



Fibre-reinforced glass/silicate composites: effect of fibrous reinforcement on intumescence behaviour of silicate matrices as a fire barrier application



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ABSTRACT

This paper is the third in series on fire-resistant laminated glass composites containing an intumescent silicate interlayer and studies the effect of reinforcing fibres on the intumescent behaviour of the silicate matrices. Two silicate matrices with different silica/metal oxide ratios ($\text{SiO}_2\text{:Na}_2\text{O}$) were reinforced by polypropylene (PP), polyamide 66 (PA66), AR-glass (ARG) and stainless steel (ST) fibres, selected because of their alkali and UVA resistant properties. Thermal degradative behaviour of fibre/silicate mixtures having 5/95 and 10/100 mass ratios were examined to understand the effect of each fibre type on the intumescence of each silicate. Fibre reinforcements of the silicate layer were either as a nonwoven web or as a woven mesh. The intumescent properties of silicates were studied by heating the composites in a furnace at 450 °C for five minutes and measuring the intumescent layer thickness. The results showed that all fibre types in nonwoven web reinforcements had a negative effect while inorganic glass fibre in a woven mesh form had a negligible effect on the overall intumescence of silicate matrices. It was proposed that while fibre type was of minor importance, the fabric structure played an important role in inhibiting intumescence. Preferred reinforcement should preferably have open mesh-like characteristics.

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1. Introduction

It is well known that glass is not combustible but on exposures to high temperatures or fire, a monolithic glass pane cracks and cannot act as an effective barrier for advancing fire and smoke to spread in adjoining compartments of a building or a ship, for example. For such applications where fire safety regulations require the use of fire barriers, fire-rated glass products capable of offering varying degree of protection ranging from 20 min to 3 h according to BS476 Part 22, are used [1,2]. The fire-rated glass acts as a passive fire protection, restricting the fire damage to a limited area.

Out of many types of fire-rated glass developed over last three decades [3–5], the most successful and widely used is a multi-layered laminated glass, where a soluble alkali-metal silicate as an intumescent interlayer is sandwiched between two glass sheets [5]. The silicate interlayer intumesces on exposure to fire, providing a degree of fire protection for a period of time. The intumescence of the alkali-metal silicate internal layer results from rapid liberation of water vapour on exposure to high temperatures. Water-soluble sodium silicate is obtained by reacting silica with sodium or potassium carbonate [6,7] or bicarbonate,

maintaining $\text{SiO}_2\text{:Na}_2\text{O}$ molar ratios (R) from 1.6 to 3.9. As the ratio R ($\text{SiO}_2\text{:Na}_2\text{O}$) increases, pH value decreases [7]. Dilution of solutions decreases the pH and high molecular mass particles are formed due to rapid polymerization and simultaneous aggregation. For greater aggregation, higher R values (>3.5) are preferred [8].

Intumescent soluble silicates consist of water in free and bound forms [9]. The bound water may be (i) hydrogen-bonded to surface silanol group of polysilicate ions, (ii) as ionic hydration, where water molecules are associated with cations and polysilicate ions, and (iii) structural water present as SiOH groups on polysilicate ions [10,11]. Free water and water hydrogen-bonded to silanol groups are released at low (<130 °C) or even at room temperatures [11]. On the other hand, ionically hydrated water is released in the temperature range 130–200 °C, where its rapid evolution generates intumescence. Structural water is released at comparatively high temperatures, ranging from 200–500 °C while the polysilicate elements begin to cross-link by condensation of SiOH groups to form $-\text{Si}-\text{O}-\text{Si}$ bonds. At approximately 900 °C the sodium silicate starts to melt to form sodium silicate glass. The overall intumescence property and solubility of silicates depends on the strength of cationic cross-links between adjacent polysilicate particles. Degrees of solubility and intumescence both decrease in the order $\text{K}^+ > \text{Na}^+ > \text{Li}^+$ i.e., the smaller the cationic atom, the lower the degrees of intumescence and solubility [11]. Thus ionic hydration is the primary mechanism for the water absorption of the silicate samples.

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The SiO₂:M₂O molar ratio also affects the dehydration and intumescence of soluble silicates [12], for which a lower ratio is more favourable. If the SiO₂:M₂O ratio is increased i.e. pH value is reduced, the polysilicate particles size increases, the surface charge density decreases and the solution becomes more colloidal. Such solutions have a higher probability of forming –Si–O–Si–bond between particles as the solution dries thus providing greater water resistance and a decrease in the intumescence property [12]. During initial fire exposure, the intumescent interlayer absorbs heat and when the interlayer reaches the required temperature, the residual water in the interlayer evaporates consequently consuming a major part of the energy released by the incident fire radiant energy. Concurrently, the interlayer expands generating a thick, tough insulating shield and hard foam, which works as a protection against smoke and flame penetration. Such glass–silicate–glass composites when exposed to fire, can produce a degree of fire protection for considerable period of time, but have limited impact resistance [13].

In our previous publications we have explored the use of some alkali-resistant fibres [14,15] as a reinforcement to increase the impact resistance of laminated glass–silicate–glass composites, which are commercially used as fire-rated glass products [5]. The results indicated that certain fibres such as polypropylene, polyamide 66, alkali resistant glass and steel fibres could significantly enhance their impact performance [15], although transparency of the matrices sandwiched between glass sheets can be affected, depending upon the reinforcement type. In this paper the effect of these reinforcing fibres on the intumescent behaviour of the silicate matrices within glass composites under thermal shock is studied. To best of our knowledge such a systematic study has not been carried out before.

