# Research report

# Project:

Analysis of asphalt shingles & SBS type elastomer membranes modified with nanosilica particles.

**Mai 2020** 





## **Context**

#### Analysis of modified and unmodified asphalt roofing products

The GONANO company has expressed an interest in analyzing the modifications made to asphalt shingles and SBS type elastomeric membranes treated with their roof protection product. To do such analysis, CNETE received roofing products made of asphalt without modifications and modifications. For each of the asphalt products, one sample was treated with the protective product that GONANO markets and one untreated. Also, a sample of the GONANO product was received to characterize its content. In total, 4 samples of asphalt shingles and elastomeric membrane were analyzed by Fourier Transform Infrared Spectroscopy (FTIR).



## **Objective**

This research aimed to identify whether there are differences between untreated asphalt and treated asphalt with the product marketed by GONANO.

This work was divided into the following stages:

- 1) Analysis technique.
- 2) Analysis of the protective product of GONANO.
- 3) Analysis of samples of asphalt roofing products.
- 4) Writing of the technical report and evaluation of the modifications.
- 5) Conclusion of modifications.

## 1. Analysis technique

#### Fourier transform infrared spectroscopy (FTIR)

Infrared (IR) spectra were obtained with a Nicolet <sup>TM</sup> FTIR spectrometer (Thermo Scientific <sup>TM</sup>) equipped with a deuterated triglycine sulfate (DTGS) detector and an iS5 Attenuated Total Reflectance (ATR) unit. The parameters of the FTIR analyzes to obtain the IR spectra carried out on the samples were 32 scans per spectrum at a resolution of 4 cm-1. IR spectra (measured in absorbance) were obtained at wavelengths between 400 and 4000 cm-1 (average IR). IR spectra were recorded with OMNIC (Thermo Scientific <sup>TM</sup> Software). For each sample, 6 spectra were produced and an average spectrum was produced and considered representative of each sample.

# 2. Analysis of the protective product

Table 1 shows the composition of the protective product. The protective product was deposited on a glass slide of the microscope and was analyzed by FTIR. The FTIR spectrum of the product is shown in Figure 1.



## 3 Sample analysis

### Analysis of asphalt shingles with or without protective product..

The IR spectra of the asphalt shingle without and with the product are shown in Figure 2 in red and blue, respectively. For asphalt shingles without product, the intensity of the IR spectrum is very low. For this sample were identified groups characteristic of organic compounds. Specifically, the IR bands in the 2960-2850 cm-1 region would correspond to the alkyl (C-H) bonds, and from 1260-1000 cm-1 to the alkoxyl (C-O) group.

On the asphalt shingle after treatment with the product that GONANO marketed, we observe functional groups' appearance specific to organosilicon compounds, such as the products shown in Table 1. More precisely, several infrared bands specific to silanes have been identified. The infrared bands of the region 3700-3690 cm-1 and 950-810 cm-1 corresponding to the Si-OH group of silica; the infrared band between 1770-1725 cm-1 (slightly marked) and 1260-1195 cm-1 corresponding to the acetoxy groups (C = O bonds of the ester groups); the bands 1385-1370 cm-1 and 1055-1030 cm-1 corresponding to the Si-alkoxy groups (Si-OCH (CH3) 2); those between 1260-1220 cm-1 corresponding to Si-CH2CH3 bonds; and the 1220-1170 cm-1 region corresponding to the alkyl groups (Si-CH2 (CH2) xCH3).

#### Analysis of the SBS membrane with or without protective product

The modified membrane, but without protective product (Figure 3, spectrum in violet color), shows absorption bands of 2960-2820 cm-1 and 1460-1380 cm-1, attributed to the CH stretches of the alkyls (CH2 and CH3). The 990-910 cm-1 absorption band is attributed to the vibration of vinyl groups (= CH), while the 750-700 cm-1 band corresponds to the vibrations of aromatic alkenes (= C-H and C = C), respectively. Then, characteristic peaks for polybutadiene at 966 cm-1 and polystyrene at 699 cm-1 were identified. These observations suggest that the asphalt membrane was modified with styrene-butadiene-styrene (SBS) polymers, as peaks characteristic of SBS were identified.



Compared to the membrane modified and treated with the protective product (Figure 3, red spectrum), a change in the chemical composition of the shingle's surface was observed during the application of the product. It has IR bands characteristic of modified asphalt shingles (mentioned below) and other absorption bands typical of the product marketed by GONANO (silane). For example, the IR absorption bands between 1770-1725 cm-1 and 1260-1195 cm-1 corresponding to the formation of ester groups with a silicon atom (Si-O-CO-CH3); absorption bands in the 1640-1600 cm-1 region characteristic of alkenes containing silicon atoms (Si — CH = CH-CH3); and the band of regions 1200-1110 cm-1 and 900 cm-1 correspondings to alkoxyl groups (Si-ORx).

#### Conclusion

Excellent dispersion of the nanosilica particles was observed in the matrix of the two roofing products. IR spectra of asphalt shingles and SBS elastomeric membranes show differences in functional groups compared to unmodified samples.

