Intumescent Gels for Fire-Resistant Glass: Thermal Insulation, UV Protection, and Durability Studies

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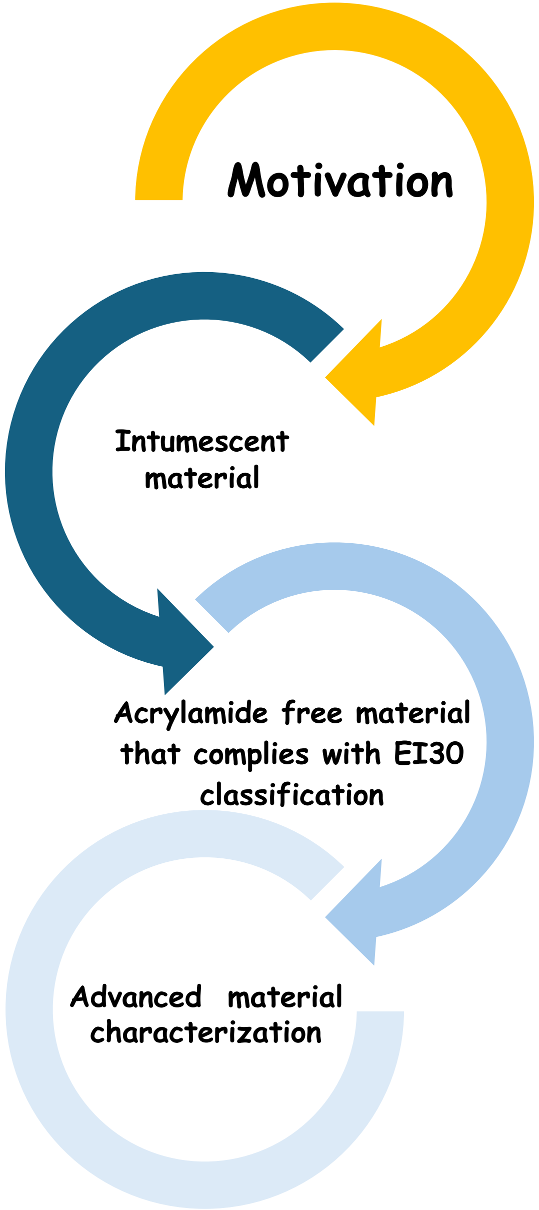
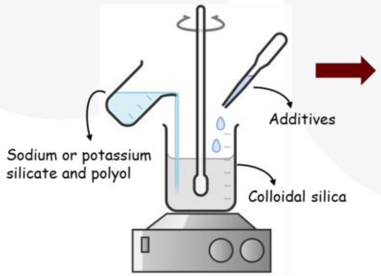
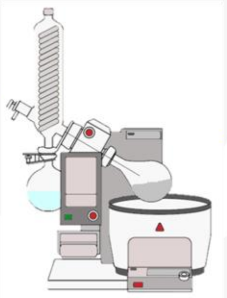
Introduction

- Despite relevant advances in fire safety strategies over recent decades, building fires remain a serious issue, causing thousands of incidents annually worldwide and resulting in injuries, property damage, and deaths.
- Fire-resistant glass is widely used in partition walls, fire doors, fire windows, escape routes, and staircases due to its excellent light transmission and protective properties ensuring compartmentation and safe evacuation.
- This type of glass contains transparent intumescent gel protective interlayers that expand when exposed to heat

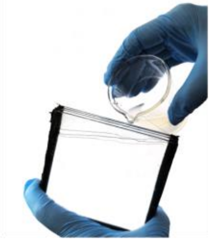


Synthesis





Optimization
- ✓ Sodium or potassium silicate
- ✓ Colloidal silicas with different characteristics (*e.g.* particle size)
- ✓ Molar ratio of SiO_2 and Na_2O or K_2O
- ✓ % of polyol
- ✓ % and type of additive
- ✓ Temperature and order of reagents mixing.
- ✓ Reaction time
- ✓ Water content



Glass laboratory prototypes:
- 2 to 4 glass plate assemblies (5x5 cm^2 or 10x10 cm^2)
- 1 to 3 mm thick intumescent layers between each pair

Cure conditions:
- T = 40-70 °C
- t = 2 - 5 h

Goal

Transparent and bubble free intumescent layers.

