Fire-resistant materials: characterization and thermal behavior of intumescent gels for fire resistant glass

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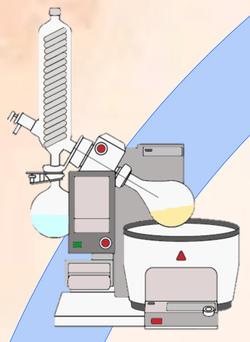
Synthesis

1 Mixing the reagents in adequate proportions

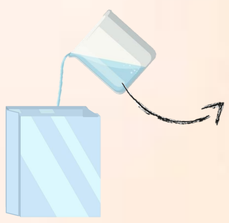


✓ Formulations with various additives and different curing conditions

✗ **Acrylamide-free**, avoiding acrylamide toxic effects on fertility and nervous system [3]

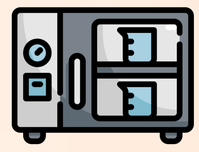
2 Assembling the glasses with intumescent interlayers

❖ 2 – 4 glasses and 1 – 3 intumescent interlayers (≤18 mm total thickness)



3 Testing different curing conditions

T = 40 – 70 °C



t = 2h
t = 3h
t = 4h
t = 5h
t = overnight

Transparent and bubble-free layers

Motivation

There are thousands of building structure fires every year worldwide, often resulting in injuries, loss of property and death. [1]

Fire safety in modern construction is extremely important.

Fire-resistant glass is commonly used in construction to restrain flames and block the smoke during a fire.

This type of glass contains **transparent intumescent gel** protective interlayers that **expand when exposed to heat**.

Intumescent material

Main goal

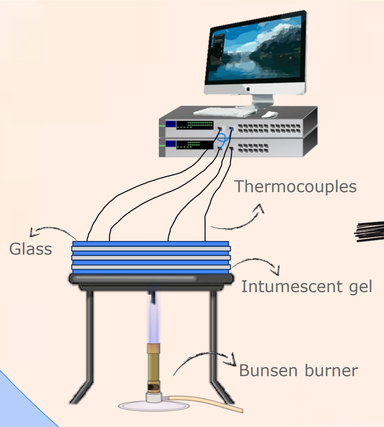
✓ Material that complies with EI 30 classification, and its characterization before and after reaction to fire

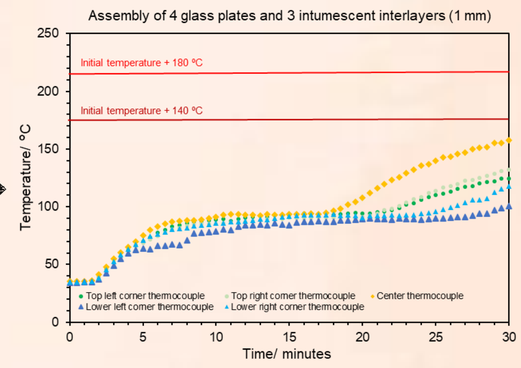
EI 30 classification, NP EN 13501-2 standard [2]

E – integrity
I – insulation
30 minutes after the onset of a fire

✓ Flames and flammable gases do not pass
✓ Limited heat transfer through conduction, convection and radiation
✓ Allows safe evacuation of people from buildings

Characterization





According to NP EN 13501-2 standard [2]:

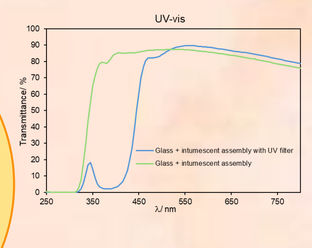
- Average temperature values must be ≤ 140 °C above the initial temperature ✓
- None of the individual temperature measurements should exceed 180 °C in relation to the initial temperature ✓

