

Continuous reaction crystallization of struvite from solution containing phosphate(V) and nitrate(V) ions

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Abstract: Research results concerning continuous removal of phosphates(V) using magnesium and ammonium ions from solutions containing 1.0 or 0.20 mass % of PO_4^{3-} ions and 0.0443 – 0.886 mass % of nitrate(V) ions are presented. Continuous struvite reaction crystallization process ran in stoichiometric conditions and at 20% excess of magnesium ions in DT MSMPR (*Draft Tube, Mixed Suspension Mixed Product Removal*) crystallizer with internal circulation of suspension in 298 K at pH 9 – 11 and mean residence time τ 900 – 3600 s. With the increase in nitrate(V) ions concentration in a feed solution mean crystal size L_m decreased by ca. 29%. Size-homogeneity within the crystal product population was not high. Increase in pH influenced product quality disadvantageously. Crystals of smaller sizes and lower homogeneity were manufactured. Elongation of mean residence time to 3600 s caused increase in crystals size even by 80% and improved size-homogeneity. Decrease of phosphate(V) ions concentration in a feed solution caused increase in mean size of struvite (by ca. 23%). Excess of magnesium ions influenced process results advantageously. Mean size of crystal products increased by more than 24%. Presence of nitrate(V) ions and process conditions in a crystallizer favored crystallization of struvite in a form of tubular crystals.

Key words: Struvite, phosphate(V) ions, nitrate(V) ions, continuous reaction crystallization, product quality, phosphorus recycling.

Introduction

Chemical removal of phosphate(V) ions from diluted aqueous solutions (e.g. from industrial, municipal wastewaters, or from liquid manure) is usually based on chemical binding of these ions in a form of sparingly soluble magnesium and ammonium salt, $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$, struvite, MAP (solubility product $\text{p}K_{\text{sp}} = 9.0 - 13.26$ (Doyle, 2002)). The reagents used in its precipitation process can be crystalline magnesium and ammonium compounds, their aqueous solutions, as well as wastewaters containing the required reagents in sufficiently high concentrations (Parsons, 2001). Providing optimal conditions for the controlled, continuous process of precipitation integrated with mass crystallization of sparingly soluble phosphate(V) salt(s) is a complex technological problem (Le Corre, 2009). Final result of this process – crystal population of determined chemical purity and size composition, produced with satisfactory yield – is dependent on many parameters precisely defining the environment in which these processes run, on these parameter values, as well as on the assumed constructional solutions (Doyle, 2002).

Struvite precipitates in ambient temperature from supersaturated water solutions of magnesium phosphate(V) in presence of ammonium ions within pH 7 – 11 range when molar ratio between the reagents $[\text{PO}_4^{3-}]_{\text{RM}} : [\text{Mg}^{2+}]_{\text{RM}} : [\text{NH}_4^+]_{\text{RM}} = 1 : 1 : 1$ is provided (Doyle, 2002). Both process course and its results are strongly influenced by pH, supersaturation, temperature, ionic strength of solution, presence of foreign substances, arrangement of crystallizer interior, mixing method and its intensity, circulation type, mean residence time of suspension in a crystallizer, spatial arrangement of reagent inlet places defining their contact method, etc. (Le Corre, 2007; Koralewska, 2009; Franke, 1995; Penicot, 1998). One of the fundamental control process parameters is pH of struvite reaction crystallization environment. The pH strongly influences struvite solubility and induction time (Bouropoulos, 2000). With the pH increase solubility of struvite decreases (its minimal value corresponds to pH 10.3 (Ohlinger, 1998) or 10.7

(Snoeyink, 1980), while its precipitation potential increases (Doyle, 2002). Also induction time indispensable for nucleation process initiation shortens (Abbona, 1982). Other factor also strongly influencing the process course, thus the removed product quality, is presence of various impurities in phosphate(V) ions solutions. These are mainly metal ions, sulphates(VI), nitrates(V), fluorides and fluosilicates. These inhibit or catalyze the precipitation reaction course, influence nucleation and crystal phase growth kinetics, modify crystal shape and eventual promote some agglomeration effects (Le Corre, 2005; Hutnik, 2011a; Hutnik, 2011b). Some of metal ions in alkaline environment of struvite continuous reaction crystallization process can also form sparingly soluble hydroxides or phosphates, what essentially deteriorates chemical composition of the product and limits the possibilities of its further applications (de-Bashan, 2004).