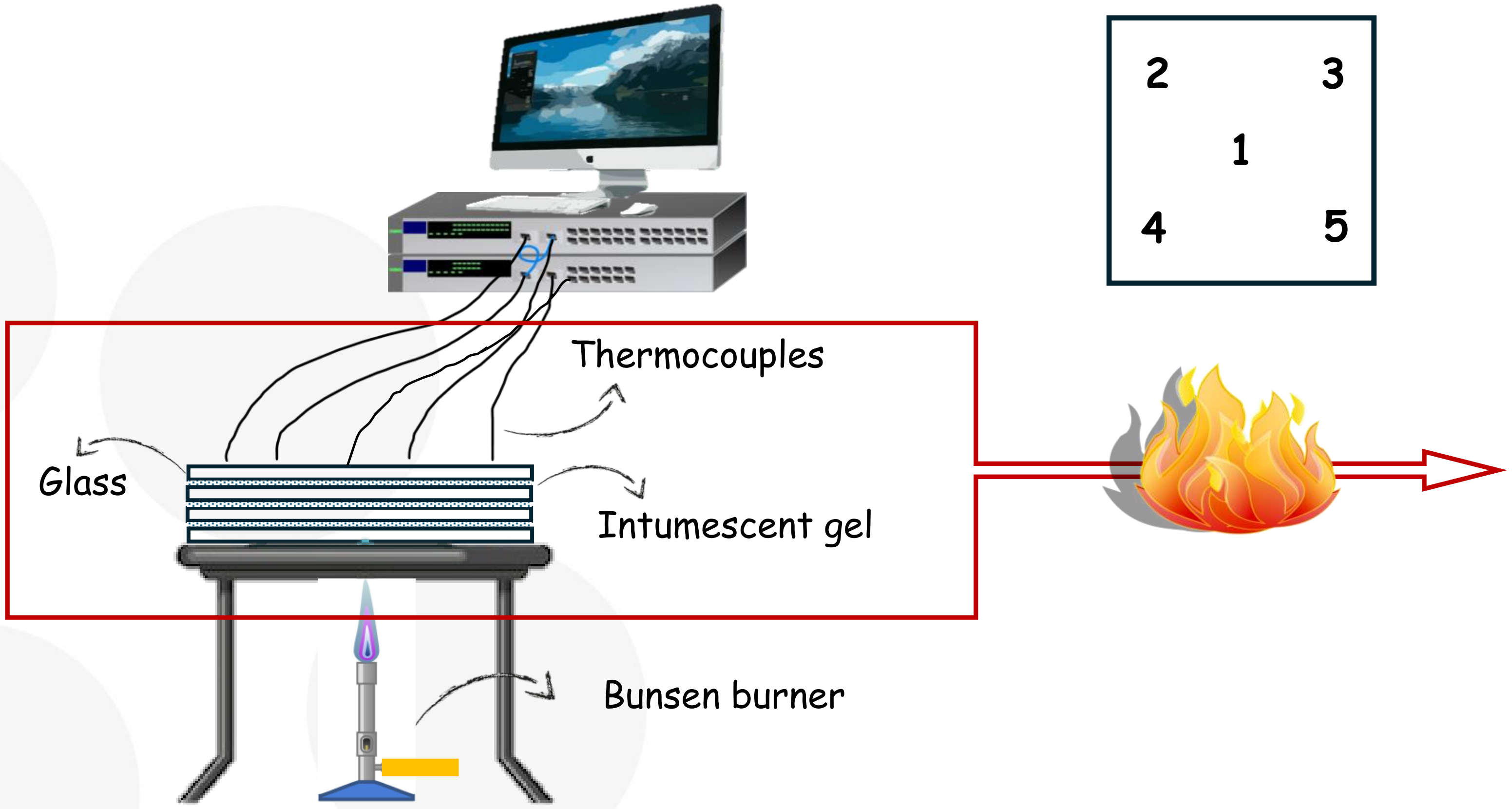
Development of intumescent formulations

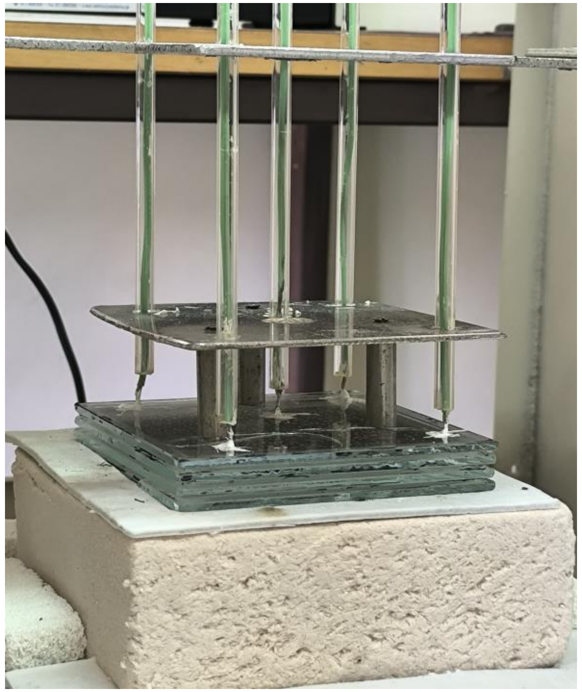
Assembling of glass prototypes

Cure

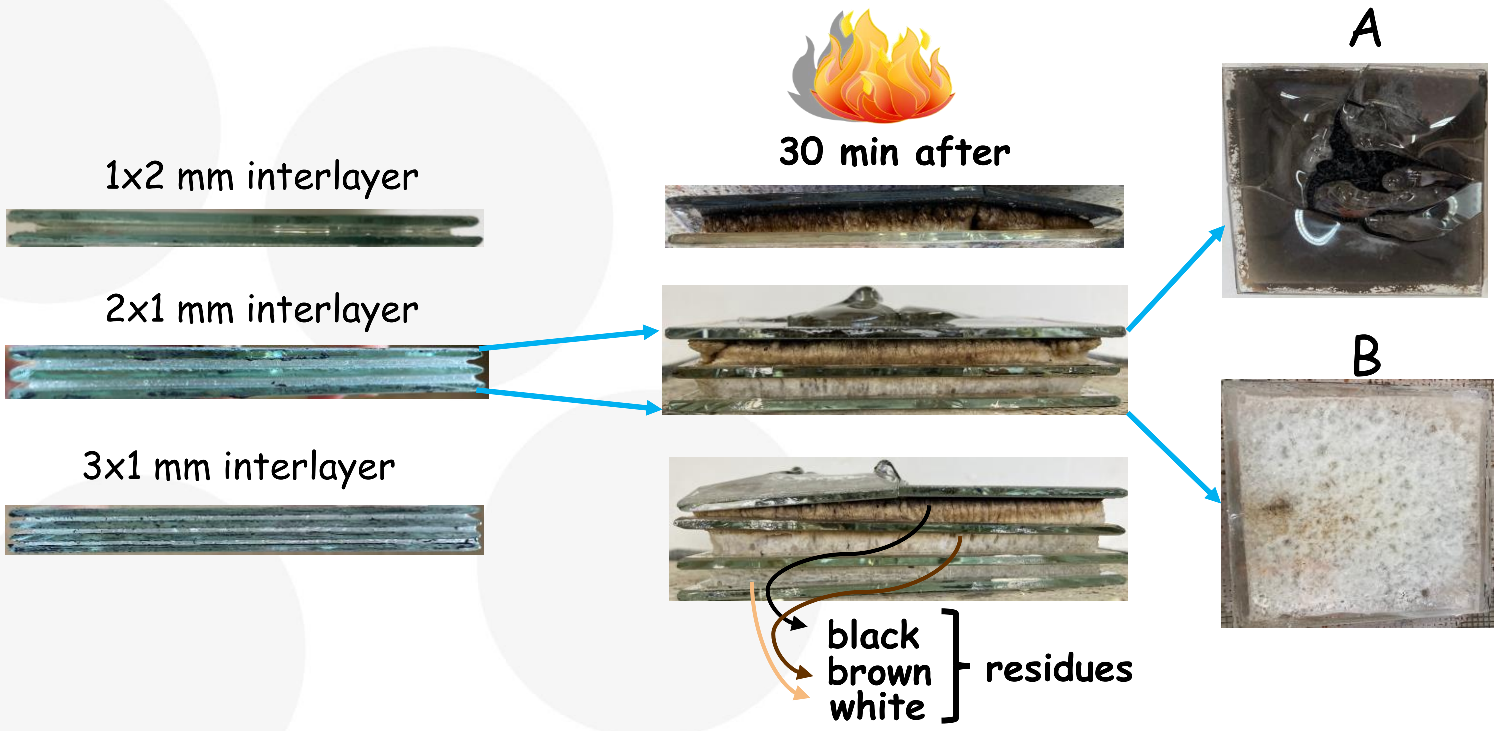
Selected formulations:
- **F4 ($Na_2O.SiO_2x$)**
- **F7, F9 ($K_2O.SiO_2x$)**

Fire tests





Fire tests



- After 30 min of fire exposure at T>1000 °C, the glass surface directly exposed to the flame (side A) exhibited breakage and partial melting. In contrast, the glass on the opposite side (side B) remained intact, maintaining its structural integrity
- The thickness of the intumescent interlayer increased significantly during fire exposure, expanding by a factor of 7 to 10

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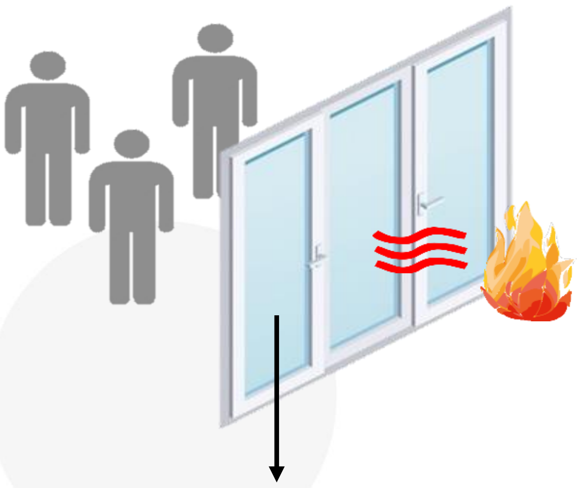
Fire tests



