

IN-VITRO TESTING OF SOLUBILITY OF THERMAL INSULATION FIBERS

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This recent work presents the results of dissolution of glass fibers in corrosion medium. The main objective of this research was to obtain the information about long-term dissolution of three types commercially produced glass fibers (E-fibers, fibers Promaglaf HTI 1200 and Promaglaf HTI 1250), which are commonly used as thermal insulation. The long-term dissolution of fibers was investigated at the temperature of 37 °C by dynamic flow-through tests in simulated lung fluid (SLF). The flow rate of SLF was 120 ml·day⁻¹. In solutions, the pH values and changes in weight loss of fibers were measured and changes in corrosion medium were evaluated by AAS. Also the values of the time dependence of the normalized leach amount of glass components (NLI) were computed and were compared with the data of NLI obtained from kinetic model of UCT Prague. The changes in appearance and composition of the fibers were evaluated with SEM-EDS techniques. This research confirmed that all fibers were dissolving in SLF, but each type differently. E-fibers dissolved nearly congruently, compared to fibers Promaglaf HTI 1200 and 1250 which were less soluble than E-fibers and dissolved incongruently. That was caused by production of precipitates – calcium phosphorous and silica layers.

INTRODUCTION

In the past asbestos was widely used as a material for thermal insulation applications for its excellent properties – resistance against corrosion and high temperatures. However, the use of asbestos has been prohibited by the World Health Organization because inhalation of its highly chemically resistant, insoluble needle-shaped fibers may cause diseases of the respiration system, from inflammations, to lung edema and pneumoconioses to lung tumors [1].

Currently, new types of glass fibers have been continually developed to replace asbestos. The producers seek to make sure that the fibers have low biopersistence, i.e. ability of the fibers aspired in the lungs to survive in the human organism, despite physiological degradation. In case of potential aspiration such fibers should dissolve in the lungs and should not be harmful to health. At the same time, the fibers should preserve properties necessary for thermal insulation applications.

Equally as glass itself, also glass fibers are subject to corrosion processes in interaction with various water solutions [2]. In the course of such processes irreversible changes occur on the glass surface and also composition of the solution changes as it is enriched with components originally contained in the glass. As mentioned before, corrosion of glass fibers in lung liquids is desirable as they degrade and they are gradually removed from the respiratory system.

In general, corrosion of glass can be described with three simultaneous processes:

- leaching of movable ions from the glass surface (interdiffusion $Me^+ - H_3O^+$),
- dissolution of SiO_2 glass network,
- back precipitation of components from glass to the solution.

Bio-solubility of fibers and their health risks are tested in practice with in-vivo and in-vitro tests [3]. *In-vivo* tests are performed on laboratory animals, especially on mice, rats and sewer rats, using either abdominal, intratracheal or inhalation pathways [4]. With those tests it is possible to directly determine potential pathogenicity of the fibers and their toxic or cancerogenic effects. However, such tests are financially demanding and therefore there have been efforts to replace them with model in-vitro tests without the use of laboratory animals. If the model conditions simulating the lung environment are selected well such tests may provide sufficient and important information about dissolution of the fibers in lungs. One of the new methods for testing of fibers is an in-vitro cellular test performed on live cultures [5] allowing direct determination of pathogenicity or genotoxicity of the fibers.

The objective of this work was to use model *in-vitro* tests in simulated extracellular lung fluid (SLF – simulated lung fluid) for dissolution of 3 types of commercially produced glass fibers designed for thermal insulation applications.

EXPERIMENTAL

The employed samples were 3 types of commercially produced fibers: E-fibers, fibers Promaglaf HTI 1200 and Promaglaf HTI 1250 declared by their producers as safe for human health and with low biopersistence.

E-fibers are white glass fibers based on silicon dioxide, calcium oxide and sodium oxide. They are used primarily for insulation and overcladding of houses and buildings. They are made by glass fiberization using a special formaldehyde-free technology called ECOSE that uses a natural binder [6]. Fibers Promaglaf HTI 1200 are white glass fibers based on silicon dioxide, calcium oxide and sodium oxide. The classification temperature, i.e. the temperature at which the value of lengthwise changes by firing is not greater than $\pm 1.5\%$, is $1200\text{ }^{\circ}\text{C}$. They are primarily used for insulation of flue ways passing through combustible building materials or insulation of fireplaces where it can be also used as a sealing material between the fireplace door and fire chamber. Fibers Promaglaf HTI 1250 are white glass fibers based on oxides of alkaline earths. The classification temperature is $1250\text{ }^{\circ}\text{C}$. They are used in high temperature aggregates or for home applications (boilers, fireplaces, furnaces, etc.) [7]. Fibers Promaglaf are made of material melted by electric arc at ca $1900\text{ }^{\circ}\text{C}$. After that the melted material is blown into fibers with compressed air or fiberized with rotating discs. The blowing method means that the melt is scattered into tiny drops that are subsequently elongated to form fibers and further processed into mats. This production method produces not only fibers but also the so-called shots (non-fiber fraction) [8].

Chemical composition of the fibers has been analyzed with X-ray fluorescence and it is provided in Table 1.

Dissolution of the fibers was tested with dynamic flow tests that appropriately simulate human lung environment. The lung liquid was simulated with modified Gamble's solution with pH 7.4 often referred to in literature [3, 9, 10] as it simulates extracellular lung fluid which is the first liquid the fibers come into contact with. Dissolution of the fibers occurred at $37\text{ }^{\circ}\text{C}$ and the flow rate of SLF was $120\text{ ml}\cdot\text{day}^{-1}$. E-fibers were tested continually for 4.5 months (135 days) and fibers

Promaglaf were tested for 60 days. The diagram of the dynamic test is shown in Figure 1. The leachates from the fibers were measured for changes of pH and concentrations of the main components (Si, Ca and Mg) using AAS methods. We also measured weight loss of the fibers and we calculated normalized quantities of the individual components that moved from the glass into the solution (NL_i). Change of the glass appearance and composition of the fibers was examined with SEM-EDS.

