

NEUTRALIZAREA ȘI INDEPĂRTAREA COMPUȘILOR CONȚINÂND IONI DE FLUORURĂ DIN SILICA GEL

NEUTRALIZATION AND REMOVAL OF COMPOUNDS CONTAINING FLUORIDE IONS FROM WASTE SILICA GEL

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In this work the possibility of neutralizing or removing compounds containing fluoride ions from waste silica gel was investigated. The elution of F⁻ ions from waste silica gel to the liquid medium was performed under static and hydrothermal conditions. It was determined that waste silica gel consists of silicon dioxide (77.77%) and other compounds containing 8.64% F⁻ and 4.03% Al³⁺ ions, which are stable during the drying process (50°C; 48 h). It was determined that under static elution conditions it is possible to remove only 6–7 wt. % F⁻ ions into a liquid medium from waste silica gel. It was found that CaO additive has a positive influence on the decomposition of AlF₃·3H₂O and the formation of CaF₂. It was found that silica gel is a prospective raw material for synthesis of Z-phase because the kinetics of compound formation is faster than in pure mixtures. A principal technological scheme has been designed for waste silica gel neutralization and utilization by using Ca(OH)₂ under static or hydrothermal conditions.

Keywords: silica gel, waste, fluoride, aluminum fluoride trihydrate, neutralization

1. Introduction

In many countries, the management of technogenic waste is a priority of the environmental protection field. Widespread attention is given to the implementation of basic waste management principles, namely waste avoidance, waste minimization, and waste disposal. At the same time, the issue of raw materials arises due to the increasing variety of industry production volumes [1, 2].

There have been many investigations of waste silica utilization from the aluminum fluoride industries [3]. Krysztafkiewicz et al. [3] explored recycling of waste silica into solutions of sodium or potassium metasilicate of various modules. Others have explored obtaining active silica that can be used as a filtration medium in the pharmaceutical, food, and other industries [4, 5]; producing average medium fillers for polymers or carriers to be used in catalytic mass preparation [6]; developing building materials with the use of chalks; and producing polishing and cosmetic pastes [7]. Sarkar et al. [8] determined the possibility of using waste silica in a normal white ware porcelain composition, replacing the quartz. It was found that the application of waste silica saves energy in the manufacture of white ware such as insulators, crockery, and tile.

Unfortunately, technogenic silica gel, due to various impurities containing fluoride ions, is not

widely used or processed, and instead it is stored in a landfill site [9, 10]. Several methods have been developed to remove fluoride ions from water: adsorption and biosorption; ion exchange; chemical precipitation, including electrocoagulation/flotation; membrane filtration; electrodialysis; and nanofiltration [11, 12]. Most methods for removing fluoride have drawbacks, including high initial cost, lack of selectivity, low capacity, and difficulty in operation or regeneration. Among these methods, adsorption is the most widely used because it is universal, low cost, and viable [13–15].

Only fragmentary data on the removal of fluoride ions from the limited solubility of solid materials and waste during hydrothermal synthesis are published in the literature [16]. Hydrothermal synthesis includes the various techniques of crystallizing substances from high-temperature aqueous solutions at high vapor pressures. It is presumed that during hydrothermal synthesis F⁻ ions in the waste silica gel will be combined into a stable compound, calcium fluoride, and this material could be used in binders and in the building materials industry.

The aim of this research was to investigate the possibility of removing compounds containing fluoride ions in waste silica gel or neutralizing them during a low-base calcium silicate hydrate synthesis.

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2. Materials and methods

In this paper the following reagents were used: - waste silica gel, i.e. a waste product of AlF_3 production (moisture content 60-65%; specific surface area by Blane $S_a = 400 \text{ m}^2/\text{kg}$; JSC Lifosa, Lithuania), dried for 7 days at room temperature (20–25°C) in the laboratory and an additional 48 hours at 50°C in the dryer; - calcium oxide which has been produced by burning calcium hydroxide (Stanchem, Poland) at 950°C for 30 minutes ($\text{CaO}_{\text{free}} = 97.68\%$).