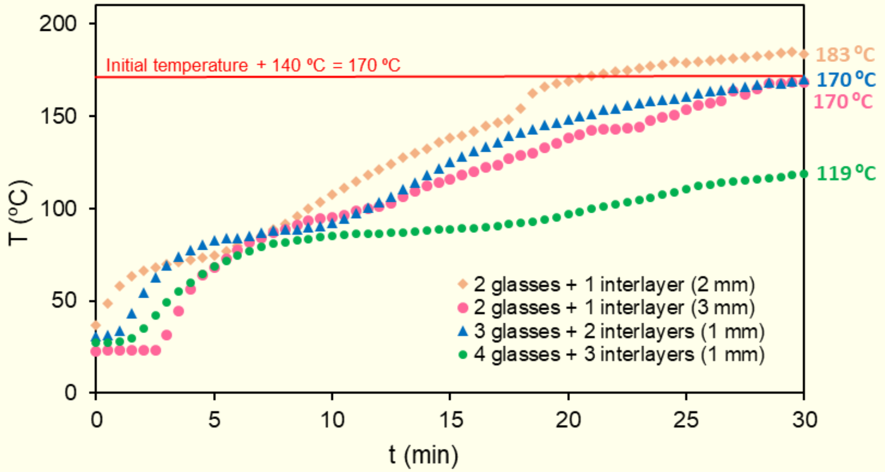
EI30 Fire-rated glass according to EN 13501-2 standard:

Integrity and insulation for 30 min.

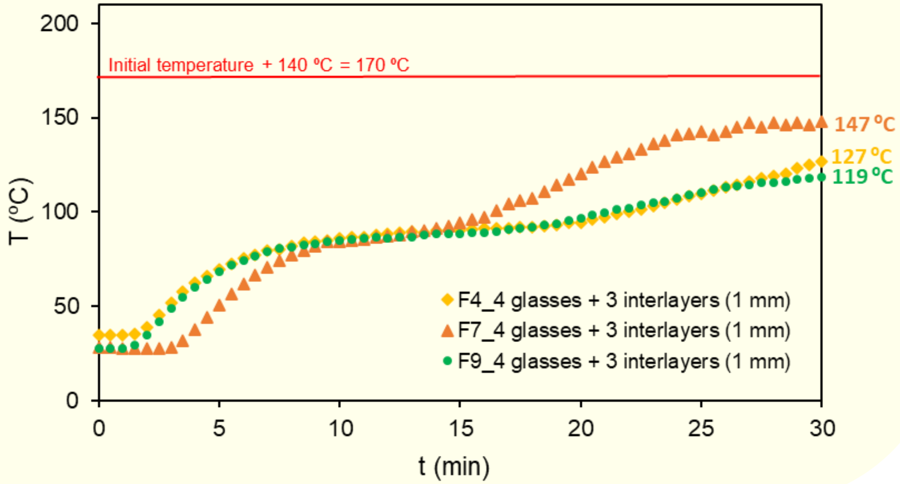
- Flames and flammable gases don´t pass.
- Temperature increase above ambient of opposite side of glass: Maximum ≤ 180 °C for each individual measurements, average temperature measurements ≤ 140 °C).

Thermal Insulation

F9



F4, F7 and F9



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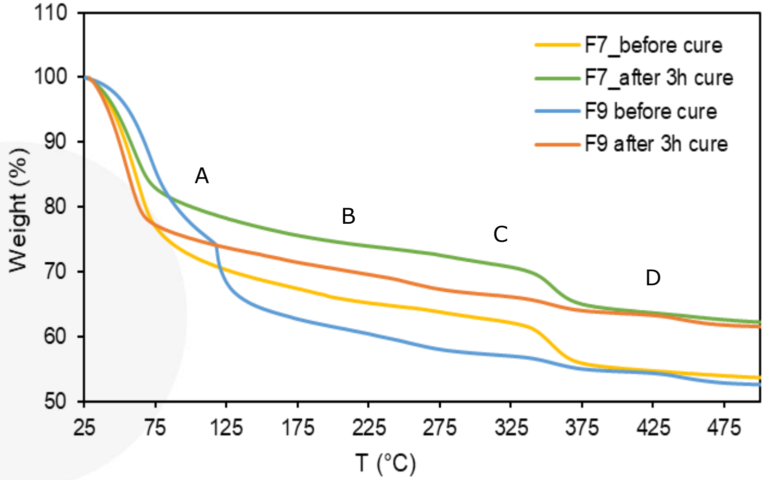
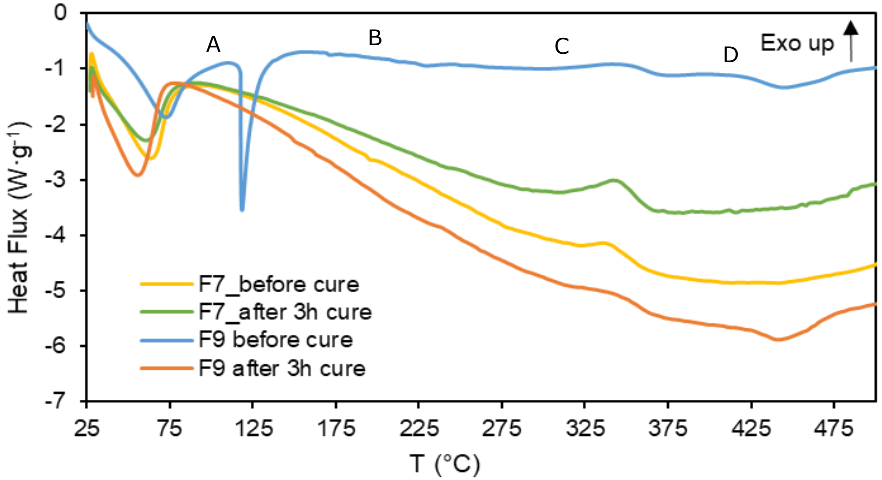
TGA/DSC