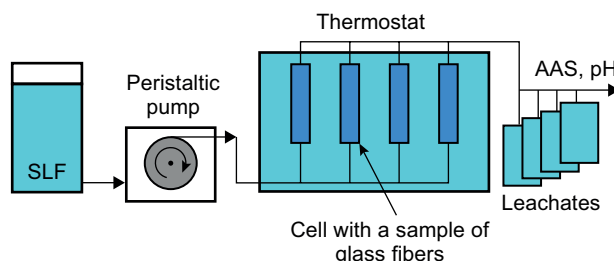


Figure 1. Schematic diagram of the dynamic test.

RESULTS AND DISCUSSION

Weight losses and kinetics of fiber dissolution

In the course of the experiment we monitored pH changes in the leachates. pH values increased with the leaching time from the initial value of 7.4 to nearly 9 after 60 days of leaching for all types of the tested fibers. The pH increase may be caused by leaching of the monitored components Ca and Mg. Weight losses of the tested fibers differed. The biggest losses were found for E-fibers which after 60 days of leaching reached over 90 % of the initial weight of the fibers. After 135 days this type of fibers was nearly dissolved (the weight loss was 99 %). On the other hand, Promaglaf HTI 1200 and 1250 fibers dissolved less. After 60 days the weight loss was 54 % of the initial weight for fibers Promaglaf HTI 1200 and 32 % for fibers Promaglaf HTI 1250.

Table 1. Chemical composition of the fibers determined by X-ray fluorescent analysis in powder samples.

Component [wt. %]	Sample type		
	E-fibers	Promaglaf HTI 1200	Promaglaf HTI 1250
SiO ₂	67.0	62.2	79.6
Na ₂ O	17.0	–	–
CaO	10.1	31.6	0.2
MgO	3.7	5.4	17.6
Al ₂ O ₃	1.3	0.6	2.3
Fe ₂ O ₃	0.4	0.1	0.1
K ₂ O	0.4	–	–

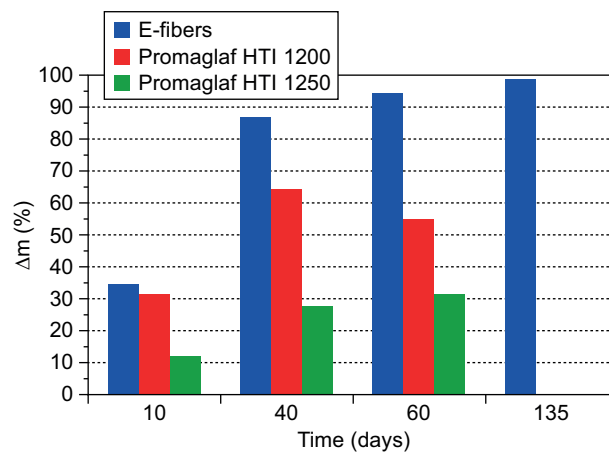


Diagram 1. Comparison of weight losses of the tested fibers.

Concentrations of the individual components Si, Ca, Mg gradually decreased with the leaching time, which is in agreement with the changing surface area of the fibers as the fibers dissolve during the tests and the surface area exposed to leaching decreases.

Concentrations of Si, Ca, Mg were used to calculate normalized amounts of the components that moved from the glass to the solution (NL_i in $[g \cdot m^{-2}]$) using the following formula:

$$(NL_i)^t = \frac{Q_i^t}{x_i \cdot S} = \frac{c_i \cdot \Delta t \cdot F + Q_i^{t-\Delta t}}{x_i \cdot S}, \quad (1)$$

where S is the surface area of the sample exposed to the corrosive solution SLF in $[m^2]$, x_i is weight mass fraction of the element i in the original material, Q_i^t in $[g]$ is the total quantity of the component i that passed into the solution SLF at the time t , c_i is the concentration of element i in the SLF leachate in $[g \cdot cm^{-3}]$, Δt is a time interval between collection of samples in $[hours]$, F is the flow rate in $[cm^3 \cdot h^{-1}]$ and $Q_i^{t-\Delta t}$ is the quantity of the element i in $[g]$ that moved into the SLF solution at the time $t - \Delta t$. In this work the calculation is related to the original surface area S_0 .

Provided the glass dissolution process is congruent the normalized amounts of NL_i should be the same and, on the contrary, provided there is reverse precipitation of some of the components, such as Si or in case of selective leaching, the quantities should be different [11, 12].

A glass corrosion model developed by the Department of Glass and Ceramics [12, 2] has been used for evaluation of kinetics of E-fibers dissolution. Even though the original model used the assumption that transfer of glass components into the solution slows down due to gradual saturation with SiO_2 it can be still used as an empirical model. The model has been adapted into the Equation 2, where the rate of transfer of the individual components into the solution can be determined by smoothing the experimental data $NL_i = f(t)$ with the following function:

$$NL_i = \frac{B}{K} [1 - \exp(-Kt)] + Wt. \quad (2)$$

In the original model, which anticipated saturation, the constants B, K and W depended both on parameters that characterize partial processes of glass corrosion and on experimental conditions of the interaction (S , V , F). In the concerned case it is necessary to take into account also a decrease of the transfer rate of the components into the solution as a result of reduced surface area of the fibers in the course of their dissolution.

The Diagrams 2, 3 and 4 show results of experimental normalized quantities of the components (NL_i) that passed from the fibers into the solution and values NL_i obtained from kinetic models developed by the Department of Glass and Ceramics of the University of Chemistry and Technology in Prague (DGC UCT in Prague) (see the Equation 2). The diagrams shown below

imply that experimental NL_i values correspond to values obtained from the kinetic model developed by DGC UCT in Prague.

The Diagram 2 indicates that E-fibers initially dissolve congruently – the individual components Si, Ca, Mg pass from E-fibers into the leachate approximately in the same ratio. Later (after 22 days), the individual quantities of the components NL start to vary which might be caused by different rates of their transfer from the fibers to the solution or by back precipitation of the leached components. However, the NL_i values subsequently stabilized and for all the components the increases were minimal, probably because most of the fibers had dissolved and therefore we could conclude that, in general, the fibers dissolved congruently.

