# DISOLUTION OF FIBERS USED IN INSULATING MATERIALS PRODUCTION


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## Abstract

Inhalation of asbestos has been associated with fibrosis, lung cancer and mesothelioma in humans and in laboratory animals. Questions have arisen regarding the safety of inhaling similarly sized particles of other types of materials, such as man-made vitreous fibers (MMVF).

This study presents the results of the dissolution rate of the glass fibers especially the effect of heat treatment on phase composition and solubility in the simulated lung fluid (SLF) using flow-through tests. Glass fibers VL1 (Superwool 607) of composition (in wt. %) $SiO_2$ 62.90, CaO 30.73, MgO 5.81, $Al_2O_3$ 0.25 and $Fe_2O_3$ 0.11 were analyzed. This type of fibers is used in the production of insulating materials.

The test was performed at 37 °C in flow-through configuration. Two solution flow rates (F) were used: 60, 120 $ml.day^{-1}$.

The NR [$g.m^{-2}.day^{-1}$] (normalized dissolution rate) for unheated and heated glass fibers was determined. This rate (NR) increased with increasing flow rate. Most probable rate controlling mechanism of glass matrix dissolution is the transport of surface reaction products through the solution boundary layer adjacent to the fiber surface. The flow-through tests determined that a dissolution rate of the unheated glass fibers is faster in SLF with pH 7.4 and for heated glass fibers is faster in SLF with pH 4.5.

The first results show that the dissolution rate of heated glass fibers is lower than that of unheated glass fibers. These results may indicate that the so called bio-soluble fibers could become less soluble and therefore potentially carcinogenic during the time of their use as a thermally insulating material.

**Keywords:** Glass fibers, dissolution rate, SLF

## 1 Introduction

In the recent years there has been increasing interest in the health effect of inhaled fibrous materials [1, 2]. Diseases from asbestos exposure take a long time to develop. Most cases of lung cancer or asbestosis in asbestos workers occur 15 or more years after initial

exposure to asbestos. The time between diagnosis of mesothelioma and the time of initial occupational exposure to asbestos commonly has been 30 years or more. Cases of mesotheliomas have been reported after household exposure of family members of asbestos workers and in individuals without occupational exposure who live close to asbestos mines.

In general, it is believed that fibers less than 0.2 µm in diameter and longer than 10 µm are most likely to induce tumors [3].

For these reasons, most of the studies have been conducted to measure the dissolution rates of various inorganic fibers using in vivo and in-vitro tests [4 to 9]. In vivo and in-vitro tests are usually executed for determination of dissolution rate of man-made vitreous fibers (MMVF) in physiological solutions. In-vivo tests are conducted on rats. There are suggested tree types of in-vivo tests: long-term inhalation, abdominal and tracheal injection. These tests are fairly expensive and time consuming. In-vitro static and flow-through tests are generally used for the testing of glass dissolution; the flow-through process (EURIMA test) was proposed and evaluated as the standard procedure for MMVF by Sebastian et al. [10].

This study presents the results of the dissolution rate of the glass fibers especially the effect of heat treatment on phase composition and solubility in the simulated lung fluid.

## 2 Experimental methods

Glass fibers VL1 of composition (in wt. %) $SiO_2$ 62.90, CaO 30.73, MgO 5.81, $Al_2O_3$ 0.25 and $Fe_2O_3$ 0.11 were analyzed (**Fig. 1**).These fibers were heated at 1100 °C for 24 hours (**Fig. 2**). X-ray diffraction detected a crystalline phase of the heated fibers. The main crystalline phase was wollastonite. The chemical composition of unexposed fibers was analyzed using X-rays fluorescence analysis (XRFA). The simulated lung fluid (SLF) with pH 4.5 (simulated intracellular lung fluid) and pH 7.4 (simulated extracellular lung fluid) was used as corrosion medium (**Tab. 1**). The tests were performed at 37 °C in flow-through configuration. These tests were carried out on the unheated glass fibers and on the heated glass fibers. Two solution flow rates (F) were used: 120 $ml.day^{-1}$ and 60 $ml.day^{-1}$. The higher one is with in the range recommended by EURIMA test guideline [10]. The concentrations of calcium, magnesium and silicon in the effluents were analyzed by AAS and spectrophotomertry. The surfaces of the fibers were observed before and after exposure by scanning electron microscope (SEM).

