

## **Influence of selected process parameters on attrition intensity in DTM type crystallizers with a jet-pump – a general neural network's approach**

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### **Abstract**

Secondary contact nucleation is a fundamental source of nuclei in large-scale industrial mass crystallization processes. Overcoming this phenomenon provides one with a possibility for a more precise process control. Facing complex interactions between constructional (DTM MSMR / MSCPR crystallizer with liquid jet-pump) and hydrodynamic factors a feedforward multilayer artificial neural network was used for creating a numerical model of this process. It enables one to predict with significant accuracy attrition intensity within NaCl crystal suspension in various technological conditions.

Keywords: DTM crystallizer, attrition degree, liquid jet-pump, neural network model.

### **1. Introduction**

For properly designed industrial-scale mass crystallization process main (and controllable) source of nuclei is a complex secondary nucleation, based mainly on attrition, breakage and abrasion phenomena (Mullin, 1993). In the most common crystallizer constructions with internal circulation of suspension this desirable (if not exceeded) effect results from the mechanical collisions between: crystal-crystal, crystal-agitator (or pump rotor) and crystal-apparatus wall (or/and other interior equipment) (Mersmann, 1995). However, in case of too intensive attrition action an excessive number of nuclei arise, causing difficulties in process control and making creation of the product of desirable crystal size distribution impossible (Gahn *et al.*, 1996; Mersmann *et al.*, 1998). Facing complex interrelations between hydrodynamic, constructional and technological factors the possibly exact prediction of attrition action in the assumed process environment is an important engineering challenge. Application of original constructions of crystallizer with a liquid jet-pump (Matynia, 1997; see Fig. 1) creates new possibilities of providing stable and intensive enough internal circulation of suspension inside the vessel simultaneously reducing the undesirable excessive

attrition effects. The construction makes practical use of injection effect while flow of working fluid with relatively high linear velocity for creation of internal circulation of processed medium. It should be noted, that only pure hydrodynamic interactions (injection, mixing) between working liquid and suspension are responsible for the transfer of kinetic energy into circulated magma, thus no mechanical parts – eventually contributing the overall secondary nucleation process – are necessary.

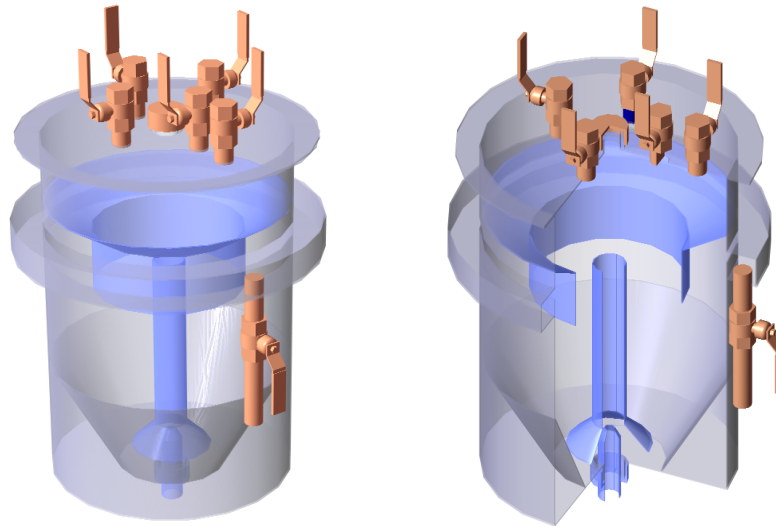


Fig. 1. Original construction of a liquid jet-pump DTM MSMPR crystallizer with internal (upward) circulation of suspension. Three main elements of a jet-pump hydraulic system are visible: feeding nozzle (situated at the crystallizer bottom), confusor and mixing chamber.

