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Mass recovery of carbonated fabrics of glass fibres after isothermal heating

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Abstract Leaching of Na⁺ ions in sodium oxide (Na₂O) and silica (SiO₂) containing glass is well investigated mainly due to its weak weathering. The object of this study was naturally (at room conditions) leached, steady state product on surface of sodium oxide-silica-alumina (Al₂O₃) glass fibers (in fabric) in a form of shell of “glyed” trona crystals as a result of interaction of leached Na⁺ ions and H₂O and CO₂ from atmosphere. There are presented results of continued former investigation of mass loss by isothermal heating of fabric and mass recovery in different atmospheres during the first phase of adsorption (at least 0.25h) without changes of state of crystals obtained during preheating at different temperatures. There are observed two ways of decomposition of trona (Na₃H(CO₃)₂·2H₂O) with its beginning at about 55-57°C and 73-75°C. The regression analysis of mass restoring in different atmospheres indicates to simultaneous and exponential mass increase by physical adsorption of CO₂ and H₂O having the different parameters of exponents vs time. Decomposition of trona is discussed in terms of parameters of exponent vs preheating temperature.

1. Introduction

Glass durability studies are generally focussed on the reaction between water and glass [1]. It has been known that the reaction produces a leached surface layer in which the alkali ions in the glass are depleted to the surface and replaced by hydrogen or hydronium ions from the water to preserve electroneutrality [1-3], that is confirmed experimentally by ion-beam etching and other techniques which determine the concentration profiles of the interdiffusing species as a function of depth in a glass [1,4-7]. The object of this study was the shell of “glued” trona (Na₃H(CO₃)₂·2H₂O) crystals on surface of sodium oxide-silica-alumina (Al₂O₃) glass fibres (in fabric) as a result of interaction of leached Na⁺ ions and surrounding atmosphere during long time (years) storing at room conditions. Another motivation is associated with the small sizes (below a μm in two directions) of crystals that increases the attitude of surface to volume, velocity of reactions, and facilitate the investigation of surface phenomena itself.

The previous investigations [8,9] of mass recovery kinetics of fabric samples showed that during at least the first 0.25 h after heating the mass (or relative mass) recovery follows ($R^2 > 0.99$) to regression:

$$A(t) = A_0 - A_1 \exp(-t/t_1) - A_2 \exp(-t/t_2) = A_1 (1 - \exp(-t/t_1)) + A_2 (1 - \exp(-t/t_2)) \quad (1)$$



The coefficients A_1 and A_2 characterize the saturated adsorbed mass volumes, when $t \rightarrow \infty$. $A_1 + A_2 = A_0$. Time constants t_1 and t_2 characterize velocity of mass adsorption. The aim of present work was to elucidate the two mass adsorption components of equation (1) by changing the content of surrounding atmosphere. The second aim was a try to evaluate decomposition of trona at different preheating temperatures by changes of mass adsorption in terms of A_1 , t_1 and A_2 , t_2 of equation (1). Additionally, there was elucidated the “disparity” of indicated by XRD the one substance of shell (trona) and by differential thermal analysis (DTA) indicated two peaks. It allows evaluate the mass of shell and glass in fabric by TG (thermogravimetry).

2. Experimental

An industrially produced, carbonized and stabilized during years in room atmosphere a glass fabric with composition of (18-22) Na_2O , (3-5) Al_2O_3 , (73-79) SiO_2 were used in experiments. The threads contained 600 glass fibres with diameter of 6 μm . To determine composition of fabric, the samples (N15;16) were heated during 2 h at 185 $^\circ\text{C}$. To evaluate the content of adsorbed water at room conditions there is used box containing a water absorber. DTA and TG of fabric were fulfilled on Shimadzu DTA-60/60H.

For isothermal experiments there are used a group of samples b, stored in room during winter (less humidity) and samples d stored in room during summer (higher humidity, temperature and its oscillations). Samples b and d were heated from 39.1 to 154.1 or 185 $^\circ\text{C}$ respectively by step of 5 $^\circ\text{C}$. Preheating time was 1h. The mass recovery of samples b was determined in three atmospheres: room, box in room at elevated RH about 70%, box in room at elevated RH 70% and CO_2 concentration from 1500 to 1700 ppm and samples d: box in room at RH 70%.

The parameters A_1 , t_1 and A_2 , t_2 of equation (1) were determined using a regression technique to recovered mass values of samples for each preheating temperature in dependence on time till 0.25h. Later this time the equation (1) is not valid because there begin the other processes which change the state of crystals [9].

