Syntactic foams consolidated with starch: modelling for pre-mould processing

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Abstract. Various manufacturing parameters involved in 'buoyancy method' were inter-related. An equation based on unit cell models for a relation between volume expansion ratio (VER) of bulk microspheres in aqueous starch and microsphere size was derived. A good agreement between the equation and experimental data was found. The inter-microsphere distance (MID) concept was introduced and it was demonstrated that the MID can be calculated numerically for microspheres with known statistical data.

Introduction

Syntactic foams are made of pre-formed hollow microspheres and binder [1]. They can be used in various structural components including sandwich composites [2, 3]. A wide range of different types of syntactic foams can be made by selecting different materials and consolidating techniques for binder and hollow microspheres. The consolidating techniques include coating microspheres [4], rotational moulding [5], extrusion [6, 7], inorganic binder solution and firing [8], dry resin powder for sintering [9-12], compaction [13, 14], liquid resin as binder [15] for in situ reaction injection moulding, and buoyancy [1, 16].

A main purpose of the present work was to investigate numerical/theoretical relationships between various manufacturing parameters of the buoyancy method for manufacturing syntactic foam consisting of ceramic microspheres and starch binder.

Materials and Method

Hollow Microspheres. Ceramic hollow microspheres supplied by Envirospheres Pty Ltd, Australia were used. Four different size groups (or commercial grades), SL75, SL150, SL300 and SL500, were employed. Particle densities for the different grades were measured to be 0.68, 0.73, 0.80 and 0.89 (g/cc) respectively and bulk densities 0.39, 0.42, 0.43 and 0.36 respectively.

Microsphere sizes were measured using a Malvern 2600C laser particle size analyser and were found to be of approximately Gaussian distribution.

Starch as Binder. Potato starch (Tung Chun Soy & Canning Company, Hong Kong) was used as binder for hollow microspheres. Particle density of the potato starch was measured using a Beckman Air Comparison Pycnometer (Model 930) and an average of three measurements was found to be 1.50g/cc. Bulk density was also measured using a measuring cylinder with a tapping device (300 taps were conducted) and an average of five measurements was found to be 0.85g/cc.

The Buoyancy Method for Manufacturing Syntactic Foams

The basic principles for manufacturing are based on the buoyancy of hollow microspheres in aqueous starch. The starch binder can be diluted for the purpose of controlling binder content in syntactic foam. Microspheres are dispersed in the binder by stirring or tumbling. The mixing container is left until microspheres float to the surface and starch settles down, forming three phases: a top phase consisting of microspheres and binder, a middle phase of water, and a bottom phase of starch, microspheres and water. The top phase is to be used for moulding. Gelatinisation of starch in the mixture can be conducted at two different points in time. One is prior to the addition of hollow microspheres to water-starch mixture and the other after moulding, which are referred to as pre- and post-mould gelatinisations respectively. In this work, pre-mould gelatinisation was employed.

Top Phase Volume Calculation

Numerical Calculation of Minimum Inter-Microsphere Distance. The top phase volume is always larger than the initial bulk volume of microspheres in air (IBVMS) as a result of expansion of IBVMS caused by starch binder between microspheres. A minimum inter-microsphere distance (MID) may be an indicator of the volume expansion of IBVMS when microspheres are dispersed in the top phase. The MID is a surface-to-surface distance. A computer program was written in MATLAB 6.5 to produce 3D models for dispersion of microspheres and to find a MID for a given volume fraction of microspheres. Microspheres with random sizes but with Gaussian distribution as measured for microspheres were randomly positioned in 3D space. Mean radii corresponding to experimental values, 26.72, 55.27, 89.09, and 179.73 µm were nominated for SL75, SL150, SL300, and SL500 respectively, with respective standard deviations of 7.06, 18.2, 29.95, and 58.83 µm.

The 3D models were created by rejecting microspheres which are closer to existing microspheres than a nominated MID and otherwise accepting microspheres until a nominated volume fraction of microspheres is reached. Iteration was conducted to find a MID corresponding to a volume fraction of microspheres experimentally given and was ended when

$$\frac{|\, v_{ms} \text{ from experiment} - \text{Calculated } v_{ms} \,|}{v_{ms} \text{ from experiment}} \leq 0.048 \qquad (1)$$

where v_{ms} is the volume fraction of microspheres.

