

Article

The Effect of Operating Variables on the Performance of Column Flotation of Silica Sand

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Abstract: This paper presents the results of a study performed on silica sand samples to determine the effect of operating parameters (collector type and dosage, solids content in the pulp, pulp pH and air flow rate) on the concentrate grade and mass recovery in laboratory-scale column flotation. Through the analysis of variance (ANOVA), it was determined that the pulp pH had a significant impact on all four of the observed grade parameters (mass contents of SiO_2 , Al_2O_3 , Fe_2O_3 and TiO_2 in the concentrate). Air flow rate had a significant impact on the mass contents of SiO_2 and Al_2O_3 . The solids content in the pulp only significantly affected the mass content of Fe_2O_3 . The mass recovery was significantly influenced by the pulp pH, with a high level of significance (p -value (p) = 0.002917), as well as by the air flow rate (p = 0.010285). On the basis of a model of the relationship between the respective grade parameters and tested operating variables, it was determined that with the use of the Aeromine 3030C collector (at a dosage of 500 g/t) the highest-grade concentrate regarding the mass contents of SiO_2 , Al_2O_3 and TiO_2 was achieved with a pulp pH within a range from 2.4 to 3 and an air flow rate above 26 L/h. The lowest mass content of Fe_2O_3 in the concentrate was achieved at a pulp pH in a range from 2.2 to 2.8 and a solids content in the pulp between 2.0% and 3.5%.

Keywords: silica sand; column flotation; operating parameters; concentrate; impurities



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1. Introduction

Silica sand is a very important non-metallic mineral raw material widely used for many industrial applications. Depending on the quality, implying specific physical and chemical properties, it is used as a basic raw material for the production of different glass types; for making moulds and cores in the foundry industry; in the ceramic, chemical and construction industries; as abrasives in sand blasting and polishing; for water filtration; for metallurgical-grade silicon production, etc. [1–4]. Different industrial uses of silica sand are based on either the chemical purity, i.e., a high proportion of silica and strictly limited amounts of certain components such as alumina, ferric and titanium oxide (Table 1) and/or physical properties of the sand such as particle-size distribution, grain shape and moisture content. Regarding the requirements of the respective industries for the chemical composition of silica sand, there are no current universal standards or industry-wide specifications; however, depending on the application, the general limit values shown in Table 1 are required. The higher proportion of silica and lower proportion of other components in the sand, the higher its quality and market value, i.e., application possibilities. Raw sand usually does not satisfy the demanding specifications and, therefore, its beneficiation is required to obtain a quartz product of wanted quality for further industrial processing [5,6].

Table 1. Quality specifications of silica sand regarding chemical composition for different industrial applications [7–14].

Component	Glass-Making (Glass Industry)	Ceramics	Foundry Moulds	Sand Filters in Water Treatment
	wt% *	wt% **	wt%	wt%
SiO ₂	>95	>93	>96	>96 (Type 1) >80 (Type 2, 3)
Fe ₂ O ₃	<0.5	<0.3	<1% total unwanted components (Fe ₂ O ₃ , K ₂ O, Na ₂ O, CaO, MgO)	<2 (Type 1)
Al ₂ O ₃	<2.5	<1.5		<3 (Type 1)
TiO ₂	<0.3	<0.15		-
Cr ₂ O ₃	<0.08	-		-
CaO + MgO	<0.6	<3.5		<1.5% CaO <2% K ₂ O
K ₂ O + Na ₂ O	<1.0	<0.5		<1.5% Na ₂ O (Type 1)
loss on ignition	<0.6	-	<0.5	-

* Depending on the type of glass manufactured (grades of silica sand). ** Depending on the type.

In the past, as a low-cost mineral raw material, silica sand was mined exclusively from high-grade deposits, which usually did not require the application of more complex beneficiation processes. However, with the development of the previously mentioned industries, the consumption of high-grade silica sands is increasing while their reserves are decreasing, and at the same time, the quality requirements are becoming stricter. Consequently, today, there is a growing need for the mining of lower-grade deposits with more complex mineral compositions and, thus, the need to include more complex beneficiation methods in process flowsheets.

Due to its mineral composition and the form of mineral intergrowth, the processing of silica sand may be very complex and often involves a series of physical and chemical beneficiation methods [15,16]. The chief processing aims are generally to achieve a suitable particle-size distribution, to increase the SiO₂ content and to reduce the content of any additional unwanted components affecting sand quality (Fe₂O₃, Al₂O₃, TiO₂, Cr₂O₃, etc.). The required particle-size distribution is obtained relatively simply by classification and washing. A basic problem is the removal of mineral impurities, bearers of unwanted components, such as heavy minerals, feldspars and micas, which can be present in different structural forms in silica sand [17–19]. A satisfactory removal of these impurities from certain types of silica sand cannot be achieved by only using simpler beneficiation methods—washing, classification and attrition scrubbing [20–22]. To obtain product quality (chemical and mineral composition) that meets the requirements of the respective industries, silica sand often needs to be additionally processed by methods such as gravity concentration [23,24], high-intensity magnetic separation [25–28], ultrasonic cleaning [29,30] or flotation. Although chemical processes (e.g., leaching), including the removal of impurities by means of various organic and mineral acids, are also available and highly efficient [4,6,31–35], the application of such beneficiation methods generally involves high equipment requirements, expensive chemicals and environmental hazards. The methods including biological treatment, such as bioleaching, have also been studied [1,36–38].

In the cases of mineral impurities of similar magnetic properties or specific gravity to quartz, e.g., feldspars, and where the desired results (silica product quality) cannot be achieved by simpler and lower-cost beneficiation methods, froth flotation may be the only solution for the removal of impurities in the form of free mineral particles [39–41]. In silica sand processing, froth flotation is primarily used to remove iron-bearing minerals and/or feldspars from the sand. This implies reverse flotation, usually under acidic conditions, using cationic amine collectors for feldspar removal and anionic petroleum sulphonate collectors for the removal of iron-bearing minerals [3,42,43], where a concentrate of im-

purities is removed as froth overflow from the flotation cell, leaving a quartz concentrate as the underflow product. Column flotation has gained great popularity in the mineral processing industry and is commonly applied as the cleaning stage of flotation circuits due to its capability for producing higher grade concentrates at good recoveries compared to the flotation in conventional mechanically agitated cells [44–48]. Even though flotation is a widely known and one of the most broadly used methods for separating valuable minerals from gangue since being introduced in the early 1900s, it is still not entirely understood [49], primarily because it is a complex process influenced by many variables, such as the pulp density and flow rate, air flow rate, pulp pH, particle-size distribution, mineralogical properties of the ore, flotation reagent type and dosage, bubble diameter and bubble size distribution, froth depth, wash water flow rate, etc. [47,49–54]. The effect of each relevant variable on the flotation process should be taken into consideration, and the combination of operating parameter values is optimised for an individual ore in terms of achievable concentrate grade and recovery. So far, only a few works dealing with the detailed analysis of the effect of relevant operating variables on the silica sand flotation performance have been published [40–43]. In his study [40], Hacifazlioglu investigates the flotation performance in removing iron-oxides from fine silica sand using the cyclojet cell developed as an alternative to the conventional cell. Mowla et al. [41] and Ibrahim et al. [39] studied the effects of operating parameters, including collector types and concentrations, type of acid, pH, solids concentration in the pulp, particle size, air flow rate, impeller speed, conditioning time and temperature on the separation efficiency of reverse flotation for the removal of iron bearing minerals from silica sand using a mechanical flotation cell. The influence of different types of flotation collectors on the grade of silica sand concentrate was investigated by Sekulić et al. and Bayat and Akarsu [42,43]. However, detailed studies on the effect of operating parameters on the performance of the flotation of silica sand in a flotation column are scarce or non-existent. In this regard, the aim of the study presented in this paper is to (1) investigate the effect of operating parameters, including collector type and dosage, pulp density (solids content in the pulp), pulp pH and air flow rate, on the performance (concentrate grade and recovery) of flotation conducted in a laboratory-scale column on silica sand samples with a significant feldspar content; (2) determine the model (non-linear regression equations) of the relationship between independent variables (operating parameters) and dependent variables (mass contents of SiO_2 , Al_2O_3 , Fe_2O_3 and TiO_2 in the concentrate and mass recovery); and (3) based on the determined model, define the optimal flotation conditions for the sand sample tested, i.e., the operating parameters value ranges within which the best results regarding grade and recovery are obtained.