One of the impurities frequently identified in industrial wastewaters are nitrate(V) ions. The experimental research results focusing on the influence of nitrate(V) ions concentration on the results of struvite $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ reaction crystallization process in a continuous laboratory DT MSMMPR (*Draft Tube, Mixed Suspension Mixed Product Removal*) type crystallizer with propeller are presented. Process yield, defined as a final concentration of PO_4^{3-} ions in mother solution, was calculated. Product crystals size distributions and their statistical parameters (L_m , L_{50} , L_d , CV) were identified. Crystal shapes (L_a/L_b) and their agglomeration were also determined.

Materials and Methods

Scheme of laboratory stand for the tests of struvite continuous reaction crystallization process in presence of nitrate(V) ions is presented in Figure 1. Steering, control and acquisition of measurement data were done with the use of computer. For precise control of measuring system working in a steady state conditions the IKA labworldsoft program was used. Working volume of the crystallizer was 0.6 dm^3 . The crystallizer used was a glass cylindrical tank (D 120 mm, H 123 mm), in a bottom part equipped with heating/cooling jacket (thermostated with external water circulation system), providing stabilization of the process temperature. In a central axis of apparatus cylindrical circulation tube (*Draft Tube*, DT, d_{dt} 57 mm, h_{dt} 53 mm) was placed, inside which three-paddle propeller (d_m 55 mm) operated. Inlet ports of reagents and alkalinizing solution, as well as product crystal suspension removal port are marked in Figure 1. The crystallizer was continuously fed with solution prepared previously in a mixer from chemically pure substances (crystalline magnesium chloride hexahydrate $\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$, ammonium dihydrogenphosphate(V) $\text{NH}_4\text{H}_2\text{PO}_4$ and sodium nitrate(V) NaNO_3 – POCh Gliwice, Poland) and deionized water (Barnstead – NANOpure DIamond). This solution was introduced into the circulation profile interior (stirrer speed: 6.6 ± 0.1 1/s; suspension movement – downward). Between the crystallizer body and circulation pipe (suspension movement – upward) aqueous solution of sodium hydroxide of concentration 20 mass % of NaOH was dosed in proportion providing the assumed, controlled pH value of struvite continuous reaction crystallization environment.

Temperature in a crystallizer was 298 ± 0.2 K. The research tests were done in stoichiometric conditions (molar proportions of reagent ions in a feed $[\text{PO}_4^{3-}]_{\text{RM}} : [\text{Mg}^{2+}]_{\text{RM}} : [\text{NH}_4^+]_{\text{RM}} = 1 : 1 : 1$) and at 20% excess of magnesium ions ($[\text{PO}_4^{3-}]_{\text{RM}} : [\text{Mg}^{2+}]_{\text{RM}} : [\text{NH}_4^+]_{\text{RM}} = 1 : 1.2 : 1$). Concentration of phosphate(V) ions in a crystallizer feed was 1.0 or 0.20 mass %, magnesium ions: 0.256 or 0.0512 mass % – in stoichiometric conditions and 0.307 or 0.061 mass % – at their 20% excess, and ammonium ions: 0.190 or 0.0380 mass %, respectively. Concentration of nitrate(V) ions varied from 0.0443 to 0.886 mass % depending on studied case. Continuous reaction crystallization of struvite ran at pH 9 (± 0.1) and for mean residence time of suspension in a crystallizer τ 900 s (± 20 s). For nitrate(V) ions concentration in a feed $[\text{NO}_3^-]_{\text{RM}}$ 0.0886 mass % also tests for pH 10 and 11, as well as for τ 1800 and 3600 s were also done.

After stabilisation of the assumed process parameter values in a crystallizer, continuous process in a steady state ran through the next 5τ . After this time whole crystallizer content was transferred onto vacuum filter. After mother solution drainage, the crystals were not washed. Crystal phase was weighed before and after drying. Also weight and volume of mother solution were determined. This way concentration of solid phase in product crystals suspension was calculated (M_T). Crystal size distribution was determined with the use of solid particle analyzer COULTER LS-230, whereas crystal shape – from individual planimetric measurements based on crystal population images from scanning electron microscope JEOL JSM 5800LV. Chemical composition of mother solution and crystal phase were identified using, among others, plasma emission spectrometer ICP-AES CPU 7000, spectrometer IR PU9712 and spectrophotometer UV-VIS Evolution 300. Accuracy of the measurement data concerning continuous

struvite reaction crystallization process in a laboratory plant was estimated to be ca. 10%.

