

## POURING OF CERAMIC SUSPENSIONS INTO POROUS MOULDS

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*Evaluation of the existing approaches to the solving of the slip casting technology (pouring of suspensions into porous moulds), in particular with respect to a quantitative description of processes taking place during this forming operation. On the basis of the generalized theory of diffusion, a general mathematical model of the formation and solidification process, taking into account transfer of the liquid phase due to concentration and pressure gradients, was worked out.*

## INTRODUCTION

The forming operations in ceramic technology constitute the basis for the manufacture of various shapes of ceramic ware. Apart from pressing, machining, extrusion on augers or piston extruders, jollying, etc., the slip casting technology finds a wide field of application thanks to its ability of forming ware of both simple and complex shapes. This technology consists of two subsequent processes, i.e. the actual forming, and the solidification of the green ceramic body. The body is understood to be the solid phase (ceramic material) resulting from reducing the content of the liquid phase in the suspension or slip. This reduction is effected by absorption of the liquid into the porous mould.

The first condition for successful slip casting is the preparation of a satisfactory suspension whose properties depend in particular on raw material composition, shape, size and size distribution of the solid particles, and on the addition of a deflocculant, i.e. an electrolyte bringing about dispergation of the particles, most frequently in an aqueous medium. Preparation of ware with the required wall thickness then requires the kinetics of body formation to be known. The quality of the body depends on its homogeneity, which in turn also depends on the moisture content field arising in the body in the course of its formation and solidification. One has also to know the time within which the body has solidified, i.e. become capable of keeping its shape and being removed from the mould. This information is of primary significance for controlling and optimizing the formation processes. It can be obtained by mathematical modelling of the slip casting technology, i.e. modelling the courses of the two processes involved.

The course of body formation has so far been described quantitatively either as a filtration or a concentration diffusion process. The latter is also employed in describing the solidification stage. The difference between the filtration and the concentration diffusion approaches is given by the different ways of describing the effects of capillary forces in the system, both in the forming and the solidification stages.

Treatment of the slip casting technology on the basis of the generalized diffusion approach, which considers transfer of the liquid phase under the effect of concentration diffusion and barodiffusion, associated with capillary suction of the liquid in menisci at the unsaturated boundary between the suspension and the porous mould, or between the green body and the porous mould, provides the necessary information for a quantitative description of body formation and solidification.

On the basis of this concept, the authors had the aim to work out a mathematical model of body formation and solidification, which would serve to calculate the data mentioned above and be needed for controlling the technology. The objective should also be achieved by working out methods for measuring material quantities which constitute the terms in transfer equations.

## PRESENT APPROACHES TO THEORETICAL TREATMENT OF SLIP CASTING

The slip casting technology of ceramic ware manufacture has been used in traditional ceramics for about 150 years. The apparent simplicity of the technology and the possibility of producing also ware of complex shapes were the reason why the technology is likewise employed in the processing of new ceramic materials based both on oxidic and non-oxidic components ( $\text{Al}_2\text{O}_3$ ,  $\text{Al}_2\text{O}_3\text{-TiO}_2$ ,  $\text{ZrO}_2$ ,  $\text{SiC}$ ,  $\text{Si}_3\text{N}_4$ , etc.) [1–10]. This is due to the low investment costs, compared to those required for new technologies such as isostatic pressing, high-temperature pressing, etc.

The slip casting technology comprises two mutually linked processes. The pouring of a ceramic suspension into a porous mould results first in the formation of a green body on the mould wall, brought about by a decrease of the content of the liquid phase. The subsequent solidification in the mould leads to a further reduction of the moisture content, an increase in green strength and the ability to retain the acquired shape during the subsequent drying process [1].

