Preparation and characterization of flame retardant cotton fabrics

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ABSTRACT
An inorganic flame retardant glass was prepared using the melt methods. The prepared glass was characterized using IR, X-ray. The x-ray patterns and IR charts show that the formation of glass without any crystals. Cotton fabric was finished using different percentage of glass 0.5, 1, 1.5 and 2 % (w/w) in the presence of citric acid as crosslinker and sodium hypophosphite as catalyst. The flame retardancy of finished cotton fabric was performed using Limited Oxygen Index (LOI) technique. The results of measurements show that the value of LOI of untreated cotton fabric equals 23.6, when cotton fabrics treated with different amounts of glass ranging from 0.5 to 2 %, the value of LOI increased to become 23.6 at 0.5% and 24.8 for the higher glass concentration. The effect of curing temperature and time on the properties and the LOI of cotton fabric was studied.

Keywords: Glass; Cotton fabric; Flame retardant; LOI; Thermal analysis; Mechanical properties.

1. INTRODUCTION
Cotton fibers have been used in different manufactures such as garments, curtains, towels, decorations and other applications owing to their outstanding properties [1]. Also, cotton fabrics have advantages of eco-friendly, biodegradability, softness, and breathability, there are some restrictions for using in special cases because it burns and produces some unacceptable gases such as CO and CO₂ beside some organic vapors [2]. Addition of flame retardant materials becomes necessary to modify the cotton fabrics to be used in the special applications such as worker's clothes, firefighter apparel, carpet, soldier clothes, transportation (airplanes, trains, cars) [3-9].

In general, the flame retardant materials may be additive or binding with the substrate materials. Also, it is classified into two broad categories, organic and inorganic materials. In most cases the organic materials are synthetic when burned give some toxic gases which are harmful to people and environment [10-12]. For several years, the halogen based flame retardants have been used to provide flame retardancy of the flammable material [13-19].

Inorganic flame retardant materials have many advantages, as it is mainly hydrated materials, it loses water through an endothermic process and forms a ceramic layer that isolates the flame from the burned materials. The most common inorganic flame retardants are the metal hydroxide such as magnesium, aluminium hydroxide, ammonium poly phosphates, phosphorus, silicates, borates and metal ores which containing the inorganic materials such as clays, zeolite [20-29].

The present work aimed to use glass material rich with magnesium and phosphorus for reducing the flammability of cotton fabric. The mechanical and physical properties of the cotton fabric loaded with different amounts of the prepared glass were tested.

2. MATERIALS AND METHODS
2.1. Materials.
Ammonium dihydrogen phosphate, sodium carbonate, magnesium oxide, citric acid and sodium hypophosphite (SHP) are of laboratory grade chemicals and were used without further purifications. Mill scoured and bleached plain weave 100 % cotton fabric weighting 240 g/m² supplied by Misr Spinning and Weaving Co., Mehalia el Kobra, Egypt. The fabric was further purified by treatment with a solution containing 2 g/l sodium carbonate and 5 g/l Egyptol (nonionic detergent) for 2 h. at boiling, after that, the fabric was washed with distilled water and left to dry in open air before finishing.

2.2. Preparation of the materials.
2.2.1. Preparation of glass.
The glass sample was prepared with chemical composition 65P₂O₅ – 15Na₂O – 20MgO. The weighed batch was inserted in an alumina crucible and melted in an SiC electric furnace (Vecstar, UK) at 1250 ± 20 °C for 60 minutes. The formed melt was gently rotated every 15 minutes to reach complete mixing and homogeneity. The melt was transferred to a warmed stainless steel mould and then immediately transferred into preheated annealing furnace regulated at 300 ± 20 °C. The annealing muffle was switched off with the glassy samples inside and left at room temperature at a rate of 24°C/hour, then the sample was grained through an agate mortar to obtain a fine homogeneous powder.

2.2.2. Preparation of glass finishing bath for cotton fabrics.
Five samples were prepared to contain 0.0, 0.5, 1, 1.5 and 2% of the prepared glass by suspending the glass weight in 100 ml distilled water and exposed to ultrasonic water bath for 20 minutes at room temperature. 1.8 gm of citric acid (CA) and 1.2 gm of sodium hypophosphite (SHP) were added to the previous mixture.
Because the prepared glass is phosphate, it is slightly soluble in water.

### 2.2.3. Finishing of cotton fabric.

Cotton fabrics were treated by pad-dry-cure method, the fabric samples were immersed in the finishing formulations and padded to a wet pick-up of calculated 100 % (w/w) then cured at different temperatures, 135°C or 160°C. The treated cotton samples were washed thoroughly with tap water and dried at room temperature overnight.