The Diagram 3 shows NL_i values for Promaglaf HTI 1200 fibers. It is obvious that, unlike E-fibers, Promaglaf HTI 1200 fibers dissolved incongruently. Ca and Mg pass to the SLF solution in approximately equal quantities but NL values for Si are somewhat higher.

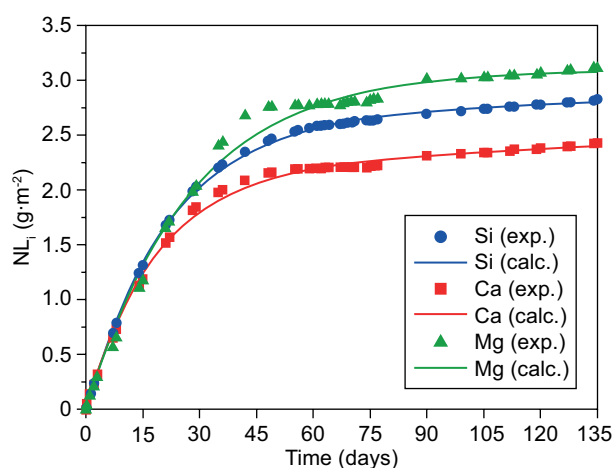


Diagram 2. Comparison of experimental and calculated NL_i values (E-fibers); exp. = experimental; calc. = calculated.

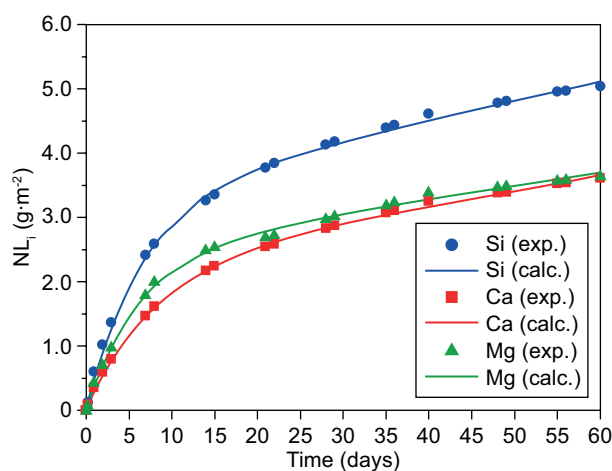


Diagram 3. Comparison of experimental and calculated NL_i values (Promaglaf HTI 1200); exp. = experimental; calc. = calculated.

The Diagram 4 with NL_i values for Promaglaf HTI 1250 fibers indicates incongruent dissolution as each component is leached in a different quantity. Ca and Mg are leached quickly. On the contrary, Si values are significantly lower, which may be caused by fast leaching of Ca and Mg from the fibers, while a poorly soluble silica layer develops on the surface of the fibers and therefore the weight losses are not significant.

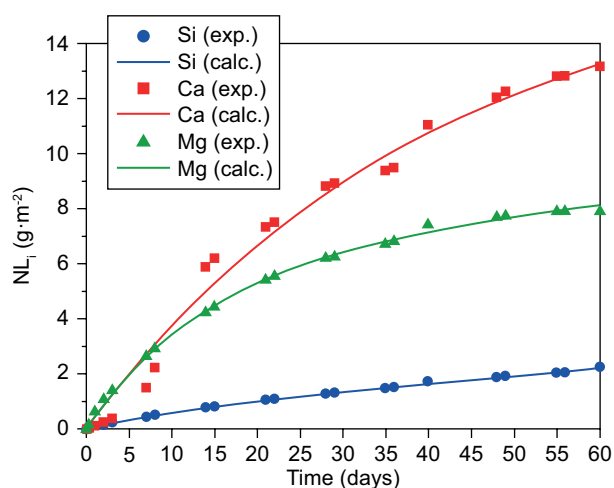


Diagram 4. Comparison experimental and calculated NL_i values (Promaglaf HTI 1250) exp. = experimental; calc. = calculated.

Evaluation of appearance and composition of fibers with SEM-EDS

Appearance and composition of all the tested fibers before and after the leaching were investigated with SEM-EDS analysis. The figures below (Figures 2-9) show changes on the fibers.

E-fibers

Figure 2 shows that the original E-fibers are smooth and intact. With advancing leaching time the fibers gradually become thinner, break down and degrade. The fibers dissolve and the overall surface area decreases. The EDS analysis (Table 2) shows that contents of Ca, Mg and alkalis gradually decrease. As mentioned above, after 135 days of leaching nearly 99 % of the fibers were dissolved; in a tiny residue of the fibers the content of Al_2O_3 increased from the original 2.4 wt. % to 16.8 wt. %, while the majority component continued to be SiO_2 . Further, the EDS analysis has shown that the composition $SiO_2-Al_2O_3-Na_2O-CaO$ in the residue is the same at different locations on the fibers surface, as demonstrated by other figures and analyses (Figure 3). One can expect potential formation of aluminum-silica complexes with lower solubility in SLF and therefore it would be useful to further investigate whether after

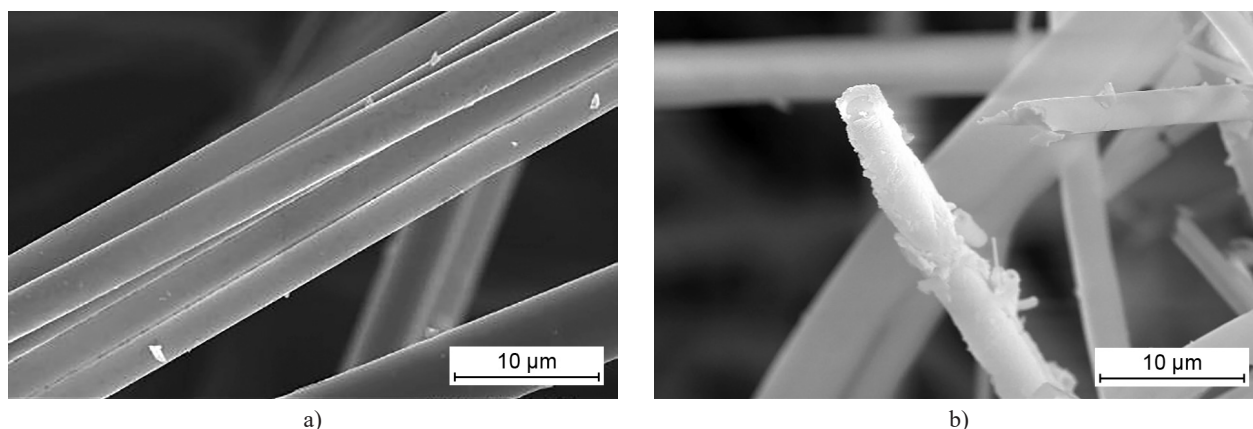


Figure 2. Original non-leached E-fibers (a), E-fibers after 135 days of leaching (b).

