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## **EVALUATION OF WASTE SILICA PRECIPITATED IN THE PROCESS OF HYDROFLUORIC ACID PRODUCTION FROM FLUOSILICIC ACID**

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Studies were performed on the potential for purification of silica, precipitated as a by-product in the course of hydrofluoric acid production, and its possible use as an elastomer filler. The process underwent in the fluosilicic acid-sulphuric acid system.

The silica surface-occluded water soluble fluoride compounds were washed off in hot water, on vacuum filters. The obtained product manifested much better physicochemical parameters (i.e., more pronounced white colour and augmented specific surface area) as compared to raw silica. Moreover, the waste silica following leaching with hot water (at its boiling temperature) exhibited a favourable particle size distribution and contained no agglomerate structures.

The purified silica was employed as a filler in mixtures containing butadiene-styrene rubber. The obtained vulcanisates showed advantageous physicomechanical parameters (high tensile strength and high modulus).

*Key words: waste silicas; purification; hydrogen fluoride production*

### INTRODUCTION

Appropriate use of waste silicas from production of fluoride compounds represents a significant economic and ecologic problem. Particular potential is associated with application of waste silicas precipitated in the course of hydrogen fluoride production (Krysztafkiewicz 1996, 1992). Hydrofluoric acid is obtained from fluosilicic acid in reaction with concentrated (around 95%) sulphuric(VI) acid.



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The HF–H<sub>2</sub>O–H<sub>2</sub>SO<sub>4</sub> mixture was filtered off the precipitate of silica and, then, separated by various means, i.a. through distillation or HF stripping using hydrocarbons. The HF containing postreactive H<sub>2</sub>SO<sub>4</sub> was returned to fluosilicic acid decomposition. The filtered off silica represented a rather troublesome waste, containing solid substance and significant volumes of water (even over 70%). Eluate of the stored waste silica and water originating from drainage of heaps exposed to atmospheric precipitation may result in serious risks of polluting open waters (with soluble fluorides). For the reasons studies are conducted on utilisation of post-fluoride silica, considering the following trends of its utilisation: transformation of waste silica to sodium, lithium or potassium metasilicate solutions (Krysztafkiewicz 1987, Jacquin 1995, Satyanarayana 1996) production of silica as a filtration medium for the difficult to filter sediments (James 1989), preparation of catalytic masses (Imelik 1994, Tanabe 1994); production of substitute construction materials containing the silica and gypsum; application in polishing pastes and in cosmetics.

Any subsequent processing of the waste silica requires that it undergoes purification by removal from its surface of occluded fluoride compounds. This is particularly significant when the waste silica is supposed to be applied as an elastomer filler. Active silicas offered on the market are relatively expensive and not in every case they have to be employed (Legrand 1998, Vansant 1995). Several composites, i.a. filled rubbers used for sealing or stoppers need not to exhaust high resistance requirements (Zaborski 1993). In such cases, the less active waste silicas can be applied, originating from production of hydrofluoric acid. In this study, results of experiments are presented on properties, purification and application of such silicas.

## EXPERIMENTAL

### MATERIALS

Waste silica was sampled directly from production of hydrofluoric acid, involving decomposition of fluosilicic acid using sulphuric(VI) acid. The silica was subjected to purification by removal of post-production contaminations (HF and H<sub>2</sub>SiF<sub>6</sub> in particular). Since hydrofluoric acid and fluosilicic acid are well soluble in water, the impurities were washed off with hot water. In this aim the waste silica was exposed to hot water on a filter, dried at 105 °C. Aqueous solutions resulting from silica washes contained dissolved fluoride compounds, which were neutralised by passing the solution through a calcium oxide-containing deposit (e.g., fine-grained lime stone or dolomite). The de-fluorised in this way water was returned to purification of raw silica.

The raw waste silica and the silica obtained after washing with hot water were subjected to physicochemical evaluation.

#### METHODS OF STUDIES

After centrifugation and drying, in the studied silicas contents of water, silica, fluorine, aluminium, calcium, magnesium and iron were estimated, their density was established and pH of 4% water suspension was determined.