2. Materials and Methods

2.1. Sample Characterisation

The raw silica sand samples used in the study were taken from the “Štefanac” deposit located in central Croatia. A bulk sample was dried, homogenised and reduced to representative smaller samples for the determination of particle-size distribution and chemical and mineral composition of the raw sand sample by standard analytical methods. The particle-size fraction of 0.63 mm to 0.1 mm, which is of interest to the glass, ceramic and foundry industries, was separated from the bulk sample by washing and classification. The results of the particle-size and chemical analyses of raw and washed sand are shown in Tables 2 and 3. As shown in Table 2, raw sand contains approximately 10.2% of particles larger than 0.63 mm, which were completely removed by sieving, and approximately 6.3% of particles smaller than 0.1 mm, mostly removed in the spiral classifier (0.8% of the class < 0.1 mm remained in washed sand). In this way, in addition to separating the required size class of sand (0.63/0.1 mm), a certain increase in the SiO_2 content and a decrease in the contents of unwanted components, i.e., Fe_2O_3 , Al_2O_3 , TiO_2 , etc., were obtained (Table 3)—explained in more detail in Section 3.1.

Table 2. Particle-size distribution of raw and washed sand.

Particle Size Class (mm)	Mass Content (%)	
	Raw Sand	Washed Sand
+0.63	10.16	-
0.63/0.5	8.12	10.3
0.5/0.355	14.98	17.1
0.355/0.25	18.30	20.8
0.25/0.18	16.15	19.9
0.18/0.1	25.98	31.1
0.1/0.063	4.08	0.8
−0.063	2.23	
Σ	100.00	100.00

Table 3. Chemical composition of raw and washed sand.

Component	Mass Content (%)	
	Raw Sand	Washed Sand (0.63/0.1 mm)
SiO ₂	88.65	90.24
Fe ₂ O ₃	0.82	0.50
Al ₂ O ₃	5.46	4.57
MgO	0.06	0.02
CaO	0.27	0.21
Na ₂ O	0.67	0.57
K ₂ O	2.62	2.57
TiO ₂	0.28	0.24
P ₂ O ₅	0.08	0.07
MnO	0.02	0.02
Cr ₂ O ₃	<0.002	<0.002
Elements *	560 ppm	500 ppm
Loss on ignition	0.5	0.6
Σ	99.48	99.66

* Ba, Ni, Sr, Zr, Y, Nb and Sc.

The following orientational mineral composition of the raw sand sample was determined by mineralogical analysis: 73 wt% quartz, 7 wt% plagioclase, 16 wt% K-feldspar and 4 wt% other minerals. Based on the microscopic analysis, it was estimated that the raw sample contains (excluding discrete feldspar and mica particles) 60% discrete (“pure”) quartz particles; 25% quartz particles containing inclusions of rutile, tourmaline or other heavy minerals; and 15% quartz particles coated with surface oxide coatings. Feldspar particles are mostly contained in the finer size classes, but they are also considerably present in coarser size classes of the sample (e.g., 36% in size class < 0.1 mm, 32% in 0.18/0.1 mm, 23% in 0.25/0.18 mm, 15% in 0.355/0.25 mm, 12% in 0.5/0.355 mm).

2.2. Experimental Procedure

The overall experimental procedure is shown schematically in Figure 1. Through the procedure of washing and classification on screens and a spiral classifier, grains larger than 0.63 mm and smaller than 0.1 mm were removed from the bulk of the raw sample, and in this way, the sand size 0.63/0.1 mm was separated for further testing. Before proceeding further, the separated class was dried in a dryer at 105–110 °C and the particle-size distribution and chemical composition of washed sand (0.63/0.1 mm) as feed material for flotation tests was determined. Reverse column flotation tests were carried out on washed sand samples in three series. Based on the results of the first (preliminary) series of tests, the appropriate flotation reagents were selected and limit values of the flotation parameters were defined. In the second series of tests, the influence of the collector type and dosage on flotation performance was investigated, and the collector and its concentration

with which the highest grade silica sand concentrate was achieved was determined and applied in the third phase of testing. The effect of solids content in the pulp, pulp pH and air flow rate on the column flotation performance was studied in the third test stage using statistical experimental design. The grade of silica sand concentrate (concentrate grade) and the mass recovery were the flotation performance indicators observed. The grade of silica sand concentrate (G) was determined as the mass content of valuable component (SiO_2) in the concentrate (1a). The mass contents of individual detrimental components (Fe_2O_3 , TiO_2 and Al_2O_3) in the concentrate were also determined as grade parameters according to Equation (1b).

$$G = \frac{m_v}{m_c} \cdot 100\% \quad (1a)$$

$$G_i = \frac{m_i}{m_c} \cdot 100\% \quad (1b)$$

where m_v is the mass of the valuable component (SiO_2), m_i is the mass of the individual detrimental component (where i is: Fe_2O_3 , Al_2O_3 or TiO_2), and m_c is the mass of the concentrate. Mass recovery (M) is calculated as a ratio of the m_c to the mass of the feed material (m_f) according to Equation (2).

$$M = \frac{m_c}{m_f} \cdot 100\% \quad (2)$$

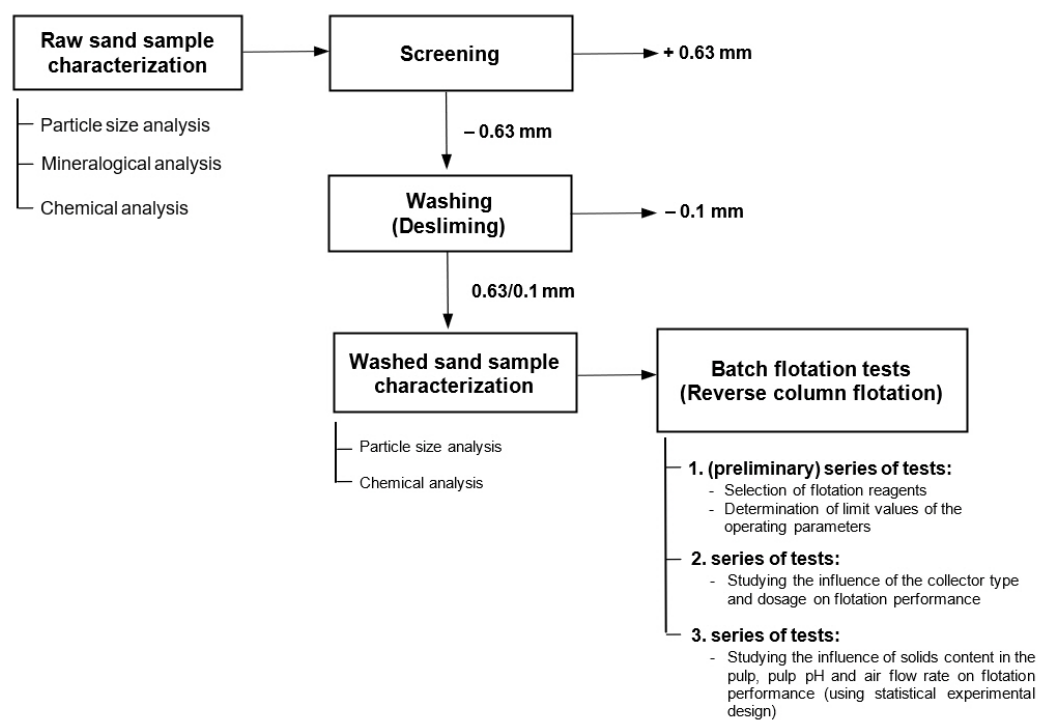


Figure 1. Simplified block diagram of the experimental procedure with the main test phases.