## 2.Experiments

Experimental tests of the attrition resistances in NaCl crystal populations (hardness via Vickers method  $16 - 24 \text{ MN m}^{-2}$ ) of initial mean size within the range of  $L_m = 0.441 - 1.52 \text{ mm}$  were done (Matynia *et al.*, 2005). Volumetric concentration of crystals in the suspension was adjusted in the  $\phi = 2 - 10\%$  range, while mean residence time of suspension in the crystallizer was changed within the  $\tau = 900 - 7200 \text{ s}$  range. Two different constructions of laboratory-scale crystallizer with a liquid jet-pump were taken under consideration: DTM MSMPR (*Draft Tube Magma Mixed Suspension Mixed Product Removal* crystallizer) (Matynia *et al.*, 1978; 1981; 1998) and a more complex design with internal hydraulic classification system – DTM MSCPR (*DTM Mixed Suspension Classified Product Removal* crystallizer) (Bechtold *et al.*, 1980; Matynia *et al.*, 1984). Both laboratory crystallizers, loaded with crystal suspension of possibly narrow, closely restricted crystal size distribution (saturated solution,  $\rho = 1198 \text{ kg m}^{-3}$ ,  $T = 298 \text{ K}$ ) were working under steady state hydraulic mode through the selected residence time. Resulting, final crystal size distributions were determined with Laser Particle Size Analyzer COULTER LS-230. Each measurement was repeated twice (repeatability test). For detailed analysis of surface structure electron microscope images were also done (Scanning Electron Microscope JEOL JSM 5800 LV).

### 3. Neural network calculations

Taking under consideration complex entirety of analyzed phenomenon (e.g. influence of hydrodynamics in macro- and microscale, etc.) a feedforward multilayer artificial neural network was used for creation a numerical model of this process based on the system information included indirectly in experimental data set (60 input–output vectors) (Tambe *et al.*, 1996).

Table 1. Artificial neural network configurations tested.

No	Configuration	Optimal number of iterations	Attrition degree, AD		$L_m$ after the process		CV after the process		
			MSMPR	MSCPR	MSMPR	MSCPR	MSMPR	MSCPR	
			RMSD						
1	3 4 6	7500	0.9158	0.5513	0.0188	0.0187	0.9681	0.9578	
2	3 6 6	8600	0.8810	0.5724	0.0145	0.0157	0.9847	0.9164	
3	3 8 6	8300	0.8905	0.5543	0.0156	0.0139	0.9959	0.9178	
4	3 10 6	8000	0.9425	0.5390	0.0198	0.0179	0.9783	0.9108	
5	3 12 6	7800	0.9349	0.5660	0.0191	0.0148	0.9533	0.9061	
6	3 14 6	8000	0.8948	0.5520	0.0172	0.0165	0.9413	0.9071	
7	3 16 6	7500	0.8811	0.5745	0.0209	0.0165	0.9758	0.9239	
8	3 18 6	8000	0.8696	0.6108	0.0229	0.0199	1.0375	0.9067	
9	3 20 6	7500	0.8783	0.5881	0.0191	0.0166	1.0341	0.9237	
10	3 22 6	7500	0.9204	0.5534	0.0235	0.0185	0.9833	0.8876	
11	3 4 2 6	7000	1.3876	0.6639	0.0279	0.0272	1.0683	0.9896	
12	3 4 4 6	6800	1.3289	0.6508	0.0296	0.0309	0.9799	0.9463	
13	3 4 6 6	7000	0.9571	0.5567	0.0184	0.0184	0.9929	0.9372	
14	3 4 8 6	6500	0.9981	0.5921	0.0233	0.0189	0.9903	0.9055	
15	3 4 10 6	6500	1.0605	0.5649	0.0241	0.0228	0.9347	0.8839	
16	3 4 12 6	7000	0.9833	0.5480	0.0199	0.0212	0.9515	0.9279	
17	3 4 14 6	6700	0.9939	0.5524	0.0248	0.0276	0.9503	0.9269	
18	3 4 16 6	6700	0.9437	0.5966	0.0243	0.0226	0.9972	0.9549	
19	3 4 18 6	6700	1.0323	0.6087	0.0267	0.0251	0.9684	0.9192	
20	3 4 20 6	6500	1.1125	0.6231	0.0238	0.0197	1.0120	0.9049	
21	3 2 2 6	6700	1.3286	0.7269	0.0449	0.0426	1.1065	1.0169	
22	3 2 4 6	10000	1.3376	0.5255	0.0384	0.0304	1.1131	0.9752	
23	3 2 6 6	7500	1.1357	0.6264	0.0516	0.0525	1.0645	0.9259	
24	3 2 8 6	6600	1.2157	0.7109	0.0324	0.0384	1.0453	0.9646	
25	3 2 10 6	6900	1.2283	0.7109	0.0298	0.0362	0.9931	0.9115	
26	3 2 12 6	6500	1.2732	0.6934	0.0294	0.0339	0.9889	0.9696	
27	3 2 14 6	6500	1.2122	0.7218	0.0306	0.0355	1.0739	0.9344	
28	3 2 16 6	6600	1.2582	0.7164	0.0308	0.0353	1.0890	0.9649	
29	3 2 18 6	6500	1.2382	0.7000	0.0317	0.0363	1.0084	0.9640	
30	3 2 20 6	6600	1.3796	0.7207	0.0349	0.0318	1.0581	0.9406	