Fire tests

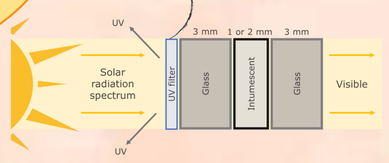
Before fire tests

Black residue

Optical characterization

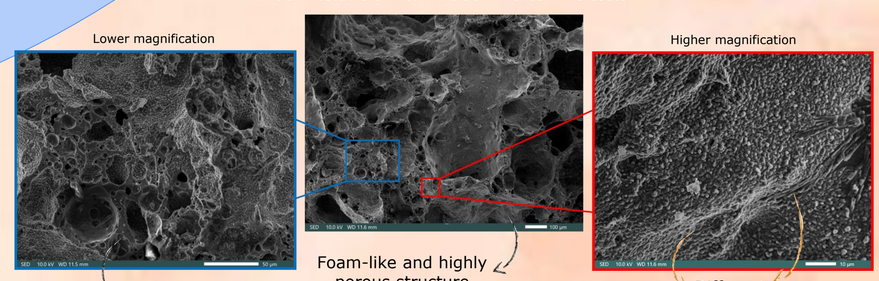


Bragg reflector composed of TiO_2/SiO_2 stacks



SEM

Black residue of Formulation 4 after fire tests



Crystalline spots in amorphous material

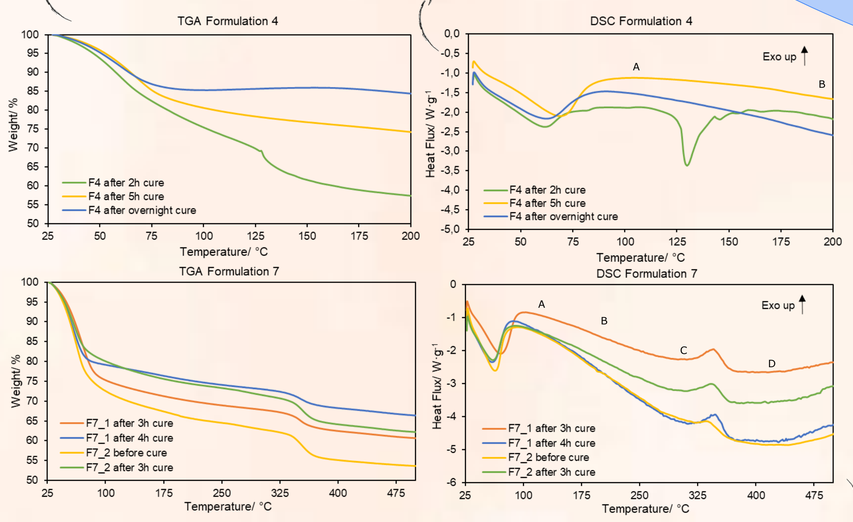
Foam-like and highly porous structure

Different shaped crystals

DSC/ TGA

Mass losses are consistent with the preparation phase and the amount of water in the formulation

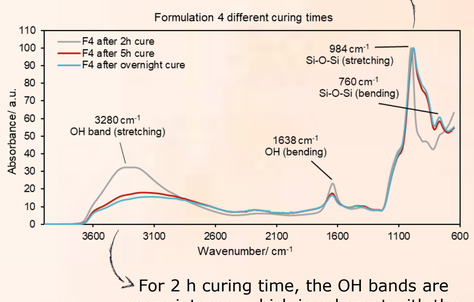
Results highly dependent on sampling



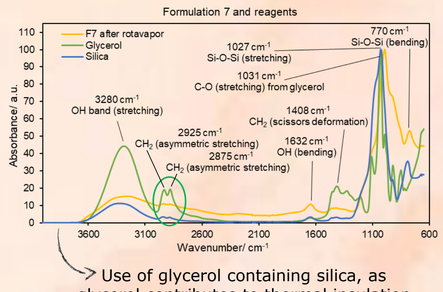
Up to A: Evaporation of free and physically adsorbed water (up to ~ 100 – 110 °C)
A – B: Initial intumescence of the sample, with water dissociation from various species, *e.g.* different forms of silicate ion (from 110 – 200 °C)
B – C: Dehydration of polysilicate silanol groups to siloxane with release of water (from 200 – 320 °C)
C – D: Exothermic peaks in the 320 – 420 °C range due to the degradation of glycerol (~350 °C)

FTIR-ATR

No significant difference of OH bands intensity and the envelope with maximum at 984 cm^{-1} after 5h curing time



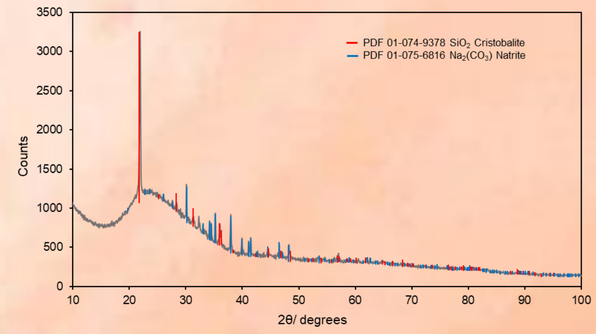
For 2 h curing time, the OH bands are more intense, which is coherent with the amount of water on the formulation



Use of glycerol containing silica, as glycerol contributes to thermal insulation

XRD

Black residue of Formulation 4 after fire tests



Concluding remarks

- ✓ FTIR-ATR and DSC/TGA results consistently confirmed the water content at each formulation stage.
- ✓ XRD of Formulation 4 after fire tests (black residue) identified **cristobalite** (tetragonal silica) and **natrite** (monoclinic sodium carbonate), which correlate well with the microstructural features observed by SEM.
- ✓ Visible light transmission ≥80% was obtained with UV protection filters, necessary for glazing durability.
- ✓ The assembled glass prototypes meet the integrity and thermal insulation requirements for **EI 30** fire-resistance classification, indicating their suitability for fire-protecting glazing applications.
- ✓ On-going studies are investigating the **intumescent gel behavior**, especially the different **multilayer structures formed during fire testing** and its role in thermal shielding performance.

References: [1] W. Liu, X. Ge, X. Zhou, Y. Tang, Thermal intumescent behavior of a gel containing silica, RSC Adv. **2015**, *5*, 33208-33211; [2] ISO 13501-2, Fire classification of construction products and building elements – Part 2: Classification using data from fire resistance and/or smoke control tests, excluding ventilation services. [3] W. Liu, X. Ge, Z. Zhang, Study of the relationship between thermal insulation behavior and microstructure of a fire-resistant gel containing silica during heating, Fire and Materials, **2018**, *42*, 44–49.

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