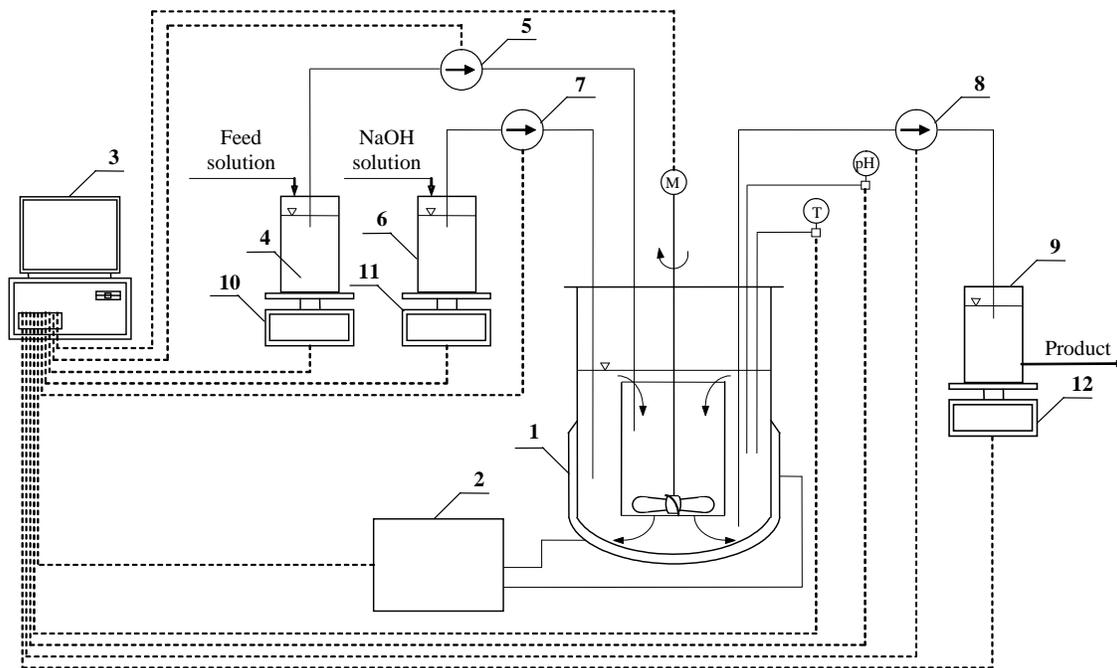


Figure 1: Laboratory test stand scheme of continuous crystallizer with propeller mixer for reaction crystallization of struvite, 1 – DT type crystallizer with internal circulation of suspension, 2 – thermostat, 3 – computer, 4 – reservoir of feed mixture: aqueous solution of $\text{NH}_4\text{H}_2\text{PO}_4$, MgCl_2 and NaNO_3 , 5 – feed proportioner (pump), 6 – alkalinity agent tank: aqueous solution of NaOH , 7 – proportioner of NaOH solution, 8 – receiver (pump) of a product crystal suspension from the crystallizer tank, 9 – storage tank of product crystal suspension, 10, 11, 12 – electronic balances, M – stirrer speed control/adjustment, pH – alkaline/acid reaction control/adjustment, T – temperature control/adjustment.

Results and Discussion

The research tests and analyses results are presented in Table 1. From these data it results, that presence of nitrate(V) ions influences both size of product struvite crystals and their size-homogeneity disadvantageously (tests No. 1 – 5 in Table 1). With the increase in NO_3^- ions concentration in a feed from 0.0443 to 0.886 mass % mean crystal size L_m decreased from 31.0 to 21.9 μm (thus by ca. 29%), similar to median crystal size L_{50} 24.0 \rightarrow 16.4 μm (Hutnik, 2009). Size homogeneity of product crystal population was not satisfactory. Coefficient of crystal size homogeneity CV varied within the 84.0 – 99.2% range (average 91.4%).