Specific surface areas of waste silicas were compared using a comparative chromatography (Paryjczak 1975). In this aim, an advantage was taken of the direct relation between the surface under a chromatographic peak and specific surface area for a standard sample. The gas (nitrogen) volume adsorbed on silica surface was calculated from the desorption peak due to its more pronounced symmetry.

Principal physicochemical parameters of silicas were also estimated, including bulk density, packing density, capacities to absorb water, dibutyl phthalate and paraffin oil. Intensity of white colour was measured by leukometry, using freshly roasted barium sulphate(VI) as a standard.

Water trapped in the structure of studied silicas was estimated using a derivatographic technique. The analysis were conducted in the OD-102 derivatograph in the following conditions: atmosphere of measurement: air, rate of heating up to the temperature of 1000 °C: 10 °C/min. Loss of weight or the amount of trapped water was estimated from thermogravimetric (TG) curve.

Studies on morphology and size of particles and on the extent of silica dispersion were conducted by transmission electron microscopy using one stage replica technique (Krysztafkiewicz 1981). In the studied, transmission electron microscope, Jeol 1200 EX II (Japan), was used.

Measurements of inter-grain porosity and of porosity linked to surface roughness were performed by the technique of mercury porosimetry using Carlo Erba, model 1540 porosimeter. Moreover, parameters typical for highly dispersed solids were established, including densifying capacity and electric conductivity. The densifying capacity was estimated in such a way that 6 g silica was added to 100 cm<sup>3</sup> butanediol – 1,4 and the time was determined after which a perfectly densified suspension was obtained. The recorded time was accepted as a measure of densifying potential.

Electric conduction was measured in 4% suspension of silica sample in water, using electric current of 500 – 1000 Hz frequency. Measurements of electric conduction were performed in special vessels equipped with platinum black-coated electrodes.

Waste silicas were applied as fillers of butadiene-styrene rubber. The mixture composition was as follows: 100 w/w of butadiene – styrene rubber, Ker 1500; 3 w/w of stearic acid, 50 w/w of silica, 5 w/w of zinc oxide, 0.8 w/w of DM accelerator (dibenzothiazole disulphide), 1.8 w/w of D accelerator (N,N'-diphenylguanidine), 2.5 w/w of sulphur. The rubber mixtures were vulcanised at 143 °C for 10 min.

Effects of fluorides occluded at the surface of waste silicas on physicochemical properties of rubber-styrene vulcanisates were observed in the course of aging studies. The vulcanisates were stored for 1 to 10 days at the temperature of 50 °C.

## RESULTS AND DISCUSSION

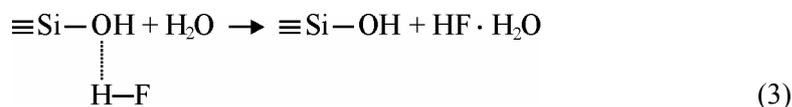
Results of chemical analysis of raw silica and water-extracted silica are presented in Table 1.

Table 1. Chemical composition of silica originating directly from production of hydrogen fluoride and of silica following extraction with water

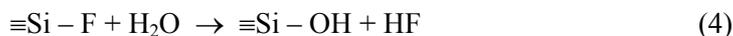
Component (% w/w)	Raw (unextracted) silica	Silica following water extraction at 60 °C	Silica following water extraction at 90-100 °C
SiO <sub>2</sub>	57.8	86.5	88.5
Humidity	30.7	5.8	3.5
Al <sub>2</sub> O <sub>3</sub>	1.235	0.687	0.213
F <sup>-</sup>	1.655	0.774	0.189
CaO	0.185	0.087	0.019
MgO	0.051	0.022	0.017
Fe <sup>+2</sup> and Fe <sup>+3</sup>	0.213	0.115	0.028
P <sub>2</sub> O <sub>5</sub>	0.125	0.043	0.012