2. Experimental details

2.1. Materials

2.1.1. Intumescent silicate matrices

Two types of water-soluble sodium silicate matrices, PS-A and PS-B were sourced from Pilkington plc (UK). These silicate solutions contain silica and sodium oxide at different molar ratios with pH 11.8 and 13.6, respectively. Their compositions are reported in Table 1.

2.1.2. Fibres

These have been fully described in our previous publications [14,15] and are summarized below and in Table 2:

- (1) Polypropylene (PP): Nonwoven web of area density, prepared from UV stable PP fibres (Fibre Vision, Denmark) using an Automatex laboratory nonwoven line.
- (2) Polyamide 66 (PA66): Nonwoven web of area density 19 g/m² from PA66 fibres (DuPont, USA), produced similarly to PP.
- (3) Alkali resistant glass (ARG) fibre: Two types of reinforcements were used; nonwoven veil (ARGV) of 44 g/m², woven mesh (ARGM) from continuous filaments (0.8 mesh/cm) of 88 g/m², both sourced from Nippon Glass, Japan.
- (4) Steel (ST): Woven mesh from continuous stainless steel filaments (12 mesh/cm; 40 g/m²), sourced from TWP Inc., USA.

Table 1
Silicate matrices and their compositions.

Silicate solutions	SiO ₂ (%)	Na ₂ O (%)	SiO ₂ :Na ₂ O (w/w)	Molar ratio	Solid content (%)	pH
PS-A	31.1	12.45	2.5:1	2.58:1	43.6	11.8
PS-B	28.4	8.60	3.3:1	3.41:1	37.0	13.6

2.2. Sample preparation

2.2.1. Silicate-fibre mixtures for thermal analysis

Mixtures of pulverized silicate matrices and fibres were used for thermal analytical studies. Dry silicate matrices were pulverized using a pestle and mortar yielding particle sizes <1 μm. PA 66, PP and ARGV fibres were pulverized also yielding particle sizes <1 μm by using a Wiley mill with a 1 mm screen. Then pulverized matrices were mixed properly with 5 and 10% (by mass) of pulverized fibres and samples are referred to as PS-A + 5% F and PS-B + 5% F (Tables 3 and 4), where F = PP, PA66 or ARG. The steel fibre was not used in this study as it was difficult to pulverize the fibres in the lab.

2.2.2. Glass–silicate–glass composite laminates

To prepare fibre-reinforced silicate layered glass composites, a fixed amount of nonwoven web or mesh (125 × 125 mm) was placed on a 3 mm thick silicate glass sheet (bottom layer of composite) with a silicon side barrier (10 mm height) fitted. The fibre content was selected based on our previous work to provide optimized mechanical properties and the transparency to these composites [15]. Then a fixed amount of silicate solution was poured very slowly over the web or mesh and the whole assembly was transferred to an oven and dried at 100 °C for 14–16 h to create 1.7 ± 0.2 mm thick silicate layer. The side barriers were cut away and another 3 mm thick glass sheet was placed on top of the dry glass/silicate sheet. A small amount of glycerol was used to wet the dry silicate surface before lowering this second glass sheet and whole assembly was further dried for 12 h at 90 °C under a load of 10 kg/m² to make sandwich-type, laminated glass–silicate–glass and glass–silicate/fibre–glass composite structures (see Fig. 1(a)). The details of the composite samples are given in Table 2, in which samples are referred to as PS-A/B + FR, where FR = fibre reinforcement, e.g., PP, PA66, ARGV, ARGM and ST.

2.3. Thermal stability and intumescence performance evaluation

2.3.1. Thermal analysis

For simultaneous DTA/TGA analysis, a TA Instrument STD2606 was used under flowing nitrogen (100 ml/min) at a heating rate of 10 °C min⁻¹. Sample mass of 10 ± 0.5 mg was used every time.

2.3.2. Intumescence performance of laminated composites

This was determined in terms of the change of silicate layer thickness and resulting foam density using a thermal shock method [16], where composite specimens (100 mm × 100 mm) were placed in a Carbolite furnace at 450 °C for 5 min. The intumescent behaviour was determined using Eq. (1) as the expansion of the silicate layer in each composite as shown in Fig. 1(b) and (c) and defined as:

$$\text{Degree of Intumescence (DI)} = \frac{T_2 - T_1}{T_1} \quad (1)$$

where, T₁ and T₂ are the thicknesses in mm of the interlayer in the composite specimens before and after thermal shock as shown by digital images of the samples before and after the test (see Fig. 1(a) and (b) and (c) respectively).

The density of intumescent layer or foam density was calculated using Eq. (2) [16]:

$$\text{Foam Density (g/cm}^3\text{)} = \frac{\text{Mass of silicate (g)}}{\text{Volume of the foam (cm}^3\text{)}} \quad (2)$$

where, the mass of silicate = (total sample mass – mass of two laminated glass sheets) and the volume of foam = composite length × composite width × thickness of the interlayer (T₂).

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