The quantitative chemical composition analysis of waste silica gel was performed by X-ray fluorescence spectroscopy (XRF) on a Bruker X-ray S8 Tiger WD spectrometer equipped with a Rh tube with energy of up to 60 keV. Powder samples were measured in Helium atmosphere and data were analysed with SPECTRA Plus QUANT EXPRESS standard less software.

The elution of F^- ions from waste silica gel to the aqueous phase was done by keeping the suspensions at different temperatures (25, 50, 75, and 95°C) for 1 h when water-to-solid (w/s) ratios were 10 and 500. The neutralization of compounds containing F^- ions in waste silica gel was performed by adding 6.5, 10, and 20 wt. % CaO to the system. The suspension was kept at 25 °C for 1 h when w/s = 10 and 500. The concentration of fluoride ions in the liquid medium and pH values were measured by using a Metler Toledo T70 potentiometer. The error of the selective electrode for F^- ions is $\pm 1 \text{ ppm}$ (0.0001%).

Application of waste silica gel to the synthesis of low-base calcium silicate hydrate was performed in mixture which molar ratio of C/S (CaO/SiO_2) was equal to 0.55. Homogenized raw materials were mixed with distilled water to obtain water/solid ratio of the suspension equal to 10. The hydrothermal synthesis was carried out in unstirred suspensions in 25 ml volume PTFE cells, which were placed in a „Parr Instrument“ (Germany) autoclave, under saturated steam pressure at 200°C, for 4 and 8 h. The resulting product was filtered, rinsed with acetone to prevent the carbonation process in the materials, dried at $50 \pm 5^\circ\text{C}$ for 24 h, and sieved through a sieve with a mesh width of 80 μm .

The X-ray powder diffraction (XRD) data were obtained with a DRON-6 X-ray diffractometer with Bragg-Brentano geometry using Ni-filtered CuK_α radiation and a graphite monochromator, operating with a voltage of 30 kV and emission current of 20 mA. The step-scan covered an angular range of 2–60° (2θ) in steps of $2\theta = 0.02^\circ$.

Infrared spectra were carried out with the help of a Perkin Elmer FT-IR Spectrum X system. Specimens were prepared by mixing 1 mg of the synthesized product with 200 mg of KBr. The spectral analysis was performed in the range of 4000–400 cm^{-1} with spectral resolution of 1 cm^{-1} .

Simultaneous thermal analysis TG/DSC was used to measure the thermal stability and phase transformation of samples at a heating rate of 15 °C/min; the temperature ranged from 30°C up to 1000°C under air atmosphere. The test was carried out on a Netzsch Instrument STA 409 PC Luxx. Ceramic sample handlers and crucibles of Pt-Rh were used.

Scanning electron microscopy (SEM) (FEI Quanta 200 FEG) was performed using an accelerating voltage of 20 kV and a working distance of 10 mm for SEM observation.

The particle size distribution of dried waste silica gel was measured using a CILAS 1090 LD grain-size analyzer, which has a sensitivity range from 0.04 to 500 μm . Water was used as the carrier liquid. The particles were dispersed by ultrasound for 2 min while the obscuration of particles in water reached 14%. The ultrasound duration during the measurement was 70 s. The standard operating procedure used was the Fraunhofer method.

3. Results and discussions

3.1. Chemical and mineralogical composition of waste silica gel

In the first stage of the investigation, the sample of waste silica gel was characterized by XRF analysis. It was obtained that waste silica gel consists of 77.77% silicon dioxide, 8.64% F, and 4.03% Al. In addition, the thermogravimetry analysis showed that mentioned sample at 30–1000°C temperature lose 9.56 wt. % (Table 1).

Table 1

Chemical composition of dried waste silica gel

Parameter	Quantity, wt. %
SiO_2	77.77
F	8.64
Al	4.03
Mass loss	9.56

It was determined that the compounds containing fluoride ions in the waste silica gel during the drying process (50°C, 48 h) remain stable because on the XRD pattern sharp peaks of $\text{AlF}_3 \cdot 3\text{H}_2\text{O}$ (*d*-spacing: 0.545, 0.386, 0.329, 0.244 nm) were identified (Fig. 1, a). Furthermore, the silicon dioxide is amorphous because a broad peak in the angle of diffraction that varied from 18° to 26° was observed. The diffraction maximums of compounds containing aluminum components (due to a small quantity) were not identified on the XRD pattern.