### 2.3. Characterization.

The amorphous nature of the prepared glasses was identified by X-ray diffraction using a Bruker AXS diffractometer (CD8 – ADVANCE) with Cu –Ka radiation operating at 40 Kv and 10 mA. The diffraction data were recorded as 20 values between 4° and 70° and the scanning rate was 10°/min. Thermal gravimetric analysis (TGA) was measured using thermogravimetric analyzer (SETRAM, Labsys TM TG-DSC16 instrument) under the heating rate of 10 °C min⁻¹, temperature range of 10 – 1000 °C and air flow rate of 20 ml min⁻¹ C using alumina as inert reference material. The attenuated total absorption Fourier transform infrared (ATR-FTIR) spectra of the prepared samples were measured at room temperature over the wavenumber 400-4000 cm⁻¹ range using by a Bruker FTIR spectrometer with a spectral resolution of ~5 cm⁻¹. The morphological textures of textile fiber together with textile samples loaded with glass were carried out by means of High resolution transmission electron microscopy (HRTEM) model Philips XL30 attached with EDX unit, using an accelerating voltage of 30 KV, magnification 10x up to 400,000x and resolution for wavelength (3.5 nm).

### Flame retarding test.

The flame retardant properties of the cotton fabrics were tested using limited oxygen index - LOI apparatus 4589-2 ASTM D 2863, Elevated- Temperature Oxygen Index ISO 4589-3, UK Naval Engineering standard NES 714 which measure the percentage of oxygen that has to be present the supported burning of the samples.

### Mechanical properties.

The Tensile strength of cotton fabrics was measured according to ASTM Test Method D5035. A Q-Test 1/5 tensile tester was used. Three specimens for each treated fabric were tested in the warp direction and the average value was recorded to represent the fabric breaking load.

### Whiteness (WI) and yellowness (YI).

WI and YI were measured using color-EyeR 3100 spectrophotometer supplied by SDL Inter England according to the standard test Method. ASTM E-313.

### 3. RESULTS

#### 3.1. X-ray diffraction (XRD).

X-ray diffraction measurement of the prepared glass, Figure 1 depicts the amorphous character of the prepared glass and gives no evidence of separation or precipitation any crystalline phase during the preparation of the conventional melting and annealing process with the proposed composition.

![Figure 1. XRD of the prepared glass.](image)

#### 3.2. Thermogravimetric analysis (TGA).

Figure 2 shows the TGA profiles of cotton fabrics coded as BL (untreated cotton fabric) and S1, S2, S3 and S4 containing different amount of glass (0.5, 1, 1.5, and 2 It is clear from TGA results that treating cotton fabric using the finishing formulation containing the prepared glass leads to a decreasing in the weight loss, the maximum weight loss of the untreated sample was about 83 % while the maximum weight loss in presence of glass the weight loss was about (65 -75 % ) depending on the concentration of glass in the finishing formulation. This finding confirms the effect of glass on the thermal behavior of the treated cotton fabrics. Also the maximum decomposition temperature was about 375°C for untreated cotton fabrics while it was about 350°C or the treated samples irrespective of the glass concentration.

![Figure 2. TGA of cotton fabric finished with different concentrations of glass.](image)

#### 3.3. Fourier Transform Infrared absorption spectra (ATR-FTIR).

The infrared absorption spectrum for pure prepared glass is shown in Figure 3. Figure 3 shows a sharp intensive peak centered at about 494 cm⁻¹ correspondings to O – P – O vibrations (PO₄³⁻) [29] and five characteristic absorption peaks in the range 709 to 1145 cm⁻¹. The absorption peak that is centered at about 709 nm may be corresponding to the symmetric stretching vibrations of P-
O-P bridging oxygens, also peaks observed at about 869 and 1041 can be attributed to both asymmetric stretching and the symmetric stretching vibrational modes of P-O-P linkages [29,30]. The including of MgO in phosphate glass causes the appearance of the orthophosphate groups with symmetric stretching modes of PO₄³⁻ centered at about 1090 cm⁻¹. These results indicate that the addition of MgO enhances the formation of unbridging oxygen (NBO), so that increasing the chemical stability of sodium phosphate glass network. On the other hand, The peak around 1145 cm⁻¹ can be assigned to the symmetric stretching vibrations of PO₄ [29-31], while the peak appeared near 1269 cm⁻¹ can be assigned to asymmetric stretching vibrations of the two non-bridging oxygen atoms bonded to phosphorus atoms, the O–P–O [31-34]. The intense peak that centered at about 1643 cm⁻¹ can be assigned to water-bending mode of P-OH bonds [35]. The peak at 2350 and 3334 cm⁻¹ is related to OH or molecular water vibrations. Figure 4 represents the infrared absorption spectra of cotton fabrics treated with different amounts of glass. The absorption peak centered at about 471 cm⁻¹ can be attributed to O–P – O vibrations (PO₄³⁻). The small peak around 547 cm⁻¹ is corresponding to P₂O₅[35]. On the other hand, a collection of characteristic bands of the cotton fabrics matched with that observed in references [36,37] and can be attributed in the bases of bands listed in Table 1.