The network was composed of three inputs (representing the most essential process factors in respect to attrition phenomena: mean residence time of suspension in the crystallizer working volume, volumetric concentration of crystals in the suspension and initial mean size of crystal population) and six output neurons, representing: mean size of crystal population after the process,  $L_m$ , attrition degree, AD (defined as the difference of mean sizes – before and after the test – with reference to the initial mean size) and final value of coefficient of variation (CV) – a set for DTM MSMPR and a set for DTM MSCPR crystallizer, respectively (indirect influence of two different hydrodynamic regimes). Before integrated learning–testing (overtraining effect prevention) procedure has started all numerical data were scaled into 0.1–0.9 range to fit within the transfer function boundaries (sigmoidal unipolar function). The 30 neural network configurations with 4 – 24 hidden neurons arranged into 1 – 2 hidden layers were a subject of statistical tests in respect to multidimensional fitting quality (quantified by RMSD – *Root Mean Square Deviation* – parameter value) – see Table 1.

The optimal configuration proved to be a 3–6–6 structure (three inputs, six output neurons and six neurons in one hidden layer), trained for 8600 iterations with learning rate set as 0.1 (momentum parameter was excluded from training–testing procedure). Its statistical accuracy was as follows: for DTM MSMPR apparatus RMSD for attrition degree (AD) was 0.8810%, for  $L_m$  – 0.0145 mm and for CV – 0.9847%. For DTM MSCPR apparatus RMSD for attrition degree (AD) was 0.5724%, for  $L_m$  – 0.0157 mm and for CV – 0.9164%.

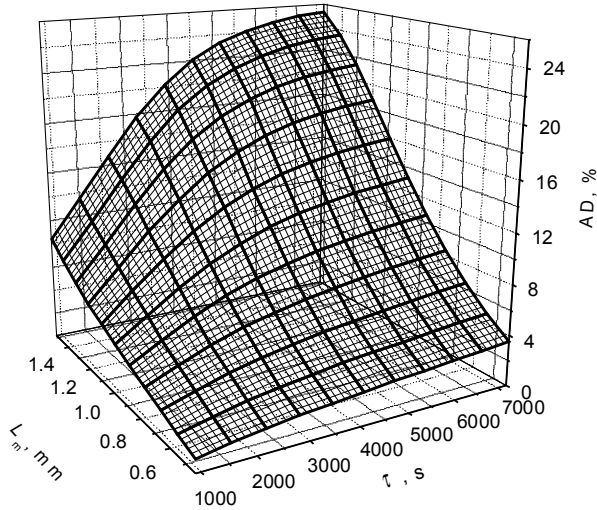
#### 4. Simulation results and discussion

This optimal configuration of neural network was then used for calculations – simulation of both crystallizers behaviour in various technological conditions (process magma service load) in respect to attrition intensity. For this work purposes an attrition degree (AD) parameter was selected as the most representative variable, fully informing about secondary nucleation magnitude. Simulations were divided into three series assuming each time a selected, constant value of one process parameter: volumetric concentration of crystals in the suspension, initial mean size of NaCl crystals and mean residence time of crystal suspension in the crystallizer working volume. Graphical presentation of numerical data generated during simulations is shown in subsections 4.1–4.3 and discussed.

##### 4.1. Assumed value of volumetric concentration of crystals in the suspension

In this set of simulations volumetric concentration of crystals in suspension was assumed to be  $\varphi = 10\%$  vol. – the highest value of  $\varphi$  from the range tested experimentally was selected in order to observe the clearest attrition effects in a form of  $AD = f(L_m, \tau)$ . Simultaneous influences of initial mean size of NaCl crystals,  $L_m$ , and mean residence time of suspension in a crystallizer working volume,  $\tau$ , on attrition degree (AD) parameter value in two different types of DTM crystallizer are presented in Fig. 2 a, b.

a)



b)