The difficulties and obstacles involved in controlling these processes are associated with the degree of

knowledge of the course of the processes, and with the preparation of slips or suspensions having the desired properties. The demands for the preparation of high-grade slips, formulated on the basis of long-term experience with traditional slip casting, include a low viscosity and high fluidity at a high solids content and a minimum content of the deflocculating agent, a high stability of the suspension with respect of settling of the solids, a short time of formation and solidification of the body, ready elimination of the deflocculant from the body during its heat treatment, minimum corrosive effects of the suspension on the porous mould, and others [10–12]. The meeting of these requirements is closely associated with the knowledge of the effect of technological parameters such as the type and amount of both plastic and non-plastic ceramic components in the suspension, the particle size and its distribution of the solids and their agglomeration, the type and amount of the deflocculating agent, the mould porosity, temperature, pressure, etc. [13].

It is virtually impossible to find such relations between the technological parameters and the properties of the suspensions, which would hold generally for all types of suspensions, the traditional and the new types alike. This is the case because one and the same change in a parameter may have different effects with various types of ceramic suspensions, in fact frequently quite opposite ones. For example, the dependence of viscosity of the suspension of a kaolinitic component on the addition of a certain deflocculant is quite different from that of a montmorillonitic component. A suspension of alumina with a low content of the liquid phase can be prepared by adjusting to  $\text{pH} < 7$ . At  $\text{pH} < 7$ , the same viscosity can only be obtained by adding more liquid phase to the suspension. Just the opposite applies to  $\text{ZrO}_2$  suspensions [12]. Similar phenomena arise in the case of the effects of technological parameters on the course of the two operations. For example, deflocculants or plastic components ensuring high-grade casting properties of a suspension may lead to the formation of bodies with a high resistance to the transfer of the liquid phase, and thus to very long times of body formation and solidification.

An optimum addition of a deflocculant, defined solely on the basis of the dependence of viscosity on the amount of deflocculant added, may lead to a suspension exhibiting satisfactory flow properties and/or stability. However, the optimum established in this way need not ensure porosity and moisture distribution homogeneity, nor the green body surface quality desired, as found in [18].

The examples given above show that optimizing the composition of a suspension on the basis of determining the respective relations is a very difficult task indeed, because a change of some technological

parameter would affect favourably certain properties, but impair others. The relationships established experimentally are often explained by means of various hypotheses on mutual interactions of particles, water films, electric double layers, formations of structures or textures, and by introducing assumptions for the system in question, which should prove the data measured correct [14–17].

#### QUANTITATIVE DESCRIPTION OF THE SLIP CASTING TECHNOLOGY

The diversity exhibited by the behaviour of ceramic suspensions, which projects itself into the diversity of courses of body formation and solidification, also leads to assumptions on a random nature of this behaviour, and to conclusions that the slip casting technology is impossible to describe in any exact way. These ideas are associated primarily with a methodical approach to theoretical treatment of the technology, based on the study of suspensions by classical ceramic methods (e.g. by establishing fluidity by measuring the rate of flow through an orifice, viscosity by a rotary viscometer, readiness of body formation and its kinetics, or the moisture content by the penetration method, and the like). There are also skeptical conclusions with respect to the accuracy and reproducibility of laboratory experiments in ceramics in general [18]. Studies [18–22] have shown that by getting rid of such an approach and efforts aimed at explaining the rules of behaviour of processes during formation and solidification, the apparently incorrect results can be understood on the basis of the phenomenological theory of transfer phenomena, as consequences of generally valid laws [23–24]. Through applying these laws to the slip casting technology, one can obtain its quantitative description. Creation of mathematical models of the operations then allows their courses to be controlled and optimized. The modelling is based on the primary balance and constitutive equations for transfer of momentum, mass and heat which have to be supplemented with material functions of the quantities arising in the equations. An analysis of the technological operation will then yield the initial and boundary conditions characterizing the given problem.