Table 2. Composition of E-fibers surface before and after leaching in SLF – dynamic flow test, 120 ml·day⁻¹, 37 °C, electron microscope – EDS analysis.

Component [wt. %]	Non leached surface	Leached surface			
		10 days	40 days	60 days	135 days
SiO ₂	69.9	72.5	73.1	69.0	73.2
Na ₂ O	13.7	11.6	9.7	13.0	8.5
CaO	9.7	10.1	6.2	3.1	0.6
MgO	4.1	3.4	2.1	0.6	0.5
Al ₂ O ₃	2.4	2.1	8.9	11.9	16.8
K ₂ O	0.2	0.3	0.1	–	–
Cl	–	–	–	2.5	–

a long period of leaching the fibers dissolve completely or whether the residues remain in the SLF solution undissolved. Practical *in-vivo* tests of biopersistence on laboratory animals usually do not monitor complete dissolution in lungs of the animals but mostly weight loss of the fibers 40 days after the fibers are introduced into the lungs. Provided the weight loss is higher than 50 % the fibers are declared safe. It is assumed that particles < 15 μm are subsequently removed from the lungs by macrophages. However, the presented results indicate that it should be useful to verify whether the fibers in the lungs are completely degradable or whether workers, who are exposed to fibers in the long term in the course of production or application of insulation systems, suffer from accumulation of less soluble residues of fibers in their lungs.

Fibers Promaglaf HTI 1200

Figure 4 shows that Promaglaf HTI 1200 fibers contained, in addition to the fibers, also shots generated during the production process of the fibers. A layer of precipitates was visible both on the fibers and on the shots (Figure 5). Although the compositions of the original non-leached fibers and shots were nearly the same (Table 3), after the leaching compositions of the layers were different. The EDS analysis has shown that after 40 days of leaching the layer developed on the fibers contained also phosphorus precipitated from SLF (the original composition of the fibers did not contain any P); on the contrary, the layer developed on the shots contained mainly SiO₂, an increased content of calcium and no phosphorus (Table 2). This means that a calcium-phosphorus layer develops on the surface of the fibers

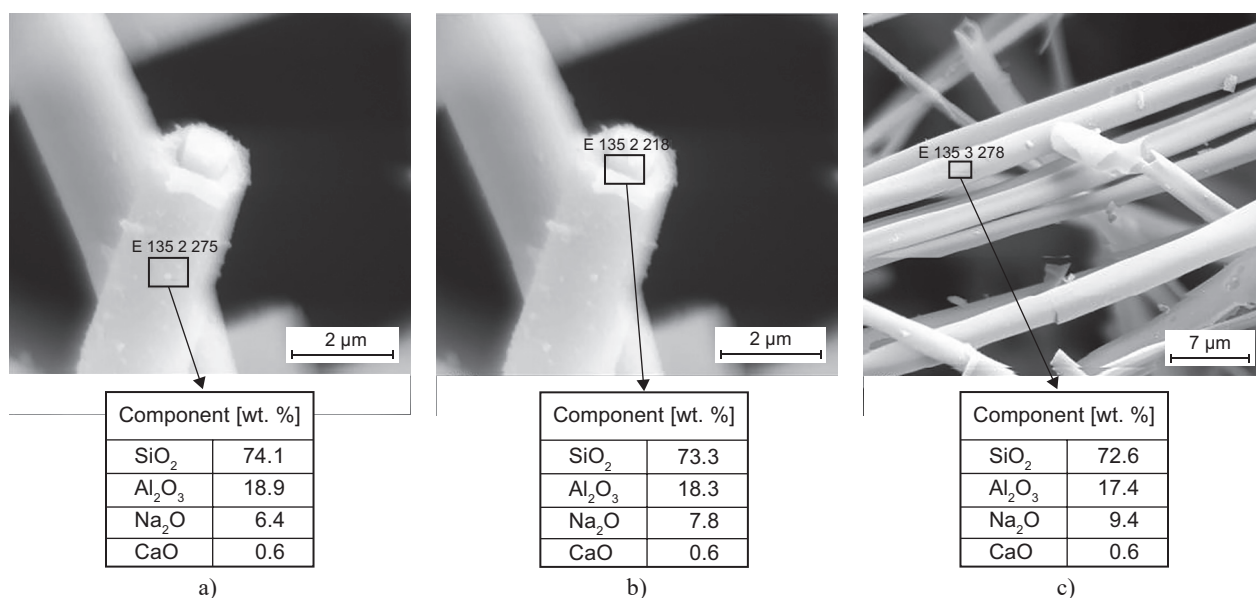


Figure 3. Comparison of analysis of residual E-fibers after 135 days of leaching in SLF – dynamic flow test, 120 ml·day⁻¹, 37 °C, EDS analysis.

