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Rotary moulding of ceramic hollow wares

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Abstract

A novel processing method for the fast and economic production of hollow ceramic components has been developed by combining *in situ* coagulation moulding with a modified version of the technique of rotary moulding[Binner, J. G. P., Al-Dawery, I. A., Tari, G. and Yan, Y., Rotary casting technique. *UK Patent application No. 0506349.0*, March 2005], the latter being adapted from the polymer industry. The process was found to require a high solids content suspension, hence development work was performed in this direction though in the end a new, commercial suspension was utilised. Of the three forming routes of gel casting, direct coagulation casting and *in situ* coagulation moulding, the latter was found to be the most promising for the new process of rotary moulding of ceramics. Due to the low value of clay-based ceramics, a new low cost coagulant was identified and the effect of lactone concentration and temperature on setting time determined.

Following substantial optimisation work, it was found that a two-speed approach to multi-axial rotation was the most successful; medium sized cream jugs could be produced in just 7 min. With respect to mould materials, the porous resin normally used for pressure casting of sanitary ware was found to be the best option, though since this is quite expensive conventional plaster-of-paris moulds were found to be a suitable material to enable companies, particularly SMEs, to become familiar with the technology whilst avoiding high costs for trials. The processed articles could be successfully fired and glazed using gas-fired kilns with no sign of any black cores.

Major advantages of the process include the ability to precisely calculate the amount of ceramic slip required, eliminating either slip wastage or the need to pour used slip back into the virgin material as currently happens with slip casting. In addition, since the precursor suspension has a very high solids content, the time and energy required to dry the green product and associated moulds has been considerably reduced.

Keywords: Rotational moulding; Gel-casting; Coagulation casting; Ceramics; Hollow ware

1. Introduction

The aim of this research work was to develop a new, sustainable ceramic production method that can replace slip casting and, in particular, the use of plaster moulds that go to landfill after as few as 40 casts. One particular goal was for the new process to be able to use potentially recyclable plastic or metal moulds having a lifetime of thousands of casts. This paper describes the key concept of the research, viz. the combination of either gel casting,[2] direct coagulation casting[3] or *in situ* coagulation moulding[4] with an adapted version of the process of rotary moulding that is used in the plastics industry. The sanitaryware and tableware industries were used as exemplars.

Rotary moulding has been used for the manufacture of a wide range of hollow plastic products, such as water tanks, kayaks and traffic cones, for more than 40 years.[5] In the process, a precursor polymer powder is rotated in a heated mould, typically across 2 axes. As the powder starts to melt it spreads along the walls of the mould where it forms a skin; after cooling the cast part can be removed. In the current work, the polymer powder has been replaced with a ceramic suspension that undergoes a setting reaction during the rotation period; *in situ* coagulation moulding (ICM) was found to be the best option.

ICM[4] is based on the progressive destabilization of ceramic suspensions to form a viscoelastic solid within which the homogeneity of the initial dispersion is maintained. One route to achieving this is via the coagulation of electrosterically dispersed, high solids content ceramic suspensions as a result of the time-dependent hydrolysis of carboxylic acid derivatives. Constraining this hydrolysis reaction within a non-porous mould leads to the formation of high density green bodies. After drying,

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the ceramic components can be sintered without special debinding operations since only a small amount of organic additive is needed.

The new *in situ* coagulation rotary moulding (ICRM) process has the potential to lead to faster production rates by a factor of 3–4 and reduced labour requirements by up to 30–40% as well as substantial savings in drying energy, material purchase and factory space requirements. Although inevitably more expensive than the plaster moulds, it was hoped that the use of plastic or metal moulds would enable the use of CAD-CAM techniques for their production in the future, significantly increasing the flexibility of the manufacturing process and reducing the time needed to introduce new product shapes. As a consequence the whole manufacturing process for hollowware items might be made considerably less wasteful and more sustainable with product costs being significantly reduced, allowing the European ceramics industry to remain competitive and increase its market share.

