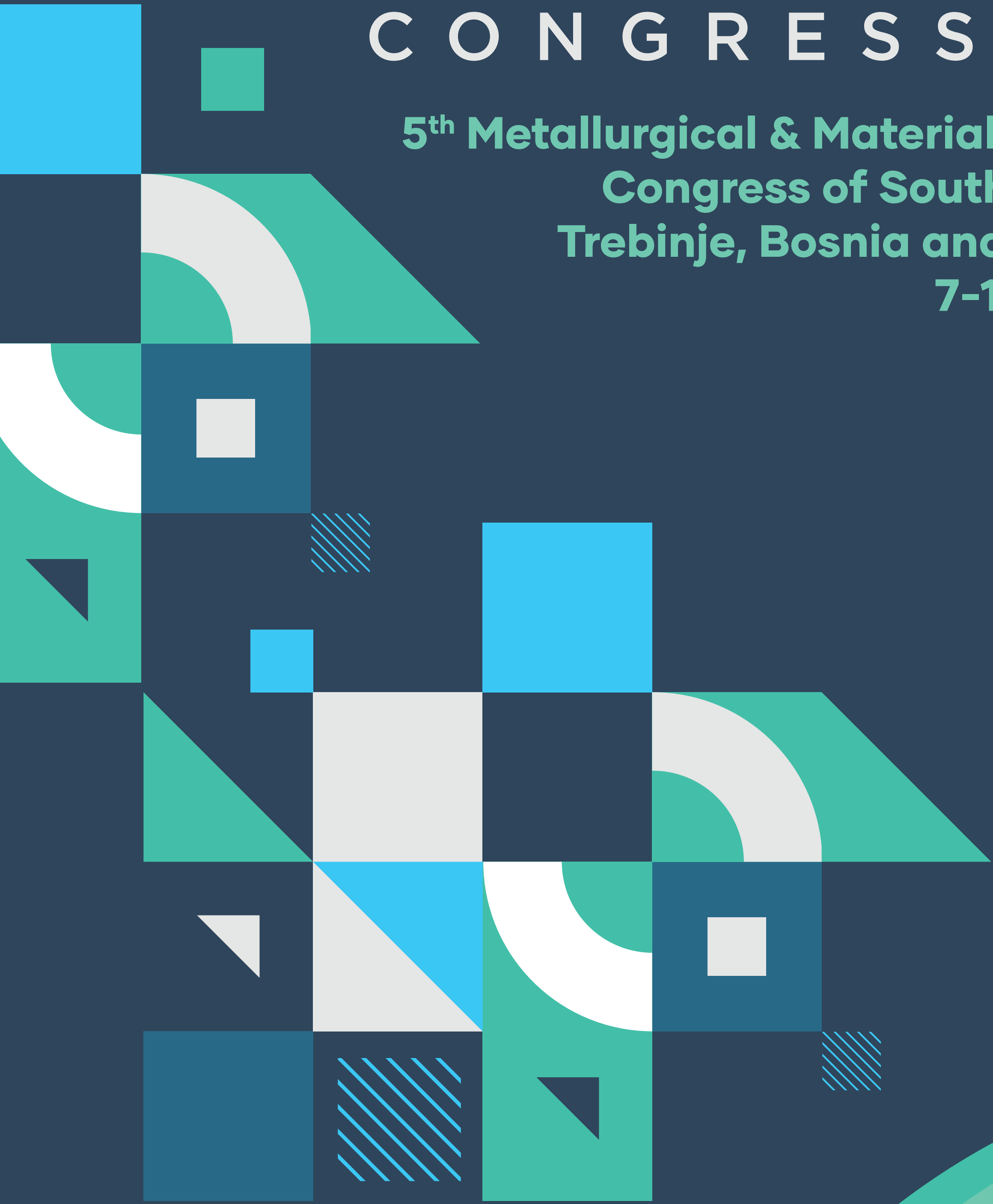
MME SEE

CONGRESS 2023

5th Metallurgical & Materials Engineering
Congress of South-East Europe
Trebinje, Bosnia and Herzegovina
7-10th June 2023



CONGRESS PROCEEDINGS

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The Faculty of Technology at the University of Banja Luka, Bosnia and Herzegovina;
The Faculty of Metallurgy at the University of Zagreb in Sisak, Croatia;
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The Faculty of metallurgy and technology at the University of Podgorica, Montenegro.

