



Functional composites based on refractories produced by pressure slip casting



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ABSTRACT

The present study investigates the sequential pressure filtration of two filter cake layers with a maximum grain size of 3 mm and 1 mm, respectively, using a water soluble additive system consisting of xanthan and guar gum. The pressure slip casting of the resulting composites was optimized with a factorial experimental design. The best combination was a lower filtration pressure for the first filter cake layer and a higher filtration pressure for the second one. Furthermore, a shorter filtration time of the first filter cake layer together with the application of a filter aid of a xanthan/guar gum solution resulted in an easy demolding of crack-free graded composites with an excellent bonding. The results of the full factorial experimental design were verified by X-ray computed tomography. The demonstrated pressure slip casting of graded refractories offers the possibility to produce functional composites with tailored compositions, microstructure and thus functionality.

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1. Introduction

Pressure slip casting is state of the art for the production of silicate ceramics with complex shapes such as whiteware and sanitary ware [1–6]. Compared with conventional slip casting, the process time for pressure slip casting is much shorter. The durability of the used polymer molds is also much longer than plaster of Paris molds and they do not need any drying between the casting cycles. Furthermore, pressure slip casting can be automated to a large extent. Thus, pressure slip casting offers a significantly higher efficiency compared to conventional slip casting [1,3–5,7,8]. Besides, technical ceramics produced by pressure slip casting also exhibit an improved microstructure with less agglomerates and superior mechanical properties [2–4,9,10].

Conventional and pressure slip casting was also repeatedly investigated to produce fine graded composites [5,11–19,20]. By changing the slip composition discrete or continuous gradients of the filter cakes can be achieved. The investigated compositions ranged from oxide ceramics including alumina–zirconia mixtures [5,11–13] over non-oxide ceramics [14–16] to ceramic-metal composites [17,18,20]. Besides changing the ceramic phase composition along the gradient in the filter cakes it is also possible to tailor other characteristics such as the porosity using layered pressure slip casting [21].

In recent years pressure slip casting was also studied as a new forming technique for coarse grained oxide ceramics such as refractories [6,22]. By combining coarse and fine grain fractions the thermal shock, corrosion, and erosion resistance of refractories is optimized. Furthermore, the sintering shrinkage is also reduced to a minimum, which allows the production of large sized components.

Klippel et al. [22] focused especially on the rheology of suitable slips and demonstrated the casting of coarse grained ceramics using a pressure filtration cell. In a further study Schafföner and Aneziris [6] reported the production of large plate shaped oxide ceramics with only minimal gradients caused by sedimentation. The addition of larger particle sizes had the effect of a reduced Young's modulus, which is an often observed feature of refractories. However, only the production of coarse grained oxide ceramics from a single slip composition was reported and the production of coarse grained composites by pressure slip casting remains unstudied.

The purpose of this study is to describe and examine the pressure slip casting of functional composites based on refractories. The approach in this study is to use sequential pressure slip casting of magnesium aluminate spinel slips. Thereby the overall aim was to produce refractories with a high quality bonding of filter cakes with different particle size distributions.

2. Experimental

In the present study graded composites made of filter cakes of the same material but different particle size distributions were

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Table 1
Batches of the slips.

Product name	Raw material	Grain size fraction	Slip S ₃	Slip S ₁
Almatis AR 78	MgAl ₂ O ₄	1–3 mm	15 wt. %	0 wt. %
		0.5–1 mm	10 wt. %	11.76 wt. %
		0–0.5 mm	30 wt. %	35.3 wt. %
		0–0.045 mm	10 wt. %	11.76 wt. %
Martoxid MR 70	Al ₂ O ₃	–	20 wt. %	23.53 wt. %
Water for binder	H ₂ O	Mass relative to dry mass		12 wt. %
Axilat RH 50 MD	Xanthan gum			0.024 wt. %
Food grade guar gum	Guar gum			0.024 wt. %
Additional water	H ₂ O		3 wt. %	4 wt. %

produced by pressure slip casting. The casting of graded composites was optimized using a full factorial experimental design.

In all experiments slip compositions similar to the ones presented by Schafföner and Aneziris [6] were applied. The used raw materials were alumina rich magnesium aluminate spinel (AR 78, Almatis GmbH, Germany) and reactive alumina (Martoxid MR 70, Martinswerke, Germany) for the finest particle fraction. For the experiments two different slip compositions with a maximum grain size of 1 mm (S₁) and 3 mm (S₃), respectively, were prepared. The batches are summarized in Table 1.