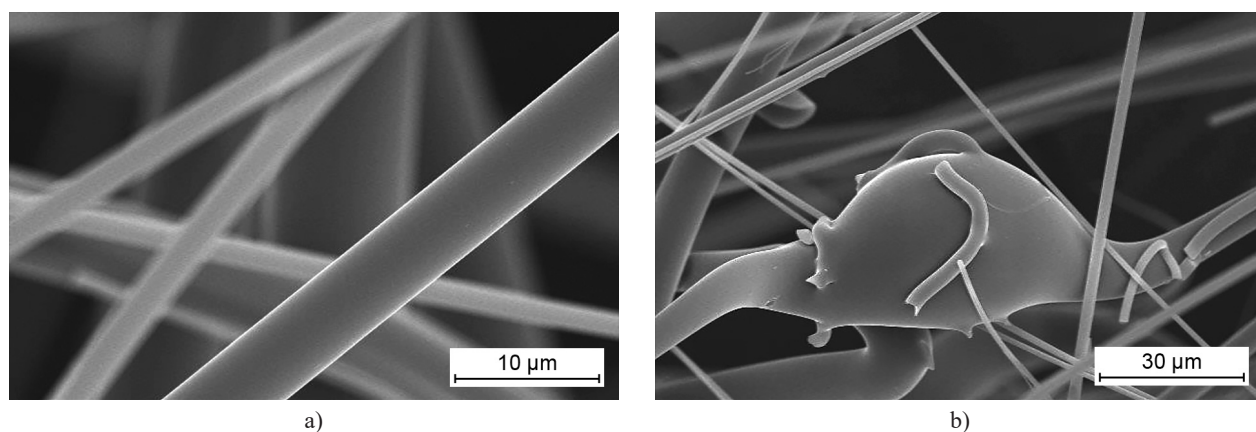


Figure 4. Original non-leached fibers (a) and shot of Promaglaf HTI 1200 (b).

but its composition in different locations demonstrates different ratios of the individual main components (SiO_2 , CaO and P_2O_5), as shown in Figure 6. The composition of the layer on the shots also varies at different locations (Figure 7) but not so distinctly as in the case of the calcium-phosphorous layer on the fibers (Figure 6).

Table 3. Composition of surface of Promaglaf HTI 1200 fibers before and after leaching in SLF – dynamic flow test, $120 \text{ ml}\cdot\text{day}^{-1}$, 37°C , electron microscope – EDS analysis.

Component [wt. %]	Non-leached fibers		Leached fibers (40 days)	
	fibers	shots	fibers	shots
SiO_2	58.9	57.5	32.1	85.7
CaO	39.6	40.9	48.6	10
MgO	1.5	1.6	1.9	1.3
P_2O_5	–	–	17.4	–
Al_2O_3	–	–	–	2.9

Variable composition of layers on the fibers and on the shots may be caused by the fact that fibers in a cell are in a so-called cluster and SLF solution flows through the fibers. The fibers on the outer surface of the cluster are more exposed to SLF than fibers inside the cluster. The EDS analysis (Figure 6) indicates that in the fibers the content of SiO_2 decreases and the content of CaO and P_2O_5 increases; on the contrary, in the shots the content of SiO_2 increases and the content of CaO decreases. The potential cause may be e.g. that surfaces of the fibers and the shots are different because of the production process and that is why the fibers and the shots dissolve by different mechanisms.

Fibers Promaglaf HTI 1250

Figure 8 shows fibers and shots made Promaglaf HTI 1250. Unlike Promaglaf HTI 1200, the original non-leached fibers and shots contain on their surface

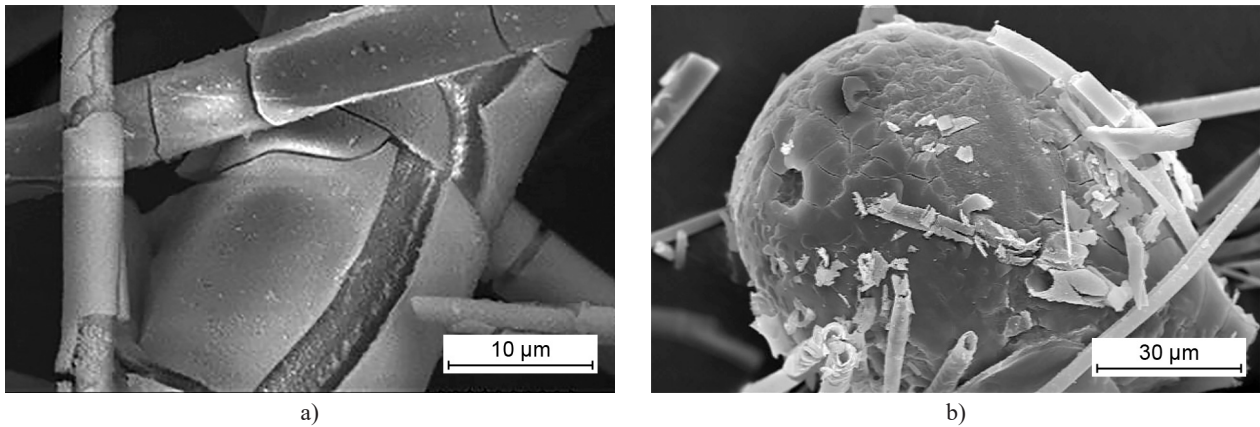


Figure 5. Fibers (a) and shot of Promaglaf HTI 1200 after 60 days of leaching (b).