Decrease of mean size L_m of product crystals with the increase in nitrate(V) ions concentration in a process system is connected with general increase in the number of the smallest struvite crystals in the whole product population. In result dominant crystal size shifts towards smaller values: L_d 30.6 \rightarrow 19.5 μm for $[\text{NO}_3^-]_{\text{RM}}$ 0.0443 \rightarrow 0.886 mass %, while number and size of the largest crystals in a product decrease. The largest size of struvite crystals produced in the presence of 0.0443 mass % of nitrate(V) ions is 170 μm , while in the presence of 0.886 mass % NO_3^- – only ca. 140 μm . Simultaneously the smallest size fraction enlarges. For $[\text{NO}_3^-]_{\text{RM}}$ 0.0443 mass % crystal fraction of sizes below 5 μm in a product was 10.2%, while in case of $[\text{NO}_3^-]_{\text{RM}}$ 0.886 mass % this fraction increased up to 19.2%, thus by ca. 90%. This trend is clearly visible in Figure 2, presenting exemplary volumetric (mass) product crystal size distributions (solid particle laser analyzer COULTER LS-230) self-established in the presence of 0.0886

and 0.443 mass % of nitrate(V) ions in a feed (pH 9, τ 900 s).

Table 1: Experimental test results concerning continuous struvite reaction crystallization process in a DT MSMRP crystallizer. Reaction crystallization process temperature: 298 K.

No.	Process parameters				Suspension in crystallizer		Crystal product characteristic ^{*)}				
	$[\text{PO}_4^{3-}]_{\text{RM}}$ mass %	$[\text{NO}_3^-]_{\text{RM}}$ mass %	pH	τ s	M_T kg crystals/m ³	$[\text{PO}_4^{3-}]_{\text{ML}}$ mg/kg	L_m μm	L_{50} μm	L_d μm	CV %	L_a/L_b -
Molar proportions of reagent ions in a feed: $[\text{PO}_4^{3-}]_{\text{RM}} : [\text{Mg}^{2+}]_{\text{RM}} : [\text{NH}_4^+]_{\text{RM}} = 1 : 1 : 1$											
1	1.0	0.0443	9	900	24.3	163	31.0	24.0	30.6	88.8	6.6
2	1.0	0.0886	9	900	24.2	147	29.4	20.3	27.6	88.5	6.6
3	1.0	0.221	9	900	24.0	148	28.7	19.2	23.0	96.6	6.6
4	1.0	0.443	9	900	24.0	150	27.9	18.0	23.8	99.2	7.6
5	1.0	0.886	9	900	24.1	145	21.9	16.4	19.5	84.0	7.8
6	1.0	0.0886	10	900	24.3	130	24.6	17.1	23.2	94.4	7.8
7	1.0	0.0886	11	900	24.7	122	17.8	12.4	10.5	96.8	8.2
8	1.0	0.0886	9	1800	24.6	141	32.9	26.3	29.3	83.1	6.8
9	1.0	0.0886	9	3600	24.9	134	52.7	40.7	54.4	86.1	7.2
10	0.20	0.0886	9	900	4.7	147	36.2	25.0	31.1	95.9	6.8
11	0.20	0.443	9	900	4.7	152	36.0	23.4	30.8	93.4	7.6
Molar proportions of reagent ions in a feed: $[\text{PO}_4^{3-}]_{\text{RM}} : [\text{Mg}^{2+}]_{\text{RM}} : [\text{NH}_4^+]_{\text{RM}} = 1 : 1.2 : 1$											
12	1.0	0.0886	9	900	24.6	23	36.6	26.9	31.6	95.6	6.7
13	1.0	0.443	9	900	24.7	22	35.0	26.0	31.1	95.2	7.8
14	0.20	0.0886	9	900	4.8	27	41.1	30.0	36.2	92.6	6.8
15	0.20	0.443	9	900	5.0	32	39.0	28.4	35.6	93.4	7.8

^{*)} Without product crystal washing

$L_m = \sum x_i L_i$, where: x_i – mass fraction of crystals of mean fraction size L_i ; L_{50} – median crystal size for 50 mass % undersize fraction; L_d – crystal mode size; $CV = 100(L_{84} - L_{16})/(2L_{50})$, where: L_{84} , L_{16} , L_{50} – crystal sizes corresponding to 84, 16 and 50 mass % undersize fractions.

In Figure 3 there are presented scanning electron microscope images of product crystal samples, which size distributions are shown in Figure 2. Differences in size and shape within the struvite crystals (L_m 29.4 and 27.9 μm , appropriately) are clearly visible. From these exemplary product images one can also conclude about lower than moderate agglomeration effects within large struvite crystals.