These results were confirmed by FT-IR data: absorption bands in the range of 1107, 930, and 802 cm^{-1} are characteristic of valence Si–O bond

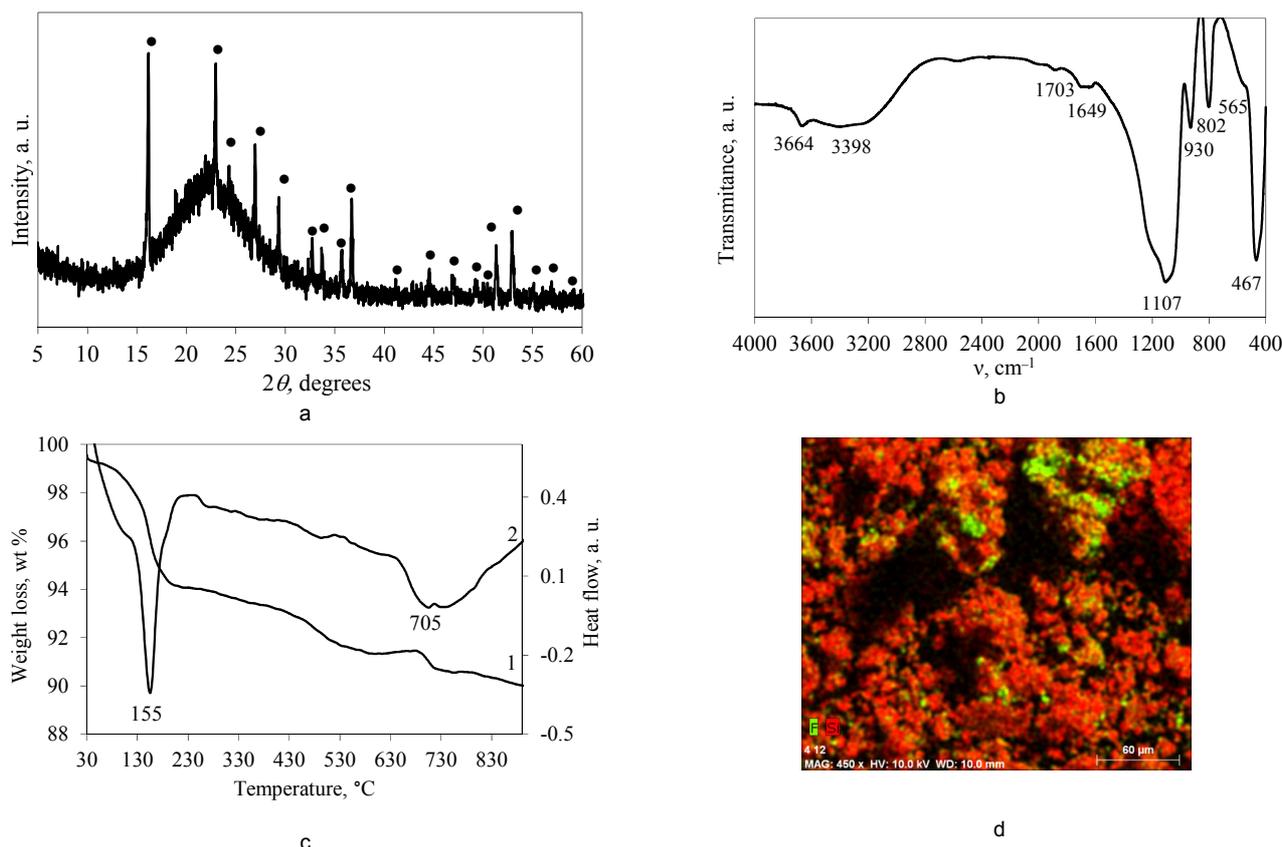


Fig. 1 - XRD pattern of waste silica gel (a), FT-IR spectrum (b), STA curves (c) and SEM micrograph (d), of dried silica gel at 50°C temperature for 48 h: 1 – TGA curve; 2 – DSC curve. Index: ● – $\text{AlF}_3 \cdot 3\text{H}_2\text{O}$.