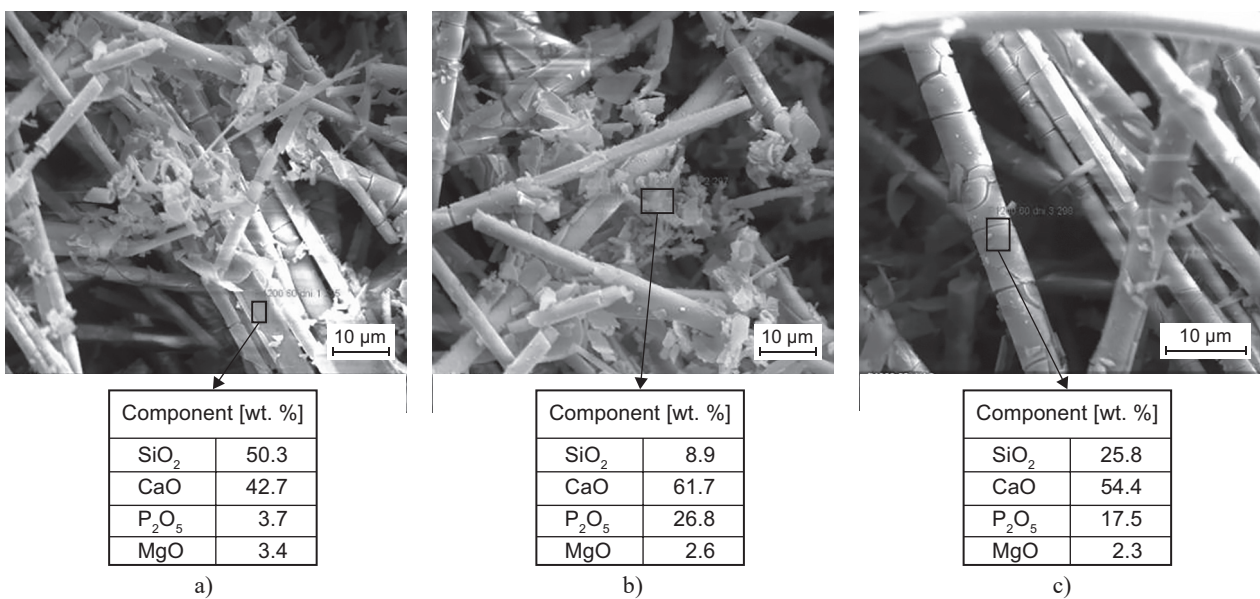


Figure 6. Comparison of analyses at different locations on the surface of fibers after 60 days of leaching in SLF (Promaglaf HTI 1200), dynamic flow test, $120 \text{ ml}\cdot\text{day}^{-1}$, 37°C , EDS analysis.

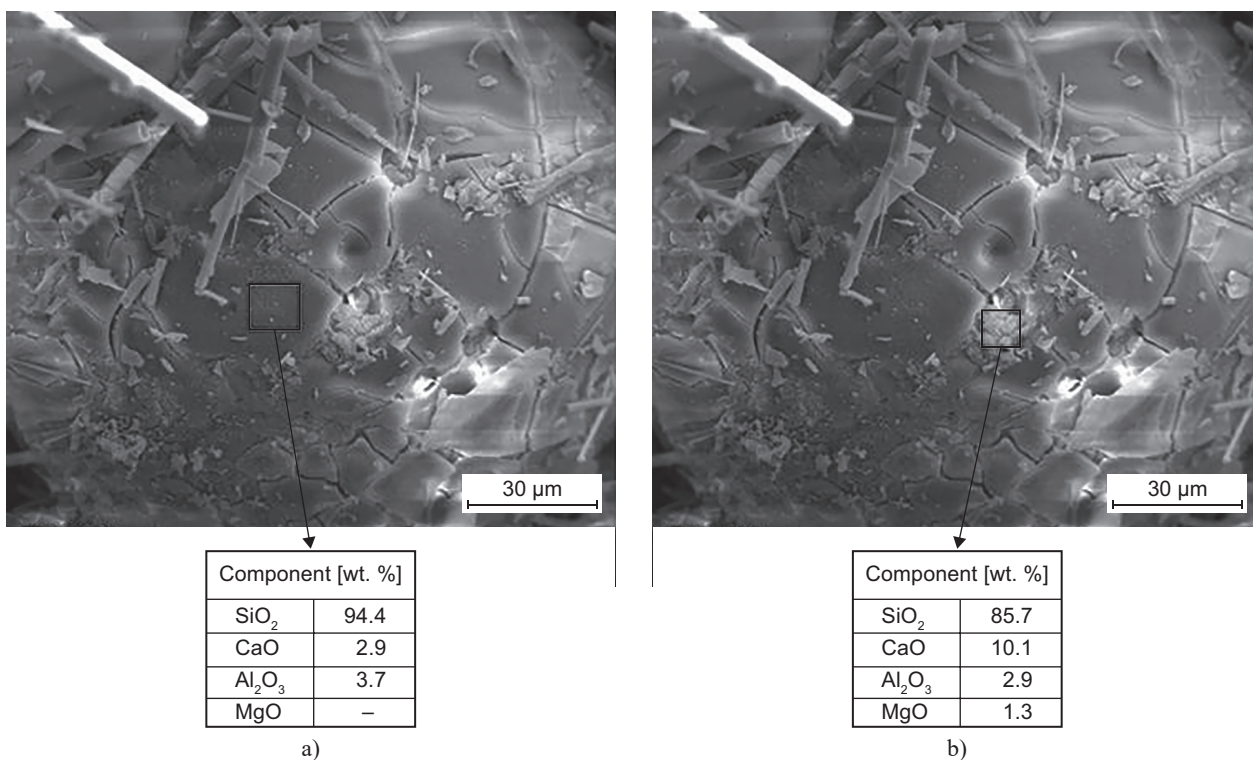


Figure 7. Comparison of chemical composition of layers at different locations on the shots after 40 days of leaching in SLF (Promaglaf HTI 1200), dynamic flow test, 120 ml·day⁻¹, 37 °C, EDS analysis.

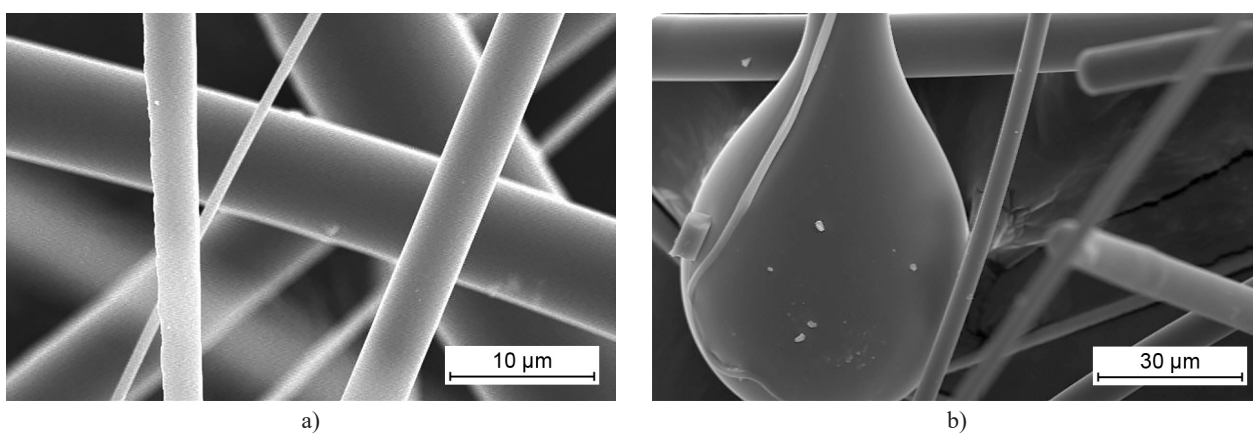


Figure 8. Original non-leached fibers (a) and shot of Promaglaf HTI 1250 (b).

