

## Study Committee D1

### Materials and Emerging Test Techniques

Paper D1-PS2-11048

# Fingerprinting and Testing Methods of RTV Silicone-Coatings for Glass Insulators

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## Motivation

- The application of Room Temperature Vulcanized (RTV) silicone coatings to toughened glass insulators is an excellent solution to prevent contamination flashovers in heavily polluted areas.
- The hydrophobic capabilities and key physical and chemical properties of the silicone rubbers are crucial to ensure their satisfactory long-term performance in service.
- The increasing number of RTV silicone rubbers in the market, with very different formulations, properties and qualities, and the lack of standardization in this specific field, results in a need for new procedures and methodologies to identify, compare and evaluate silicone coatings from a quantitative point of view.
- This paper presents a research carried out to develop methodologies and test procedures to assess and compare six RTV silicone coatings.



Figure 1. Silicone coated glass insulators

## Experimental Approach

- **Test objects:** six different RTV silicone rubbers available on the market were tested.
- **Fingerprinting methods:** Fourier transform infrared spectroscopy (FTIR), thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC).
- **Physical features:** viscosity, density, solids content and drying times of the liquid silicones before application were studied.
- **Testing methods:** hydrophobicity transfer tests and low molecular weight (LMW) content.
- **Further research** was carried out to optimize the LMW fluid extraction methodology, by means of n-hexane solvent immersion, investigating the influence of test parameters which is of key importance for samples extracted from insulators in service.

## Fourier Transform Infrared Spectroscopy (FTIR)

- Analytical technique used to determine the chemical functional groups in the composition of the silicone.
- It can detect the type of the polymer and the presence of alumina trihydrate (ATH) filler, which is typically requested by utilities to improve the tracking and erosion resistance of the silicone.

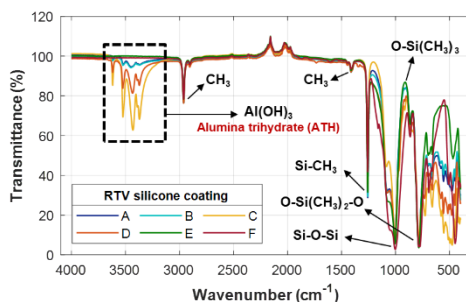


Figure 2. FTIR transmittance spectra

## Thermogravimetric Analysis (TGA)

- TGA measures the percent weight loss of a test sample while the sample is heated at a uniform rate in a controlled and specific nitrogen atmosphere.
- It is primarily used to determine the composition of the sample, including volatiles and inert filler, as well as indications of thermal stability.
- The TGA method gives information about the filler weight content. The percentage of ATH filler can be estimated from the TGA curves since it is related to the first mass loss observed in the thermograms.

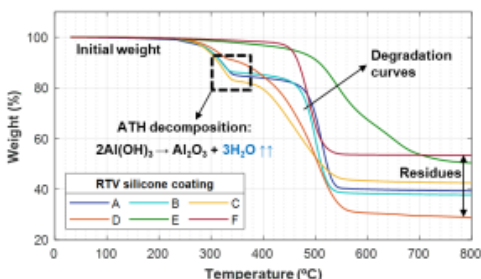


Figure 3. TGA analysis of the RTV silicone coatings

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### Differential Scanning Calorimetry (DSC)

- Qualitative and quantitative technique that provides a thermal fingerprint of a material.
- The difference in heat flow energy inputs into a tested material and a reference material is measured as a function of temperature while the tested material and the reference material are subjected to a controlled temperature program.
- This method was successful in determining the type of polymer, based on characteristic temperature (e.g., melting point and glass transition temperature)

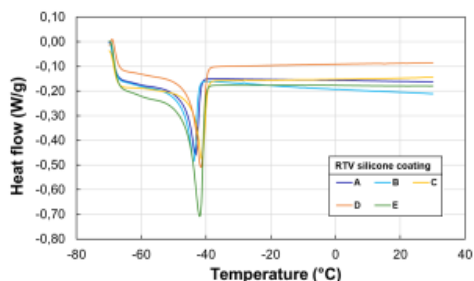


Figure 4. DSC analysis of the RTV silicone coatings

### Physical Features of the Coatings

- The characteristics of the liquid silicone material before application may affect the coating process.
- Density, viscosity, non-volatile-matter (solids) content, and drying times –among others- are key features of the material that affect the industrial performance of coating insulators.

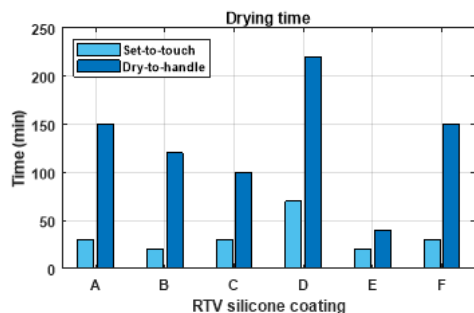


Figure 5. Evaluation of the drying times

### Hydrophobicity Transfer Test

- Methodology based on covering the silicone coating with a controlled pollution layer of nano silica.
- Then, the contact angle of a water droplet in touch with the coating is measured at fixed time intervals.
- These curves allow a quick evaluation of the hydrophobicity transfer capabilities of new silicones.