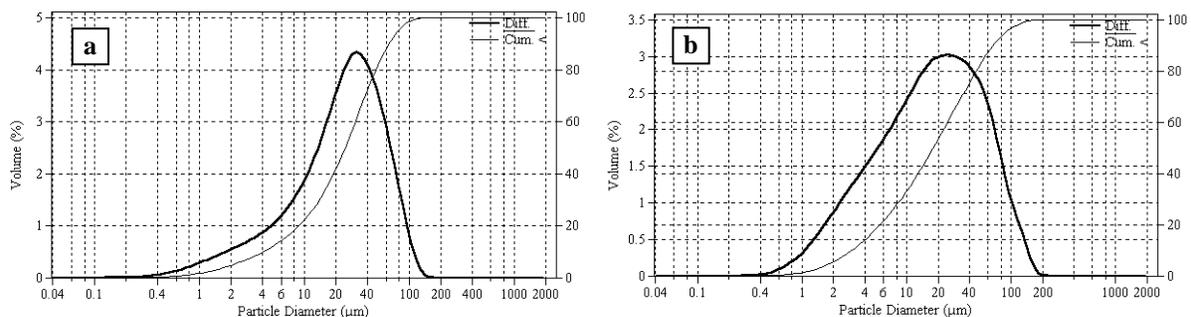


Figure 2: Exemplary differential (left scale) and cumulative (right scale) volumetric (mass) size distributions of

crystals produced in continuous struvite reaction crystallization process in presence of 0.0886 mass % (a) and 0.443 mass % (b) nitrate(V) ions in a feed (pH 9, τ 900 s, corresponding to No. 2 and 4 in Table 1).

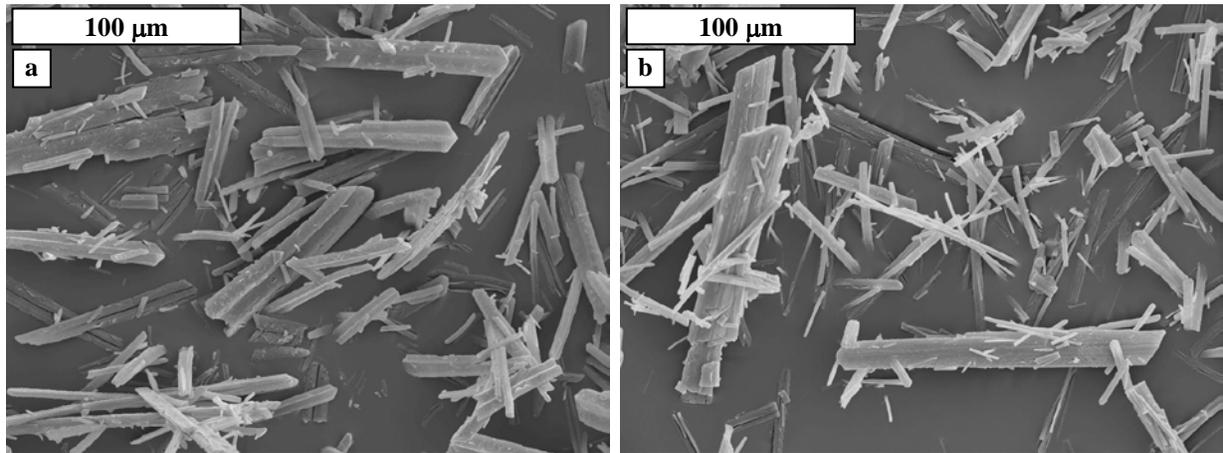


Figure 3: Scanning electron microscope images of struvite crystals produced in a continuous DT MSMPR crystallizer in presence of 0.0886 mass % (a) and 0.443 mass % (b) nitrate(V) ions in a feed (corresponding to Figure 2 cases). Magnification: 500 \times .

With the increase in pH of struvite continuous reaction crystallization process environment product crystal size decreased, as well (tests No. 2, 6 and 7 in Table 1). Raise of pH from 9 to 11 (τ 900 s) caused, that mean crystal size L_m decreased by ca. 40%: from 29.4 to 17.8 μm at concentration 0.0886 mass % nitrate(V) ions in a feed. Increase in crystallizer environment's pH did not favour homogeneity of the manufactured product. The CV coefficient increased up to 96.8%. Dominant crystal size L_d shifted towards smaller values: from 27.6 μm (pH 9) to 10.5 μm (pH 11). In a crystalline product both number and size of the largest struvite crystals decreased. Their largest size at pH 9 was ca. 160 μm (Figure 2a), while at pH 11 – only 80 μm (Figure 4a). Simultaneously fraction of the smallest size particles enlarged. At pH 9 crystal fraction of sizes below 5 μm in a product was 12.1%, while at pH 11 it raised up to 18.9%. In result crystal mean size L_m decreased significantly from 29.4 to 17.8 μm . In Figure 5a there is presented scanning electron microscope image of product crystals, which size distribution is shown in Figure 4a. Smaller sizes of struvite crystals produced at pH 11 are clearly observable (compare Figures 3a and 5a).