TGA



DSC



- Up to Point A (~100–110 °C): Evaporation of free and physically adsorbed H_2O occurs
- Between A and B (110–200 °C): The onset of intumescence is observed, associated with the dissociation of H_2O from various chemical species, including different forms of silicate ions
- Between B and C (200–320 °C): Dehydration of polysilicate silanol groups takes place, leading to the formation of siloxane bonds and the release of H_2O
- Between C and D (320–420 °C): Exothermic reactions are detected, attributed to the thermal degradation of polyol components, with a peak around 350 °C

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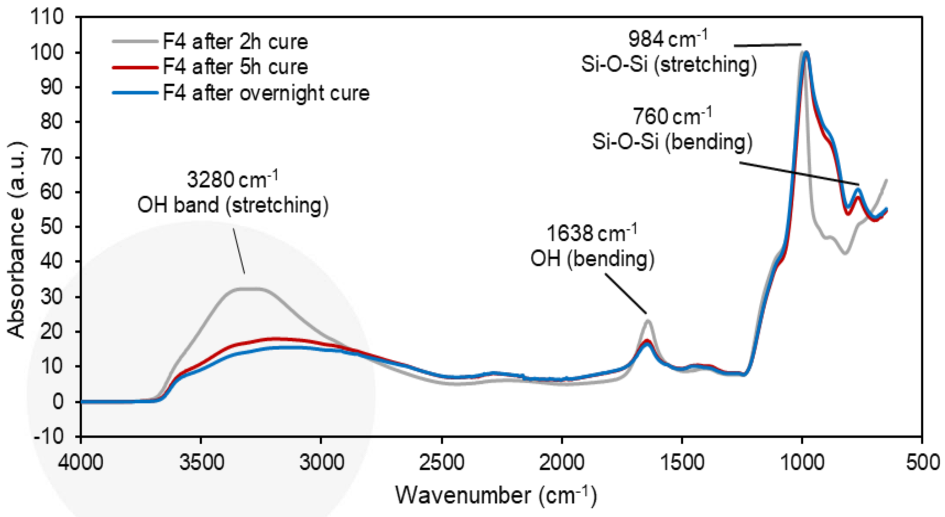
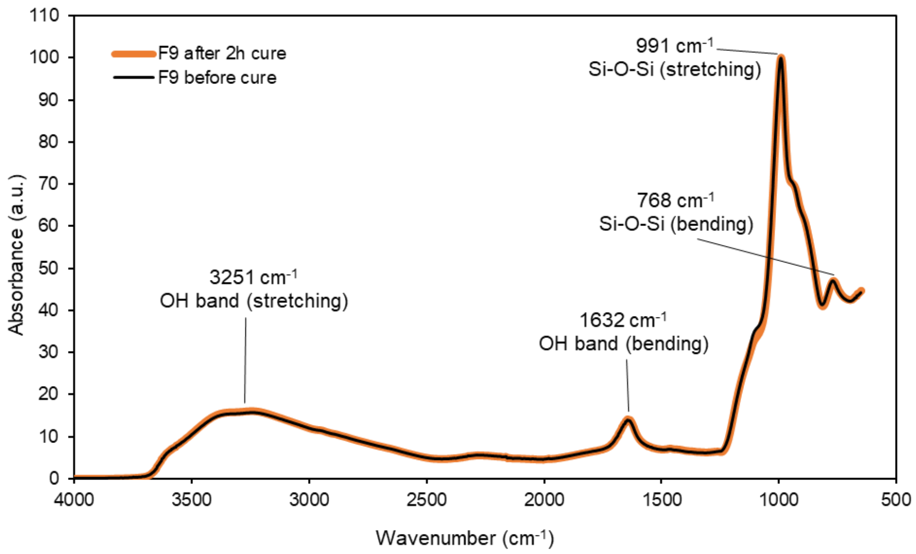
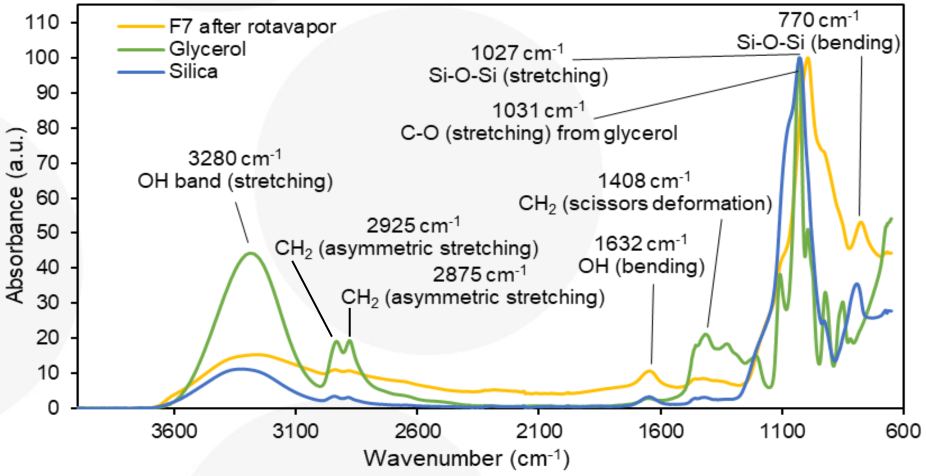
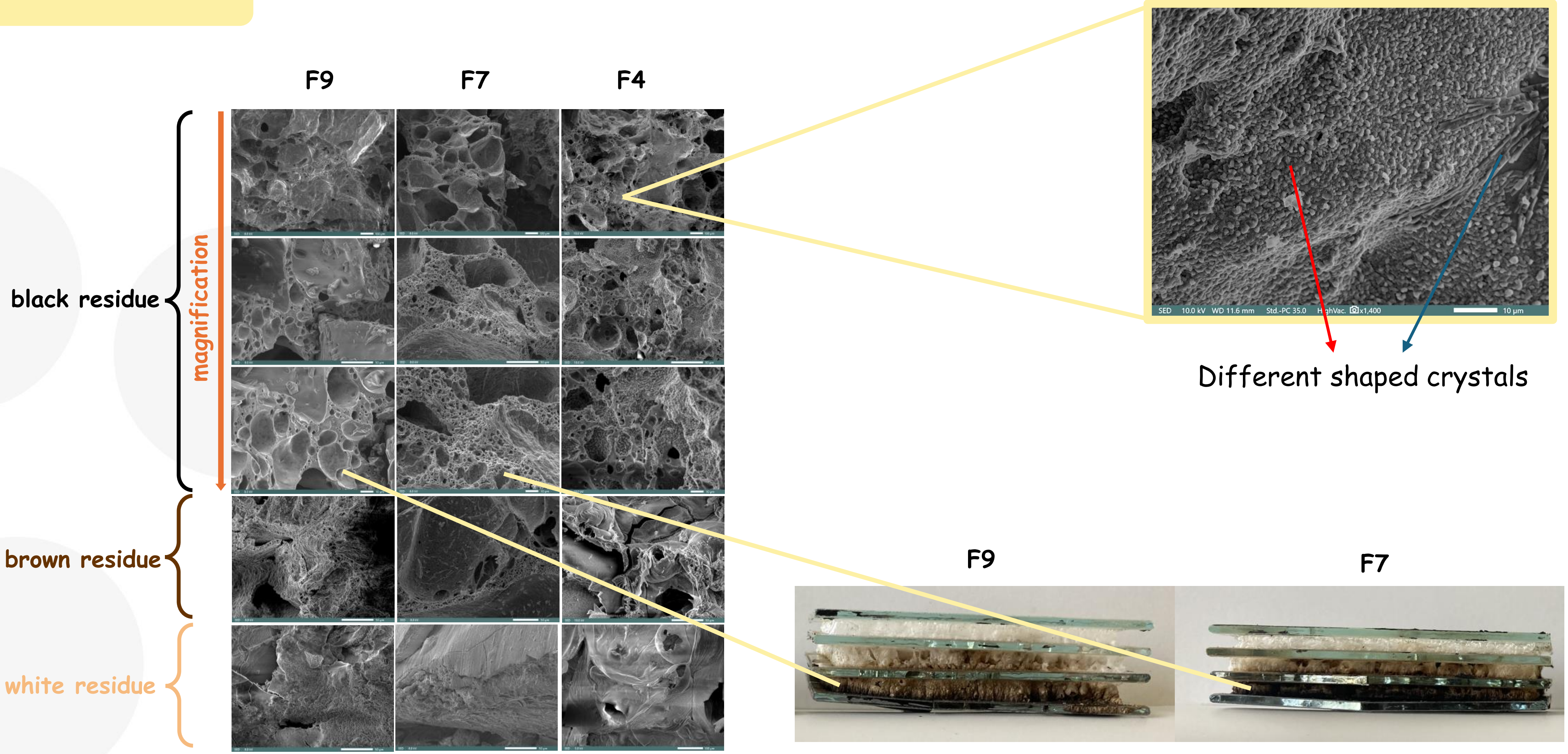
FTIR-ATR


F4 after 2h cure
F4 after 5h cure
F4 after overnight cure
984 cm-1
Si-O-Si (stretching)
760 cm-1
Si-O-Si (bending)
3280 cm-1
OH band (stretching)
1638 cm-1
OH (bending)
Absorbance (a.u.)
Wavenumber (cm-1)