In principle, one can distinguish two approaches to describing the formation and solidification of the green ceramic body. The first regards both operations as a transfer of the liquid phase through a layer of porous material and the quantitative description is then based on the filtration theory, where a constant pressure gradient is usually considered as the main driving force [18, 24, 25, 29]. The other is based on the concentration diffusion theory and makes use of Fick's laws in describing phenomenologically the transfer of the liquid phase due to a concentration gradient [20, 21, 26–28].

## DESCRIPTION OF POURING BY THE FILTRATION THEORY

The filtration approach is based on the idea that regardless of the type of the porous mould and that of the suspension, the physical principle of body forming is given by removal of water from the suspension until a state corresponding to the critical concentration of the solid component is attained. In the course of the process, the system is therefore concentrated by passage of the liquid into the porous mould. During the subsequent body solidification, the same mechanism causes the system to be further dehydrated, and the liquid left behind in the resulting body determines its porosity. Both processes result in the formation of an incompressible layer of granular material on an incompressible porous mould, by filtration under a constant pressure difference. A quantitative description of the liquid flow through a porous system then follows from Darcy's equation having the form [18]:

$$K \text{ grad } p = \mu q, \quad (1)$$

where  $K$  is the permeability of the porous system,  $p$  is pressure,  $q$  the filtrate flow density and  $\mu$  is viscosity.

The rate of body formation is then given by the following equation obtained by resolving equation (1) [7, 18]:

$$L^2/t = 2pK_1\Phi K_2(\beta\mu\Phi K_2 + K_1)^{-1}, \quad (2)$$

where  $t$  is time,  $L$  the body thickness,  $p$  the capillary suction,  $K_1$  and  $K_2$  are the permeability of the body and mould respectively,  $\Phi$  is the mould porosity,  $\mu$  is the viscosity of the liquid phase in the suspension, and  $\beta$  is a factor given by the concentration of the system and the body porosity.

The dependence of body thickness  $L$  on time follows from equation (2):

$$L = K_\infty t^{1/2} \quad (3)$$

where the constant  $K$  sums up all the constants determining the body formation rate.

The quantitative description of the kinetics of body formation is doubtlessly a significant contribution of this approach, because experimental verification of equation (3) in the casting of both traditional and new types of slips has proved it to be justified [10, 17, 30, 31]. Likewise, the concept that filtration corresponds to the physical principle of the body formation process can be regarded as being justified. The quantities arising in equation (2), namely  $K_1$ ,  $K_2$ ,  $\beta$  and  $\Phi$  are difficult to define in an exact way in the case of ceramic dispersions (polydisperse ceramic materials with particles of various shapes), and moreover depend to a significant degree on the technological parameters. From the standpoint of optimizing and controlling the slip casting technology, one would have to

know the kinetics of body formation. In addition to this, the quality of the final green body is also decided on by the distribution of the liquid phase throughout the body. It is associated in particular with the uniformity of particle orientation, porosity and shrinkage in the body volume. One has therefore to know also the concentration fields in the body, in the course of its formation as well as solidification. Information on the concentration fields also constitutes the entry condition for exact solving of the subsequent drying process [18]. The filtration theory either does not provide such information at all, or in the case of its generalization, it would be difficult to establish experimentally the material parameters required. A direct solving of the body solidification process is ruled out for the same reason.

## DESCRIPTION OF THE SLIP CASTING TECHNOLOGY BY THE DIFFUSION THEORY

The diffusion approach can be applied to the transfer of a liquid through a ceramic mix during ceramic body formation and solidification on the assumption that both the suspension and the body conform to the conditions of a binary mixture of incompressible components, i.e. the ceramic material and the liquid, or electrolyte (a mixture of the liquid phase with the deflocculant). Under isothermal and isobaric conditions, the calculation of concentration fields by the diffusion approach is then based on the mass balance:

$$\delta_t C + \text{div } \mathbf{h} = 0, \quad (4)$$

where  $\delta_t$  is differentiation with respect to time,  $C$  is the concentration of the liquid by volume and  $\mathbf{h}$  is the diffusion flow for which it holds that