Raw silica was contaminated with fluoride compounds, occluded at its surface. After intense extraction with water at temperatures approaching boiling temperature of water a very high extent of extraction of the fluoride compounds was achieved. The water-soluble fluorine compounds, occluded at the silica surface could be removed using hot water. The reactions followed the listed below mechanisms:

- HF might be bound to silica surface by hydrogen bonds between silanol groups and HF molecule. Hot water might promote disruption of the relatively weak bonds:



- hydrogen fluoride intensely dissolves silica and, therefore, at surface of the latter some silanol groups may substituted by fluorine. Extraction with hot water promotes release of hydrogen fluoride and reconstruction of active silanol groups at the silica surface:



Temperature exerted a significant effect on efficiency of fluorine compound removal. High extent of fluorine compound extraction could have been obtained following purification conducted at temperatures approaching boiling temperature of water (at the silica surface have remained permanently occluded fluorine compounds in amounts of around 0.1%). Extraction with water has exerted also a significant effect on stripping of the remaining contaminations (compounds of aluminium, calcium, magnesium, iron and phosphorus).

Principal physicochemical parameters of studied silicas (the raw and the water-extracted one) are presented in Table 2.

Table 2. Physicochemical properties of waste silicas before and after purification in hot water

Physicochemical parameter	Raw (unextracted) silica	Silica following water extraction at 60 °C	Silica following water extraction at 90-100 °C
Appearance	White powder	White powder	White powder
Structure	Traces of crystallites	Amorphous	Amorphous
pH of aqueous dispersion	4.5 – 5.0	6	6
Density, g/cm <sup>3</sup>	2.18	2.05	2.05
Bulk density, g/dm <sup>3</sup>	190	175	150
Packing density, g/dm <sup>3</sup>	290	250	220
Capacity to absorb water, cm <sup>3</sup> /100g	300	350	350
Capacity to absorb dibutyl phthalate, cm <sup>3</sup> /100g	320	370	375
Capacity to absorb paraffin oil, cm <sup>3</sup> /100g	350	400	420
Extent of whiteness, %	68.4	81.0	83.2
Specific surface area, m <sup>2</sup> /g	20.5	24.8	30.5

Extraction with hot water significantly influenced physicochemical parameters of the silica. Following the extraction (and following extraction at the temperature of boiling water) the silica manifested most favourable parameters. It manifested the highest capacity to absorb paraffin oil and dibutyl phthalate as well as the highest specific surface area. This pointed to augmented activity of the silica. Waste silicas were distinguished also by their amorphous character, which permitted to include them to the group of hydrated precipitated silicas, widely used as fillers. Low bulk density represents an advantageous character of such silicas and in the case of a well extracted product it did not exceed 150 g/dm<sup>3</sup>. Water extraction markedly improved whiteness of the silica: following the procedure conducted at the boiling temperature the extent of whiteness reached 83.2% (for the raw silica the extent was 68.4%). Water released upon silica heating at temperatures of 100 – 1100 °C has been formed by condensation of silanol, ≡Si–OH groups and formation of siloxane, ≡Si–O–Si≡ groups.

As indicated by the thermogravimetric data, studied silica contained similar amounts of water and upon heating at 120 °C lost weightwise the mean of 6 – 7% water (this was mostly physically-bound water). The remains of the so called trapped water could have been removed only by heating at higher temperatures. Silica heating at temperatures above 800 °C has resulted in an almost complete loss of the capacity of binding water. The first, clear decrease in silica weight at the temperature of 120 °C has been followed by subsequent drop in weight at 400 – 500 °C. Above the

temperature of 500 °C a mild, gradual decrease in weight could have been noted till the end of the sample heating.

Results of dispersion testing are presented in Table 3. The studies have shown that particles of diameters below 1 µm (for raw silicas and silicas extracted with water) have been most numerous.