vibrations and are typical for silica (Fig. 1, b). A sharp absorption band near the 467 cm^{-1} frequency range is characteristic of deformation vibration of Si–O–Si. Moreover, in the FT-IR spectrum, the absorption band found at 3664 cm^{-1} and the broad absorption band found at 3398 cm^{-1} describe the vibrations of hydroxyl groups connected to each other by hydrogen bonds. The deformation vibration of crystallization water is characterized by an absorption band at 1649 cm^{-1} . In addition, the absorption band in the 565 cm^{-1} frequency range was assigned to the F–Al–F vibrations and confirmed the presence of a compound containing fluoride ions on waste silica gel.

The DSC curve showed a significant endothermic peak at 155 $^{\circ}\text{C}$ that was mainly due to the removal of crystallization water from the $\text{AlF}_3 \cdot 3\text{H}_2\text{O}$ structure (Fig. 1, c). During this process, the waste silica gel sample mass loss was about 5.50 wt. %. The stoichiometric calculations of $\text{AlF}_3 \cdot 3\text{H}_2\text{O}$ show that only 5.8% of fluoride ions are combined into the structure, and the remaining portion of them are adsorbed by the silica gel (Fig. 2, curve 1). The distribution of fluoride ions in the silica gel structure was confirmed by SEM analysis (Fig 1, d). Removal of the crystallization water in the waste silica gel structure occurs at the higher thermal treatment temperature. The blurred endothermic effect was identified at 705 $^{\circ}\text{C}$, which characterizes the aluminum trifluoride phase

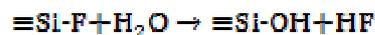
transition (α -; β -).

The SEM micrograph showed an amorphous mass of $\text{SiO}_2 \cdot n\text{H}_2\text{O}$ and tetragonal crystals of $\text{AlF}_3 \cdot 3\text{H}_2\text{O}$ with a size of 2.5–3 $\mu\text{m} \times 4$ –8 μm (Fig 1, d). According to the results of particle size distribution analysis, the diameter of the waste silica gel particles varied between 1 to 155 μm . In addition, one fraction of particles, with size of 38–60 μm , are dominant in this waste.

In the next stage of investigation the possibility of F^- ion removal from waste silica gel to a liquid medium was examined.

3.2. F^- ion elution from waste silica gel to a liquid medium

The water-soluble fluoride compounds occluded at the waste silica surface could be removed by using hot water. Extraction with hot water promotes release of hydrogen fluoride and reconstruction of active silanol groups at the waste silica surface:



Temperature has a significant effect on efficiency of fluoride removal [7]. For this reason, the elution of F^- ions from waste silica gel to the liquid medium at temperature range 25 $^{\circ}\text{C}$ –95 $^{\circ}\text{C}$ was performed.

It was determined that the mineralogical composition of waste silica gel slightly varies

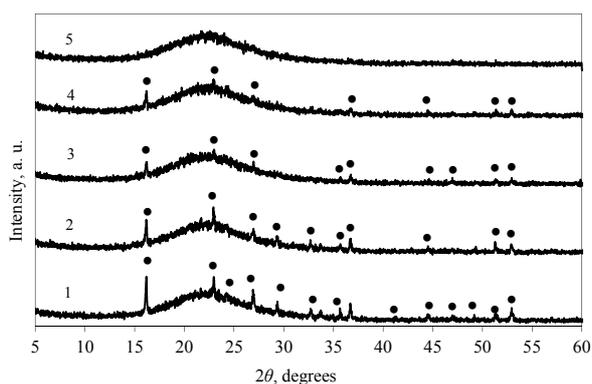


Fig. 2 - XRD diffraction patterns of the waste silica gel when w/s ratio was equal to 10 (curves 1–4) and 500 (curve 5) after 1 h treatment in different temperatures, °C: 1 - 25; 2 - 50; 3 - 75; 4 - 95; 5 - 25. Index: ● – AlF₃·3H₂O.