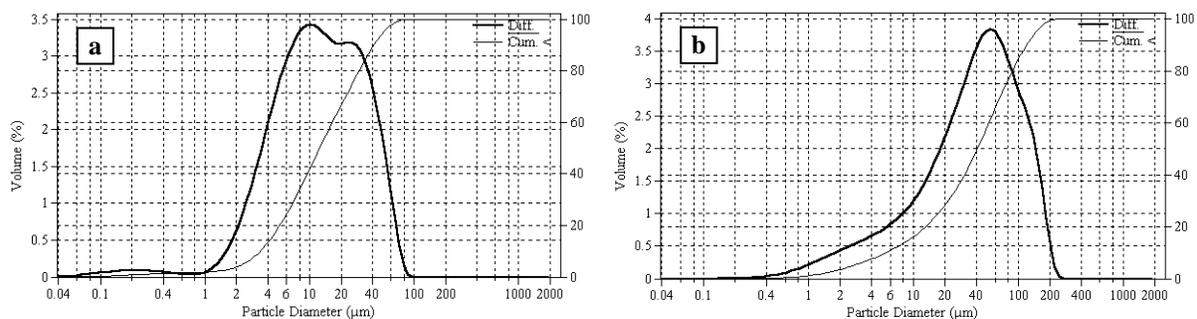


Figure 4: Exemplary differential (left scale) and cumulative (right scale) volumetric (mass) size distributions of crystals produced in a continuous reaction crystallization process in presence of 0.0886 mass % of nitrate(V) ions in a feed: a) pH 11, τ 900 s and b) pH 9, τ 3600 s (corresponding to No. 7 and 9 in Table 1).

Elongation of mean residence time of suspension in a crystallizer up to 3600 s caused, however, increase in product crystal size even by ca. 80% (tests No. 2, 8 and 9 in Table 1). In presence of 0.0886 mass % nitrate(V) ions struvite crystals reached mean size L_m 52.7 μm (pH 9, τ 3600 s). Elongation of mean residence time produces decrease

of solution supersaturation, thus decrease of process kinetic component values: nucleation rate of struvite crystals, as well as their linear growth rate.

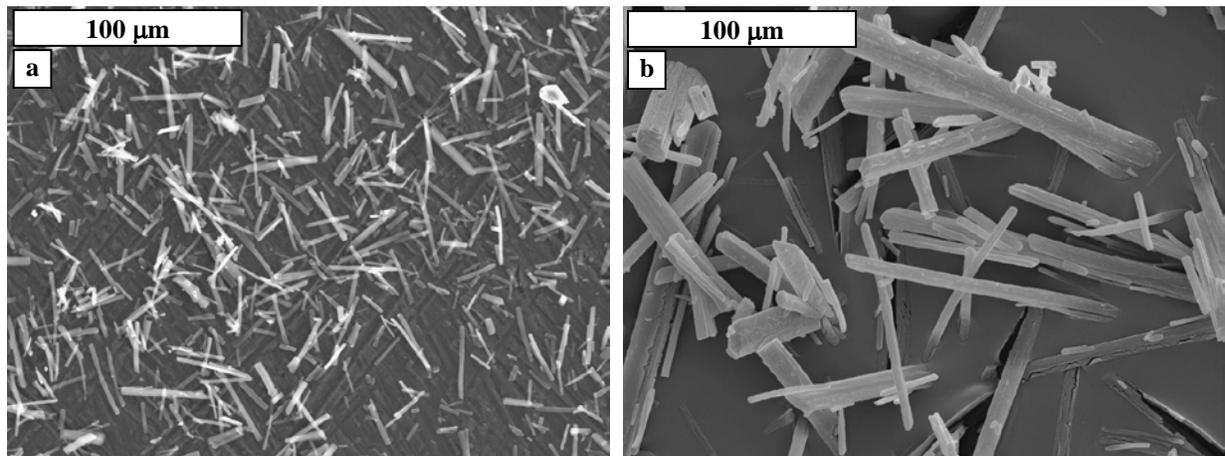


Figure 5: Scanning electron microscope images of struvite crystals produced in a continuous DT MSMPR crystallizer in the presence of 0.0886 mass % of nitrate(V) ions in a feed. Process parameters: a) pH 11, τ 900 s, b) pH 9, τ 3600 s (corresponding to Figure 4 cases). Magnification: 500 \times .