Flotation tests aimed at determining the effect of solids content in the pulp, pulp pH and air flow rate on concentrate grade and mass recovery were carried out according to the central composite design, and the test results were processed statistically by the analysis of variance (ANOVA) in the *Statistica* software system version 13.5.0 by TIBCO Software Inc. (Palo Alto, CA, USA). The analysis of variance determined which of the individual independent variables (solids content in the pulp, pulp pH and air flow rate) and which of the interactions of two out of three independent variables tested had a significant effect on dependent variables (mass contents of SiO_2 , Fe_2O_3 , Al_2O_3 and TiO_2 in the concentrate and mass recovery), and also, the magnitude of this effect was estimated. Using the multiple

regression method in the same program, the coefficients in non-linear Equation (3) of the relationship between dependent variables (response) and independent variables (factors) were determined. The general form of this equation is [55–57]

$$Y = b_0 + \sum_{i=1}^k b_i x_i + \sum_{i=1}^k \sum_{j=i+1}^k b_{ij} x_i x_j + \sum_{i=1}^k b_{ii} x_i^2 \quad (3)$$

and in the case of three independent variables, it has the following form (4):

$$Y = b_0 + b_1 x_1 + b_2 x_2 + b_3 x_3 + b_{11} x_1^2 + b_{22} x_2^2 + b_{33} x_3^2 + b_{12} x_1 x_2 + b_{13} x_1 x_3 + b_{23} x_2 x_3 \quad (4)$$

where

Y —dependent variable

x_1, x_2, x_3 —independent variables

b_0 —constant (*Intercept*)

b_1, b_2, b_3 —coefficients that describe the linear effect of independent variables x_1, x_2 and x_3 on dependent variable Y

b_{11}, b_{22}, b_{33} —coefficients that describe the non-linear (quadratic) effect of independent variables x_1, x_2 and x_3 on dependent variable Y

b_{12}, b_{13}, b_{23} —coefficients that describe the interactions of independent variables x_1, x_2 and x_3 with dependent variable Y

In the case of two independent variables, the regression equation represents a surface in three-dimensional space and has the following form (5):

$$Y = b_0 + b_1 x_1 + b_2 x_2 + b_{11} x_1^2 + b_{22} x_2^2 + b_{12} x_1 x_2 \quad (5)$$

Based on the model (non-linear regression equations) of the relationship between each observed dependent variable and the independent variables (Equations (6)–(10)) and the graphical interpretation of the analysis results, the ranges of operating parameter values within which the best results regarding grade and recovery are obtained for sand sample testing was determined.

2.3. Analytical Methods

A particle-size analysis of the sand samples was carried out by wet sieving with standard laboratory test sieves, followed by drying and weighing. The chemical compositions of all samples were determined by inductively coupled plasma emission spectrometry (ICP-ES), while the mineral composition of the raw sand sample was determined by X-ray diffraction (XRD) analysis. Structural characteristics, i.e., particle shape and structural relations between quartz grains and mineral impurities (impurities in the form of coatings or inclusions) were defined by microscopic analysis.

2.4. Batch Flotation Tests and Equipment

The batch flotation tests were conducted in a laboratory flotation column made of approx. 3.5 mm thick glass, with the main parts and dimensions shown in Figure 2. The pulp was prepared in a conditioning tank (4) with an agitator (3) whose rotating speed can be varied from 0 to 1500 rpm. After agitation, the pulp was transferred to a flotation column through the valve (6) and column feed port (5). The concentrate of impurities overflowed into a concentric launder (1), from which it was continuously drained (2) by gravity. A compressor (11) of a capacity of 190 L/min with a container of 24 L and pressure of up to 8 bar was used as an air source. The air flow rate was controlled by a valve (10) and measured by a flowmeter (9), enabling flow adjustment in the range from 0.05 to 0.6 L/min. Air was introduced into the column through a sparger (8) made of sintered glass with an opening of 10 μm . The regulated operating parameters and their limit values in flotation tests are shown in Table 4.

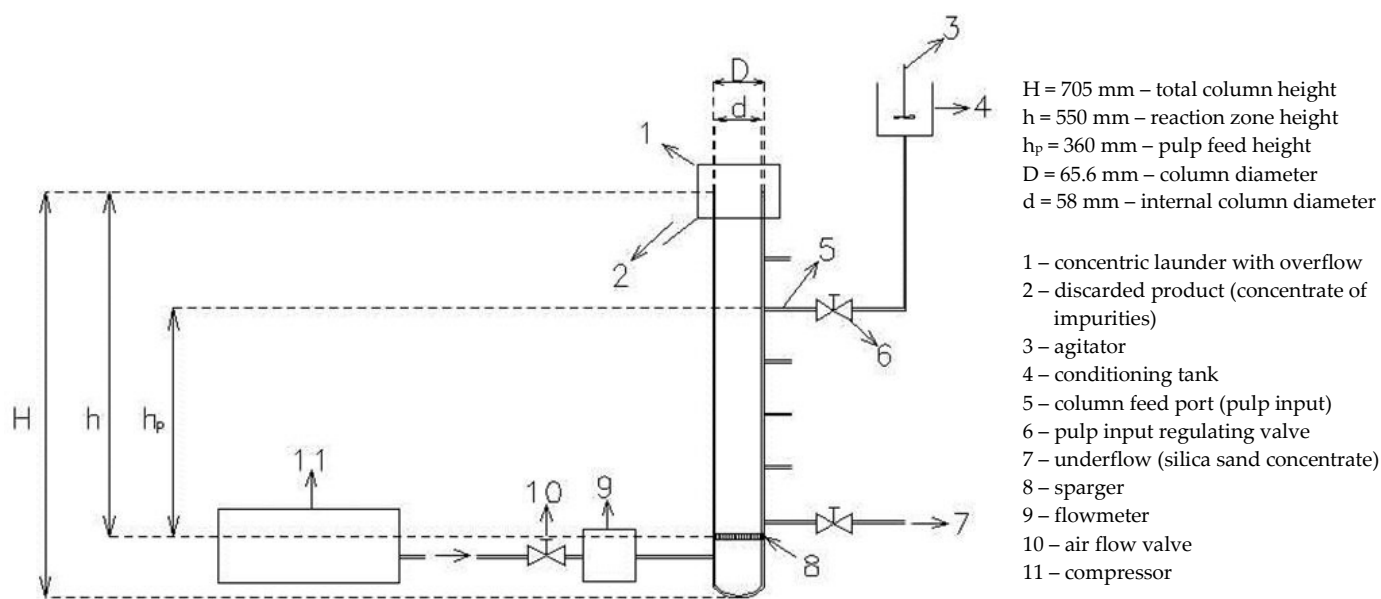


Figure 2. Laboratory flotation column scheme [58].

Table 4. Operating parameters and limiting conditions in column flotation tests.

Solids Content in the Pulp (wt%)	Reagent Type and Dosage (g _{reagent} /t _{sand})	Pulp pH	Air Flow Rate (L/h)	Conditioning/ Flotation Time (min)
Conditioning: 50–70%	Collector dosage: 200 do 900 g/t Anionic collectors *: Aero 869F, Aero 864, Aero 801, Custofloat CR1 Cationic collectors *: Aeromine 3030C, Custofloat CR3, Armoflote 14, 21, 64 Armoflote 820	2 do 4 (flotation in acidic pulp)	12–36 L/h	Conditioning: Total: 6–8 min (1–2 min with H ₂ SO ₄ +4 min with collector +2 min with frother)
Column flotation: 2–10%	Frother **: 200 do 500 g/t Aerofroth 65 Pulp pH regulator: H ₂ SO ₄	7 (flotation in neutral pulp)		Column flotation: 10 min

* Collector chemical composition: Aero 869—petroleum sulphonates, butanol, 2-ethylhexanol, triethylene glycol and monobutyl ether; Aero 864—petroleum sulphonates, 2-ethylhexanol; Aero 801: petroleum sulphonates, mineral oil, water; Custofloat CR3—mixture of fatty acids; Aeromine 3030C—amines, alkyl acetates, 2-ethylhexanol; Armoflote 14—alkyldiamine acetate; Armoflote 21, 64 and 820—alkylamine acetate. ** Frother chemical composition: Aerofroth 65—polyglycol mixture.

