Syntactic foams consolidated with starch: mixing behaviour for post-mould processing

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Abstract. Syntactic foam manufacturing method involving 'post-mould processing' was studied for potential building material applications. It was found starch particles tend to adhere to hollow microspheres during mixing, forming agglomerations. A transition in formation, after phase separation, of mixture volumes in water was also found to take place at a calculated relative density value of 1 for an agglomeration consisting of multiple starch particles and one microsphere. Starch-microsphere inter-distance was discussed and considered to be an important parameter affecting starch content in an agglomeration.

Introduction

Syntactic foams are particulate composites made of pre-formed hollow microspheres and binder [1]. A wide range of different types of syntactic foams can be made by selecting different materials and consolidating techniques for binder and hollow microspheres. As previously discussed in reference [2], the buoyancy method has recently been demonstrated to be capable of control of a wide range of binder contents at low costs, widening applicability of syntactic foams. Also it allows us to use starch as binder for manufacturing potential building materials such as interior wall boards, ceiling panels, etc. It has some advantages over other binders such as epoxies, phenolics, etc, being readily available, environmentally friendly, and inexpensive renewable a polymeric binder.

A main purpose of the present work was to investigate mixing behaviour of starch particles and ceramic hollow microspheres in 'post-mould processing' [2, 3] for starch particle quantitative formation in syntactic foams.

Constituent materials for syntactic foams

Hollow Microspheres. Ceramic hollow microspheres supplied by Envirospheres Pty Ltd, Australia were used. Two different size groups (or commercial grades), SL75 and SL300, were employed.

Microsphere and starch particle sizes were measured using a Malvern 2600C laser particle size analyser and mean sizes were found to be 53, 178, and 47 for SL75, SL300, and starch respectively. They were found to be of approximately Gaussian distribution as previously shown in reference [2]. Particle densities and bulk densities of the four hollow microsphere groups were also measured using a Beckman Air Comparison Pycnometer (Model 930) and a measuring cylinder (capacity 250cc) respectively. Three hundred taps were conducted for each bulk density measurement. An average of five measurements was taken for each size group and is available in reference [2].

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. SEM images of starch particles employed was shown elsewhere [2]. A gelatinisation temperature range for starch was measured to be 64-69ºC.

The buoyancy method for manufacturing syntactic foams

The basic principles for manufacturing of syntactic foams containing starch as binder are based on the buoyancy of hollow microspheres in aqueous starch binder [2-3]. The starch binder (= starch particles + water) can be diluted for the purpose of controlling binder content in syntactic foam. When microspheres are dispersed in binder in a mixing container as a result of tumbling/stirring, the mixing container is left until microspheres float to the surface, forming three phases i.e. top phase consisting of microspheres, starch particles and water, middle phase of water only, and bottom phase of microspheres, starch particles and water. The top phase is to be used for moulding. Gelatinisation of starch in the mixture was conducted after moulding, which is referred to as 'post-mould gelatinisation' as opposed to 'pre-mould gelatinisation' [2-3].

Phase volume measurement

Measuring cylinders (500cc, 50mm in diameter) were used for observation of phase separation and measurements of phase volumes at a room temperature ranging 17 - 20°C. A wooden lid was used on the top of each cylinder to minimise evaporation of water from the mixture. A constant volume of 400cc for binder in each cylinder was used. Measurements for phase volumes were taken in every 30 minutes until a constant value was found in three successive readings and the final constant value was used. Measurements for phase volumes were made prior to adding microspheres. Starch particles (only two phases in this case, top phase contains water only and bottom phase contains starch particles and water) were found to settle down in 1 to 2 hours in binder, depending on starch content in binder. After adding microspheres to binder, phase separation took another 1 to 2 hours in binder, depending on microsphere size and starch content in binder. Stirring of mixture containing microspheres was conducted after sealing by tumbling each measuring cylinder upside-down and back up for 20 times manually.

Results and discussion

The total volume change rate after mixing in water (TVCRAM) (= [top and bottom phase volumes after mixing] / [microsphere and starch bulk volumes in water before mixing]) is given as a function of starch volume fraction before mixing (SVFBM) (= [starch bulk volume in water before mixing] / [microsphere and starch bulk volumes in water before mixing]) in Fig. 1. It appears to be dependant upon microsphere size. As the microsphere mean size decreases, TVCRAM increases. This indicates large gaps between microspheres and starch particles exist for small microspheres. Also, the maximum TVCRAM occurs at a starch volume fraction as indicated with filled and open arrows respectively for SL75 and SL300 . (The position of the arrow will be further discussed below.) Further, the TVCRAM does not much appear to be affected by initial bulk volume of microspheres (IBVMS).