Longer residence time of crystals in the supersaturated mother solution made, however, that final effect of the process was advantageous. Crystal phase grew slower, however more stable and longer, thus reached larger final sizes. Their population homogeneity (CV 86.1%) improved. Elongation of mean residence time up to 3600 s made that dominant size L_d of struvite crystals shifted towards higher values: from 27.6 μm (τ 900 s) to 54.4 μm (τ 3600 s). Size of the largest crystals in a whole product crystal population reached 160 μm (τ 900 s, Figure 2a) and 250 μm (τ 3600 s, Figure 4b). Fraction of the smallest-size crystal decreased visibly. Crystal population of size $L < 5 \mu\text{m}$ in a product represented 12.1 and 9.2%, respectively. Electron microscope image of crystals produced at τ 3600 s is presented in Figure 5b. The number of small size crystals also decreased. In a microscope's „field of vision” one can notice original in shape tubular crystals. Struvite crystals are damaged. One can notice slightly deformed edges and surface defects.

Simultaneous influence of nitrate(V) ions concentration in a crystallizer feed ($0.0443 \leq [\text{NO}_3^-]_{\text{RM}} \leq 0.886$ mass %), pH of process environment ($9 \leq \text{pH} \leq 11$) and mean residence time of suspension in a working volume of the crystallizer ($900 \leq \tau \leq 3600$ s) on mean product crystals size L_m (in μm) can be presented in a form of empirical correlation, Eq. (1):

$$L_m = 5.374 \cdot 10^2 [\text{NO}_3^-]_{\text{RM}}^{-0.098} \text{pH}^{-2.665} \tau^{0.400} \quad (1)$$

with correlation coefficient R^2 0.941 and mean relative error $\pm 8.6\%$. Correlation (1) is based on 11 sets of experimental data (Table 1).

The 5-time decrease of phosphate(V) ions concentration in a feed (from 1.0 to 0.20 mass %) produces increase in struvite mean crystal size L_m by 23% (tests No. 2, 4, 10 and 11 in Table 1). Local supersaturation at the crystallizer inlet decreased. Thus struvite nucleation rate, very strongly dependent on supersaturation, decreased as well. It caused decrease of nuclei number, producing thus larger mean size of the crystals removed from the crystallizer.

Excess of magnesium ions in relation to phosphate(V) and ammonium ions content in a crystallizer feed (molar proportions $[\text{PO}_4^{3-}]_{\text{RM}} : [\text{Mg}^{2+}]_{\text{RM}} : [\text{NH}_4^+]_{\text{RM}} = 1 : 1.2 : 1$) influenced the process results advantageously (tests No. 12 – 15 in Table 1). Mean struvite crystal size L_m enlarged from 36.2 to 41.1 μm (by 13%) and from 36.0 to 39.0 μm (by 8%) for $[\text{PO}_4^{3-}]_{\text{RM}}$ 0.20 mass % in presence of nitrate(V) ions of concentration 0.0886 and 0.443 mass %, respectively. Higher supersaturation in respect to magnesium ions in a discussed process environment generally influenced all partial phenomena creating complex reaction crystallization process advantageously. Net effect (among others higher nucleation rate, however also higher crystal growth rate) caused more favourable product crystal size distribution.

From the analysis of scanning electron microscope images of crystal products (Figures 3 and 5) it results, that

struvite crystals habit was not identical. Based on planimetric measurement results covering 50 crystals randomly selected from three scanning electron microscope images (from each product crystals population) it was concluded, that mean ratio of their length L_a to their width L_b varied from 6.6 to 8.4 depending on the process parameter values (Table 1). Increase in nitrate(V) ions concentration in a feed caused that geometrical simplex L_a/L_b varied from 6.6 to 7.8. The crystals became shorter and thinner (Figure 3). Increase in process environment's pH resulted that crystal length L_a and width L_b decreased, however width L_b decreased more: $L_a/L_b = 6.6, 7.8$ and 8.2 – appropriately for pH 9, 10 and 11 (τ 900 s and $[\text{NO}_3^-]_{\text{RM}}$ 0.0886 mass %, Figures 3a and 5a). With the elongation of mean residence time of suspension in a crystallizer both crystal length L_a and their width L_b enlarged, however not in a proportional way. The L_a/L_b simplex value enlarged from 6.6 to 7.2 (Figures 3a and 5b). Lower concentration of phosphate(V) ions in a feed and magnesium ions excess in a process system did not introduce essential changes in mean values of L_a/L_b .