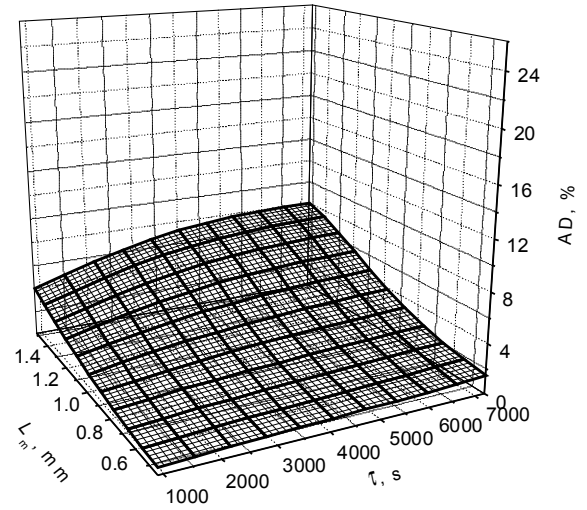


Fig. 2. Simultaneous influence of initial mean size of NaCl crystals,  $L_m$ , and mean residence time of suspension in a crystallizer vessel,  $\tau$ , on attrition degree (AD) parameter value (volumetric concentration of crystals in suspension  $\varphi = 10\%$  vol., (a) – DTM MSMPR crystallizer, (b) – DTM MSCPR crystallizer).

#### 4.2. Assumed value of initial mean size

In this set of simulations initial mean size of NaCl crystals was assumed to be  $L_m = 1.52$  mm – the highest value of  $L_m$  from the range tested experimentally was selected in order to observe the clearest attrition effects in a form of  $AD = f(\tau, \varphi)$ . Simultaneous influences of volumetric concentration of crystals in suspension,  $\varphi$ , and mean residence time of suspension in a crystallizer working volume,  $\tau$ , on attrition degree (AD) parameter value in two different types of DTM crystallizer are presented in Fig. 3 a, b.

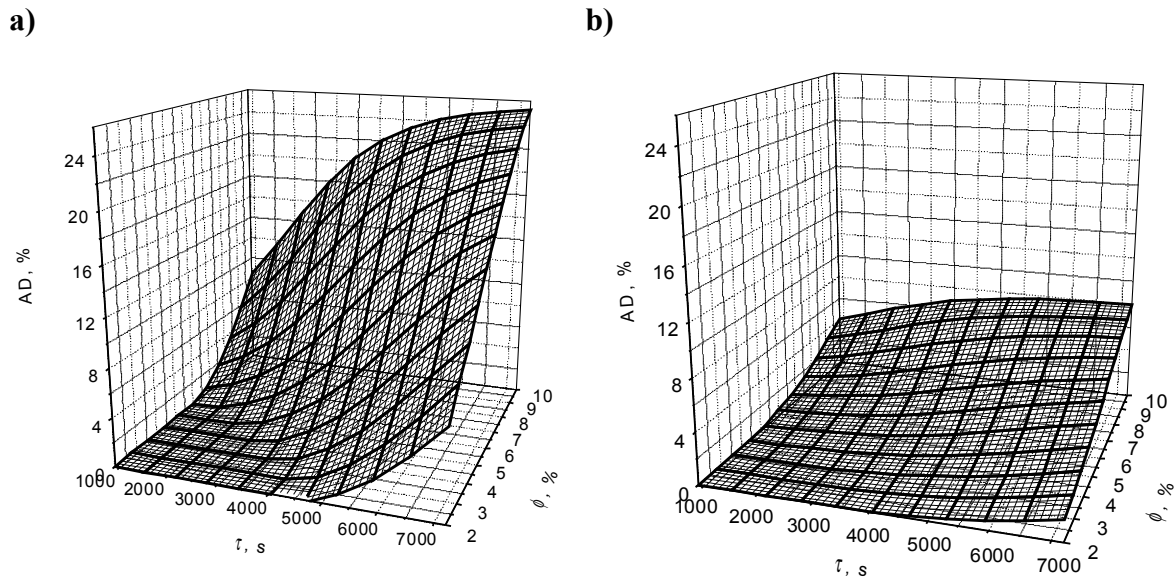


Fig. 3. Simultaneous influence of volumetric concentration of crystals in suspension,  $\phi$ , and mean residence time of suspension in a crystallizer vessel,  $\tau$ , on attrition degree (AD) parameter value (initial mean size of NaCl crystals,  $L_m = 1.52$  mm, (a) – DTM MSMPR crystallizer, (b) – DTM MSCPR crystallizer).