During the phase separation in a measuring cylinder after tumbling/stirring of aqueous mixture, starch particles tend to settle down to form sediment but microspheres to float to the top due to their densities. Also, some interaction between microspheres and starch particles take place i.e. some starch particles are carried by microspheres to form the top phase with microspheres, and microspheres are carried by starch particles to form the bottom phase with starch particles. To quantify this phase separation, bottom phase volume fraction after mixing (BPVFAM) (= [bottom

phase volume after mixing] / [top and bottom phase volumes after mixing]) is plotted as a function of SVFBM as shown in Fig. 2. If there were no such interaction between microspheres and starch particles, all the data points would have been on the dashed line shown in each plot in Fig. 2. Data points under the dashed line indicate that starch particles are trapped in the top phase but those above the dashed line indicate that microspheres are trapped in the bottom phase. However, those that are close or on the dashed line do not necessarily mean that microspheres are not trapped in the bottom phase nor starch particles are trapped in the top phase. Thus, those data points close to the dashed line provide only a necessary condition (not sufficient condition) for the case where no interaction between microspheres and starch particles exists. Further, a physical transition (not graphical) in each size group is seen to occur at the similar starch volume fraction to that for TVCRAM (see Fig. 1) as indicated with filled and open arrows for SL75 and SL300 respectively.
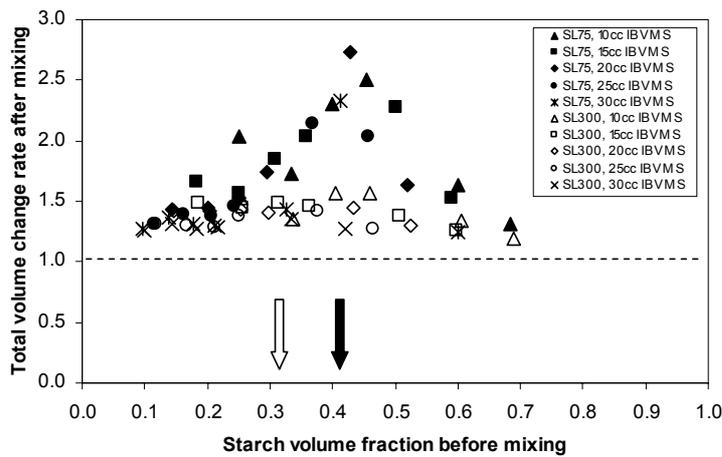

Figure 1 Total volume change rate after mixing (TVCRAM) as a function of SVFBM: (a) SL75 and (b) SL300.
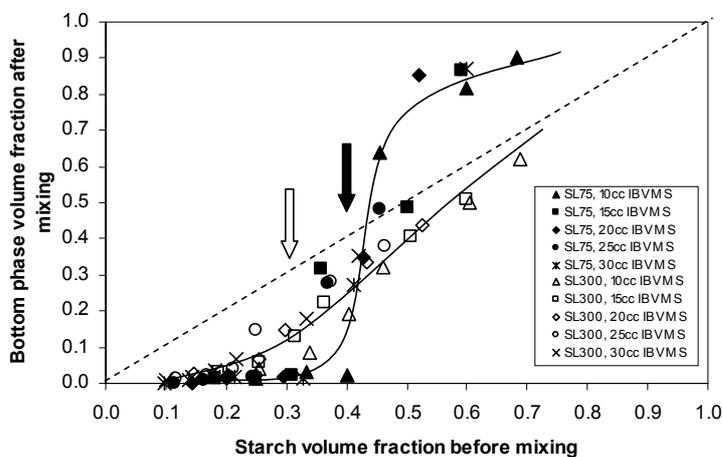

Figure 2 Bottom phase volume fraction after mixing (BPVFAM) as a function of SVFBM: (a) SL75 and (b) SL300.

To identify the interaction between microspheres and starch particles, direct observations using a microscope were made and found that starch particles tend to adhere to microspheres. When a microsphere settles down, starch particles that already adhered to the microsphere do not easily separate from the microsphere. However, starch particles settled down on top of other starch particles adhered to a microsphere readily separate from each other when the microsphere changes from translational motion to rotational motion. This indicates the attractive force between a starch particle and a microsphere is stronger than that between starch particles. Agglomerations are hence formed due to the presence of starch particles, acting as glue, between microspheres. (An agglomeration consists of microspheres and starch particles.) The buoyancy of each agglomeration depends upon fractions of constituent particles – the more starch particles the heavier. Volume fraction of starch particles on a microsphere making a relative density of 1.0 (VFSMRD) were

calculated to be 0.41 and 0.31 for SL75 and SL300 respectively. For the calculations, each mean diameter of microsphere group was used and it was assumed that starch particles are spherical for approximation. It is important to note that the values of VFSMRD correspond to the points indicated by arrows in Fig. 1 and Fig. 2. Thus, the VFSMRD appears a good indicator for both the maximum TVCRAM and the transitional points.

It can be explained about the VFSMRD as the transitional point indicator. The phase separation may be a stochastic process to a large extent. When a volume fraction of starch is lower than VFSMRD, the density of an agglomeration would have more chance to be lower than 1, allowing more number of agglomerations to float to form the top phase. When a density of an agglomeration, however, is higher than 1, more number of agglomerations tends to settle down to form the bottom phase. It can be further explained about the high abruptness of the transition (Fig. 2) for small microspheres as follows. It is a truism that a small starch-microsphere inter-distance allows more chance for starch particles to rapidly adhere to microspheres than a long starch-microsphere inter-distance. Thus, as microsphere size decreases in a given space, the starch-microsphere distance increases for a given microsphere bulk volume. As a result, agglomeration rapidly occurs and its size tends to be large for small microspheres. Consequently, relatively not many individual particles or/and small agglomerations are formed from small microspheres. Such individual particles or/and small agglomerations are the ones that causes smoothness of the transition because their densities are not much affected and tend to follow the dashed line in Fig. 2.

Conclusions

Post-mould processing for manufacturing syntactic foams using starch as binder has been studied. A transition in carrying starch particles by microspheres during phase separation was found and explained using a calculated relative density value of 1 for an agglomeration consisting of multiple starch particles and one microsphere.

Acknowledgement

References

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