Table 3. Extent of dispersion of studied silica particles

Particle diameter (µm)	% w/w of the fraction (mean)		
	Raw silica	Silica following water extraction at 60 °C	Silica following water extraction at 90-100 °C
Below 0.10	30.2	50.4	61.5
0.10 - 1.0	25.5	24.5	23.8
1.0 - 5.0	22.5	9.4	6.1
5.0 - 10.0	9.1	9.2	5.3
above 10.0	12.6	6.5	3.3

Following extraction with hot water (at the boiling temperature) has shown the most advantageous particle size distribution (most numerous particles of diameters below 0.10 µm – 61.5%, least numerous particles of diameters above 10 µm: as few as 3.3%). Action of hot water most probably has promoted destruction of silica agglomerate structures which automatically has improved dispersion structure of the silica. The observed extensive spread of particle sizes might be explained by phenomena which accompany primary and secondary agglomeration and by presence of slight amounts of contaminants.

Pore volumes for the unwashed silica and silica extracted with water at its boiling temperature are listed in Table 4.

Data of the Table 4 unequivocally indicate that the water-extracted silica has manifested higher total pore volume expressed in cm<sup>3</sup>/g than the silica which has not been subjected to the cleaning procedure. Nevertheless, both silicas have exhibited low total pore volumes (below 0.5000 cm<sup>3</sup>/g), which indicates their low surface activity, confirmed by the low values of specific surface area (of 30 m<sup>2</sup>/g order).

The obtained physicochemical data have been confronted with electron-microscopical patterns of studied silicas. Such studies represent a modern way of collecting data on morphology and structure of particle surface, structure of individual particles and agglomerate character. Moreover, they allow to appraise dispersion of the raw silica and of the silica extracted with hot water.

The raw waste silica has manifested polydispersity and strong tendency to form large accumulations of particles (agglomerates), as demonstrated in Figs.1 and 2. In the drawings of TEM patterns the poorly developed surface (smooth surface) can be seen.

Table 4. Distribution and volume of pores of radius range  $50\text{Å} \leq R \leq 75000\text{Å}$

P (atm)	R (Å)	Hg (cm)	Raw silica			Silica following water extraction at 90-100 °C		
			Scd (cm)	V <sub>p</sub> (%)	Σ V <sub>p</sub> (cm <sup>3</sup> /g)	Scd (cm)	V <sub>p</sub> (%)	Σ V <sub>p</sub> (cm <sup>3</sup> /g)
1	75000	0	0	100	0	0	100	0
2	37500	0	1.36	45.2	0.2348	1.2	48.8	0.2509
10	7500	0	1.78	28.2	0.3074	1.6	30.7	0.3190
100	750	0	1.98	20.2	0.3419	1.8	23.1	0.3602
200	375	0	2.00	19.4	0.3454	2.0	21.5	0.3713
300	250	0.06	2.02	18.5	0.3489	2.0	20.3	0.3798
500	150	0.1	2.08	16.1	0.3592	2.0	19.2	0.3873
800	93.75	0.18	2.16	12.9	0.3730	2.1	17.8	0.4005
1000	75	0.24	2.24	9.7	0.3868	2.2	11.5	0.4094
1200	62.5	0.26	2.34	5.7	0.4041	2.3	8.8	0.4222
1400	53.6	0.3	2.44	1.6	0.4214	2.4	2.3	0.4431
1500	50	0.32	2.48	0.0	0.4283	2.5	0.0	0.4680

Extraction of the silica with water at 90 – 100 °C has resulted in the product of a lower tendency to agglomerate but its particles have continued to form accumulations of an irregular shape. This has been confirmed by the electron microscopical patterns of the purified silica (Fig.3). Inquisitive analysis of the photographs allows to conclude as to the favourable effect of water extraction on dispersion, decomposition of agglomerates and shape of the particles.