3. Results and discussion

The SEM picture of fibres with shell of trona crystals (XRD) are seen in Figure 1. During carbonation the fabric becomes thicker and less flexible, because crystals of shell are “glued” together and with surface of glass. We do not know the possible intermediate reactions on surface and how much time its take. The crystals of shell belong to trona (XRD) that have been converted to soda (Na_2CO_3 , XRD) by heating of fabric. The known equation of trona thermal conversion to soda is



The curve of DTA signal in Figure 2 during conversion trona to soda of “glued” fabric (in contrary to powder of trona [10]) contains two peaks and corresponding two steps in TG curve (not shown), caused by high thermal conductivity of “glued” system of trona crystals and glass. If “glued” system by pressing the fabric in spherical mortar is broken, the thermal conductivity of system decreases and crystals outward of thread reach the conversion temperature earlier, but inside a thread reach it later, that leads to widening of DTA peak to compare with its “glued” state, as it seen in Figure 2. Effect increases as pressing level increases. The decrease of mass at about 40 $^\circ\text{C}$ in Figure 2 could be caused partly by desorption of crystal water of trona (before decomposition of its bicarbonate content [11]).

It is assumed that mass content of fabric before decomposition of trona (P_{bd}) consists of mass of glass (p_{glass}), the steady state mass of adsorbed water at room conditions (p_{water}) and trona crystals:

$$P_{bd} = p_{\text{glass}} + p_{\text{water}} + p_{\text{trona}} \quad (3)$$

As it proceeds from reaction equation (2), the mass of trona before conversion is

$$p_{trona} = \frac{2M_{trona}}{M_{CO_2} + 5M_{H_2O}} \Delta \uparrow = 3.3714 \cdot \Delta \uparrow \text{ mg} \tag{4}$$

where M_{trona} , M_{CO_2} un M_{H_2O} are molar weights and $\Delta \uparrow$ is loss of mass by heating and evolving of CO_2 and H_2O . Then the composition of fabric is: humidity in range of 0.98-0.99%, trona about 43.8%, leached glass 55.2% from the equilibrium mass of fabric sample at about 22°C and 55% RH.

The relative mass loss (samples b) after/before heating P_1/P_0 of fabric after long and dry storing in room during winter (Figure 3, left curve) indicates the beginning of trona decomposition at about 57°C to compare with 73°C after more humid storing (samples d) in summer, that coincide with observations given for example in [12,13].

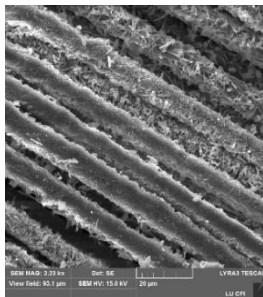


Figure 1. SEM image of shell of trona crystals on fibres of glass

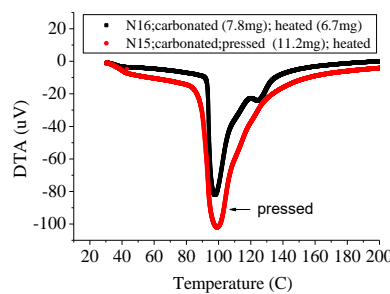


Figure 2. DTA signal of trona decomposition for normal and pressed glass fabrics

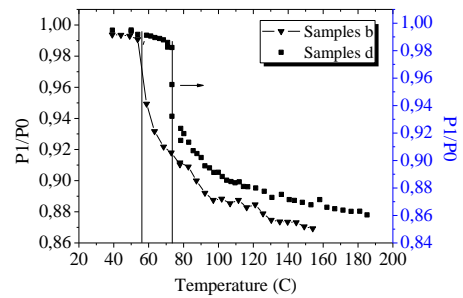


Figure 3. Mass loss during heating(1h) of fabrics stored at dry (left and more humid atmosphere

As it seen in Figure 4, 5 the relation A_1/A_0 increases with concentration of CO_2 and relation A_2/A_0 with humidity of air to compare with its values at normal room conditions. It indicates that the first part of equation (2), having time constant $t_1 \ll t_2$ (Figure 6) and $A_1/P_0 < A_2/P_0$ (Figures 4, 5), characterizes the mass adsorption of CO_2 and the second part- the water adsorption from atmosphere. The mass adsorption of H_2O is higher to compare with CO_2 (Figures 4, 5), but adsorption of CO_2 is faster (Figure 6).

The preheating temperature of the increases of CO_2 and H_2O adsorption in Figure 4, 5 coincides with beginning temperature of trona decomposition in Figure 3 at about 57°C, that could be caused with distortion of surface of crystals by evolving H_2O and CO_2 and increase its area and number of adsorption sites. The dissipation of points in Figure 4-6 is associated with dissipation of temperature and humidity during its measurements. Increase the both of A/P_0 with decrease of temperature (not shown) and increase of concentration of CO_2 and H_2O in atmosphere (Figure 4, 5) indicates to physical adsorption and its simple accumulation during at least 0.25h after those were lost during trona decomposition. The correlation between three mass desorption peaks during decomposition in Figure 3 (left curve) and three peaks of water adsorption (Figure 5) may indicate that adsorption sites are part of the same desorption sites during decomposition, because adsorption is unsaturated and determined by number of adsorption sites that explains also the high selectivity of adsorption.

