150
Thickness,mm	t	94.9	94.9	95.0	94.9	95.0	95.0
Dry mass,g	A	3898.4	3908.3	3903.5	3900.5	3907.2	3905.0
SSD mass, g	B	3919.6	3931.0	3923.4	3922.2	3928.9	3928.0
Mass in water,g	C	2283.9	2296.9	2291.1	2287.5	2296.2	2291.6
Volume, cc (B-C)	E	1635.7	1634.1	1632.3	1634.7	1632.7	1636.4
Bulk Sp Gravity (A/E)	F	2.383	2.392	2.391	2.386	2.393	2.386
Max Sp Gravity	G	2.562	2.562	2.562	2.562	2.562	2.562
% Air Voids (100(G-F)/G)	H	7.0	6.6	6.7	6.9	6.6	6.9
Vol Air Voids (HE/100)	I	114.1	108.6	108.7	112.3	107.6	112.2
Load,N	P	<del> </del>	<del> </del>	<del> </del>	15938.9	15489.0	15609.1
<b>Saturated/Conditioned</b>							
Thickness,mm	t <sup>I</sup>	94.8	94.9	94.9			
SSD mass, g	B <sup>I</sup>	3984.9	3989.3	3984.6			
Mass in water,g	C <sup>I</sup>	2349.1	2354.9	2352.1			
Volume, cc (B <sup>I</sup> -C <sup>I</sup> )	E <sup>I</sup>	1635.8	1634.4	1632.5			
Vol Abs Water, cc (B <sup>I</sup> -A)	J <sup>I</sup>	86.5	81.0	81.1			
% Saturation (100J <sup>I</sup> /I)		75.8	74.6	74.6			
% Swell (100(E <sup>I</sup> -E)/E)		0.006	0.018	0.012			
Load,N	P <sup>I</sup>	14468.6	15807.0	15248.9			
Dry Str. (2000P/(tDp))	Std				712.8	692.0	697.3
Wet Str. (2000P <sup>I</sup> /(t <sup>I</sup> Dp))	Stm	647.7	706.9	682.0			
Visual Moisture Damage (0 to 5)		1	1	1	<i>slight stripping observed</i>		
Cracked/Broken Aggregate ?		< 2%	< 2%	< 2%	<i>slightly cracked aggregates</i>		
Average Dry Strength (kPa)		700.7					
Average Wet Strength (kPa)		678.9					
TSR,%		96.9					

Table 8- determination of water sensitivity, Canada.

The first one presents the results with an aminic additive, where it is possible to conclude that the index of retained strength has a lower percentage compared with the second table which shows the results of Nanostructured Bitumen.

Should we have a look deeply in the figures, where we can conclude that the results after immersion are notably better with our Bitumen, where the average is 739,4 KPa.

In terms of TSR % the value achieved with Nanostructured Bitumen (104,2 %) is again a fantastic conclusion of the amelioration. Thus, regarding pavement life, this means the elongation of life several times more.

For sure, if it is compared with a pavement without additive, Nanostructured Bitumen could involve a huge improvement of roads life. Therefore there is no doubt that these figures indicate the reason of using the new bitumen.

Of course, the amount of money saved using Nanostructured Bitumen could be a great stimulus to begin the use of it. Because, as it has been said before, the more longer life of pavement, the less money used in its conservation.

### Moisture Sensitivity Data

AASHTO T-283

Mix: Ditecpesa/AME SP12.5FC1 D12 Cat D

exp. Global Number: 276720-1

Sample Remarks: Lab. Crushed Stone - 0.05% Ditecpesa Compound

Date sampled: July 31, 2017.