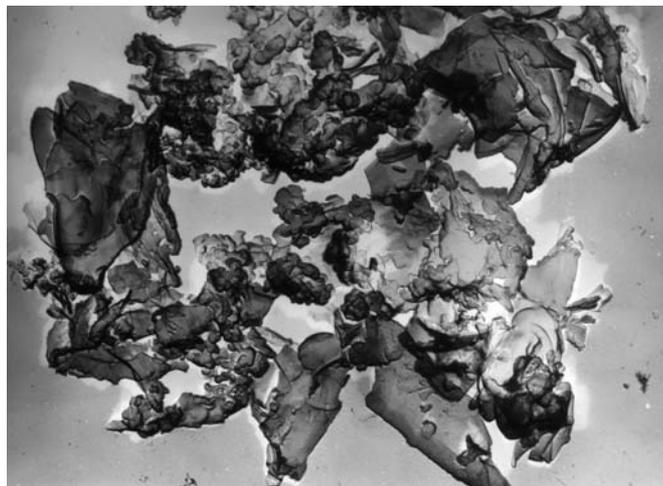


Fig. 1. TEM of raw silica (~ 13,000x)

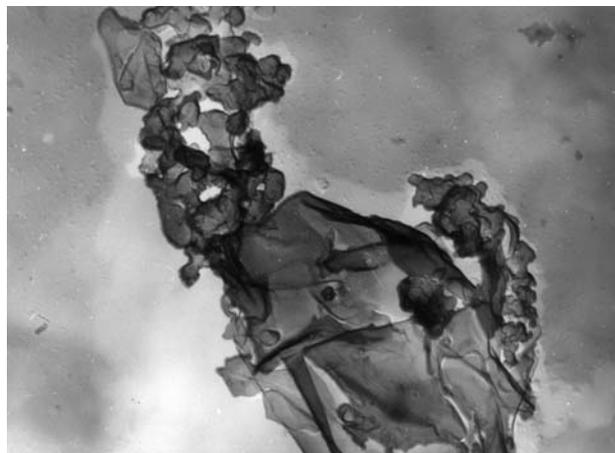


Fig. 2. TEM of raw silica (~ 26,000x)

Electric conductance and duration of their densifying activity in several cases allow to conclude on character of silica. The densifying activity (in s) and electric conductance (in  $\mu\text{S}$ ) of various pyrogenic, precipitated and the studied waste silicas are compared in Figs. 4 and 5.

In the pattern of Fig.4, the butanediol-1,4 densifying activity can be noted to be limited to 45 s.

Pyrogenic silicas (Aerosil and Cab-O-Sil) have shown densifying activity which has markedly exceeded 45 s while precipitated and waste silicas have demonstrated such densifying activity below 45 s. Clearly the lowest duration of the densifying activity has been demonstrated by the raw waste silica (as low as 15 s). Extraction with water has only insignificantly improved the parameter (after water extraction at 90 – 100 °C duration of the densifying activity has increased to 20 s).

The short duration of butanediol-1,4 densification by the waste silicas has reflected their heterogeneity and presence of large agglomerates, as confirmed by electron microscopy.

Also in the case of electric conductance, a threshold could be drawn between pyrogenic silicas on one hand and precipitated or waste silicas on the other. The threshold has corresponded to conductance of 100  $\mu\text{S}$ . Electric conductance of 4% dispersion of Aerosil 200 in water amounts to 95  $\mu\text{S}$  and, thus, pyrogenic silicas always manifest electric conductance values below 100  $\mu\text{S}$ . Definitely the highest electric conductance, 202  $\mu\text{S}$ , has been demonstrated by the waste silica from decomposition of fluosilicic acid under effect of sulphuric (VI) acid. The so high electric conductance of the waste silica reflects presence of contaminants, occluded at the silica surface. After extraction with water the silicas have shown a clear decrease in electric conductance (e.g., silica pre-washed with water at 90° C exhibits electric conductance of 158  $\mu\text{S}$ ).

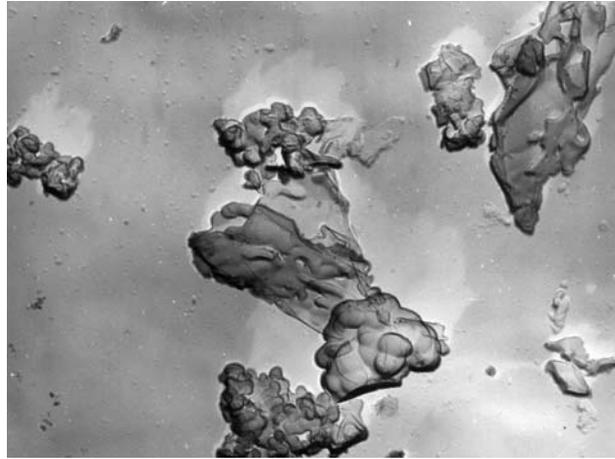


Fig. 3. TEM of raw silica following water extraction at 90-110 °C (~ 26,000x)