CONGRESS PROCEEDINGS - MME SEE 2023
5th Metallurgical & Materials Engineering Congress of South-East Europe

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PREFACE

On behalf of the Scientific and Organizing Committee, it is a great honor and pleasure to wish all the participants a warm welcome to the Fifth Metallurgical & Materials Engineering Congress of South-East Europe (MME SEE 2023) which is being held in Trebinje, Bosnia and Hercegovina, 07 - 10 June 2023.

The MME SEE 2023 is a biannual meeting of scientists, professionals, and specialists working in the fields of metallurgical and materials engineering. The aim of the Congress is to present current research results related to processing/structure/property relationships, advances in processing, characterization, and applications of modern materials. Congress encompasses a wide range of related topics and presents the current views from both academia and industry: Future of metals/materials industry in South-East European countries; Raw materials; New industrial achievements, developments and trends in metals/materials; Ferrous and nonferrous metals production; Metal forming, casting, refractories and powder metallurgy; New and advanced ceramics, polymers, and composites; Characterization and structure of materials; Recycling and waste minimization; Corrosion, coating, and protection of materials; Process control and modeling; Nanotechnology; Sustainable development; Welding; Environmental protection; Education; Accreditation & certification.

The editors hope that Congress will stimulate new ideas and improve knowledge in the field of metallurgical and materials engineering. The Congress has been organized by the Association of Metallurgical Engineers of Serbia, with the co-organization of the Institute for Technology of Nuclear and Other Mineral Raw Materials, Belgrade, Serbia, Faculty of Technology and Metallurgy, University of Belgrade, Serbia, Faculty of Technology, University of Banja Luka, Bosnia and Herzegovina; the Faculty of Metallurgy, University of Zagreb, Sisak, Croatia; the Faculty of Natural Sciences and Engineering, University of Ljubljana, Slovenia; and the Faculty of Metallurgy and technology, University of Podgorica, Montenegro.

Financial support from the Ministry of Science, Technological Development and Innovation of the Republic of Serbia to researchers from Serbia for attending the congress is gratefully acknowledged. The support of the sponsors and their willingness to cooperate have been of great importance for the success of MME SEE 2023. The Organizing Committee would like to extend their appreciation and gratitude to all sponsors and friends of the conference for their donations and support.

We would like to thank all the authors who have contributed to this book of abstracts and also the members of the scientific and organizing committees, reviewers, speakers, chairpersons, and all the conference participants for their support of MME SEE 2023. Sincere thanks to all the people who have contributed to the successful organization of MME SEE 2023.

On behalf of the 5th MME SEE Scientific and Organizing Committee

Miroslav Sokić, PhD

DISSOLUTION OF POLYPHOSPHATE GLASS: IMPACT OF pH ON DIFFUSION COEFFICIENTS OF MODIFYING CATIONS AT LOW TEMPERATURES

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This paper shows the dissolution behavior of polyphosphate glass in demineralized water and 2% citric acid solution at temperatures: 15, 20, 30, 37 and 50 °C. The influence of the solvent type on the diffusion coefficients of the modifying cations, under static conditions, is also presented. The diffusion coefficients, determined from concentration profiles of the modifying cations, are calculated to be in the range of 0.27 to 350x10^{-14} m^2/h for dissolution in demineralized water, and in the range of 3.6 to 148x10^{-12} m^2/h for the dissolution in 2% citric acid solution. The values of activation energy of cations diffusion in demineralized water are in the range from 68.32 to 105.41 kJ/mol, while for 2% citric acid solution they are in the range from 39.28 to 55.34 kJ/mol.

Keywords: polyphosphate glass, diffusion coefficient, dissolution ;

Introduction

Phosphate glasses are very interesting materials for use in agriculture as controlled release fertilizers. These materials have low chemical durability, as provided by easily hydrated P-O-P bridges, induced by water in a humid environment. The knowing of the chemical processes that occurs at the contact surface between glasses and aqueous solution may help in the designing and production of materials that can be used for protection of the natural environment. Three main processes have to be considered during glass alteration: hydration reaction, ion-exchange reactions in which modifier cations such as alkali (or alkaline-earth) are replaced by H^+ ions and hydrolysis, where water reacts with metal-oxygen bonds in the glass to form hydroxyl groups (Labbilta et al. 2023; Çelikbilek Ersundu, Kuzu, and Ersundu 2022; Bouabdalli et al. 2023). Ion exchange reactions, takes place parallel to the hydrolysis of the covalent bonds in the glass network, have been identified as key processes of glass dissolution. Ion exchange is referred to the exchange between hydrogen or hydronium ions (H^+ or H_3O^+) from solution and glass network-modifying cations e.g. cations bonded to non-bridging oxygens and their diffusion through the glass. Dependence of the extracted cations concentration on square root of time indicates the diffusive nature of this process. A mathematical description of cation release into solution is based on a number of simplifying assumptions (Mrabet, Cherbib, and Khattech 2020; Fournier et al. 2018; Poluektov et al. 2017; Melling and Allnatt 1980; Tošić et al. 2013) The rate of release of the i^{th} cation into solution via diffusion-controlled ion exchange r_{ei} (g/m^{2}h) is given by magnitude of the flux species (J_i) through the surface of glass i.e. at the glass-solution interface:

$$J_i = \rho x_i \sqrt{\frac{D_i}{\pi t}} \tag{1}$$

where D_i is the effective diffusion coefficient, ρ is the density of the glass, t is the time, x_i is the mass fraction of i^{th} cations in the glass.

The total amount of leached ions, *i* dissolved per unit area of interface at the time *t* is:

$$M_{(it)} = \int_0^t D_i \left(\frac{\partial c_i}{\partial y} \right)_{y=0} \cdot dt \tag{2}$$

where c_i is the concentration leached ion at distance *y* from the moving glass-solution interface at time *t*. To compare the leaching of different cations, the normalized i^{th} element mass release from a unit area (f_i) was used:

$$f_i = \frac{M_{(it)}}{x_i \cdot S} \tag{3}$$

For initial time of dissolution (short time):

$$f_i = 2\rho \cdot \sqrt{\frac{D_i \cdot t}{\pi}} \tag{4}$$

where D_i has an Arrhenius temperature dependence that can be represented by the following equation:

$$D_i = D_{oi} \exp\left(-\frac{E_{ai}}{RT} \right) \tag{5}$$

where D_{oi} is the pre-exponential term, *T* is the absolute temperature, *R* is the molar gas constant and E_{ai} is the activation energy of effective diffusion. According to Doremus (Doremus 1982). approach, interdiffusion coefficient of H^+ ion in glass can be represented as $D_i \equiv D_{iH}$. Hence pre-exponential term can be expressed as a function of solution's pH value, e.g. $D_{oi} \cong \xi_i \cdot D^*_{oH} \cdot 10^{-pH}$, where D^*_{oH} is pre-exponential term of the diffusion coefficient for protons in the glass and ξ_i is the dimensionless parameter. The rate of cations release via ion-exchange mechanism is given by:

$$r_{ei} = \rho \cdot x_i \sqrt{\frac{\xi_i D^*_{oH}}{\pi \cdot t}} \cdot 10^{\frac{-pH}{2}} \, exp\left(-\frac{E_{ai}}{2RT} \right) \tag{6}$$

It can be noticed (Eq (6)) that ion-exchange occurs preferentially in acidic solutions. Increase in pH value leads to decreases of ion-exchange rate. In addition, the rate of ion-exchange depends on the type of anions involved. The ion-exchange reaction leads to gradual depletion of cations content in the near surface layers (Melling and Allnatt 1980; Tošić et al. 2013; Doremus 1982).

The test of the chemical stability of the examined glass was carried out in order to predict the deterioration of phosphate glass during its application to the soil. Also, the rate of cations release from the glass is very significant parameter because these ions represent very useful nutrients for the plants.

Materials and methods

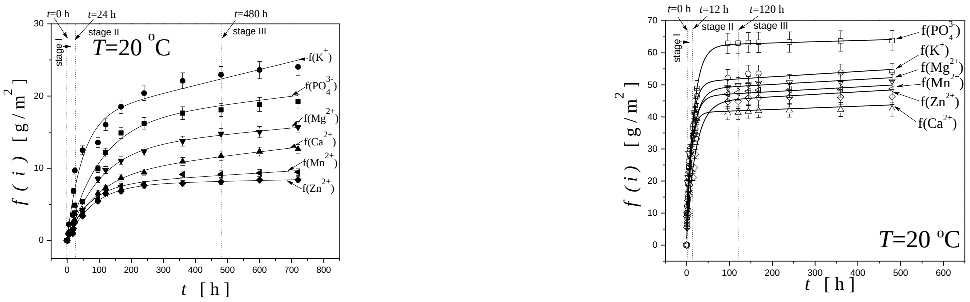
The appropriate glass batches compositions were prepared from reagent grade raw materials $(NH_4)_2HPO_4$, K_2CO_3, $CaCO_3$, SiO_2, MgO, ZnO and MnO_2 in an open crucible. The glasses mixture was melted at T = 1230 °C for t = 1h in an electric furnace and the melt was quenched on a steel plate. Powder X-ray diffraction (XRD) analysis confirmed the quenched melt to be vitreous (data not shown). Leach tests were conducted in demineralized water (pH=6.93) and 2% citric acid solution (pH=2.41). The experiments were performed with glass powders particle with size of 0.3-0.65 mm (specific surface area is 0.0178 m^2/g). Glass powder (1 g) was placed into volumetric flasks and then 50 ml of water or citric acid was added. Closed flasks were placed in a water bath. The experiments were performed at temperatures of 15, 20, 30, 37 and 50°C for different times in the range from 0.5 to 720 h.