$$\mathbf{h} = -D \text{ grad } C \quad (5)$$

where  $D$  is the diffusion coefficient. Joining equations (4) and (5) yields the liquid concentration balance for calculating the concentration field, in the form

$$\delta_t C = \text{div } (D \text{ grad } C) \quad (6)$$

which, if  $D$  is not a function of concentration, acquires the form

$$\delta_t C = D \text{ div grad } C. \quad (7)$$

The transfer of the liquid in the process of body formation and solidification is then regarded as diffusion of the liquid through a suspension, body as well as the porous mould. A quantitative description of these processes involves determination of the respective material quantities, i.e. diffusion coefficients of the liquid in the suspension, the body and the mould. Further introduced are concentrations characterizing the properties of the system. The coagulation concentration  $C_k$  is that at which the suspension of initial

concentration  $C_0$  coagulates and forms the body. This concentration is at the boundary between the body and the suspension. In the course of body formation, at the boundary between the body and the porous mould the concentration is  $C_s$  and depends on the properties of the body and those of the porous mould, and is in equilibrium with the surface concentration of the liquid in the mould,  $C_g$ . The concentrations defined then constitute the boundary conditions for the solving of transfer equation [11, 12].

Resolving of the balance equation (7) has expressed the relationship e.g. for calculating the time development of the concentration profile for unidirectional transfer of the liquid in a ceramic suspension during body formation, in the form [26]

$$C(x, t) = C_0 \left\{ B + \sqrt{(D_g/D_s)} \Theta \left[ x/2\sqrt{(D_s t)} \right] \right\} \left\{ B + \sqrt{(D_g/D_s)} \right\}^{-1} \quad (8)$$

where  $B = C_s/C_g$  is the separation coefficient of the liquid and  $\Theta$  is Gauss's error coefficient. Similar solutions were effected in the calculation of time development of concentration in the body or in the porous mould [26]. The solving of the diffusion equations also proved the validity of equation (3) where in the given case, coefficient  $K$  is a function of the diffusion coefficients and concentrations  $C_0$ ,  $C_s$ ,  $C_g$  and  $C_k$ .

Studies [18, 32, 34] used the diffusion approach to deal with the uni-directional body solidification. Resolving of equation (7) for boundary conditions, assuming knowledge of concentrations  $C_k$  and  $C_s$ , the initial cosine concentration distribution of the liquid in the body, the authors found that both the local and the mean concentrations of the liquid in the body decreased in an exponential way. An equation for calculating the time required for satisfactory solidification was also derived. Development of the diffusion transfer theory allowed study [38] to contribute to elucidating the effect of adding a deflocculant on body formation, and to define the conditions necessary for determining the technological minimum of the deflocculant content in the suspension. The relations between the rate of body growth and the share of the non-plastic component and deflocculant addition were also expressed.

The results of treating the slip casting technology by means of the diffusion theory indicate that this approach allows the characteristic quantities of the body formation process, analogous to those of the filtration theory, to be expressed. In addition to this, however, it is also capable of providing information on the processes and technological parameters affecting the quality of the final green body, including those required for dealing with the subsequent operation, i.e. drying [11, 26, 27].

## GENERALIZATION OF THE DIFFUSION THEORY FOR DESCRIBING THE CASTING PROCESS

The approach to the description of processes involved in slip casting of ceramics, involving the theory of concentration diffusion, is based on the concept that the concentration gradient is the sole driving force of liquid transfer during body formation and solidification. Even though this is just a phenomenological description of processes based on the diffusion theory, it does not refute the objection of the filtration theory that the liquid transfer is associated with capillary forces acting in the system. This apparent disagreement between the two approaches can be eliminated by introducing the concept that under isothermal conditions, transfer of the liquid is a diffusion process driven by the concentration gradient and the capillary pressure gradient. In other words, the diffusion transfer of the liquid is effected jointly by concentration diffusion and barodiffusion. The latter is brought about by capillary suction of the liquid in pores at the unsaturated boundary, e.g. that between the body and the porous mould.