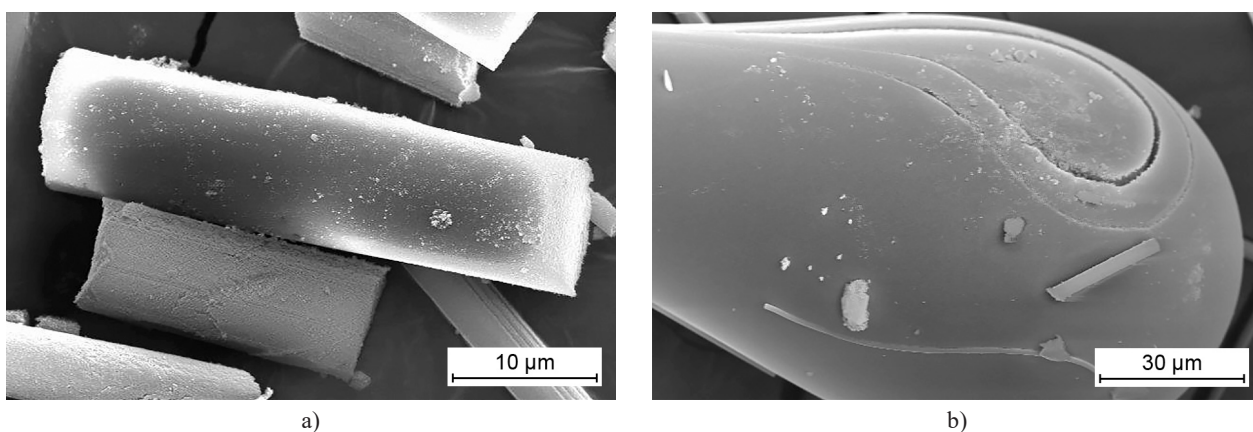


Figure 9. Fibers (a) and shot of Promaglaf HTI 1250 after 60 days of leaching (b).

high quantity of SiO₂ (over 90 wt. %) and the rest is only MgO (ca 8 wt. %) and CaO (ca 2 wt. %) (Table 4). In model tests of fiber dissolution Mg and Ca components are dissolved in SLF solution and a layer with high silica content (ca 98 wt. % of SiO₂, Table 4) develops on the surface of the fibers and of the shots which may slow down further dissolution. An analysis of the surface of the fibers after 40 days of leaching has also shown presence of Al which was not detected in the original non-leached surface but it was found in the volume of the fibers in a powder sample (Table 1). This fact suggests that even the surface of the fibers and the shots with high content of silica gradually dissolves and exposes sub-surface structures of the glass. Unlike Promaglaf HTI 1200, the composition of the surface of the fibers and the shots after 60 days of leaching is nearly the same which might mean that surfaces of the fibers and the shots have

the same character and structure and therefore one can expect that the fibers and the shots dissolve by the same mechanism.

Effects of surface changes on the rate of dissolution

Development of a model with *S* as a function of time

A model based on the Equation 2 was used for basic description of kinetics of transfer of glass components, originally created with the assumption that the dissolution rate of the glass network slows down as a result of saturation of the solution. However, it is obvious that slower dissolution may be also affected by gradual reduction of the contact surface between glass and solution. A simple mathematical model has been derived to estimate effects of the diminishing surface of the glass.

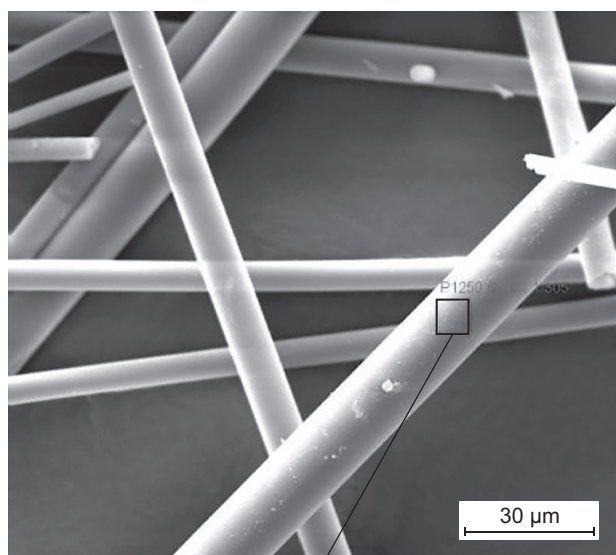
The model is based on the weight balance with the assumption that the concentration in the leachate $c \rightarrow 0$, i.e. no precipitation occurs

$$\frac{d\Delta m_{\text{glass}}}{S dt} = k \cdot c_s \quad (3)$$

The model also anticipates that the fibers have a cylindrical shape, they dissolve only in the radial direction (i.e. their length does not change) and their

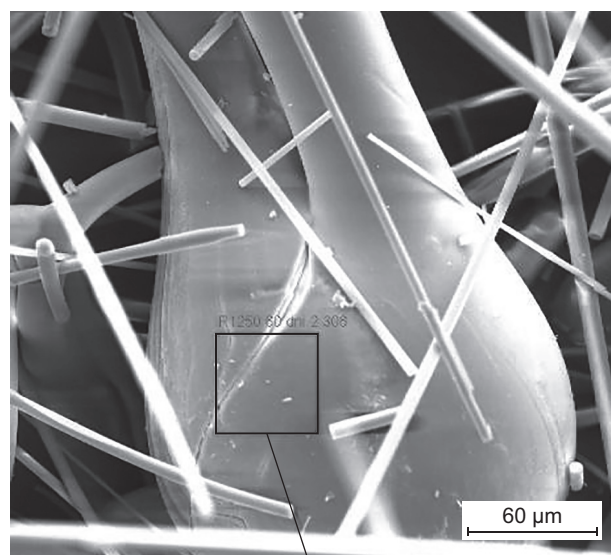
Table 4. Composition of surface of Promaglaf HTI 1250 fibers before and after leaching in SLF – dynamic flow test, 120 ml·day⁻¹, 37 °C, EDS analysis.