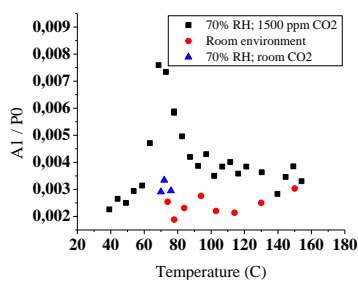


Figure 4. A_1/P_0 at the three atmospheres vs preheating temperature (samples b)

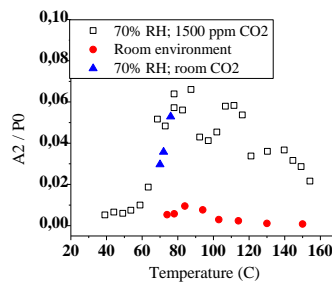


Figure 5. A_2/P_0 at the three atmospheres vs preheating temperature (samples b)

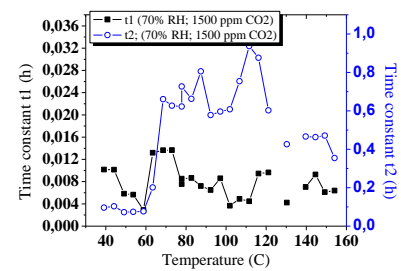


Figure 6. Time constants t_1 and t_2 vs preheating temperature (samples b)

Group of samples d before heating were stored at higher humidity, temperature and they fluctuations (summer) that increase the dissipation of points in Figures 7, 8 compared to that in a case of samples b. Mass after heating was restored at room conditions and increased RH to about 70%. The beginning of decomposition in this case is at 73°C in Figure 3 (right curve), about three times decreased water adsorption compared to samples b (Figure 5), and there is not seen the profiling of curve (Figure 3 right). Adsorption of water A_2/P_0 in Figure 8 also begins at the same temperature as decomposition. Adsorption peak of CO_2 looks similar to that in Figure 4, but wider. The height is the same in spite of lower concentration CO_2 compared to samples b (Figures 4 and 7). It indicates, that crystals of trona remember the earlier storing conditions in character of its decomposition and restoring that are quite different and may contain the new information.

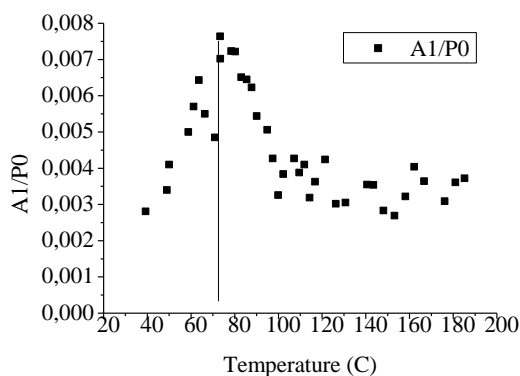


Figure 7. A_1/P_0 vs heating temperature (Samples d, RH 70%)

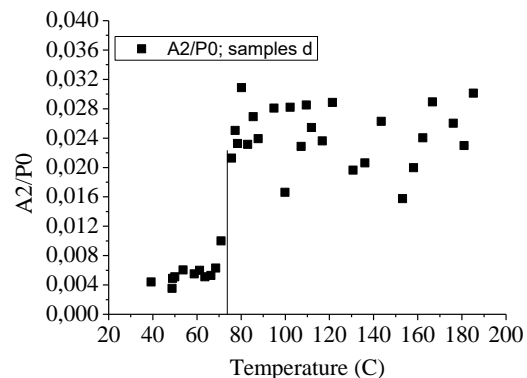


Figure 8. A_2/P_0 vs heating temperature (Samples d, RH 70%)

4. Conclusions

The first stage (at least up to 0.25 h) of mass restoring after thermal decomposition of trona crystals is associated with accumulation of mass by physical adsorption of CO_2 and H_2O from atmosphere and approximates with sum of two exponents with different values of its parameters. The first exponent with less time constant of equation (1) characterizes the physical adsorption of CO_2 and the second one - H_2O . Later this stage the mass adsorption moves from exponents.

The increase of fabric adsorption, heated over beginning of decomposition of trona is associated with destruction of surface of crystals, increased adsorption area and number of adsorption sites.

The non-isothermal decomposition of trona in glass fabric has the double and short DTA signal peak, caused by good thermal contact in system of “glued” trona crystals and glass fibre.

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