The silica sand samples for each individual column flotation test were prepared (conditioned) in the following way:

1. Mixing the appropriate amount of water and a sample of washed sand (0.63/0.1 mm) in order to obtain 50–70 wt% solids (sand) content in the pulp (depending on the collector type used);
2. Adding 2M H₂SO₄ to the pulp to achieve the desired pulp acidity (pH = 2–4) when using collectors (anionic: Aero 801, 864, 869F, Custofloat CR1, and cationic: Aeromine 3030C, Armoflote 14, 21, 64, 820) that are effective in an acidic pulp. Acid was not added in tests using collectors (anionic: Aero 704, Aero 845 and Custofloat CR3) that

are effective in a neutral pulp. When the desired pH is reached, the mixing of the pulp continues for 1–2 min;

3. Adding the required amount of collector (200–900 g/t of sand depending on the test and type of collector). Mixing continues for 4 min;
4. Adding the frother Aerofroth 65 (200–500 g/t of sand). Mixing continues for 2 min;
5. Diluting the pulp with water to a 2–10% solids concentration (20–110 g/L of water) and adding the required amount of H_2SO_4 to maintain the desired pulp acidity ($\text{pH} = 2\text{--}4$) during flotation tests under acidic conditions.

After the described preparation procedure, the compressed air valve was opened and the air flow was adjusted to values ranging from 12 to 36 L/h, depending on the test. The conditioned pulp was gradually introduced into the column. The total flotation time per test was 10 min, and during this time, the froth with separated mineral impurities (bearers of Fe, Al, Ti, etc.) was skimmed off the pulp surface and collected in containers in the form of overflow. After flotation was completed, the silica sand concentrate was discharged in the form of underflow at the bottom part of column. The flotation products (silica sand concentrates) were analysed for chemical composition by ICP-ES. The optimal collector concentrations, solids content in the pulp, pulp pH and air flow rate were determined based on the results of the second and third series of flotation tests.

For a given column configuration, depending on the test conditions (air flow rate, solids content in the pulp, etc.), the estimated gas holdup ranged from 3.6% to 7.9%, the mean bubble diameter from 0.39 mm to 0.72 mm and the corresponding superficial bubble surface rate from 20 s^{-1} to 31.7 s^{-1} .

3. Results and Discussion

3.1. Preliminary Test Results

By removing the size class $< 0.1 \text{ mm}$ through the washing process, a certain improvement in the quality of the silica sand sample was achieved (Table 3). In relation to raw sand, the mass content of SiO_2 in the washed sand was increased by approximately 1.8%, while the mass content of Al_2O_3 decreased by approximately 16%, and the mass content of Fe_2O_3 decreased by 39% (relative change in percentages). However, the obtained chemical composition does not comply with the requirements of the glass, ceramic or foundry industries for silica sand quality [4,14].

The results of the preliminary series of flotation tests with washed sand samples showed that the acceptable operation of the flotation column under conditions of performed tests was achieved with air flow rates ranging between 15 L/h and 30 L/h and with solids content in the pulp between 2 wt% and 6 wt%. It was found out that with the addition of a frother, more froth was produced, and slightly better results were achieved, but these differences became negligible with increasing frother concentrations above approximately 300 g/t. It was also established that under identical flotation conditions, the best results in terms of sand concentrate grade and mass recovery were achieved using the Aero 864, Aero 869F, Custofloat CR3 and Aeromine 3030C collectors. A significant improvement in the quality of the concentrate in comparison to the feed (washed) sand was achieved especially in the test with the cationic collector Aeromine 3030C. For example, at a 500 g/t dosage of that collector, an increase in the mass content of SiO_2 by approximately 5.3% was achieved, along with a decrease in the mass contents of Fe_2O_3 and Al_2O_3 by approximately 50% and a decrease in the mass content of TiO_2 by approximately 33% in comparison to washed sand (relative change in percentages). In the same test, the mass contents of Na_2O and K_2O were significantly reduced (by approximately 65% and 47%, respectively), which, along with the significantly lower mass recovery than in tests with other collectors (Table 5), indicates that during flotation tests with this collector, there was a significant separation of feldspar from the sand, which is expected considering the application of amine collectors [3,42,59].

Table 5. The effect of collector type and concentration on the concentrate grade and mass recovery.

Flotation Performance Indicators		Collector Type and Concentration (g/t)											
		Aero 869F			Aero 864			Aeromine 3030C			Custofloat CR3		
		300	500	700	300	500	700	300	500	700	300	500	700
Mass content in the concentrate (%)	SiO ₂	90.30	91.09	90.88	91.05	91.21	91.23	92.95	95.04	95.50	90.72	91.06	91.37
	Fe ₂ O ₃	0.40	0.23	0.20	0.47	0.43	0.31	0.37	0.26	0.23	0.32	0.22	0.21
	Al ₂ O ₃	4.79	4.33	4.61	4.58	4.37	4.46	3.39	2.31	2.25	4.69	4.29	4.05
	TiO ₂	0.12	0.02	0.02	0.02	0.02	0.02	0.16	0.16	0.15	0.03	0.02	0.02
	Na ₂ O	0.63	0.50	0.60	0.52	0.51	0.50	0.46	0.20	0.19	0.63	0.49	0.64
	K ₂ O	2.71	2.47	2.72	2.46	2.53	2.43	1.74	1.37	1.38	2.71	2.43	2.32
Mass recovery (yield) (%)		97.80	95.80	95.40	97.40	96.30	95.20	88.30	81.40	76.90	97.00	96.70	96.20

3.2. The Effect of Collector Type and Concentration

Table 5 and Figures 3 and 4 show the influence of the dosage and type of the mentioned collectors on the quality of the concentrate and mass recovery at unchanged values of other operating parameters (frother concentration: 250 g/t, mass content of sand in the pulp: 4%, pH: 2.5–3 or 7 if using the Custofloat CR3, air flow rate: 24 L/h, flotation time: 10 min). Even though, independently from the collector concentration, the highest-grade concentrate in terms of SiO₂, Al₂O₃, Na₂O and K₂O contents was achieved with the Aeromine 3030C collector, a slightly better result in terms of a decrease in the Fe₂O₃ content and a significantly better result in terms of a reduction in the TiO₂ content was achieved with the Aero 869F and Custofloat CR3 anionic collectors. In the flotation tests with the Aero 869F and Custofloat CR3 collectors, very similar results were obtained in terms of the mass content of Fe₂O₃, Al₂O₃ and TiO₂ in the concentrate (therefore, Figure 4 does not show the flotation results for the Aero 869F collector). The concentrate grade increases and the mass recovery reduces by increasing the collector dosage; however, the concentrate grade change is slight with an increase in collector dosage from 500 g/t to 700 g/t, regardless of the applied collector type. By increasing the dosage of the Aeromine 3030C collector from 300 g/t to 500 g/t, the concentrate grade is significantly increased (the mass content of SiO₂ in the concentrate increased by 2.3%, and the mass contents of detrimental components decreased as follows: Fe₂O₃ by ca. 30%, Al₂O₃ by ca. 32%, Na₂O by ca. 57% and K₂O by ca. 21%), along with an 8% decrease in the mass recovery. In the case of Aero 864 and Custofloat CR3 collectors, by increasing their dose from 300 g/t to 500 g/t, the mass contents of the unwanted components in the concentrate remained practically unchanged. As can be seen in Table 5, regardless of the applied type of collector, in the tests conducted with collector dosages of 300 g/t and 500 g/t, a relatively high mass recovery was achieved (81.4%–97.8%; calculated according to Equation (2)), which was expected considering the small concentrations of undesirable components that should be separated from the silica sand. The significantly lower mass recoveries in flotation tests carried out with the Aeromine 3030C collector are understandable, regarding the feldspar content in the tested sand sample determined by mineralogical analysis and the chemical composition of the concentrate obtained by flotation with this collector (a significant reduction in the mass content of Al₂O₃, Na₂O and K₂O).