#### The following conclusions can be drawn:

- 1. The addition of nanosilica increases and regenerates the flexibility of two asphalt roofing products by creating new chemical bonds similar to the SBS type membrane.
- 2. The stability of the new chemical bonds of the nanosilica indicates a strong reaction with the asphalt products due to the coupling agent used and the many new bonds created by the product mixture.
- 3. Nanosilica make the two roofing product more resistant to high-temperature attributed to the improved dynamic modulus. On the other hand, at low temperatures, the bituminous binder modified with nanosilica will remain unchanged.
- 4. The results of the FTIR spectra demonstrated that the nanosilica used in the solution would delay and decrease the oxidative ageing process. This was confirmed by the major reduction of carbonyl groups in asphalt products. This decrease is attributed to the large specific surface area



of the nanosilica and the presence of new hydroxyl groups. Nanosilica particles are a known modifier for inhibiting oxidative reactions in products composed of organic materials.

5. Due to coupling agents' presence, hydroxyl groups on inorganic nanoparticles convert to various organic functional groups. The reaction causes the nanosilica to transform the hydrophilic surface of the asphalt into a hydrophobic surface. These modifications to the matrix will help the evacuation of liquids on the roofs by reducing their absorbances.

The application of the protection product for asphalt roofs marketed by GONANO demonstrates superior performance to the original products tested. Therefore, the anti-ageing performance and the fatigue cracking performance of GONANO modified asphalt mix are greatly improved. Also, the rutting resistance and anti-peel property of the nanosilica modified asphalt mix are improved.



# FTIR spectra results

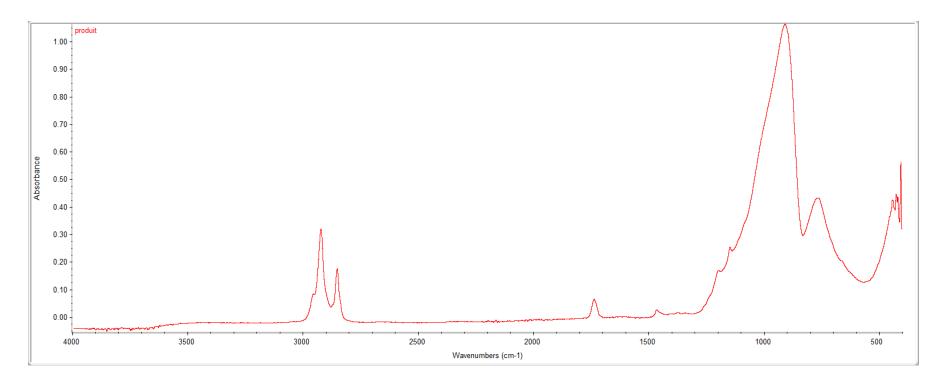


Figure 1. IR spectrum of the protective product used by GONANO.



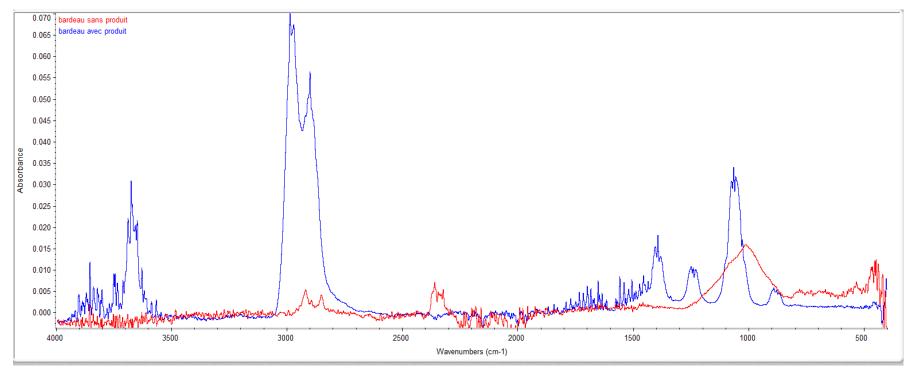


Figure 2. IR spectra of asphalt shingles with and without GONANO protective product.



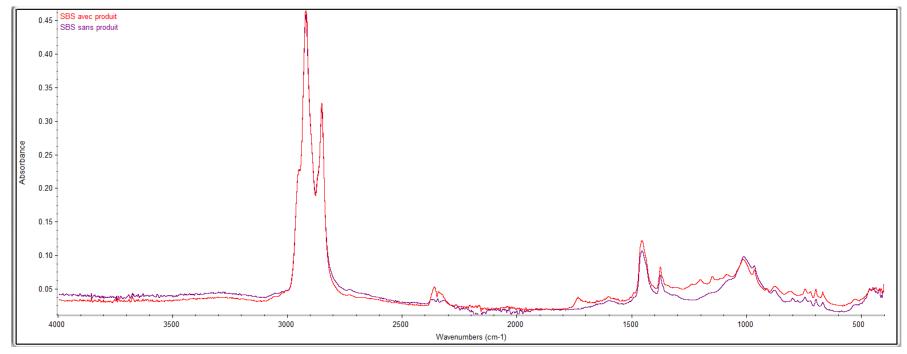


Figure 3. IR spectra of the elastomeric membrane (SBS) with product and without GONANO protective product.



### Références

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