

# Problemy przenoszenia skali w krystalizatorach z rurą cyrkulacyjną i mieszadłem

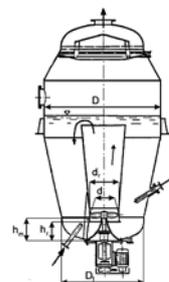
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## Streszczenie

W artykule przedstawiono analizę zagadnienia przenoszenia skali na przykładzie jednego z typów krystalizatora powszechnie stosowanego w aplikacjach przemysłowych. Zdefiniowano istotny parametr procesowym, mający duży wpływ na powtarzalność technologii. Określono wymagania z punktu widzenia kinetyki procesu podczas przenoszenia skali oraz, biorąc pod uwagę najczęściej stosowaną procedurę, przeanalizowano możliwości i ograniczenia tego sposobu.

**Słowa kluczowe:** przenoszenie skali, krystalizator DTM, czas pierwotnej cyrkulacji



Przenoszenie skali

# Scale-up problems in crystallizers with the draft tube and propeller

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## Introduction

While considering a scale-up problem in crystallization from solution it is necessary to decide which parameters describing the fluid dynamics in the apparatus are the most important. Apart from unit power input influencing mixing conditions and thus conditions of nucleation, growth, pumping efficiency and crystal size distribution, there is one more parameter, namely primary circulation time (PCT)  $\tau_c$ . It is directly connected to mixing intensity ( $l_c = l/\tau_c$ ) and macromixing conditions [1].

The above mentioned PCT is particularly important in chemical processes performed in stirred tanks without hydraulic classification. It is consistent with isothermal crystallization from solution conducted in DTM (**D**raft **T**ube **M**agma) or FC (**F**orced **C**irculation) apparatus. In both mentioned types of devices, the renewal of supersaturation generated in thin layer right below the liquid table depends mainly on the frequency of fresh superheated solution delivery in this very spot. The frequency is in this case inversely proportional to the primary circulation time and the solution supersaturation is the driving force for nucleation and growth of crystals.

PCT can be defined as the mean time for liquid element to perform full loop around crystallizer [2]. It is of main importance in devices in which the more supersaturated zone is in their upper part (e.g. vacuum DTM). When the bed is more uniform this is of secondary matter. The extension of PCT decreases the contact frequency between

subsequent portions of suspension and supersaturation layer. Despite the fact that PCT applies to contact frequency of subsequent portions of crystals with a narrow supersaturated solution zone, its influence on the process outcome cannot be neglected.

The significance of PCT as well as the small usefulness of preserving full geometrical similarity while scaling-up of crystallizers was noted by Myerson [1]. Also Srilatha [3] stated that constant unit power input and constant PCT without geometrical similarity preserved are applied in practice.

Literature review have shown that the PCT increases with increasing volume of the apparatus [1, 4]. It is mainly the result of the longer path of suspension movement as well as the smaller pumping efficiency as a consequence of scale-up. Despite the strong dependence of PCT on pumping efficiency, its increase does not compensate the influence of volume increase [5].

In this paper, the influence of scale on PCT change was analyzed for crystallizer with inner suspension circulation known as Draft Tube Magma (DTM). The scheme of such apparatus is presented in Figure 1. In such crystallizer the PCT influences:

- The frequency of mixing of circulating suspension
- The transportation time of superheated mother liquor to the supersaturation generation zone.

The primary circulation time  $\tau_c$  is defined as:

$$\tau_c = \frac{V}{V_p} \quad (1)$$

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Where  $V$  is the working volume of the apparatus and  $V_p \propto nd^3$  is pumping capacity. The working volume can be further expressed by the production rate  $m_{cr}$  and the residence time  $\tau_{re}$  related to the magma density  $m_T$  [6, 7]:

$$V = \frac{m_{cr} \tau_{re}}{m_T} \quad (2)$$

The magma density can be described by the relation [8, 9]:

$$m_T = \rho_{cr} (1 - \varepsilon^*) = \rho_{cr} \phi \quad (3)$$

where  $\varepsilon^*$  is bed porosity,  $\phi$  is volumetric concentration of solid in suspension and  $\Delta_{cr}$  is crystal density.