Component [wt. %]	Non-leached fibers		Leached fibers			
	fibers	shots	40 days		60 days	
			fibers	shots	fibers	shots
SiO ₂	90.2	91.0	97.4	99.1	98.5	98.3
CaO	1.7	1.2	–	–	–	–
MgO	8.2	7.8	0.7	0.9	0.5	0.8
Al ₂ O ₃	–	–	1.6	–	1.0	0.9



Component [wt. %]	
SiO ₂	98.4
Al ₂ O ₃	0.9
MgO	1.7

a)



Component [wt. %]	
SiO ₂	98.4
Al ₂ O ₃	0.9
MgO	1.7

b)

Figure 10. Comparison of chemical composition of surface of the fibers and the shots after 60 days of leaching in SLF (Promaglaf HTI 1250), dynamic flow test, 120 ml·day⁻¹, 37 °C, EDS analysis.

bases have negligible surface in comparison with the lateral surface. The processes of back precipitation and saturation of the solution are disregarded and congruent dissolution is assumed. This means that the relation between the surface and weight of the fibers is:

$$\frac{m}{m_0} = \left(\frac{S}{S_0}\right)^2 \quad (4)$$

$$S = S_0 \sqrt{1 - \frac{\Delta m}{m_0}} \quad (5)$$

where Δm is the weight of the dissolved glass, S_0 and S are glass surfaces at the time $t = 0$ and t , m is the weight of undissolved glass, m_0 is the initial weight of the glass, k is the reaction rate constant and c_s is the saturated concentration.

After substituting the Equation 3 to the Equation 5 we will get:

$$\frac{d\Delta m}{S_0 \sqrt{1 - (\Delta m/m_0)}} = k \cdot c_s \cdot dt \quad (6)$$

This model can be simply solved numerically by replacing differentials with differences for very small time intervals. Therefore, when using the initial condition $t = 0$, $\Delta m = 0$, for $\Delta m < m_0$

$$\Delta m_t = \Delta m_{t-\Delta t} + \Delta t \cdot k_1 \sqrt{1 - \frac{\Delta m_{t-\Delta t}}{m_0}} \quad (7)$$

where $k_1 = k \cdot c_s \cdot S_0$.

If the numerical calculation shows that $\Delta m > m_0$ then $\Delta m = m_0$ shall apply for the given point and all the following points.

The Diagram 5 shows a comparison of weight losses of E-fibers obtained using the proposed mathematical model which includes a change of the fibers surface area. The model described the experimental data very well and it has shown that the change of the surface alone leads to a plausible model of congruent dissolution. In a follow-up work it would be useful to try models that anticipate saturation of the solution and a change of the surface.

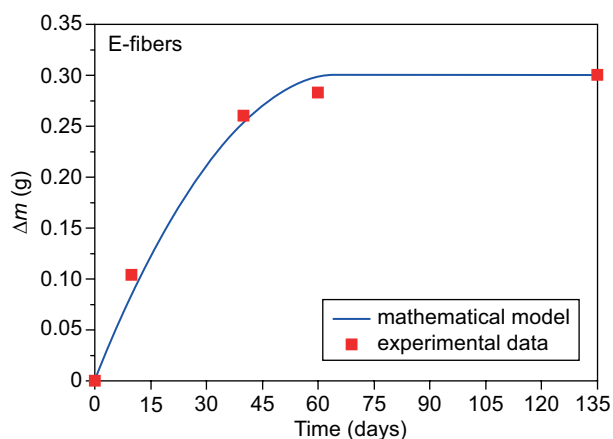


Diagram 5. Comparison of actual weight losses (■ points) and losses calculated with a mathematical model (— curve).

CONCLUSION

This work investigated dissolution of 3 types of glass fibers used commercially for thermal insulation. The dissolution process was monitored for 135 days (E-fibers) and for 60 days (Promaglaf HTI 1200 and 1250 fibers) with dynamic flow tests in simulated lung fluid with pH 7.4 at 37 ± 0.5 °C, the flow rate of SLF was $120 \text{ ml} \cdot \text{day}^{-1}$.

The results have shown that all types of the tested fibers dissolve in the model SLF. The dissolution was the fastest for E-fibers (after 60 days over 90 wt. % of the fibers were dissolved and after 135 days nearly 99 wt. % of the fibers were dissolved); in general, E-fibers dissolve nearly congruently and therefore they can be considered safe for human health in case of aspiration into lungs. The Promaglaf fibers dissolved less – after 60 days of leaching the weight loss of HTI 1200 fibers was 54 wt. %, and the weight loss of HTI 1250 fibers was 32 wt. %; both types of the fibers demonstrated incongruent dissolution. In case of the Promaglaf HTI 1200 fibers this is probably caused by precipitation of components from the solution and by development of a calcium-phosphorous layer on the surface of the fibers; in case of the Promaglaf HTI 1250 fibers it was probably caused by fast leaching of Ca and Mg from the surface and by development of a chemically resistant layer with high silica content on the surface.

The experimental time dependence of NL_t corresponds to the data calculated from the kinetic model developed by DGC UCT in Prague. A mathematical model (7) taking into account a change of the surface area corresponds with the weight losses of E-fibers as they dissolve congruently, the solution is not saturated and back precipitation does not occur. This model was not appropriate for the other 2 types of glass fibers as they developed layers on their surfaces.

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