The starting balance and constitutive equations for the given situation can be derived using the rational thermodynamic approach [20, 23]. This approach allows constitutive equations, defining the types of materials in which arbitrary irreversible processes take place, to be chosen. In the case of a thermokinetic process in a homogeneous material it holds that

$$\dot{\rho} = \rho_0 [T, v, x_i, \dots, x_{n-1}] \quad (9)$$

and the constitutive equation for mass flow has the form

$$\dot{j}_i = \dot{j}_i(\rho_0). \quad (10)$$

By restricting to continuous functions  $\dot{j}_i$  and their developing into a Taylor series around the homogeneous state, by considering only linear terms and taking into account volume inversion with respect to pressure, flow equation (10) acquires the following form for a binary mixture:

$$\dot{j}_i = -\rho D \text{grad } x - \rho D_T \text{grad } T - \rho D_p \text{grad } p. \quad (11)$$

where the coefficients  $D$ ,  $D_T$ ,  $D_p$  are functions of  $T$ ,  $p$ ,  $x_i$ , and the mass flow of the  $i$ -th component is defined with the use of convection  $v$ , i.e.

$$\dot{j}_i = \rho_i (v_i - v). \quad (12)$$

$D$ ,  $D_T$  and  $D_p$  are the coefficients of diffusion, thermodiffusion and barodiffusion respectively,  $v_i$  is the rate of flow of the  $i$ -th component, and  $\rho_i$  is the density of the  $i$ -th component, while for the density of the mixture it holds that

$$\rho = \sum \rho_i. \quad (13)$$

Under isothermal conditions, equation (11) has the form

$$\mathbf{j}_i = -\rho D \text{grad } x_i - \rho D_p \text{grad } p \quad (14)$$

where  $x_i$  is a mass fraction defined by the equation

$$x_i = \rho_i / \rho. \quad (15)$$

Local changes of volume take place in the course of some processes in ceramic materials, so that the so-called volume averaged rate  $\mathbf{u}$  is introduced in place of the convection rate:

$$\mathbf{u} = \sum C_i \mathbf{v}_i \quad (16)$$

where  $C_i$  is the volume share of the  $i$ -th component in the mixture:

$$C_i = \rho_i \mathbf{v}_i. \quad (17)$$

For the volume share it holds that

$$\sum C_i = 1. \quad (18)$$

As

$$\mathbf{v} = \sum x_i \mathbf{v}_i \quad (19)$$

then the volume flow  $\mathbf{h}_i$  of the  $i$ -th component is defined

$$\mathbf{h}_i = C_i (\mathbf{v}_i - \mathbf{u}). \quad (20)$$

For a binary mixture, the flow equation has the form

$$\mathbf{h} = -D \text{grad } C - D_p \text{grad } p. \quad (21)$$

Transfer of a liquid in a ceramic mix can be described quantitatively if the process can be regarded as diffusion in a binary mixture of incompressible components.

The moisture balance has the following form in the case of a non-reactive medium:

$$\delta_t C + \mathbf{u} \text{grad } C + \text{div } \mathbf{h} = 0. \quad (22)$$

If the components are in motion but the medium is stationary, so that

$$\mathbf{u} = 0, \quad (23)$$

then the moisture balance has the form

$$\delta_t C + \text{div } \mathbf{h} = 0. \quad (24)$$

and equation (21) holds for  $\mathbf{h}$ .