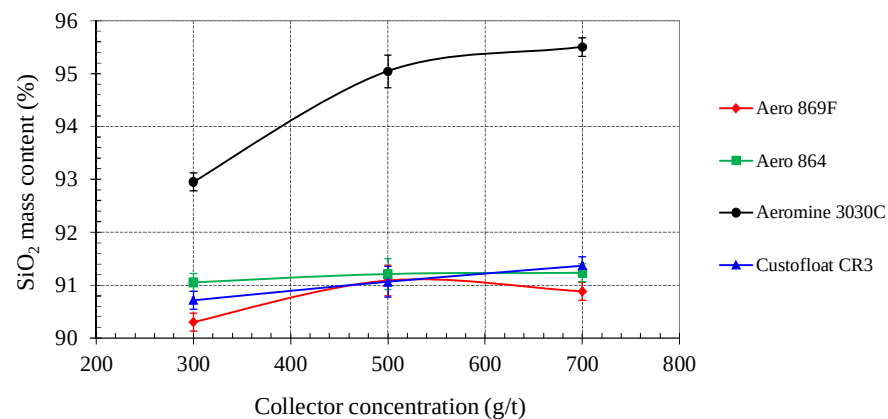


Figure 3. SiO₂ content in the concentrate as a function of collector type and concentration.

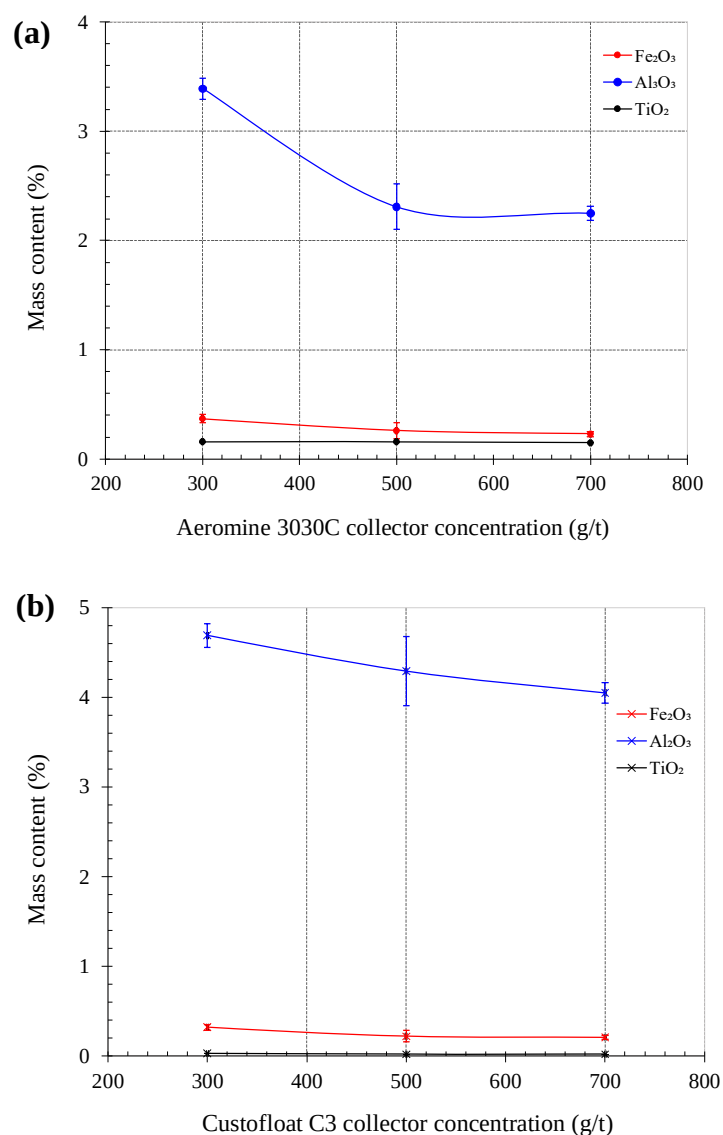


Figure 4. *Cont.*

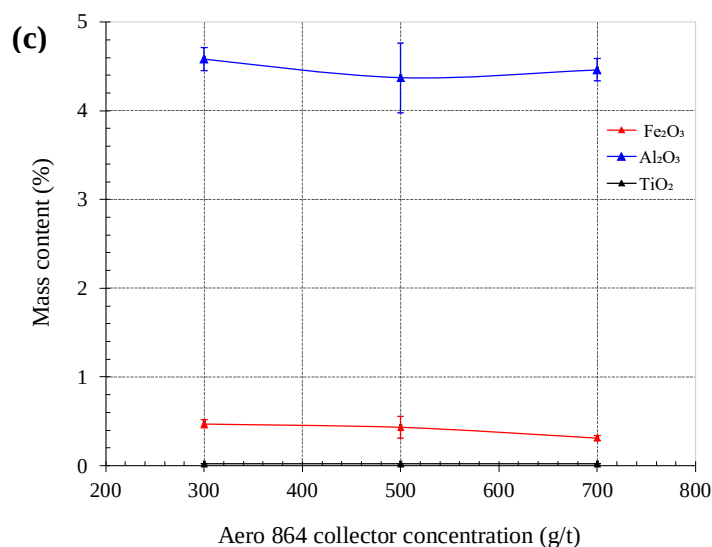


Figure 4. The content of impurities in the concentrate as a function of collector type and concentration; collectors: Aeromine 3030C (a), Custofloat CR3 (b) and Aero 864 (c).

3.3. The Effect of Solids Content in the Pulp, pH and Air Flow Rate

With regard to the previously presented results, in the phase of studying the influence of solids content in the pulp, air flow rate and pulp pH on the column flotation performance using statistical experimental design, the Aeromine 3030C collector was used at a concentration of 500 g/t and a flotation time of 10 min. Table 6 shows the results of the conducted tests, i.e., the achieved mass contents of the main components in the concentrate and mass recovery (dependent variables) for each combination of values of solids content in the pulp, pulp pH and air flow rate in accordance with the central composite design. The test results were processed statistically by the analysis of variance for each of the observed dependent variables. The results of variance analysis for the mass content of SiO₂ in the concentrate are shown in Table 7, and the analysis results were obtained in the same way for the other dependent variables (mass contents of Fe₂O₃, Al₂O₃ and TiO₂, as well as mass recovery). The effect significance is determined on the basis of the *p*-value (*p*-level or level of significance) in the fifth column of the table. The ANOVA effect estimates are shown in second column of tables (labelled *Effect*). The second to last column shows the value of the constant (*Intercept*, first row in the table) and the values of the coefficients in the regression equation (Equation (4)) for the coded values of independent variables and their interactions. The last column of the table shows the value of the constant (*Intercept*) and the values of the coefficients in the regression equation for the actual (uncoded) values of independent variables and their interactions. *Intercept* represents the constant b_0 in regression Equation (4), i.e., the mean value of the dependent variable when all of the independent variables in the equation are equal to zero.