### Table 1. ATR-FTIR absorption peaks of cotton fabric treated with glass.

| Reference | Assignment                                      | Peak position (cm⁻¹) |
|-----------|-------------------------------------------------|----------------------|
| 8&9       | C-OH bending vibrations                          | 668                  |
| 8&9       | COC,CCO and CCH stretching vibrations           | 898                  |
| 8&9       | C-C, C-OH, C-H stretching vibrations            | 1018                 |
| 8&9       | C-O – C asymmetrical stretching vibrations      | 1160                 |
| 8&9       | (H-C-C, H-C- O, and H-O-C bend)                 | 1324                 |
| 8&9       | H- C- H and O – C – H bending vibration         | 1434                 |
| 8&9       | OH bending of absorbed water                    | 1641                 |
| 8&9       | C–H symmetrical stretching vibrations           | 2899                 |
| 8&9       | OH stretching vibrations                        | 3293                 |

#### 3.4. SEM and EDAX.

Figure 5 shows the Scanning Electron Microscopic (SEM) images and EDAX. The surface texture of the untreated cotton fabrics shows straight fibers and arranged in parallel directions, no layers formed on the surface (Figure 5 a1). Loading glass on the cotton fabric, a characteristic layer was observed on the surface (Figure 5 b1) and the intensity of the formed layer increases with increasing the content of glass in the finishing bath. The observed layers which formed on the surface were analyzed by EDAX (Figures 5 a and b) technique to give an evidence of the attachment of glass on the textile surface.

![Figure 5](image-url)  
**Figure 5.** Surface morphology of pure cotton fabric and cotton fabric loaded with glass where (a) and (a1) are the SEM and EDX of pure cotton fabric; (b) and (b1) are the SEM and EDX of treated cotton fabric respectively.

#### 3.5. Flame retarding properties.

Table 2 shows the effect of curing temperature and concentration of glass on the flame retarding properties of the treated fabrics expressed as limited oxygen index.

The obtained data show that the value of L.O.I of the untreated cotton fabrics is 19.6, treating cotton fabrics with 0.5% and 1% glass and curing at 135°C is accompanied by increasing LOI to 23.6 and 24.8 respectively and any increase of the glass concentration over 1% in the range studied has no effect on LOI values. It is concluded that 1% is enough to be added to cotton fabric. Increasing curing temperature to 160°C leads to increasing the values of LOI to 23.8 at 0.5% glass concentration and about 25.5 at 1% glass concentration and also no need to use a higher concentration of glass higher than 1%. Increasing LOI at higher curing temperature can be explained by the ability of citric acid to crosslink cellulose chains and form a 3D network at high...
temperature that may capture the glass inside it leading to more effect on the LOI.

Table 2. LOI of the treated cotton fabric samples padded for a pick-up 100%, cured for 5 minute followed by washing and drying.

| Conc. (g/100ml) | Limiting oxygen Index (L.O.I) |
|-----------------|-------------------------------|
|                 | Curing at 135°C | Curing at 160°C |
| ---             | 19.6            | 19.6            |
| 0.0             | 21.8            | 21.8            |
| 0.5             | 23.6            | 23.8            |
| 1               | 24.8            | 25.5            |
| 1.5             | 24.8            | 25.7            |
| 2               | 24.8            | 25.7            |

3.6. Mechanical and physical properties of treated cotton fabrics.

Table 3 gives an idea about the effect of the treatment on the mechanical and physical properties of the finished cotton fabrics. It is obvious that increasing the concentration of glass leads to a marginal decrement in the whiteness index and tensile strength of the treated samples while increases the yellowness and elongation at break is almost unchanged. The effect of the treatment on the mechanical and physical properties may be attributed to the acidity of citric acid and high curing temperature which has an adverse effect on the mechanical and physical properties.

Table 3. Effect of finishing treatment on some physical and mechanical properties of cotton fabrics

| Conc. (g/100ml) | W1 | Y1 | Tensile strength (Kg.f) | Elongation at break (%) |
|-----------------|----|----|------------------------|------------------------|
|                 | 70.18 | 4.99 | 40 | 17 |
| 0.0             | 68.63 | 5.43 | 37 | 15 |
| 0.5             | 66.16 | 6.36 | 38 | 16 |
| 1               | 60.76 | 8.30 | 39 | 14 |
| 1.5             | 60.77 | 8.24 | 39 | 15 |
| 2               | 60.13 | 7.79 | 38 | 16 |

4. CONCLUSIONS

An inorganic flame retardant glass was prepared using the melt methods. The structure of prepared glass was confirmed using IR, X-ray. The x-ray patterns and IR charts show that the formation of glass without any crystals. Cotton fabric was finished using different percentage of glass 0.5, 1, 1.5 and 2 % (w/w) in the presence of citric acid as crosslinker and sodium hypophosphite as catalyst. From the results obtained it can be concluded that the flame retardancy of treated cotton fabrics increased by increasing the amount of glass used from 0.5 % to 1 % and any increase of glass has a marginal effect on flame retardancy. The LOI was found to be 19.6 and 23.6 when untreated cotton fabric and cotton fabric treated with 5 % glass and LOI reached about 24.6 and 25.7 when 1% of glass was used. Increasing curing temperature from 135°C to 160°C enhanced the LOI. Also, it can be said that the treatment has a marginal effect on the mechanical and physical properties of the treated cotton fabrics.

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