A comparison of equations (6) and (21) then indicates that on basing the treatment of body formation and solidification on the concentration diffusion theory, the diffusion coefficient in equation (6) has the significance of the effective diffusion coefficient  $D_{ef}$ , which also takes into account the transfer of liquid by

the effect of capillary barodiffusion. For  $D_{ef}$  it then holds that

$$D_{ef} = D + D_p \text{grad } p / \text{grad } C. \quad (25)$$

The concept described above made it possible to elucidate and describe quantitatively e.g. the processes involved in liquid transfer in a ceramic green body in the course of drying, that is when a liquid-unsaturated boundary arises in the material (the liquid-saturated ceramic body and the drying environment, or the boundary between the liquid-saturated green body and the unsaturated one) [22].

A phenomenological description of body formation and solidification based on the concept that the transfer of the liquid in the course of both processes is driven at the same time by the concentration gradient and the capillary pressure gradient, is then based on the balance equation

$$\delta_t C = \text{div} (D \text{grad } C + D_p \text{grad } p). \quad (26)$$

Equations (21), (25) and (26) can be resolved when one knows  $D$  and  $D_p$  or their dependences  $D = D(C, T, p, X_i)$  and  $D_p = D_p(C, T, p, X_i)$  where  $X_i$  are technological parameters. The initial and boundary conditions determining the processes of body formation and solidification must also be known. The purely concentration diffusion models of body formation and solidification are resolved for type I boundary conditions, i.e. those assuming knowledge of liquid concentration at the boundaries. The concentrations are generally variable in terms of time, but are usually regarded as being constant [35]. When taking into account the difficulties involved in experimental determination of the liquid concentrations at the boundaries, e.g. on the body surface during its formation at the body-mould interface, then it is only the relative simplicity of resolving the transfer equations which is in favour of these conditions. Measurement of the surface flow of the liquid in the suspension-porous mould closed system is experimentally more viable. In addition to the fact that the liquid flow is closely associated with the entire process, as indicated by the above analysis, the type II boundary conditions are more suitable for describing the processes in question. They have the following form:

(i) Body formation:

$$\begin{array}{llll} t > 0 & x = 0 & \mathbf{h} = \mathbf{h}(t) & p = p_i(t) \\ & x = H & \delta_x C = 0 & p = p_0 \end{array} \quad (27)$$

where  $H$  is the suspension head.

(ii) Body solidification:

$$\begin{array}{llll} t > 0 & x = 0 & \mathbf{h} = \mathbf{h}(t) & p = p_i(t) \\ & x = L & \delta_x C = 0 & p = p_0 \end{array} \quad (28)$$

The initial conditions have the following form:

(i) Body formation:

$$t = 0 \quad x > 0 \quad C = C_0 \quad p = p_0 + \rho g(H - x)$$

(ii) Body solidification:

$$t = 0 \quad x \in (0; L) \quad C = C(x) \quad p = p_0 + \rho g(H - x) \quad (29)$$

Transfer equations (21) and (26), jointly with the indicated conditions (27, 28, 29) represent a general mathematical model of body formation and solidification during the slip casting process.

The model represents a basis for the authors' approach to the resolving of the slip casting technology, that is the processes of body formation and solidification in a porous mould. Determination of material quantities arising in the transfer equations is a necessary prerequisite for practical use of the mathematical model. The authors intend to apply the model to the resolving of slip casting of ceramic bodies of simple shapes, including experimental verification of the results yielded by the model. The course of the continuing study will be described in subsequent papers.

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#### LITÍ KERAMICKÉ SUSPENZE DO PORÉZNÍ FORMY

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Zhodnoceny jsou současné přístupy k řešení kvantitativního popisu technologie lití keramické suspenze do porézní formy. Jsou porovnány modely získané z přístupů založených na teorii filtrace a difúze.

Na základě posouzení obou přístupů je vypracováno řešení kvantitativního popisu technologie lití zobecněnou difúzní teorií plynoucí z racionální termodynamiky. Je vyjádřen matematický model procesu tvorby a zatuhování střepe, uvažující přenos kapalné fáze vlivem gradientu koncentrace a tlaku. Jsou diskutovány počáteční a okrajové podmínky řešení.