during elution at 25°C for 1 h when the ratio of w/s = 10 (Fig. 2, curve 1). It was ascertained that the increase of temperature positively affects the decomposition of AlF₃·3H₂O: the intensities of this compound's diffraction peaks decreased 1.5 times when the process temperature was equal to 95°C (Fig. 2, curve 4). Meanwhile, the data for the chemical analysis of the liquid medium showed that the amount of F⁻ ions remained approximately the same: ~0.002 wt. % in the temperature range 25–95 °C (Table 2). Because of the acidic nature of AlF₃·3H₂O, the pH value of the liquid medium also decreased. Therefore, in order to completely destroy the structure of the mentioned compound, the ratio of waste silica gel to the liquid medium was increased to 500. In this case, all crystalline structure compounds were decomposed in the temperature range 25–95°C (Fig. 2, curve 5). Only visible is a broad peak typical of amorphous SiO₂ in the angle of diffraction that varied from 18° to 26° on the XRD curve. Unexpected results were obtained after chemical analysis of the liquid medium: although AlF₃·3H₂O decomposes the released amount of F⁻ ions into the medium was equal to 6–7 wt. % from the total amount of F⁻ ions in the waste silica gel (Table 2). It should be noted that the negligible decrease of F⁻ ion concentration can be explained by the strong adsorption properties of the waste silica gel when the

temperature of liquid medium was increased (Table 2). This fact was confirmed by the slight increase of the liquid medium pH value.

Thus, it can be stated that fluoride ions removal into the liquid medium depends on such factors as dissociation, solubility, w/s ratio and adsorption properties of the waste silica gel. In order to reduce the adsorption properties of the waste silica gel and the mobility of F⁻ ions, in the next stage of research the additive CaO was used.

3.3. The influence of CaO additive on F⁻ ion mobility in the waste silica gel

Precipitation reactions are often used to remove F⁻ ions from wastewater because of the formation of CaF₂ precipitate. In order to reduce the F⁻ ion stability in the waste silica gel, in elution experiments, different amounts of CaO additive (6.5, 10, 20 wt. %) were used at 25°C for 1 h when the ratio of w/s = 500.

An amount of 6.5 wt. % CaO additive was used because, according to stoichiometric calculations, it is a sufficient amount of Ca²⁺ ions to bind all F⁻ ions in the waste silica gel. It was determined that CaO stimulates the formation of CaF₂ because low-intensity peaks of the latter compound were observed (Fig. 3, curve 1). Also, the concentration of F⁻ ions in the liquid medium decreased to 0.19 wt. % (Table 3). Therefore, by adding a larger amount of CaO additive (10 wt. %, 20 wt. %) into waste silica gel samples, the intensities of the CaF₂ diffraction peaks noticeably increased (Fig. 3, curves 2, 3). Also, the concentration of F⁻ ions in the liquid medium decreased almost 3 times, to 0.06 wt. % (Table 3). One of the main reasons may be the formation of CaF₂ and/or the reduction of SiO₂ adsorption properties in the alkaline medium (pH = 10.81). In addition, in samples with 20 wt. % of CaO additive, the formation of calcite (*d*-spacing: 0.304 nm) was observed.

Table 2

Concentration of F⁻ ions and pH values of liquid medium after 1 h in different temperatures when w/s = 10, 500

Temperature, °C	F ⁻ ion concentration · 10 ⁻² in the liquid medium wt.%		pH		F ⁻ ion concentration in the silica gel wt.%	
	w/s = 10	w/s = 500	w/s = 10	w/s = 500	w/s = 10	w/s = 500
25	2.4	61.3	2.80	5.15	8.62	8.03
50	2.3	59.7	2.84	5.32	8.62	8.04
75	2.1	53.3	2.89	5.38	8.62	8.11
95	2.0	52.5	2.90	5.76	8.62	8.12