**Fig. 1** Glass fiber VL1

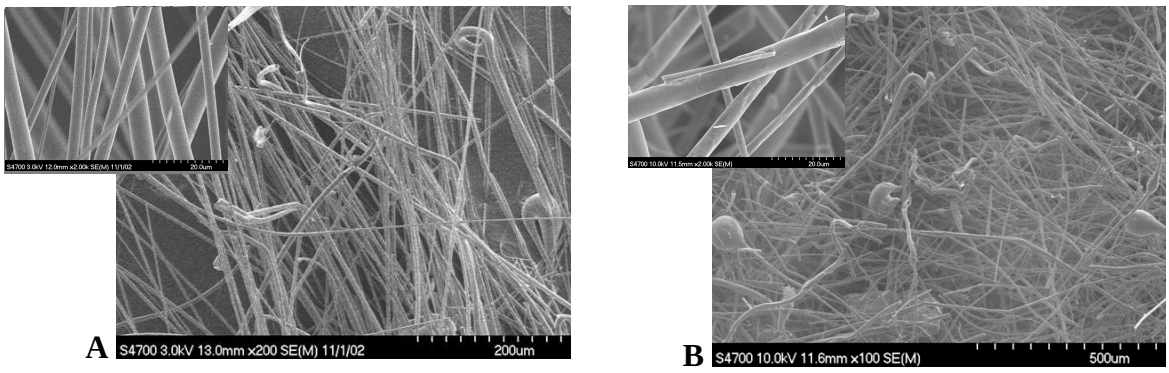


**Fig. 2** Scanning electron micrograph of **A** unheated – amorphous fibers **B** heated fibers–crystalline

**Tab. 1** Composition of simulated lung fluid (SLF) denotes SLF with pH=4.5 [a], SLF with pH=7.4 [b]

| Component | Conc. [mg/l] | Component | Conc. [mg/l] |
|---|---|---|---|
| NaCl | 7120 | $H_2NCH_2CO_2H$ (glycine) | 118 |
| $NaHCO_3$ | 1950 | $Na_3C_6H_5O_7 \cdot 2H_2O$ (citrate) | 152 |
| $CaCl_2$ | 22 | $Na_2C_4H_4O_6 \cdot 2H_2O$ (tartrate) | 180 |
| $Na_2HPO_4 \cdot 12H_2O$ | 373 | $NaC_3H_3O_3$ (pyruvate) | 172 |
| $Na_2SO_4$ | 79 | $C_3H_6O_3$ lactic acid (90%) | 156 |
| $MgCl_2$ | 99 | HCl (diluted 3:5) [ml] | 4.1[a]/0.04[b] |

## 3 Results and discussion

At the beginning of the interaction for and the unheated and the heated fibers, the selective leaching Ca and Mg was observed in SLF4.5, which was caused by $Me^{2+}$ - $H_3O^+$ interdiffusion in fiber surface. On the other hand for the unheated (amorphous) fibers, the lower $NL_{Me}$ values then the $NL_{Si}$ ones indicate the back precipitation of Ca and Mg at longer times of interaction in both solutions. The lower $NL_{Me}$ values then the $NL_{Si}$ were indicated for the heated (crystalline) fibers in SLF (4.5, 7.4) only for flow rate 60 [ml.day$^{-1}$] but for the higher one the opposite is true.

The normalized dissolution rate of the fiber $NR_{Si}$ [g.m$^{-2}$.day$^{-1}$] was determined as the slope of linear fit of experimental data. The lower rates of dissolution at pH=4.5 are in good agreement with minimum of glass dissolution rates observed in slightly acidic solutions (e.g. [11]). The higher dissolution rate of the heated fibers in the SLF which had a pH of 4.5 was determined. $NR_{Si}$ of the unheated fibers was higher than of the heated fibers in both of the simulated lung fluids. The normalized dissolution rate increased with the increasing flow rate (Fig. 3). The higher dissolution rates at higher solution flow rates can be explained using a simple mathematical model [12].

The normalized dissolution rate of glass can be calculated according to equation (1):

$$NR_{Si} = \frac{dm_{Si}}{x_{Si} S dt} = \frac{k^+ D/h}{k^+ + D/h}\left(c_s - c\right) \tag{1}$$

there $k^+$ is the surface reaction rate constant, D is the diffusion coefficient of surface reaction products through the solution boundary layer, h is the thickness of this layer and cs and c are saturated and actual concentration of Si in the solution. Constant $NR_{Si}$ values allow the assumption that the steady state with constant difference cs-c was achieved very

soon. The measured concentrations in the effluents were higher of the unheated fibers than of the heated fibers. The thickness of the diffusion layer was decreased with the increasing flow rate. This assumption is in agreement with the chemical engineering models considering the decrease of boundary layer thickness with increased solution flow rate.

The corrosive products and corroded layers on the surfaces of the exposed fibers were determined by SEM-EDS (**Fig. 4**).