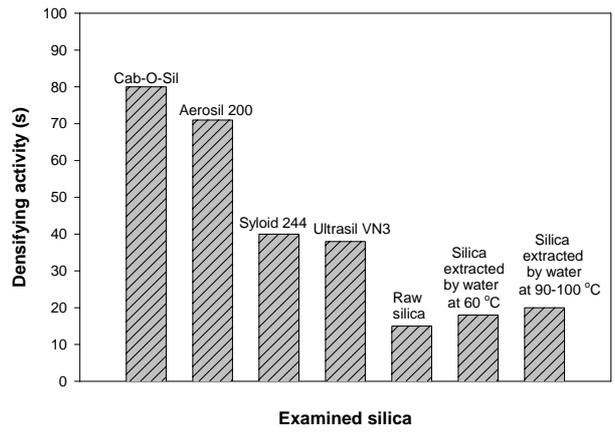


Fig. 4. Comparison of densifying activity compared for various silicas produced worldwide and for the tested waste silicas

Most important strength parameters of vulcanisates obtained following filling them with the studied waste silicas are shown in Table 5. Very interesting results have been obtained, which permit to apply the waste product in specific technologies. Application of the waste silica as a filler, following its extraction with water at 60 – 90 °C, deserves particular attention. The silica allows to obtain vulcanisates of best strength parameters: their tensile strength exceeds 12 MPa while the module amounts to 4.5 MPa. Data of Table 5 indicate also how far strength parameters can be improved by extraction of soluble fluorine compounds from silica surface.

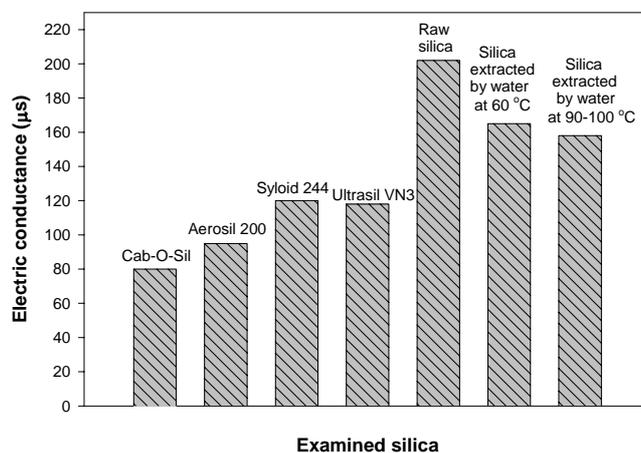


Fig. 5. Comparison of electric conductance of various silicas

Table 5. Strength parameters of butadiene-styrene vulcanisates filled with waste silicas

Parameter	Raw silica	Silica following extraction with water at 60 °C	Silica following water extraction at 90-100 °C
Tensile strength, MPa	8.2-8.8	9.5-11.0	10.1-12.9
Modulus (M-300), MPa	2.8-3.1	3.7-3.9	4.5-4.6
Relative elongation, %	400-420	435-470	458-505
Hardness, °Sh	60	62	62

Strength parameters of vulcanisates containing the unpurified waste silica have clearly deteriorated under effect of aging (depending upon duration of sample aging, tensile strength has decreased by around 35-50% as compared to the value measured directly after vulcanisation). The vulcanisates containing water-extracted silicas have demonstrated a much lower tendency for deterioration of strength parameters in the course of aging. A significant effect has been exerted by the temperature of waste silica extraction. Tensile strength as well as the M-100 modulus of vulcanisates filled with silicas extracted at the temperature of 60°C have decreased by 15-35% (depending upon duration of vulcanisate aging). On the other hand, vulcanisates filled with silicas extracted with water at the temperature of 90-100 °C have demonstrated advantageous physicochemical properties even following aging. Their tensile strength and modulus have decreased by as little as 5-15% depending upon duration of accelerated aging to which the samples have been exposed.