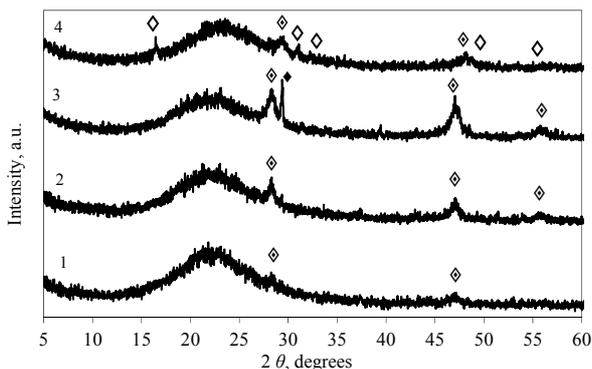


Fig. 3 - XRD patterns of waste silica gel after interaction with CaO additive at 25°C at a water-to-solid ratio of 500 and at the following CaO contents (wt. %): 1 - 6.5; 2 - 10; 3 - 20; 4 - 6.5 (w/s = 10). Indexes: ◇ - $\text{Al}_2(\text{OH})_{2.76}\text{F}_{3.24}\cdot\text{H}_2\text{O}$; ◊ - CaF_2 ; ◆ - CaCO_3 .

Table 3

Concentration of F^- ions and pH values of liquid medium after 1 h of interaction of waste silica gel with CaO additive at 25°C when w/s = 500

CaO additive, wt. %	F^- ion concentration in the liquid medium, wt. %	pH
6.5	0.19	7.07
10	0.17	8.81
20	0.06	10.81

It should be noted that the elution of F^- ions from the waste silica gel and CaO sample was not effective when the ratio of water-to-solid was equal to 10: the concentration of F^- ions in the liquid medium was higher, and the typical diffraction peaks of $\text{Al}_2(\text{OH})_{2.76}\text{F}_{3.24}\cdot\text{H}_2\text{O}$ together with CaF_2 were identified in the XRD curve (Fig. 3, curve 4).

Thus, the decomposition of $\text{AlF}_3\cdot 3\text{H}_2\text{O}$ and the formation of CaF_2 is more intensive when the amount of CaO additive in the waste silica gel samples increases. However, a small amount (0.5–0.6 wt. %) of fluoride ions remained in the liquid medium under all experimental conditions. For this reason, in the next experimental stage the application of waste silica gel to the formation of a

low-base calcium silicate hydrate under hydrothermal synthesis conditions was performed.

3.4. Application of waste silica gel to the synthesis of low-base calcium silicate hydrate

It was determined that in CaO–waste silica gel– H_2O mixture when the molar ratio of $\text{CaO}/\text{SiO}_2 = 0.55$ after 4 h of isothermal curing at 200°C, semi-crystalline calcium silicate hydrate, C-S-H(I) ($d - 0.304, 0.278, 0.183 \text{ nm}$), and hydrogarnet ($\text{Ca}_3\text{Al}_2\text{O}_6(\text{H}_2\text{O})_6$, $d - 0.513, 0.443, 0.336 \text{ nm}$) were formed (Fig. 4, a, curve 1). In addition, the main compound in the waste silica gel containing F^- ions, $\text{AlF}_3\cdot 3\text{H}_2\text{O}$, was unstable and decomposed. Thus, the F^- ions actively interacted with calcium ions and formed calcium fluoride ($d - 0.315, 0.193, 0.165 \text{ nm}$), the structure of which is stable under hydrothermal synthesis conditions. By prolonging the duration of isothermal curing to 8h, the main synthesis product, a high-crystallinity, low-base calcium silicate hydrate, Z-phase ($d - 0.155, 0.419, 0.382, 0.304, 0.183 \text{ nm}$), the stoichiometric composition of which corresponds to the molar ratio $\text{CaO}/\text{SiO}_2 = 0.55$ of the initial mixture, was already formed (Fig. 4, a, curve 2). Moreover, calcium fluoride and hydrogarnet remained stable during the hydrothermal synthesis process (Fig. 4, a, curve 2). It should be underlined that the crystallization of calcium silicate hydrates proceeds faster by using waste silica gel compared to pure mixtures [17–18].