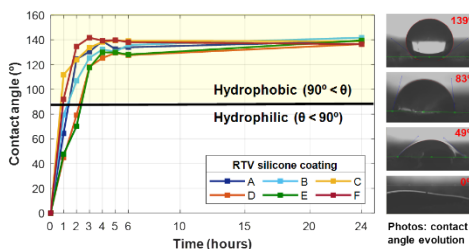


Figure 6. Hydrophobicity transfer tests results

### Low Molecular Weight (LMW) content

- Hydrophobicity is closely related to the diffusion of low molecular weight (LMW) siloxanes from the bulk to the surface of the coating.
- The LMW fluid extraction was carried out by means of n-hexane solvent immersion. Quantification of the LMW fluids is a key parameter of the hydrophobicity.
- The methodology was optimized investigating the influence of test parameters such as the immersion time and the influence of the sample morphology which is key for silicone samples taken from insulators in service.

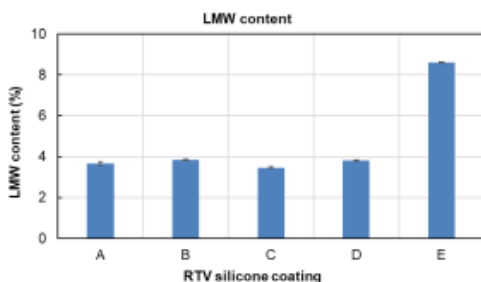


Figure 7. LMW content in different RTV silicone coatings

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### LMW – Sample Thickness

- Test performed using samples with thicknesses in the range between 50 and 400  $\mu\text{m}$  with an approximate mass of 0,5 g.
- Immersion time in n-hexane: 168 hours.
- Results: no thickness influence was observed in extracted LMW silicones content since it remained constant independently of the thickness of the sample.

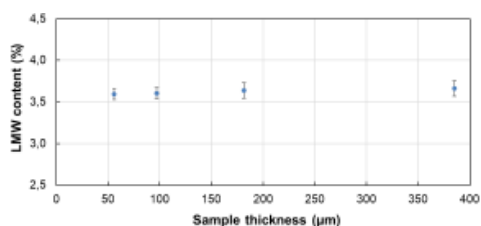


Figure 8. Sample thickness influence in LMW content

### LMW – Sample Mass

- Test performed using samples with masses between 0,1 and 10 g and a constant thickness of 380  $\mu\text{m}$ .
- Samples with masses around 0,5 g and thickness of 380  $\mu\text{m}$  can be used to test new RTV silicones since no influence of these two parameters was observed in LMW silicones extraction. In both cases standard deviations were low too.
- It should be noted that in case of aged samples where such sample control is not as accurate as with new samples different masses and morphologies could be used.

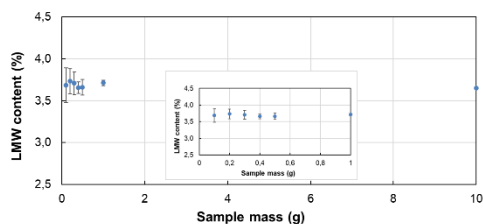


Figure 9. Sample mass influence in LMW content

### LMW – Immersion time

- Influence of immersion time in n-hexane was evaluated from 1 to 336 hours.
- Conducted tests showed that 168 hours is time enough to extract silicones LMW content.

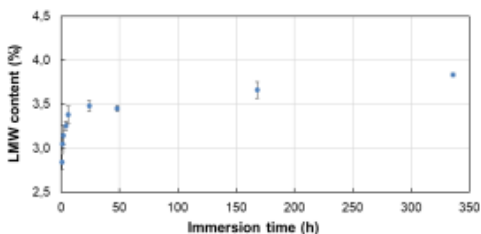


Figure 10. Immersion time influence in LMW content

## Conclusions

- The analyses of six different RTV silicone coatings showed that fingerprinting methods are a useful tool for the proper identification of the silicone material.
- Physical features of the silicones before application showed significant differences among them that may affect the application process as well as the coating performance.
- Hydrophobicity transfer properties were tested following a method based on the use of nano silica powder. This testing method delivers interesting results to evaluate the hydrophobicity performance of silicone coatings when new, but deeper analyses and long-term evaluations are still required to reach conclusions about the actual performance in the field.
- Finally, the method optimization carried out to extract LMW content in silicones was an important procedure to establish comparison between samples and get reliable results. As for the results obtained, it should be noted that no influence of sample morphology in terms of size and thickness is observed. However, immersion time should remain constant in every analysis. 168 hours have proven to be an effective time frame for LMW content extraction.