#### 4.3. Assumed value of mean residence time

In this set of simulations mean residence time of suspension in a crystallizer vessel was assumed to be  $\tau = 7200$  s – the highest value of  $\tau$  from the range tested experimentally was selected in order to observe the clearest attrition effects in a form of  $AD = f(L_m, \phi)$ . Simultaneous influences of volumetric concentration of crystals in suspension,  $\phi$ , and initial mean size of NaCl crystals,  $L_m$ , on attrition degree (AD) parameter value in two different types of DTM crystallizer are presented in Fig. 4 a, b.

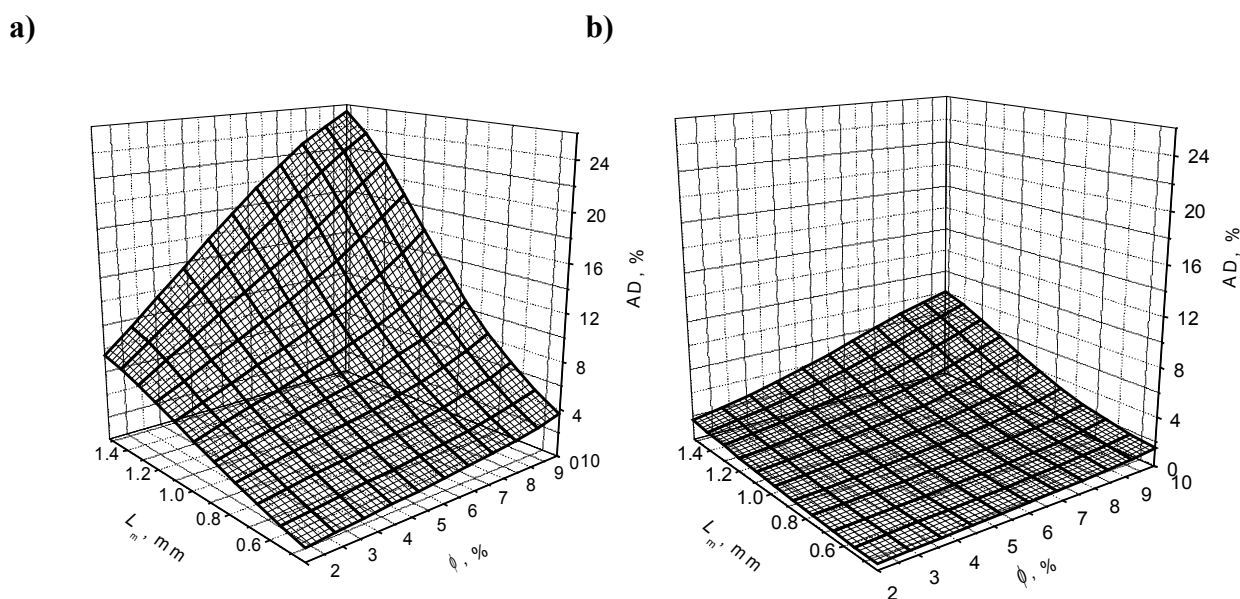


Fig. 4. Simultaneous influence of initial mean size of NaCl crystals,  $L_m$ , and volumetric concentration of crystals in suspension,  $\phi$ , on attrition degree (AD) parameter value (mean residence time of suspension in a crystallizer vessel  $\tau = 7200$  s, (a) – DTM MSMPR crystallizer, (b) – DTM MSCPR crystallizer).

The data presented graphically in Figs. 2–4 suggest that in a DTM MSCPR crystallizer construction a considerable lower attrition effects are observable compared to a DTM MSMPR variant. It results from different hydrodynamic regimes inside both crystallizer interiors. In MSCPR solution some sedimentation/hydraulic classification effects are required, thus vigorous internal circulation of suspension – like in MSMPR configuration – is confined significantly. Resulting mechanical interactions between crystals and crystal–internal elements of the apparatus are thus less intensive compared to MSMPR construction. It should be also noted, that absence of rotational elements (e.g. mixer) in both laboratory crystallizers additionally contributes the relatively low value of attrition degree (AD), compared to the systems with helicoidal (Matynia *et al.*, 2004) or propeller agitators (Bechtold *et al.*, 2004).

In all figures, presenting the neural network simulation results, one can notice the same qualitative trends – however fully developed in MSMPR configuration while significantly dumped in case of MSCPR variant.