F9 after 2h cure
F9 before cure
991 cm-1
Si-O-Si (stretching)
768 cm-1
Si-O-Si (bending)
3251 cm-1
OH band (stretching)
1632 cm-1
OH band (bending)
Absorbance (a.u.)
Wavenumber (cm-1)


F7 after rotavapor
Glycerol
Silica
770 cm-1
Si-O-Si (bending)
1027 cm-1
Si-O-Si (stretching)
1031 cm-1
C-O (stretching) from glycerol
3280 cm-1
OH band (stretching)
1408 cm-1
CH2 (scissors deformation)
2925 cm-1
CH2 (asymmetric stretching)
1632 cm-1
OH (bending)
2875 cm-1
CH2 (asymmetric stretching)
Absorbance (a.u.)
Wavenumber (cm-1)

SEM



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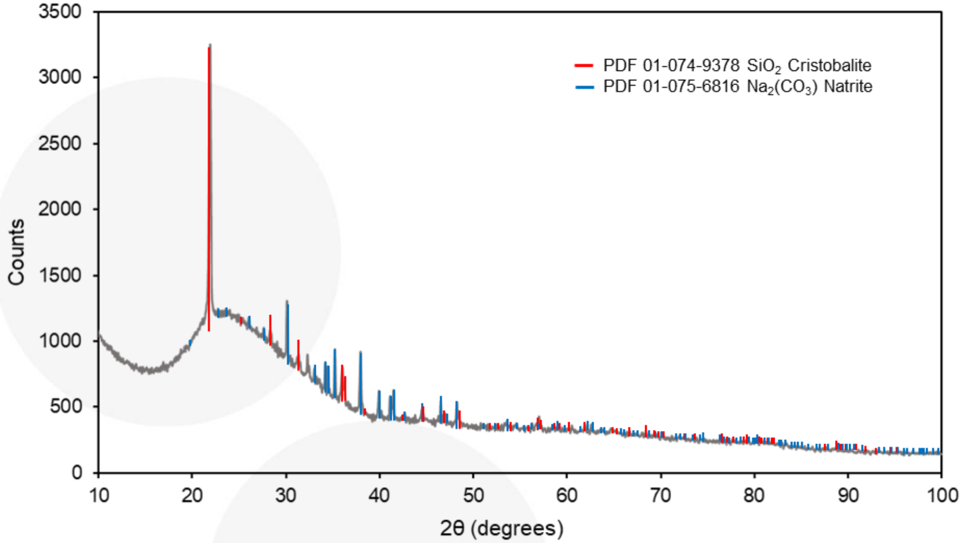
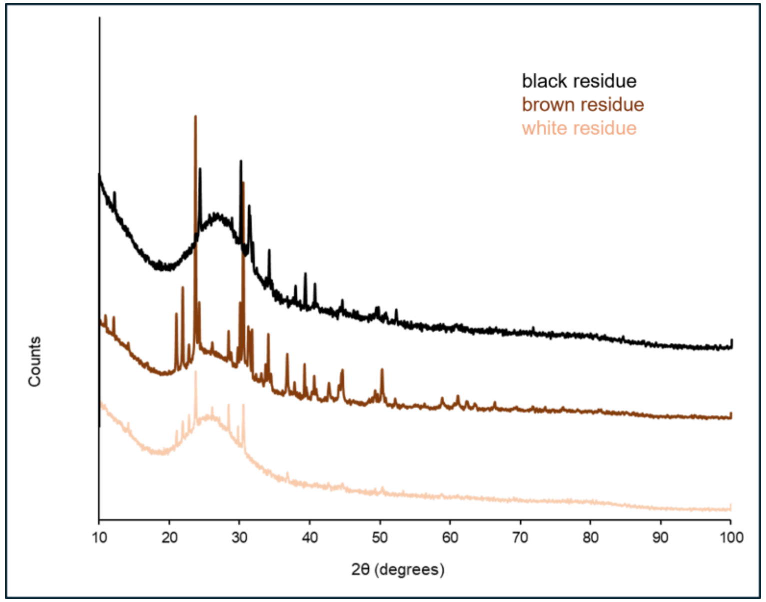
XRD

F4



Different shaped crystals detected in the SEM images of F4 black residue fire, were identified in XRD as cristobalite (tetragonal silica) and natrite (monoclinic sodium carbonate).

F9



XRD peaks correspond to metastable phases, which are different for each residue.