Thus, results of the studies on aging have confirmed the definitely negative effect of fluoride ions on strength parameters of the vulcanisates. Following extraction of fluorides, waste silicas can, to a limited extent, be employed in production of rubber as moderately active fillers.

Table 6. Aging studies on butadiene-styrene vulcanisates filled with waste silicas ( $R_r$  - tensile strength, M-100 modulus)

Duration of aging (days)	Raw silica				Silica following water extraction at 60 °C				Silica following water extraction at 90-100 °C			
	$R_r$ (MPa)	$\Delta R_r$ (%)	M-100 (MPa)	$\Delta(M-100)$ (%)	$R_r$ (MPa)	$\Delta R_r$ (%)	M-100 (MPa)	$\Delta(M-100)$ (%)	$R_r$ (MPa)	$\Delta R_r$ (%)	M-100 (MPa)	$\Delta(M-100)$ (%)
0	8.3	-	2.9	-	9.7	-	3.8	-	11.2	-	4.5	-
1	5.2	-37.4	1.9	-34.5	8.1	-16.5	3.2	-15.8	10.6	-5.4	4.2	-6.7
3	4.3	-48.2	1.8	-37.9	7.5	-22.7	3.0	-21.1	10.0	-11.7	4.0	-11.1
5	3.8	-54.2	1.6	-44.8	7.1	-26.8	2.8	-26.3	9.8	-12.5	3.8	-15.6
10	3.5	-57.8	1.4	-51.7	6.9	-28.9	2.5	-34.2	9.6	-14.3	3.7	-17.8

## CONCLUSIONS

Effect of temperature of water, used for purification of waste silica, was noted on extraction of fluorine compounds occluded on the silica surface and on principal physicochemical parameters (in particular on the extent of whiteness, bulk density, specific surface area and capacity to absorb paraffin oil).

After extraction with hot water (at the boiling temperature), waste silica demonstrated most favourable particle size distribution and contained no large agglomerate structures in contrast to raw silica, which exhibited polydispersity and tendency to form secondary agglomerates.

Waste silicas representing by-products from hydrogen fluoride production might find application as semiactive fillers of rubber vulcanisates. In their presence, products were obtained of adequate physicochemical parameters. A particular strengthening effect was observed when silica extracted with water at its boiling temperature was used as a filler. The obtained vulcanisates manifested high tensile strength and adequate modulus.

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**Krysztafkiewicz A., Świt Z., Jesionowski T.**, *Badania odpadowej krzemionki stracanej w procesie produkcji kwasu fluorowodorowego z kwasu fluorokrzemowego*, Physicochemical Problems of Mineral Processing, 39 (2005) 165-176 (w jęz. ang).

Przeprowadzono badania nad możliwością oczyszczania i ewentualnego wykorzystania jako napełniacza elastomerów krzemionki wytracającej się jako odpad w produkcji kwasu fluorowodorowego. Proces ten przebiega w układzie kwas fluorokrzemowy-kwas siarkowy.

Zaokludowane na powierzchni krzemionki rozpuszczalne w wodzie związki fluoru wmywano w gorącej wodzie na filtrach próżniowych. Uzyskano produkt o zdecydowanie lepszych parametrach fizykochemicznych (m.in. o zwiększonej białości oraz powierzchni właściwej) w porównaniu z krzemionką surową. Ponadto, krzemionka odpadowa po ługowaniu gorącą wodą (w temp. wrzenia) odznacza się korzystnym rozkładem wielkości cząstek i nieobecnością struktur aglomeratowych.

Oczyszczoną krzemionkę stosowano jako napełniacz mieszanki zawierającej kauczuk butadienowo-styrenowy. Uzyskano wulkanizaty o zadawalających parametrach fizykochemicznych (duża wytrzymałość na rozciąganie i wysokie moduły).