In Fig. 2 there is presented simultaneous influence of initial mean size of NaCl crystals,  $L_m$ , and mean residence time of suspension in a crystallizer working volume,  $\tau$  on a resulting AD parameter value. Strong nonlinear relations are observed, suggesting some synergistic effects between  $L_m$  and  $\tau$  – each of them shows stronger influence on AD value for higher values of other parameter. Thus, the clearest effects are observable for the largest  $L_m$  (1.52 mm) and the longest  $\tau$  (7200 s). It should be noted, that the difference between attrition degree (AD) parameter values corresponded to the most and to the least significant results ( $L_m = 0.44$  mm,

$\tau = 900$  s) attains ca.  $\Delta AD = 25\%$ . Nonlinear changes in  $AD = f(\tau)_{L_m = \text{const}}$  or  $AD = f(L_m)_{\tau = \text{const}}$  are also visible. Similar trends, however very suppressed, are observable for MSCPR apparatus. The points corresponding to the most and the least significant AD results are located in identical points; however, the difference between these extreme AD values reaches in this case  $\Delta AD = \text{ca. } 8\%$  only.

Similar trends can be observed in Fig. 3, presenting simultaneous influence of mean residence time of suspension in a crystallizer working volume,  $\tau$ , and volumetric concentration of crystals in suspension,  $\phi$ , on AD value. As it can be expected, the mildest process conditions in respect to secondary nucleation effects correspond to “thin suspension” ( $\phi = \text{ca. } 2\%$  vol.) and relatively short mean residence time ( $\tau = 1000\text{--}4000$  s). These conditions enable one to limit the collision probability in process space ( $\phi$ ) and in process time ( $\tau$ ). In case of MSMR configuration it is possible to modify (reduce) in restricted range an AD value within 0–26% range by appropriate changes in  $\phi$  and  $\tau$  values. In case of MSCPR variant similar activity can result in the  $\Delta AD$  changes within the 0–8% range only. Some area of negligible attrition effects is, however, observable in both crystallizer constructions ( $\phi = 2\text{--}4\%$ ,  $\tau = 1000\text{--}4000$  s).

Similar situation is also presented in Fig. 4, where a space relation  $AD = f(L_m, \phi)$  is presented for both constructional variants. According to theoretical facts, “thin suspensions” ( $\phi = \text{ca. } 2\%$ ) of fines ( $L_m = 0.44$  mm) are relatively resistant to mechanical attrition because the values of kinetic energy exchanged between individual particles are strongly limited (size-effect) while relatively long distances between individual particles cause lower probability of collisions ( $AD = \text{ca. } 1\%$ ). Contrary, opposite process conditions ( $\phi = 10\%$ ,  $L_m = 1.52$  mm), where a more convenient environment for the development and exchange of higher values of kinetic energy in the microsystem exists, lead to observed intensification of attrition results (up to  $AD = \text{ca. } 25\%$ ). In case of MSCPR variant diversification between minimum and maximum values of AD does not exceed ca. 8%.

## 5. Conclusions

Numerical neural model of the attrition process in two crystallizer constructions enables one to predict (and eventually control) with the significant accuracy intensity of secondary (contact) nucleation within NaCl crystal suspension in diversified technological conditions, including: mean size of the particles creating the population, volumetric concentration of solid phase in suspension and mean residence time of suspension in the apparatus working volume. Based exclusively on experimental data, thus devoid of any simplifying assumptions, the model can correctly render all possible hidden, strongly nonlinear intrinsic interrelations and feedbacks between these three process factors, which – with various intensity – modify the specific hydraulic regime originally resulting from the selected geometrical arrangement of the vessel interior of two different constructional solutions (mixed and classified product removal). Experimental and simulated data, however connected strictly quantitatively only with the laboratory-scale crystallizers used in this study (thus of relatively small working volumes), can provide one with – at least – qualitative information about attrition behavior within NaCl crystal suspensions in liquid jet-pump crystallizers of diversified internal hydraulic regimes, making the comparative study, as well as selection of the optimal process conditions possible.



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## **Symbols**

AD	– attrition degree, %
CV	– coefficient of variation of crystal sizes, %
$L_m$	– mean size of crystals, m
$T$	– process temperature, K
$\varphi$	– volumetric concentration of crystals in suspension, % vol.
$\rho$	– solution density, kg m <sup>-3</sup>
$\tau$	– mean residence time of suspension in a crystallizer volume, s
DTM	– Draft Tube Magma crystallizer
MSCPR	– Mixed Suspension Classified Product Removal crystallizer
MSMPR	– Mixed Suspension Mixed Product Removal crystallizer
RMSD	– Root Mean Square Deviation

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