Based on the results obtained by the analysis of variance, i.e., the *p*-value and the *Effect*, the following can be concluded:

- All three operating parameters have a significant impact on the concentrate grade, with the pH of the pulp significantly affecting all four grade parameters (mass contents of SiO₂, Al₂O₃, Fe₂O₃ and TiO₂), air flow rate significantly affecting the mass contents of SiO₂ and Al₂O₃, and the solids content in the pulp only affecting the mass content of Fe₂O₃;
- Air flow rate has the greatest effect on the mass contents of SiO₂ and Al₂O₃ in the concentrate with a high level of significance ($p = 0.000067$ and $p = 0.001922$, respectively), and pulp pH has a slightly lower effect but also with a high significance. With a probability of error of only 5.9%, the quadratic effect of solids content in the pulp on the SiO₂ content in the concentrate can be taken into account as well. Given that the estimates of the effect of pulp pH on the SiO₂ content in the concentrate and the effect of air flow rate on

the Al_2O_3 content are negative (a negative *Effect* value), it follows that an increase in the pulp pH reduces the SiO_2 content, and an increase in the air flow rate decreases the Al_2O_3 content in the concentrate. Both solids content in the pulp and pulp pH have a significant (linear) effect on the mass content of Fe_2O_3 , of which the effect of solids content in the pulp is greater, while only pulp pH has a significant (both linear and quadratic) effect on the mass content of TiO_2 . The quadratic effect of pulp pH on the SiO_2 and Al_2O_3 contents in the concentrate is also significant. The linear effect of solids content in the pulp on the contents of SiO_2 , Al_2O_3 and TiO_2 and the linear effect of air flow rate on the contents of Fe_2O_3 and TiO_2 are not significant.

- Mass recovery is significantly affected by pulp pH, with a high level of significance ($p = 0.002917$), as well as by air flow rate ($p = 0.010285$). A negative value of the effect estimates of these two independent variables means that their increase results in a decrease in mass recovery. Solids content in the pulp does not have a significant impact on mass recovery.
- The interactions between operating variables do not have a significant impact neither on any of the observed grade parameters nor mass recovery. By comparing the p -values, it can be noted that the interaction between solids content in the pulp and air flow rate has the greatest impact on the mass contents of SiO_2 and Al_2O_3 in the concentrate, and the interaction of pulp pH and air flow rate has the greatest impact on the Fe_2O_3 content, while the mass content of TiO_2 and mass recovery are virtually unaffected by any of the interactions.

Table 6. Plan and results of laboratory flotation tests (collector: Aeromine 3030C, 500 g/t; flotation time: 10 min).

Test No.	Operating Parameter (Independent Variable)			Mass Content in the Concentrate (%)				Mass Recovery (Yield) (%)
	Solids Content in the Pulp (%)	Pulp pH	Air Flow Rate (L/h)	SiO_2	Fe_2O_3	Al_2O_3	TiO_2	
1	3.0	2.5	20.0	93.74	0.23	2.37	0.17	83.5
2	5.0	2.5	20.0	93.54	0.37	2.55	0.20	84.2
3	3.0	3.5	20.0	92.65	0.28	3.31	0.28	79.1
4	5.0	3.5	20.0	91.82	0.36	3.89	0.27	78.9
5	3.0	2.5	28.0	95.78	0.23	1.25	0.20	77.8
6	5.0	2.5	28.0	95.90	0.29	1.11	0.18	77.0
7	3.0	3.5	28.0	93.89	0.28	2.68	0.29	68.0
8	5.0	3.5	28.0	94.15	0.39	2.44	0.29	72.5
9	4.0	3.0	24.0	95.04	0.26	2.31	0.16	81.4
10	4.0	3.0	24.0	95.60	0.31	1.86	0.18	78.2
11	2.3	3.0	24.0	94.63	0.28	2.44	0.27	76.4
12	5.7	3.0	24.0	94.14	0.35	2.21	0.20	80.9
13	4.0	2.2	24.0	94.09	0.24	2.93	0.24	88.6
14	4.0	3.8	24.0	92.22	0.41	3.63	0.33	59.9
15	4.0	3.0	17.3	93.40	0.28	2.82	0.29	82.6
16	4.0	3.0	30.7	95.79	0.30	1.53	0.16	67.2
17	4.0	3.0	24.0	94.63	0.24	2.32	0.22	76.4

Substituting appropriate coefficient values (the last column in the tables for actual values), obtained by analysis for each of the observed dependent variables, into Equation (4) gives the regression Equations (6)–(10), i.e., models of non-linear relationship between the respective grade parameters or mass recovery (Y) and solids content in the pulp ($Ir-192\rho$), pulp pH (k) and air flow rate (p):

- (a) Equation of the dependence of the mass content of SiO_2 in the concentrate on the operating parameters ($Ir-192\rho$, k , p)

$$Y = 57.65 + 1.28Ir-192\rho + 17k + 0.76p - 0.26Ir-192\rho^2 - 2.78k^2 - 0.012p^2 - 0.123Ir-192\rho \cdot k + 0.044 Ir-192\rho \cdot p - 0.052k \cdot p \quad (6)$$

(b) Equation of the dependence of the mass content of Fe_2O_3 in the concentrate on the operating parameters

$$Y = 1.275 - 0.03Ir-192\rho - 0.506k - 0.03p + 0.014Ir-192\rho^2 + 0.07k^2 + 0.003p^2 - 0.0025Ir-192\rho \cdot k - 0.0016Ir-192\rho \cdot p + 0.0069k \cdot p \quad (7)$$

(c) Equation of the dependence of the mass content of Al_2O_3 in the concentrate on the operating parameters

$$Y = 14.04 + 0.47Ir-192\rho - 8.68k + 0.028p + 0.02Ir-192\rho^2 + 1.43k^2 - 0.0021p^2 + 0.075Ir-192\rho \cdot k - 0.036Ir-192\rho \cdot p + 0.03k \cdot p \quad (8)$$

(d) Equation of the dependence of the mass content of TiO_2 in the concentrate on the operating parameters

$$Y = 1.657 - 0.07Ir-192\rho - 0.676k - 0.031p + 0.013Ir-192\rho^2 + 0.124k^2 + 0.0006p^2 - 0.005Ir-192\rho \cdot k - 0.0013 Ir-192\rho \cdot p + 0.0013k \cdot p \quad (9)$$

(e) Equation of the dependence of mass recovery on the operating parameters

$$Y = 65.72 - 8.09Ir-192\rho + 19.5k + 2.19p + 0.41Ir-192\rho^2 - 4.6k^2 - 0.058p^2 + 1.1Ir-192\rho \cdot k + 0.1 Ir-192\rho \cdot p - 0.29k \cdot p \quad (10)$$

Table 7. Effect estimates and second-order regression coefficients for SiO_2 mass content in the concentrate as a function of solids content in the pulp, pulp pH and air flow rate.

Independent Variable (Factor)	Effect Estimates; Dependent Variable: % SiO_2 Central Composite Design (3 Factors, 1 Block, 17 Tests)					
	Effect ¹	Std. Err. ²	t(7) ³	p ⁴	Coeff. ⁵	Coeff. ⁶
Mean / Intercept ⁷	95.09308	0.222897	426.6227	0.000000	95.09308	57.64587
(3) Flow rate (L)	1.75582	0.209349	8.3870	0.000067	0.87791	0.75575
(2) pH (L)	−1.40515	0.209349	−6.7120	0.000274	−0.70257	17.00388
pH (Q)	−1.38950	0.230419	−6.0303	0.000526	−0.69475	−2.77900
Solids content (Q)	−0.51976	0.230419	−2.2557	0.058701	−0.25988	−0.25988
Flow rate (Q)	−0.37127	0.230419	−1.6113	0.151155	−0.18563	−0.01160
1L by 3L	0.35250	0.273528	1.2887	0.238450	0.17625	0.04406
(1) Solids content (L)	−0.21587	0.209349	−1.0312	0.336768	−0.10794	1.28111
2L by 3L	−0.20750	0.273528	−0.7586	0.472852	−0.10375	−0.05187
1L by 2L	−0.12250	0.273528	−0.4479	0.667792	−0.06125	−0.12250

¹ Estimates of the effect of independent variables and their interactions listed in the first column of the table; ² Standard error of effect estimate; ³ Test value t where the number (7) indicates the so-called degree of freedom, which is a parameter of the T-distribution that affects the values of the dependent variable (the t-test is performed to test the basic hypothesis); ⁴ Significance level of the performed test (p-level); ⁵ Coefficients in the regression equation (Equation (3)) for coded values of independent variables and their interactions; ⁶ Coefficients in the regression equation for the actual values of the independent variables and their interactions; ⁷ The constant (intercept) in the regression equation of the second degree (constant b_0 in Equation (3)) for the actual values in the last and for the coded values of the independent variables in the penultimate column of the table; (L)—linear effect of independent variable; (Q)—quadratic effect of independent variable; 1L by 2L, 1L by 3L, 2L by 3L—linear effects of the interactions of independent variables.