After completion of experiment, the flask content was filtered and the concentration of ions in the obtained leachate were determined. The chemical composition of glass as well as flask content was determined by gravimetric (Si) and spectroscopic methods, *i.e.*, by AAS using a PERKIN ELMER 703 instrument (Ca, K, Mg, Cu, Zn) and UV/VIS spectroscopy using a PHILIPS 8610 spectrophotometer for phosphorus determination.

Results and discussion

The chemical composition of glass is 45.4P_2O_5 3.1SiO_2 25.6K_2O 14.5CaO 9.3MgO 1.2ZnO 0.9MnO [mol%]. This glass belongs to the polyphosphate glasses, consists of phosphate chains containing 32 tetrahedra, of which 30 have two bridging oxygens and two, the terminal tetrahedra, have one bridging oxygen. The modifier ions are placed in the cavities between the entangled phosphate chains (a detailed analysis of the structure of the examined glass is presented in the paper (Tošić et al. 2013)).

Fig. 1 shows the dependence of normalized i^{th} element mass release from a unit area (f_i), on time for T=20 °C, where three stages can be distinguished. At the beginning, for shorter dissolution times (stage I), the changes of f_i are linear with time for all cations, suggested the highest rates of glass dissolution under the given conditions. Under static leaching conditions, this linear phase is followed by an intermediate phase (stage II) where cations mass release becomes slower with increasing dissolution time. In stage III, it can be proposed that dissolution time does not influences f_i significantly.



a) *b)*

Figure 1 The time dependence of f_i for different ions at a temperature of 20 °C: a) demineralized water and b) *2% citric acid solution*

With increasing time, the rates of dissolution are several orders of magnitude lower than the initial rates. With temperature increase, the time intervals corresponding to the stage I and II are shortened, while that of stage III expand (data not shown). Also, from Fig. 1, it can be seen that the time intervals for all the three stages are smaller when the examined glass is dissolved in a 2% citric acid solution (Fig. 1b) with respect to time intervals corresponding to glass dissolution in demineralized water (Fig. 1a), at the same temperature. In the case of glass dissolution in demineralized water (Fig. 1a), stage I can be observed in the time interval from 0 to 24 h, stage II from 24 to 480 h and stage III over 480 h. In an acidic medium (Fig. 1b), the stage I can be observed from 0 to 12 h, stage II from 12 to 120 h and stage III over 120 h. The initial dissolution rates are several orders of magnitude higher when dissolving in an acidic medium with respect to the dissolution in demineralized water, while for longer dissolution time, the dissolution rates are of the same order of magnitude for the same cations in both solvents.

The ion exchange reaction involves interdiffusion and exchange of the cations in the glass with protons from the water that results in formation of an interdiffusion flux of cations replaced by the H^+ ions. Diffusive nature of the ion exchange appears as a crucial phenomenon during initial moments of glass leaching.

By using Eq. (4) and the measured concentrations of each ions in solution for short times (initial step of the glass/water reaction where the ion exchange reaction dominates), the values of the effective diffusion coefficients for all ions at all experimental temperatures were determined. Cation diffusion coefficients were calculated for a time of 5 h for demineralized water and for a time of 1 h for a 2% citric acid solution. Tables 1 and 2 shows the calculated cations' diffusion coefficients at all experimental temperatures for both solvents.