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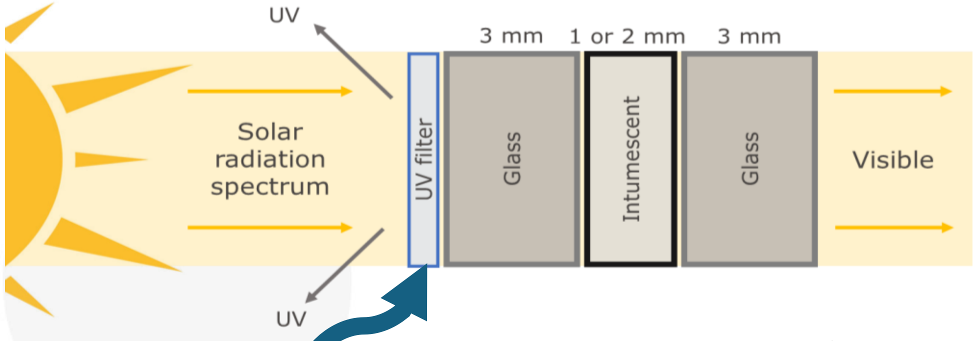
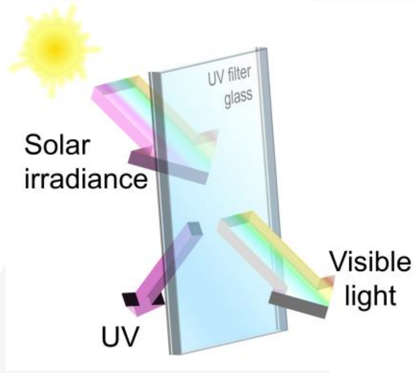
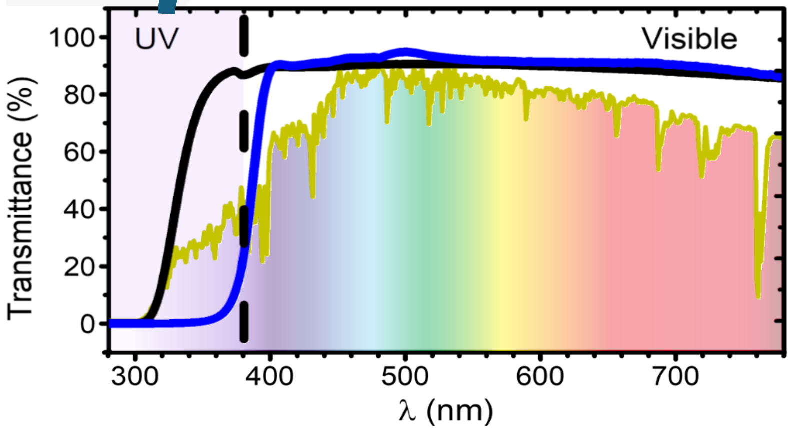
UV Protection and Durability Studies

Application of UV filters to prevent interlayers degradation by UV radiation



Bragg reflector composed of TiO_2/SiO_2 stacks





Durability tests according to ISO 12543-4:2021

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Radiation test- exposure to Xenon arc source (336 h)

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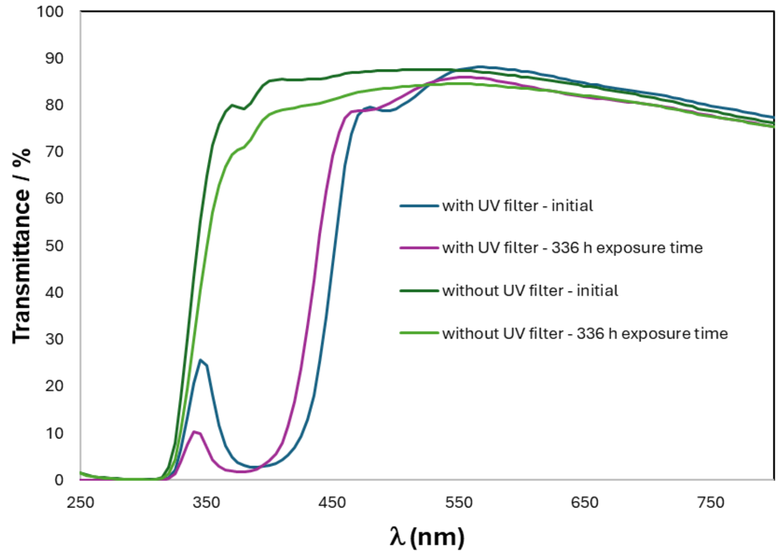
UV-Vis



- Multilayer Bragg Reflector (MBR) coating to the outermost surface proved effective for UV protection.

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Concluding remarks

- While all tested formulations complied with the normative thermal insulation requirements, the potassium silicate formulation F9 demonstrated superior efficiency compared to F4 and F7.
- TGA/DSC confirmed the water content at various formulation stages, as well as the temperature ranges associated with the degradation of organic components.
- Preliminary durability tests of the glass assemblies indicated improved UV protection, attributed to the application of a MBR coating on the outermost surface.
- SEM micrographs revealed that the black residue of formulation F9 exhibited larger pore sizes, correlating with the greater expansion (interlayer thickness) observed after fire exposure, when compared to the residues of F4 and F7.
- XRD analysis of the F4 black residue (post-fire) revealed the presence of cristobalite (tetragonal silica) and natrite (monoclinic sodium carbonate), findings that align well with the microstructural features observed by SEM.
- Ongoing work aims to further optimize the formulations and optical filters, and to complete the comprehensive materials characterization and durability testing.

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r2u modular systems

PRR Plano de Recuperação e Resiliência

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