Figures 5–10 show the graphical interpretation of analysis results, i.e., contour plots (response profiles) of dependent variables defined by Equation (5) for SiO_2 mass content in the concentrate and mass recovery. On the coordinate axes of each of the independent variables, actual values are marked. Figures 5–7 show the dependence of SiO_2 mass content in the concentrate on every combination of two of the three tested independent variables (operating parameters). In the same way, the dependence of the other grade parameters (mass contents of Fe_2O_3 , Al_2O_3 and TiO_2 in the concentrate) on independent variables is shown graphically (figures available as Supplementary Material). These graphical representations also show areas of the independent variable values that result in the highest

grade of concentrate, i.e., the highest mass content of SiO_2 and the lowest mass contents of Al_2O_3 , Fe_2O_3 and TiO_2 in the concentrate.

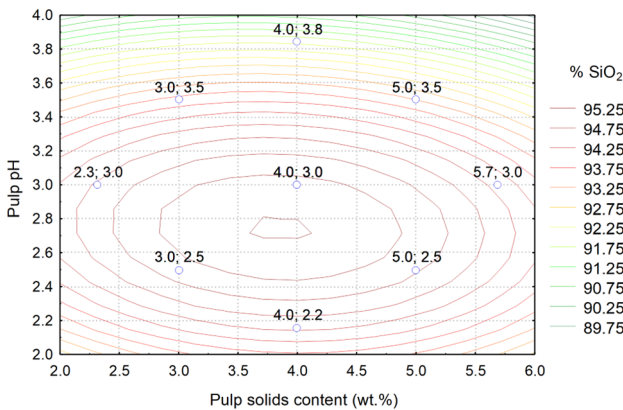


Figure 5. Contour plot of SiO_2 mass content in the concentrate as a function of pulp solids content and pH.

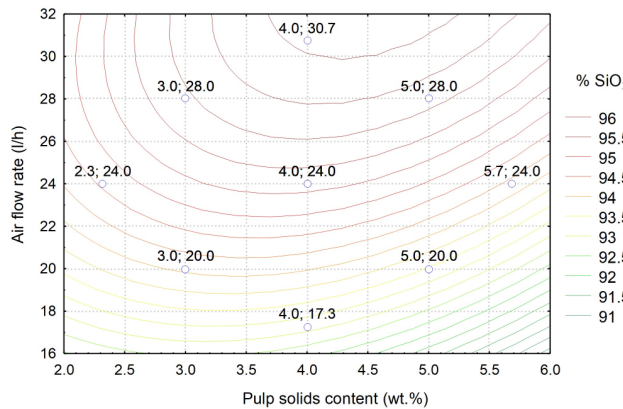


Figure 6. Contour plot of SiO_2 mass content in the concentrate as a function of pulp solids content and air flow rate.

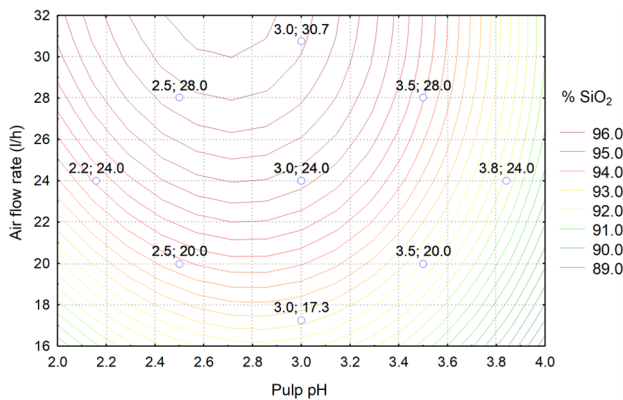


Figure 7. Contour plot of SiO_2 mass content in the concentrate as a function of pulp pH and air flow rate.

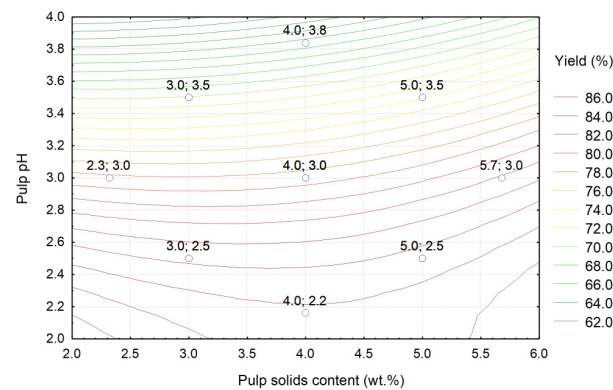


Figure 8. Contour plot of mass recovery (yield) as a function of pulp solids content and pH.

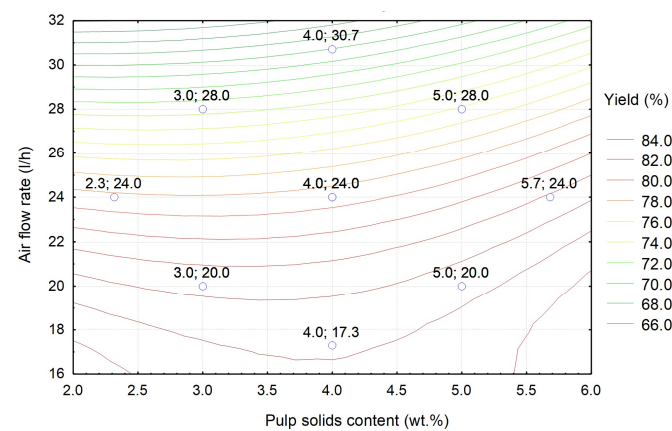


Figure 9. Contour plot of mass recovery (yield) as a function of pulp solids content and air flow rate.

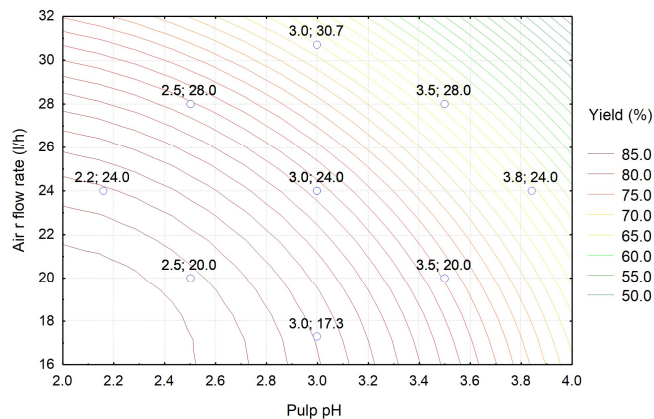


Figure 10. Contour plot of mass recovery (yield) as a function of pulp pH and air flow rate.

As can be seen in Figures 5–7, and according to the regression Equation (6), the highest mass content of SiO_2 in the concentrate is achieved at a pulp pH in the range from 2.4 to 3.0 and an air flow rate greater than 26 L/h. At these values of pulp pH and air flow rate, it is possible to obtain a 95%–96% mass content of SiO_2 in the concentrate. By increasing the pH above 3.2 and reducing the air flow rate below 22 L/h, the mass content of SiO_2 decreases significantly (e.g., at pH = 3.6 and an air flow rate of 20 L/h, a content of approximately 92 wt% SiO_2 can be expected). Changes in solids content in the pulp, as already established by the analysis of variance, do not significantly affect the mass content of SiO_2 (Figures 5 and 6) nor the mass contents of Al_2O_3 and TiO_2 .