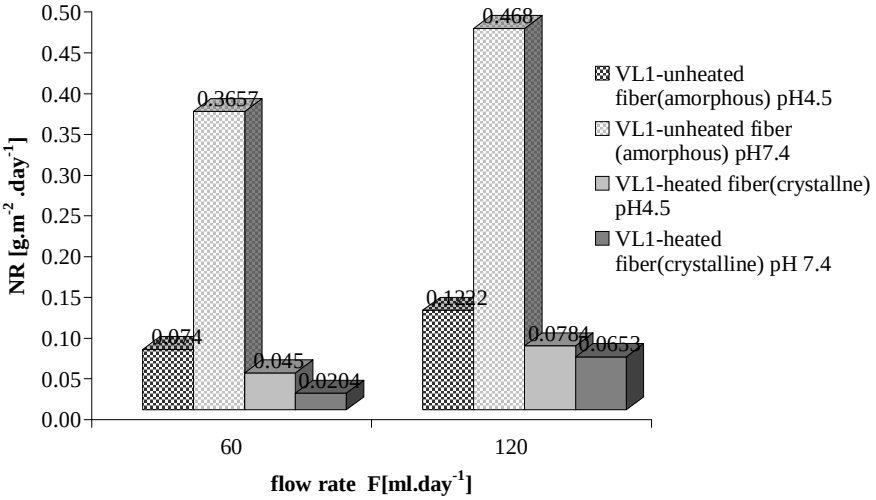


**Fig. 3** Comparison of normalized dissolution rates (NR) of the unheated (amorphous) and heated (crystalline) glass fibers

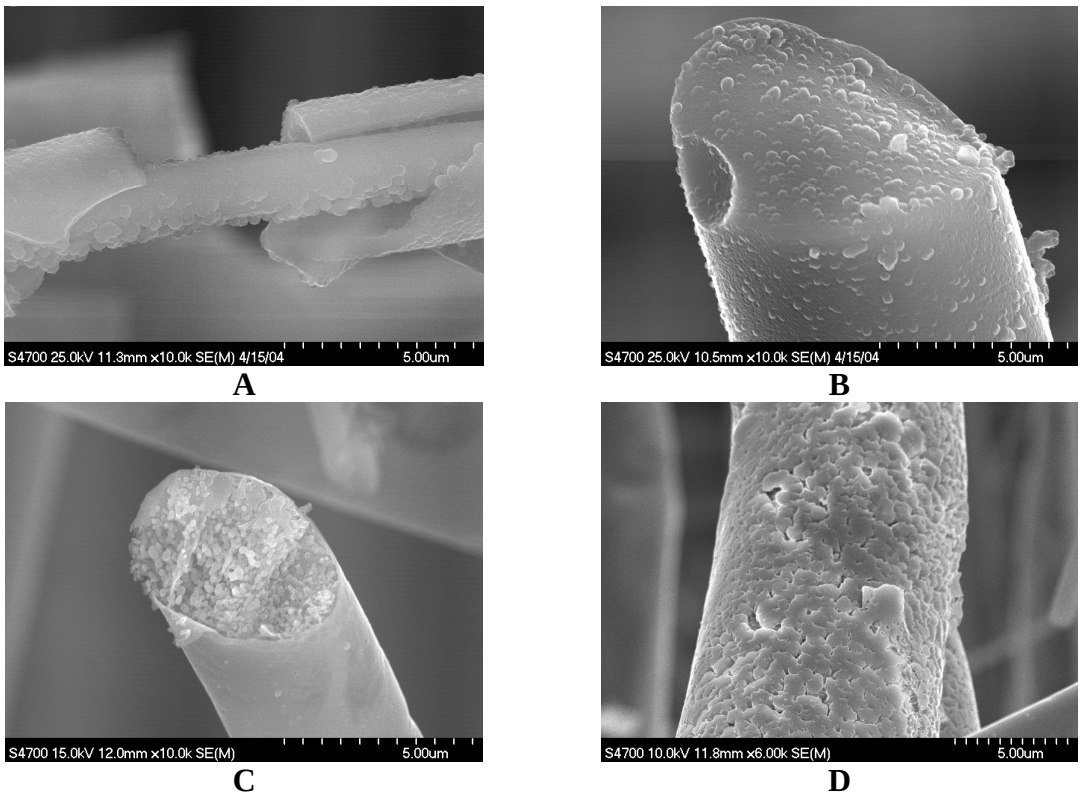


**Fig. 4** SEM micrograph of exposed fibers after 7 days in SLF **A**: amorphous fibers pH 4.5 **B**: amorphous fibers pH 7.4 **C**: crystalline fibers pH4.5 **D**: crystalline fibers pH7.4

## 4 Conclusions

The tested glass fibers dissolve incongruently. Most probable controlling mechanism of dissolution is the transition of surface reaction products through the stationary layer of the solution. The most important finding of this study is that there is some change in the solubility of the bio-soluble fibers cause by heat treatment. The phase composition of these fibers has high effect to the solubility in the SLF. These results may indicate that the so called bio-soluble fibers could become less soluble in a lung fluid and therefore potentially carcinogenic during the time of their use as a thermal insulating material.

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