In Table 1 there are also presented analytically determined concentrations of phosphate(V) ions in a postprocessed mother solution. It may be practically assumed, that this concentration stabilised on a relatively constant and low level 144 ± 20 mg $\text{PO}_4^{3-}/\text{kg}$ in stoichiometric conditions and 26 ± 5 mg $\text{PO}_4^{3-}/\text{kg}$ in the presence of magnesium ions excess. Average efficiency of phosphate(V) ions removal from a feed solution ranged thus from 92 to 99% depending on process technological parameters. Presence of nitrate(V) ions in a continuous struvite reaction crystallization system does not influence essentially the residual level of phosphate(V) ions concentration in a postprocessed mother solution. These concentration values, however, decreased systematically with the pH increase and with elongation of mean residence time of struvite crystals suspension in a crystallizer. It can be explained by decrease of struvite solubility with the increase in reactive mixture pH, while longer contact time of crystal phase with supersaturated solution in a crystallizer provides more thorough discharge of the generated supersaturation. Working supersaturation in a crystallizer, thus in a postprocessed mother solution can not be, however, calculated with satisfactory high precision. In majority of published works solubility product of struvite pK_{sp} 13.26 (Ohlinger, 1998) is assumed, where corresponding equilibrium concentration of phosphate(V) ions is 3.6 mg/kg. In reality this value is unknown, since it depends on many process, technological and even constructional factors, and differs significantly from the value provided by Ohlinger et al. (1998). Excess of magnesium ions with reference to phosphate(V) and ammonium ions concentrations influenced thus the process yield advantageously. Concentration of phosphate(V) ions in a postprocessed mother solution was distinctly smaller (from 5- to 7-time) than in stoichiometric conditions technological case.

Conclusions

The research results concerning continuous removal of phosphate(V) ions from the diluted aqueous solutions ($[\text{PO}_4^{3-}]_{\text{RM}}$ 0.20 and 1.0 mass %) with the use of magnesium and ammonium ions in the presence of nitrate(V) ions are shown. The research was carried out in a DT MSMPR type crystallizer with internal circulation of suspension driven by propeller mixer both in stoichiometric conditions and at 20% excess of magnesium ions with relation to phosphate(V) and ammonium ions contents. It was concluded, that with the increase in feed concentration of nitrate(V) ions from 0.0443 to 0.886 mass %, mean size of crystal product L_m decreased by ca. 29% (from 31.0 to 21.9 μm at pH 9 and τ 900 s, $[\text{PO}_4^{3-}]_{\text{RM}}$ 1.0 mass %). Homogeneity within the product crystals population was not high (average CV ca. 91%). Increase in pH of continuous struvite reaction crystallization process environment influenced crystalline product quality disadvantageously. Crystals of smaller sizes (L_m 17.8 μm at pH 11, τ 900 s and feed concentration of nitrate(V) ions 0.0886 mass %) and of lower homogeneity (CV ca. 97%) were produced. Contrary, elongation of mean residence time of suspension in a crystallizer (up to 3600 s, pH 9), responsible however for decrease of working supersaturation in mother solution, caused enlargement of product crystals size even by 80% (L_m ca. 53 μm). Homogeneity within crystal population improved (CV ca. 86%). In process conditions characterized by relatively long mean residence time of suspension in a crystallizer higher quality product is formed. However, under these process conditions unit process yield is small, thus lower economical efficiency of a whole production plant is reported.

Based on product crystal images (scanning electron microscope) it was concluded, that crystal agglomeration effects were less than moderate. Properly shaped struvite crystals were produced. Presence of nitrate(V) ions in the reaction crystallization system and process conditions established in a crystallizer favoured struvite crystallization in a characteristic form of tubular crystals.

Decrease of phosphate(V) ions concentration in a feed (to 0.20 mass % of PO_4^{3-}) caused increase in mean size of struvite crystals (by ca. 23%). Similarly, excess of magnesium ions in relation to phosphate(V) and ammonium ions content in this solution influenced the process results advantageously. Mean size of product crystals enlarged by more than 24%.

Excess of magnesium ions in a process system definitely influenced the continuous struvite reaction crystallization process yield advantageously. Concentration of phosphate(V) ions in a postprocessed mother solution decreased to 22 – 32 mg/kg depending on the feed composition, what can be regarded as a very good result of their removal from solution.

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