Based on the regression Equations (7)–(9) and the graphical interpretation of the analysis results (available as Supplementary Material), it can also be concluded that by column flotation using 500 g/t Aeromine 3030C collector:

- The lowest mass content of Al_2O_3 in the concentrate is also achieved at a pulp pH in the range of 2.4 to 3.0 and an air flow rate greater than 26 L/h. At these values of pulp pH and air flow rate, it is possible to obtain a 0.9%–1.8% mass content of Al_2O_3 in the concentrate. By increasing the pH above 3.2 and reducing the air flow rate below 22 L/h, the mass content of Al_2O_3 increases (e.g., at pH = 3.6 and an air flow rate of 20 L/h, a content of approximately 3.9 wt% Al_2O_3 can be expected);
- The lowest mass content of Fe_2O_3 in the concentrate is obtained at a pulp pH in the range from 2.2 to 2.8 and the solids content in the pulp from 2.0% to 3.5%, while the lowest mass content of TiO_2 is obtained at a pulp pH in the range from 2.4% to 3.0% and the solids content in the pulp from 3.5% to 5.5% (changes in the air flow rate have no significant impact on either of those two dependent variables). At these values, the mass content of Fe_2O_3 is approximately 0.25% and of TiO_2 is approximately 0.18%. However, by increasing or decreasing the operating parameters beyond the mentioned limits, the mass contents of Fe_2O_3 and TiO_2 do not change considerably, i.e., any change in the combination of values of the observed independent variables within the observed limits results in changes in the mass content of Fe_2O_3 and TiO_2 in the concentrate within relatively narrow limits (approximately from 0.2% to 0.5% Fe_2O_3 and from 0.1% to 0.4% TiO_2 under the performed test conditions) and they are substantially less significant compared to changes in the mass contents of SiO_2 and Al_2O_3 .

Figures 8–10 show the contour plots of mass recovery as a function of every combination of two of the three tested operating parameters and areas of operating parameter values where the highest mass recovery is achieved. As can be seen from these figures, and according to the regression Equation (10), the highest mass recovery (>84%) is obtained at a pulp pH in the range from ca. 2.0 to 2.5 and air flow rates from ca. 16 L/h to 20 L/h. Mass recovery decreases with an increase in pulp pH and air flow rate. This change is more pronounced with an increase in pH approximately above 3 and air flow rate above 24 L/h. The changes in solids content in the pulp also do not significantly affect the mass recovery.

4. Conclusions

The research presented in this article determined the effect of the collector type and dosage, solids content in the pulp, pulp pH and air flow rate on the concentrate grade and mass recovery in laboratory-scale column flotation conducted on silica sand samples, as well as the value ranges of flotation operating parameters within which the highest concentrate grade and mass recovery are obtained.

Regardless of the collector dosage, the highest-grade concentrate in terms of the mass contents of SiO_2 , Al_2O_3 , Na_2O and K_2O was obtained using the cationic collector Aeromine 3030C. A slightly better result in terms of a decrease in the mass content of Fe_2O_3 and a significantly better result in terms of TiO_2 content reduction were achieved with the Aero 869F and Custofloat CR3 anionic collectors. Regardless of the applied collector type, increasing the collector dosage increases the concentrate grade and reduces the mass recovery; however, the change in concentrate grade is inconsiderable with an increase in collector dosage from 500 g/t to 700 g/t.

The results of the analysis of variance for each of the dependent variables in the flotation tests conducted with the Aeromine 3030C collector showed that all three tested independent variables (operating parameters) have a significant impact on the concentrate grade. Pulp pH significantly affects all four grade parameters, i.e., the mass contents of SiO_2 , Al_2O_3 , Fe_2O_3 and TiO_2 in the concentrate, air flow rate significantly affects the mass contents of SiO_2 and Al_2O_3 , and the solids content in the pulp only affects the mass content of Fe_2O_3 . Air flow rate has the greatest effect on the mass contents of SiO_2 and Al_2O_3 in the concentrate with a high level of significance, while the pulp pH has a slightly smaller

effect on these contents but also with a high level of significance. Solids content in the pulp and pulp pH have a significant linear effect on the mass content of Fe_2O_3 in the concentrate, while pulp pH is the only observed parameter that has both a significant linear and quadratic effect on the mass content of TiO_2 . Mass recovery is significantly affected by pulp pH with a high level of significance and by air flow rate. The interactions between operating parameters did not have a significant effect on any of the observed grade parameters nor on the mass recovery.

Using multiple regression, the coefficients in the regression equations, i.e., a model of the relationship between the respective performance indicators (SiO_2 , Fe_2O_3 , Al_2O_3 and TiO_2 contents in the concentrate, mass recovery) and the operating parameters (solids content in the pulp, pulp pH and air flow rate) was determined. On the basis of that model and its graphical interpretation (regression surfaces of dependent variables and their contour plots), it can be concluded that by column flotation of the tested silica sand with the use of the Aeromine 3030C collector (at a dosage of 500 g/t): (1) the highest concentrate grade in terms of mass contents of SiO_2 , Al_2O_3 and TiO_2 is obtained at a pulp pH in the range from 2.4 to 3.0 and an air flow rate greater than 26 L/h, while the effect of solids content in the pulp is minor (increasing pH above 3.2 and decreasing air flow rate below 22 L/h, the mass content of SiO_2 decreases considerably and the mass content of Al_2O_3 increases); (2) the lowest mass content of Fe_2O_3 in the concentrate is obtained at a pulp pH in the range from 2.2 to 2.8 and a solids content in the pulp in the range from 2.0% to 3.5%, while air flow rate has no significant effect on Fe_2O_3 content; (3) any change in the combination of values of the observed independent variables within the observed limits results in changes in the mass content of Fe_2O_3 and TiO_2 in the concentrate within relatively narrow limits, substantially narrower in comparison to changes in the mass contents of SiO_2 and Al_2O_3 ; (4) mass recovery decreases with an increase in pulp pH and air flow rate, and this change is more pronounced with an increase in pH above ca. 3 and air flow rate above 24 L/h, while changes in solids content in the pulp do not significantly affect mass recovery. The highest mass recovery (>84%) is achieved at a pulp pH in the range from ca. 2.0 to 2.5 and air flow rates in the range from ca. 16 L/h to 20 L/h. However, satisfactory mass recovery (approximately 80%) is also achievable at air flow rates greater than 26 L/h, at which the highest concentrate grade is obtained, if the pulp pH is less than approximately 3.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/min14040341/s1>, Figure S1. Contour plot of Fe_2O_3 mass content in the concentrate as a function of pulp solids content and pH; Figure S2. Contour plot of Fe_2O_3 mass content in the concentrate as a function of pulp solids content and air flow rate; Figure S3. Contour plot of Fe_2O_3 mass content in the concentrate as a function of pulp pH and air flow rate; Figure S4. Contour plot of Al_2O_3 mass content in the concentrate as a function of pulp solids content and pulp pH; Figure S5. Contour plot of Al_2O_3 mass content in the concentrate as a function of pulp solids content and air flow rate; Figure S6. Contour plot of Al_2O_3 mass content in the concentrate as a function of pulp pH and air flow rate; Figure S7. Contour plot of TiO_2 mass content in the concentrate as a function of pulp solids content and pH; Figure S8. Contour plot of TiO_2 mass content in the concentrate as a function of pulp solids content and air flow rate; Figure S9. Contour plot of TiO_2 mass content in the concentrate as a function of pulp pH and air flow rate.

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Data Availability Statement: The original data presented in the study are contained within the article. Further inquiries can be directed to the corresponding author.

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