The XRD analysis data were confirmed by the results of DSC (Fig. 4, b). An endothermic effect, typical to crystallization water removal from C-S-H(I) and Z-phase structure, in the 111–133°C temperature range was observed (Fig. 4, b). An endothermic effect in the 319–325°C temperature range characterize decomposition of $\text{Ca}_3\text{Al}_2\text{O}_6(\text{H}_2\text{O})_6$, whereas C-S-H (I) recrystallization to wollastonite occurs at 812°C.

Thus, during the hydrothermal synthesis, the F^- ions in the waste silica gel were combined into a stable compound: calcium fluoride. The data of the liquid medium analysis showed that fluoride ions were not released to the liquid medium after the hydrothermal synthesis process.

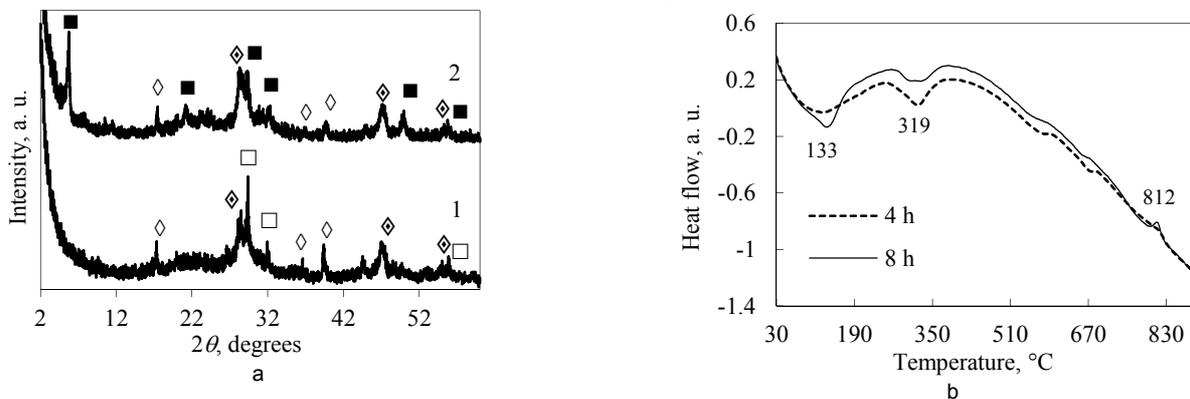


Fig. 4 - XRD patterns (a) and DSC analysis curves (b) of hydrothermal synthesis products after different isothermal treatment of mixtures with waste silica gel: 1 - 4 h; 2 - 8 h. Indexes: ◇ - hydrogarnet ($\text{Ca}_3\text{Al}_2\text{O}_6(\text{H}_2\text{O})_6$); □ - CSH(I); ◊ - CaF_2 ; ■ - Z-phase.

Thus, the experimental studies showed that reduction of mobility and/or removal of the F^- ion from the waste silica gel can be carried out using $Ca(OH)_2$ at static or hydrothermal conditions.

Based on the data obtained in this study, a principal technological scheme for the waste silica gel neutralization or utilization has been designed (Fig 5.). The required silica gel and $Ca(OH)_2$ weighed by the weight dispensers (3) are supplied to the mixer (5) in which the required amount of water is added from the reservoir (2) by using the volumetric dispenser (4). The mixture of raw materials is supplied by the pump to the adsorber (6) or to an autoclave (7), depending on the chosen conditions of neutralization. Stable compounds are formed during the hydrothermal treatment of waste silica gel with $Ca(OH)_2$ (CaO/SiO_2 ratio is equal to 0.55) in the autoclave (7), when the water vapor temperature is $200^\circ C$. After hydrothermal treatment, the product is placed into a filtration unit (8) and, later into a pulverous dryer (10), where 0.75–1.00 mm diameter pellets with a moisture content of 1.5–2% are produced.