Sample		1	2	3	4	5	6
Diameter,mm	D	150	150	150	150	150	150
Thickness,mm	t	95.0	95.0	95.0	95.0	94.9	94.9
Dry mass,g	A	3900.2	3904.3	3906.8	3902.2	3905.5	3905.7
SSD mass, g	B	3923.0	3928.1	3928.2	3923.9	3928.1	3927.1
Mass in water,g	C	2293.0	2292.1	2293.4	2288.0	2294.0	2294.3
Volume, cc (B-C)	E	1630.0	1636.0	1634.8	1635.9	1634.1	1632.8
Bulk Sp Gravity (A/E)	F	2.393	2.386	2.390	2.385	2.390	2.392
Max Sp Gravity	G	2.562	2.562	2.562	2.562	2.562	2.562
% Air Voids (100(G-F)/G)	H	6.6	6.9	6.7	6.9	6.7	6.6
Vol Air Voids (HE/100)	I	107.7	112.1	109.9	112.8	109.7	108.3
Load,N	P	X	X	X	15938.9	15741.0	15938.9
<b>Saturated/Conditioned</b>							
Thickness,mm	t <sup>l</sup>	94.9	94.9	94.9			
SSD mass, g	B <sup>l</sup>	3981.2	3989.6	3989.7			
Mass in water,g	C <sup>l</sup>	2350.8	2353.2	2354.6			
Volume, cc (B <sup>l</sup> -C <sup>l</sup> )	E <sup>l</sup>	1630.4	1636.4	1635.1			
Vol Abs Water, cc (B <sup>l</sup> -A)	J <sup>l</sup>	81.0	85.3	82.9			
% Saturation (100J <sup>l</sup> /I)		75.2	76.1	75.4			
% Swell (100(E <sup>l</sup> -E)/E)		0.025	0.024	0.018			
Load,N	P <sup>l</sup>	16532.5	16268.7	16796.3			
Dry Str. (2000P/(tDp))	Std				712.1	704.0	712.8
Wet Str. (2000P <sup>l</sup> /(t <sup>l</sup> Dp))	Stm	739.4	727.6	751.2			
Visual Moisture Damage (0 to 5)		1	1	1	<i>slight stripping observed</i>		
Cracked/Broken Aggregate ?		< 2%	< 5%	< 3%	<i>slightly cracked aggregates</i>		
Average Dry Strength (kPa)		709.6					
Average Wet Strength (kPa)		739.4					
<b>TSR,%</b>		<b>104.2</b>					

Table 9- determination of water sensitivity, Canada.

## 2. IMPROVEMENT OF PROPERTIES

Most importantly, it is possible to recap the following improved properties:

- 2.1. Increasing around 10 times saline attack resistance; due to the covalent chemical union which makes more difficult bitumen-aggregate shifts on the surface, which could be attacked.
- 2.2. Immersion-Compression test; through chemical union are achieved the highest values what could being expect to this property.

Additionally, there are further improved properties, as well as are pointed below:

- 2.3. High resistance to fissure reflection; result of last generation polymer use, incorporated by chemical ways into the bitumen and chemical union by nanomaterial.
- 2.4. Longer useful life, better waterproofing; Useful life will be longer, consequence of more favorable fatigue. In terms of waterproofing, it will be an equivalent factor which is in function of granulometric dosage in the asphalt mix and bitumen content.
- 2.5. Less economic cost and environment improvement; thanks to pavement life elongation, it is no necessary to make pavement reinforcements at regular periods.
- 2.6. Analysis Life Cycle, present values are higher than the conventional ones due to the delay of pavement reinforcements necessity.
- 2.7. Faster Compaction; Are needed less passes to obtain the designed compaction, so the use of Nanostructured Bitumen reduces time and money due to over compaction, having the warranty of reach the designed compaction.

### 3. CONCLUSIONS

We have develop a new bitumen with better mechanical properties and for extreme climates; pavements with high Winter maintenance or beach areas in warm climates with high levels of humidity and salinity.

In fact, this Nanostructured Bitumen presents general improved properties and mainly a big resistance at salt chemical attack which is the majority element use to de-ice, at Winter maintenance. On the other hand, also helps in areas with important salt concentration closed to the sea.

Furthermore, better mechanical properties are achieved, as water sensitivity tests show a higher RCTI% with Nanostructured Bitumen, which means that water resistance is greater.

Moreover, it is an important technological advance with a financial impact, hence Nanostructured Bitumen increases box flow return taxes of concessional big projects in the long time, where pavement reinforcement could be delayed in time.

In summary, with Nanostructured Bitumen the compaction design is reached before that with conventional bitumen, hereby it is a huge amelioration regarding quality pavements and the reduction of laying times.