In order to prevent a sedimentation or segregation of the coarse grain fraction, dissolved xanthan (Axilat RH 50 MD, C.H. Erbslöh GmbH & Co. KG, Germany) and food grade guar gum were used as stabilizers because these two hydrocolloids show a strong positive interaction regarding their viscosity [23]. As a rule the added amount was 0.024 wt. % for both stabilizers regarding the raw material dry mass.

The xanthan/guar gum solution did not only act as a stabilizer to prevent a possible sedimentation, but also acted as a binder because the viscosity of this hydrocolloid solution depends strongly on the water content. During the filtration of the slips, the water content decreases which hence increases the viscosity of the binder/water solution. Thereby a sufficient green strength of the filter cakes can be attained.

Prior to mixing the slip, the binder solution was prepared using 12 wt. % water regarding the dry mass by carefully adding the binders separately to half of the water using a high shearing laboratory mixer (RZR 2102 control, Heidolph Instruments GmbH & Co. KG, Germany). Afterwards, the two separate binder solutions were mixed together.

In all experiments 3 kg of dry mass were used for each slip. For the slip preparation the dry mass was first mixed for 4 min using a standard concrete mixer (ToniMIX, Toni Technik Baustoffprüf-systeme GmbH, Germany). The slips were in general thixotropic, therefore the mixer was kept at the lowest power level to avoid a strong stirring and hence decrease in viscosity of the slips.

After the dry mixing, the binder solution was added in three steps to ensure a homogeneous mixture. After each adding the slip was stirred for 3 min followed by a careful scrapping of the mixer walls to remove any lumps. Finally an amount of 3 wt. % and 4 wt. % of distilled water was added to the S₁ and S₃ slips, respectively. The slips were then stirred for 3 min for a last time. Shortly after preparing the slips, the water content was controlled using a thermobalance (MA 30, Sartorius AG, Germany) in order to ensure a comparable slip composition in the experiments.

For all the filtration experiments a laboratory pressure filtration cell was applied. The pressure filtration cell operated similar to a pressure casting machine using pressurized air as the pressure medium. It allows the testing of a wide range of factors before performing upscaling experiments. The filter medium of the pressure filtration cell was poly(methyl methacrylate) (PMMA), which was identical to the usual filter medium material used in commercial pressure slip casting machines. The filter medium had a filtration

area of 33.7 cm² and was supported by a porous metal plate with a relative open area of 40.2%.

In all cases the slips were first filled into the pressure filtration cell, then sealed before the pressure was abruptly increased using a lever valve. The pressure was held constant during the whole filtration time.

In preliminary experiments the filtration properties of both slips were first separately investigated. For that purpose the slips were filled with a height of 20 mm into the pressure filtration cell and were then filtrated with a pressure of 5 bar and 7 bar, respectively, until the dewatering of the filter cake started. The dewatering starts at the point when the cake filtration of the slip ceases, i.e. the filter cake is completely built up and no slip remains on the filter cake surface. With the beginning of the dewatering the remaining water of the filter cake is then displaced by air [22,24,25]. The start of the dewatering was clearly perceivable by the time that a fizzling sound became evident, which was caused by the breakthrough of pressurized air. The start of the filter cake dewatering was defined as the termination criterion for the filtration and the necessary time to reach the dewatering was thus recorded for later experiments at each pressure.

The following experiments regarding the graded filter cake composites were mainly conducted using a 2⁴ full factorial experimental design because factorial designs have several advantages. They allow the simultaneous study of several factors, i.e. independent variables, and also of so called interactions, i.e. the influence of one factor on the other, with only a minimum of experiments [26]. The overall aim was to achieve a composite of high quality which can be easily demolded at the same time. The factorial experimental design is summarized in Table 2 in conjunction with Table 3. All of the following statistical analyses were conducted with the software package R [27].

The general procedure for the casting of graded filter cakes was always to filtrate the slip S₁ (d_{max} = 1 mm) on top of a filter cake of the S₃ slip (d_{max} = 3 mm).

In the following the chosen factors of the factorial experimental design will be described. The first factor (A) was the use (+1) or non-use (−1) of a so called binder filter aid. This binder filter aid had the aim to improve the demolding of the filter cake and to prevent the penetration and thus possible clogging of the filter medium

Table 2
Factors of the 2⁴ full factorial experimental design.

Factor	Identifier	Lower level (−1)	Higher level (+1)
Binder filter	A	Without	With
Filtration pressure of first (coarse) slip S ₃	B	5 bar	7 bar
Filtration pressure of second (finer) slip S ₁	C	5 bar	7 bar
Filtration time relative to beginning of filter cake dewatering	D	75%	100%

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