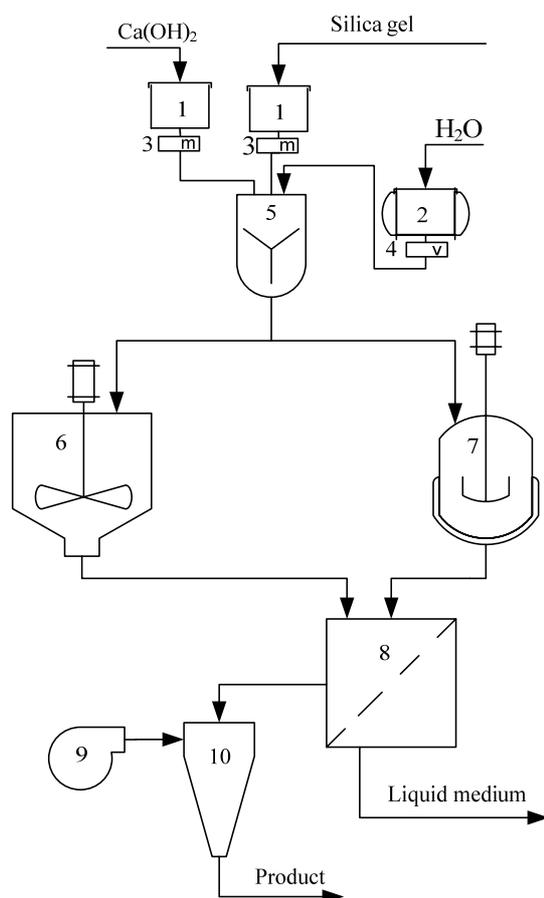


Fig. 5 - Principal technological scheme of waste silica gel neutralization and utilization: 1 – bunkers of raw materials, 2 – reservoir of water, 3 – weight dispensers, 4 – volume dispenser, 5 – mixer, 6 – adsorber, 7 – autoclave, 8 – unit of filtration, 9 – heater, 10 – pulverous dryer

4. Conclusions

1. It was determined that waste silica gel from aluminum fluoride production consists of silicon dioxide (77.77%) and other compounds containing 8.64% F^- and 4.03% Al^{3+} ions, which are stable during the drying process ($50^\circ C$; 48 h).

2. It was determined that under static elution conditions it is possible to remove only 6–7 wt. % F^- ions into the liquid medium from waste silica gel. In addition, the amount of waste silica gel to the liquid medium (w/s) affects the stability of $AlF_3 \cdot 3H_2O$ which decomposes when w/s is equal to 500. Also, it was found that CaO additive has a positive influence on the decomposition of $AlF_3 \cdot 3H_2O$ and the formation of CaF_2 . However, a small amount (0.5–0.6 wt. %) of fluorine ions were still remain in the liquid medium under all experimental conditions.

3. It was found that waste silica gel is prospective raw material for the synthesis of Z-phase because the kinetics of compound formation is faster than in pure mixtures. Also, during the hydrothermal synthesis, the F^- ions were not released to the liquid medium but combined into a stable compound, calcium fluoride. A principal technological scheme has been designed for waste silica gel neutralization and utilization by using $Ca(OH)_2$ under static or hydrothermal conditions.

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MANIFESTĂRI ȘTIINȚIFICE / SCIENTIFIC EVENTS



3rd International RILEM Conference on Strain Hardening Cementitious Composites (SHCC3-Delft) 03 November 2014 to 05 November 2014, Villa Augustus, Dordrecht, The Netherlands

Strain-Hardening Cement Composites (SHCC) are classes of fibre-reinforced cement-based composites in which crack formation in the cement-based matrix is controlled by fibres bridging cracks to the extent that multiple, closely spaced fine cracks form at increasing tensile deformation and force. RILEM TC-FDS is focussing on the durability afforded to the structural system in service conditions. This must be appropriately modelled and characterised to enable recommendation of rational design guidelines for durability design with SHCC. In addition the TC is dealing with a standard test procedure for such characterisation to verify compliance of a material to specified durability requirements.

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The conference covers the latest findings and research works related to cement based composites with a strain hardening behaviour under mechanical loading in the following topics:

- Test methods for mechanical characterization
- Structural design and performance
- Durability characterization and design
- Theoretical considerations and computational methods
- Practical Applications

The conference is the third in the series of SHCC-conferences. The first conference was organised by prof. Van Zijl in Stellenbosch, South Africa in 2009 and the second conference was in 2011 in Rio de Janeiro, Brazil organised by prof. Toledo Filho.

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