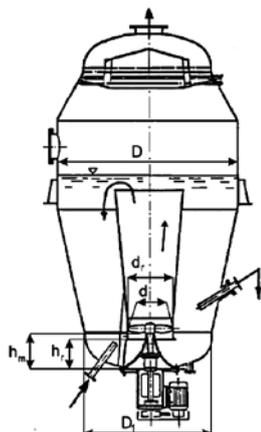


Fig. 1. The DTM crystallizer [10]

The pumping capacity  $V_p$  can be calculated using design relation [10]:

$$V_p = \frac{m_{cr}}{\Delta C_s} \quad (4)$$

in which  $\Delta C_s$  is supersaturation of the solution.

The last formula describes the dependence between the required level of the real mean solution supersaturation in the apparatus  $\Delta C_s$  (kinetic process conditions), production rate  $m_{cr}$  (mass balance conditions) and the necessary pumping capacity (fluid dynamics conditions).

### Hydraulic conditions

According to equation (1) the primary circulation time is defined by the ratio of volume and pumping capacity in the apparatus. The pumping capacity in DTM crystallizers can be expressed as a product of pumping capacity number  $K_p$ , stirrer diameter  $d$  and number of revolutions  $n$  [10, 11]:

$$V_p = K_p n d^3 \quad (5)$$

This equation was derived with the assumption that working volume of the vessel is proportional to the stirrer diameter in third power and the pumping capacity is proportional to the product of revolution number and working volume of the tank. The proportionality coefficient in this case is pumping capacity number  $K_p$  in which the influence of shape and geometrical configuration as well as stirrer type on pumping effect is embedded [10]. Knowing that  $K_p$  depends very little on the crystallizer volume one can write:

$$\tau_c \propto \frac{D^3}{d^3 n} = \left(\frac{D}{d}\right)^3 \frac{1}{n} \quad (6)$$

When full geometrical similarity is preserved during scale-up the change in PCT can be expressed as:

$$\frac{\tau_{c,in}}{\tau_{c,lab}} \propto \frac{n_{lab}}{n_{in}} \quad (7)$$

Using simple relations valid for scale-up one can derive the change in PCT with increasing scale of apparatus for most commonly recommended scale-up criteria, i.e. constant unit power input  $\varepsilon$  and constant tip speed  $u_t$ , as:

$$\text{For } \varepsilon = \text{const: } \frac{\tau_{c,in}}{\tau_{c,lab}} \propto \frac{d_{in}^{2/3}}{d_{lab}^{2/3}} \quad (8)$$

$$\text{For } u_t = \text{const: } \frac{\tau_{c,in}}{\tau_{c,lab}} \propto \frac{d_{in}}{d_{lab}} \quad (9)$$

This means that value of PCT will increase during scale-up with preserved full geometrical similarity several times. The same situation is for other scale-up criteria encountered in the literature.

### Kinetic requirements

The kinetics of crystallization process should be mimicked while scaling up the laboratory measurements to industrial scale. If so, how the increase in apparatus volume will affect the PCT from the kinetic point of view? Let's analyze some examples.

The first possible scale-up criterion is the unit production rate defined as  $m_{cr}/V$ . When such parameter is to be constant with increasing scale then primary circulation time can be defined as:

$$\tau_c = \frac{V}{V_p} = \left(\frac{V}{m_{cr}}\right) \Delta C_s \quad (10)$$

Usually suspension supersaturation is maintained constant during scale-up, so the change in PCT should follow the relation:

$$\frac{\tau_{c,in}}{\tau_{c,lab}} = \frac{\frac{V_{in} \Delta C_s}{m_{cr}}}{\frac{V_{lab} \Delta C_s}{m_{cr}}} = 1 \quad (11)$$

This means that for unit production rate as a scale-up criterion value of PCT should be kept constant [12].

The next possibility to present primary circulation time in terms of kinetic parameters is to use the definition of the crystallizer volume presented in equation (2). The combination of the mentioned equation (2) and equation (10) will give:

$$\tau_c = \frac{m_{cr} \tau_{re}}{m_{cr}} = \frac{\tau_{re}}{m_T} \Delta C_s \quad (12)$$

The latter equation shows that the primary circulation time is proportional to residence time and solution supersaturation and inversely proportional to magma density. Taking into account the discussed criterion, scale-up process should be performed in a way where change in PCT is consistent with kinetic conditions of crystallization ( $\tau_{re}$ ,  $\Delta C_s$  and  $m_T$ ). Assuming constant value of solution supersaturation, it can be described as:

$$\frac{\tau_{c,in}}{\tau_{c,lab}} = \frac{\frac{\tau_{re,in}}{m_{T,in}}}{\frac{\tau_{re,lab}}{m_{T,lab}}} \quad (13)$$

The change of PCT can be determined by analyzing influence of  $\left(\frac{\tau_{re}}{m_T}\right)$  in equation (13). One can distinguish three cases [12]:

1. When  $\left(\frac{\tau_{re}}{m_T}\right) = \text{const}$  then  $\left(\frac{\tau_{c,in}}{\tau_{c,lab}}\right)$  should not change.