2. Experimental and results

2.1. Optimisation of suspension compositions

The first goal was to modify existing clay-based suspensions used to slip cast items so that they were suitable for either gel casting, direct coagulation casting or *in situ* coagulation moulding, techniques that have been reviewed recently by Tari.[6] The aim was to determine which was best suited for the modified rotary moulding technique.

Following initial trials it became apparent that an extremely high solids content, ideally >80 wt%, was critical for achieving solidification, rather than simply suspension thickening, from the relevant chemical reactions. To maximize the solids content, the zeta potential versus pH characteristics were initially measured for aqueous dispersions of each of the raw material powders used as a function of surfactant addition to determine the optimum levels of the latter so that the individual powder components all displayed similar zeta potential behaviour. Once these had been identified, the surfactants were added to each powder individually so that they could be combined at a fixed zeta potential value; this resulted in maximum deflocculation and hence low viscosity.[7]

It is well known that increasing suspension viscosity increases the time required to de-air a slip; in the ceramic industry around 4% of production loss is currently associated with parts being rejected due to entrapped air bubbles and so anything that increases the viscosity of the initial slip is not desirable. Therefore limiting the suspension viscosity was a very important parameter in the development of the suspension.

Having optimised the zeta potential, the particle size distribution of the components was also investigated and optimised via grinding to maximize the solids loading further. In general, a broad particle size distribution allows the solids loading to be increased without a commensurate increase in viscosity.[8] Throughout the development work it was found to be essential to blunge the mixture for 1 h, in a shear mixer. Eventually, a suspension with a solids content of >80 wt% was achieved whilst retaining a pourable consistency. The key steps were the understanding and control of the zeta potential and the optimisation of the raw material particle sizes.

Significant attention was given to keeping costs to a minimum since the traditional ceramics industry is very competitive (a major factor behind their support for the work) and hence works with extremely tight margins. Thus a number of options that could have been investigated for a purely scientific study were considered inappropriate, e.g. the introduction of especially fine powders as part of the particle size optimization stage - they would have resulted in an increase in both production costs and health and safety issues in the factories. In addition, every component and additive had to be carefully considered to ensure that it did not result in thermal expansion differences between the body and the glaze, necessitating the development of a new glaze formulation.

A newly developed, commercial high solids content, low viscosity clay suspension with a density of $1.98\,\mathrm{kg\,l^{-1}}$ was subsequently introduced to the research programme.[1] In trials it outperformed the suspension developed above in terms of rheological behaviour and was also found to offer the additional advantage that more of the coagulation agent for the *in situ* coagulation moulding process could be added, allowing acceleration of the coagulation reaction, which in turn reduced the time needed for this stage of the process (albeit at the expense of requiring a longer time for burnout, by a factor of 3, at the start of the sintering schedule). Hence the commercial suspension was used for all the subsequent work.

2.2. Consolidation methods

Work was focused on investigating the three different solidification techniques to determine which would be most suitable for rotary moulding. It rapidly became obvious that controlling the viscosity versus time behaviour as a function of temperature during solidification was crucial. Preliminary work focused on the formation of simple bars ($120\,\mathrm{mm} \times 12\,\mathrm{mm} \times 5\,\mathrm{mm}$), this was subsequently extended to cylinders using a very crude, single axis rotary moulding facility. It is believed that the study was the first time that any of the three techniques outlined in Section 2.1 had been investigated with clay bodies.

2.2.1. Gel-casting

The gel casting work, based on the use of the acrylamide monomers,[2] revealed that the order of mixing the components was very important and that continuous mixing of the slurry was vital to ensure homogeneity of properties in the final product, probably because of dealing with a multicomponent system. For reasons not yet understood, it was possible to gel cast clay bodies with a lower monomer content, but slightly more initiator and catalyst, compared to the gel casting of alumina ceramics.

Whilst it was possible to gel cast satisfactory clay bodies in <1 min, there were significant problems associated with burning

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