Idealised Mono-Sized Particle Dispersion Models. Idealised mono-sized particle dispersion models i.e. simple cubic (SC) unit cell, face centred cubic (FCC) unit cell, and body centred cubic (BCC) unit cell were used for derivation of volume expansion ratio (VER) of bulk microspheres in the top phase being defined as (top phase volume) / IBVMS.

The VER based on the three models (independent of model type) was derived to be

$$VER = \left(1 + \frac{d_e}{2r + d_0}\right)^3, \qquad (2)$$

where r is the radius of microsphere, d_0 is the initial *MID*, and (d_0+d_e) is the *MID* after expansion. For a practical microsphere dispersion in the top phase, equivalent values for d_0 and d_e can be found. The d_0, when d_e = 0, in Eq. 2 was calculated for each microsphere size group using the packing factor (= microsphere bulk density / microsphere particle density) of bulk microspheres for a given mean radius of microspheres.

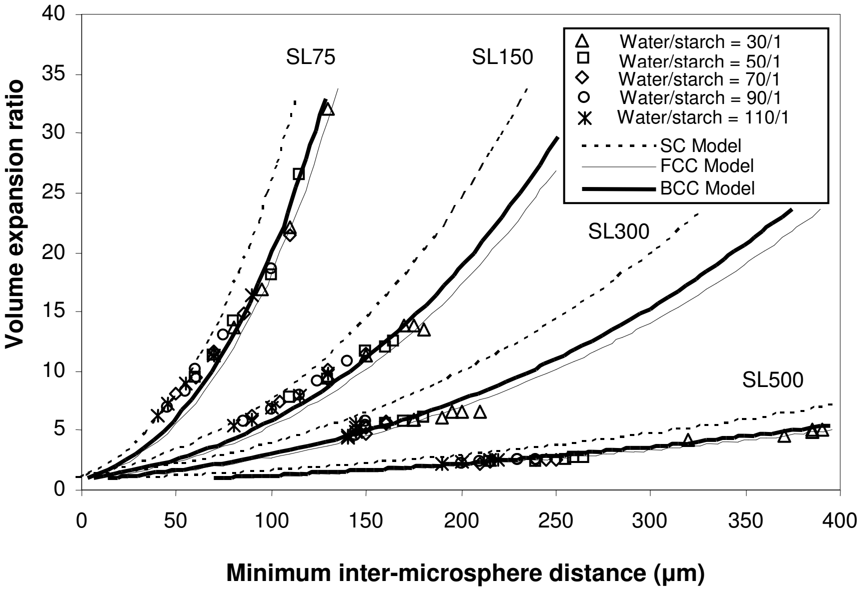

Figure 1 Experimental volume expansion ratio (VER) versus numerically calculated minimum inter-microsphere distances (MID = d_0+d_e) in comparison with theoretical curves generated according to Eq. 2. The initial bulk volumes of microspheres (10cc, 15cc, 20cc, 25cc and 30cc) for each water/starch ratio are seen such that the higher VER the lower IBVMS.

Results and Discussion

VER of IBVMS in the top phase after tumbling/stirring for a granule starch volume fraction in binder ranging from 0.005 to 0.022 was measured. It was found to be high for small sized microspheres (SL75) and high starch content but low IBVMS. The effect of IBVMS on the expansion ratio seems to be due to the buoyant force because the smaller the IBVMS, the lower the buoyant force, giving smaller squeezing force and hence larger inter-microsphere distances. Also, it is a truism that the volume expansion is caused by increase in distance between microspheres. Once microspheres are wetted with binder, inter-particle distance would be affected by various factors such as starch content and IBVMS. It was assumed that a MID exists in the top phase for a given manufacturing condition. Also, the top phase can be assumed as being formed through random positioning of microspheres after tumbling/stirring. The experimental VER versus numerically calculated MID (= d_0+d_e) is shown in Fig. 1 with theoretical curves generated according to Eq. 2. The theoretical curves based on BCC and FCC models appear to be in a good agreement with data. Meanwhile, SC model appears to be in a relatively poor agreement with data compared to the other models and has unrealistic values for d_0 being negative. The predictions based on BCC and FCC would be useful for practical design of mixing containers for different microsphere sizes and eventually for optimization of manufacturing system.

Conclusions

Various parameters for syntactic foam manufacture based on the 'buoyancy method' have been studied. An equation (Eq. 2) based on unit cell models with MID concept for a relation between VER and microsphere size is derived and successfully used to predict experimental data.

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