- When  $\left(\frac{\tau_{re}}{m_T}\right) \downarrow$  decreases then  $\left(\frac{\tau_{c,in}}{\tau_{c,lab}}\right) \downarrow$  should also decrease.
- When  $\left(\frac{\tau_{re}}{m_T}\right) \uparrow$  increases then  $\left(\frac{\tau_{c,in}}{\tau_{c,lab}}\right) \uparrow$  should also increase.

The first case could theoretically take place but would require performing the measurements at relatively high solid concentrations in suspension and residence time close to those of industrial scale. Such conditions make it difficult to deal with in laboratory equipment. For those reasons the most often case encountered is the second one. The desire to achieve high production efficiency from unit volume is the main reason for such state. It is because of the increase in  $m_T$  is much greater than increase in  $\tau_{re}$ . The third case is strictly theoretical – it leads to decrease in unit efficiency of the apparatus.

One has to keep in mind that change of primary circulation time can be induced only by change in hydraulic conditions of suspension flow in the apparatus. Those dynamic conditions are independent of crystallization process kinetics.

### Conclusions

The presented theoretical analysis of a DTM crystallizer scale-up showed that increase in PCT will unfavorably affect reaction kinetics. The scale-up possibilities with preserved full geometrical similarity do not allow to maintain constant PCT. Furthermore the discrepancies between process requirements and possibilities resulting from hydraulic and geometrical criteria are very large. Those relations are presented in Figure 2. In each analyzed cases ( $u_t = \text{const}$  and  $\varepsilon = \text{const}$ ) full geometrical similarity of the crystallizers excludes satisfaction of constant PCT condition. Those findings question the full geometrical similarity concept during scale-up and point to the necessity of hydraulic conditions optimization in industrial designs in order to limit the PCT increase.

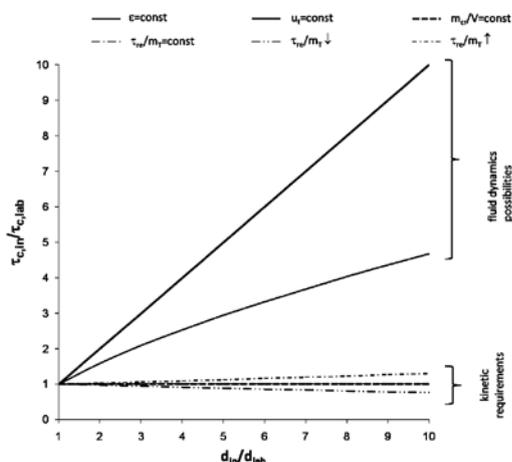


Fig. 2. Possibilities and requirements of primary circulation time value conservation during scale-up [12]

### Nomenclature

- $\Delta c_s$  [kg/m<sup>3</sup>] mean real solution supersaturation  $m_c$  [kg/h] production rate  
 $d$  [m] stirrer diameter,  $m_T$  [kg/m<sup>3</sup>] magma density  
 $D$  [m] crystallizer diameter,  $n$  [1/s] number of revolutions  
 $G_a$  [m/s] attrition rate,  $u_t$  [m/s] tip speed  
 $G_{ef}$  [m/s] effective linear crystal growth rate,  $V$  [m<sup>3</sup>] crystallizer working volume  
 $G_{kin}$  [m/s] kinetic crystal growth rate,  $V_p$  [m<sup>3</sup>/s] pumping capacity  
 $I_c$  [1/s] circulation intensity,  $\varepsilon$  [W/kg] unit power input  
 $K_p$  [-] pumping efficiency number,  $\tau_c$  [s] primary circulation time  
 $L$  [m] mean crystal size,  $\tau_{re}$  [s] residence time

### Indices:

- lab* – laboratory scale      *in* – industrial scale

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