Table 1 The cations coefficients diffusion for dissolution in demineralized water

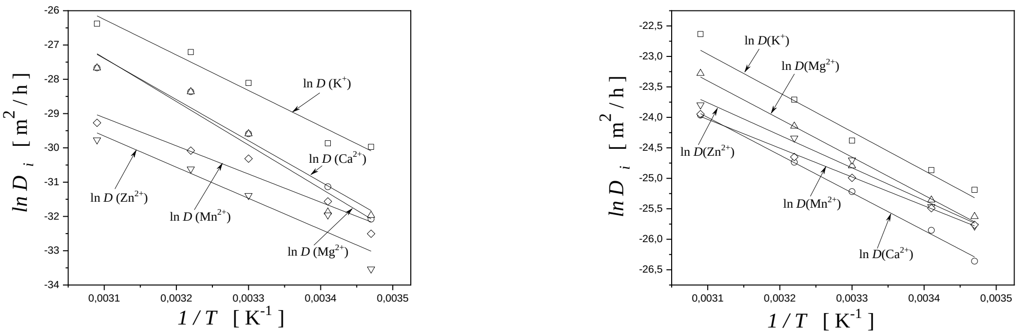
T [°C]	D_i [m^2/h] x 10^{-14}				
	i				
	K^+	Ca^{2+}	Mg^{2+}	Zn^{2+}	Mn^{2+}
15	9.62	1.17	1.31	0.27	0.76
20	10.7	3.01	1.44	1.32	1.96
30	62.1	14.1	14.1	2.33	6.83
37	153	48.1	48.1	5.05	8.63
50	350	96.4	96.4	11.8	19.4

Table 2 The cations coefficients diffusion for dissolution in *2% citric acid solution*

T [°C]	D_i [m^2/h] x 10^{-12}				
	i				
	K^+	Ca^{2+}	Mg^{2+}	Zn^{2+}	Mn^{2+}
15	11.5	3.6	7.4	6.4	6.5
20	15.9	5.9	9.8	8.7	8.5
30	25.8	11.2	17.1	18.8	14.0
37	50.5	19.0	32.7	26.9	19.7
50	148.0	39.2	77.7	46.3	39.8

By comparing certain values of the diffusion coefficients of the same cations from Tables 1 and 2, it can be observed that the diffusion coefficients of cations are two orders of magnitude higher in 2% citric acid solution compared to the corresponding ones in demineralized water, which clearly indicates the influence of pH (type of solvent) on cations diffusion, i.e. that ions exchange is significantly faster in an acidic environment (Eq. (6)).

According to Eq. (5), the diffusion coefficients of cations and the rate of their release by diffusion show an Arrhenius temperature dependence. Dependence of ln D_i on $1/T$, for all cations in both solutions are represented in Fig. 2. The activation energies of cations diffusion ($E_{ad,i}$) were determined from the slopes of these lines.



a) *b)*

Figure 2 *The dependences of ln D_i to 1/T:* a) demineralized water i b) *2% citric acid solution*.

Table 3 shows the calculated values of activation energies, $E_{ad,i}$, for present cations for both solvents. The values of activation energies of diffusion ($E_{ad,i}$) of the same cations are significantly lower when dissolved in a 2% citric acid solution with respect to the values obtained when dissolved in demineralized water, i.e. the influence of temperature on the cations diffusion during dissolution in an acidic environment is smaller compared to demineralized water.

Table 3 The calculated activation energies of diffusion of cations during dissolution in demineralized water (d.w.) and *2% citric acid solution (c.a.)*

	$E_{ad,i}$ [kJ/mol]	
i	d.w.	c.a.
K^+	86.13	55.34
Ca^{2+}	99.67	51.44
Mg^{2+}	105.41	51.99
Zn^{2+}	75.451	44.36
Mn^{2+}	68.32	39.28

Conclusion

Dissolution of the polyphosphate glass in demineralized water and 2% citric acid solution was investigated under static conditions in the temperature range T=15–50 °C for different times using glass powder sample with particle in the range 0.3–0.65 mm. The time dependence of the concentration of elements in solution was analyzed. The cation diffusion coefficients, for dissolution in demineralized water, were calculated to be in the range of 0.27 to 350x10^{-14} m^2/h, while for the dissolution in 2% citric acid solution the values of the diffusion coefficients were calculated to be in the range of 3.6 to 148x10^{-12} m^2/h. The values of cations diffusion coefficients are two orders of magnitude higher in the case of 2% citric acid solution compared to the corresponding ones in demineralized water. The values of activation energy of cations diffusion in demineralized water are in the range from 68.32 to 105.41 kJ/mol, while for 2% citric acid solution they are in the range from 39.28 to 55.34 kJ/mol. Such values indicate that the influence of temperature on the cations diffusion during dissolution in an acidic environment is lower compared to demineralized water.

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