

Manufacture of syntactic foams using starch as binder: post-mold processing

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Abstract

Syntactic foam manufacturing method, ‘post-mold processing’, based on the buoyancy of hollow microspheres was studied for potential building material applications. The post-mold processing involves mixing starch particles and ceramic hollow microspheres in water. It was found starch particles tend to adhere to hollow microspheres, forming agglomerations, during mixing. It was also found that ‘volume fraction of starch particles on a microsphere making a relative density of 1.0’ (VF_{SMRD}) is an indicator for mixture volume transitions. Both the maximum total volume expansion of mixture and a transition in formation, after phase separation, of mixture volume in water referred to as ‘top phase’ in a mixing container were taken place at a calculated VF_{SMRD}. It was found that hollow microsphere size effect on attracting starch particles was relatively high but IBVMS effect was not significant. Also, no effect of water volume for a given diameter of cylindrical container was found. Starch-microsphere inter-distance was discussed and considered

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to be an important parameter affecting starch content in an agglomeration. A Simple Cubic cell model for the starch-microsphere inter-distance was adopted to quantitatively explain various effects on starch content in agglomeration such as hollow microsphere size, initial bulk volume of hollow microspheres (IBVMS) and water volume. Further, the following were found for manufactured syntactic foams: (a) volume fraction of starch in foam is of linear relation with starch content before mixing for a given experimental data range and (b) shrinkage is relatively high for small hollow microspheres with high starch content.

1. Introduction

Syntactic foams are particulate composites made of pre-formed hollow microspheres and binder (1). They have been used in sandwich composites (2-4) and areas where low densities are required e.g. undersea/marine equipment for deep ocean current-metering, anti-submarine warfare (5-9). The densities of syntactic foams in the past, however, have been relatively high compared to the traditional expandable foams, limiting their applications.

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 (10), rotational molding (11), extrusion (12) and ones that use inorganic binder solution and firing (14), dry resin powder for sintering (15-18), compaction (19, 20), liquid resin as binder (21) for in situ reaction injection molding, and buoyancy principle (1, 22-26). The last method (buoyancy) 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. Starch has not been well known as binder even though it has been used in plasterboards (27). It has some advantages over other binders such as epoxies, phenolics,

etc, being readily available, environmentally friendly, and inexpensive renewable a polymeric binder.

In this paper, ‘post-mold processing’ (25, 26) which allows better dimensional control than ‘pre-mold processing’ (25, 26) is studied for manufacturing syntactic foams consisting of ceramic hollow microspheres and starch. A main purpose of the present work was to investigate mixing behaviour of starch particles and ceramic hollow microspheres for understanding of starch particle quantitative formation as binder in syntactic foams.

2. Constituent materials for syntactic foams

2.1. Hollow Microspheres

Ceramic hollow microspheres (composed of silica 55-60%, alumina 36-40%, iron oxide 0.4-0.5% and titanium dioxide 1.4-1.6%) supplied by Envirospheres Pty Ltd, Australia were used. Four different size groups (or commercial grades), SL75, SL150, SL300 and SL500, were employed. Scanning electron microscope (SEM) images of hollow microspheres were given elsewhere (24).

Microsphere sizes were measured using a Malvern 2600C laser particle size analyser and were listed in **Table 1**. They were found to be of approximately Gaussian distribution as previously shown in reference (24). 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 measurements are listed in **Table 2**.

Table 1 Mean size and standard deviation of hollow ceramic microspheres and starch particles.

Hollow microspheres and starch	Mean diameter or size (μm)	Standard deviation (μm)
SL75	53	14
SL150	111	36
SL300	178	60
SL500	359	118
Starch	47	24

Table 2 Particle and bulk densities of hollow ceramic microspheres.

Hollow microspheres	Particle density (g/cc)	Bulk density (g/cc)
SL75	0.68	0.39
SL150	0.73	0.42
SL300	0.80	0.43
SL500	0.89	0.36

2.2. 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 (24). Size of starch particles was measured using a Malvern 2600C laser particle size analyser and was found to be of approximately Gaussian distribution as shown in **Figure 1**. A gelatinisation temperature range for starch was measured to be 64-69°C.

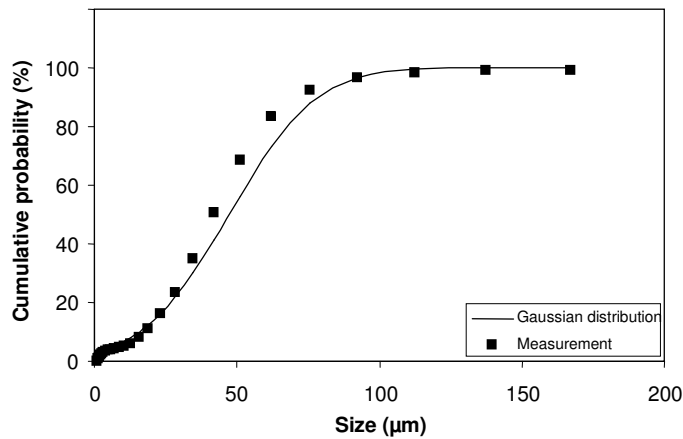


Figure 1 Size distributions of potato starch particles with a cumulative Gaussian distribution curve.

Mean size and standard deviation of potato starch particles measured by Malvern 2600C laser particle size analyser are listed in **Table 1**.

3. 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 (22, 23). 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 three phases are schematically shown in **Figure 2**. The top phase is to be used for molding. Gelatinisation of starch in the mixture was conducted after molding, which is referred to as ‘post-mold gelatinisation’ as opposed to ‘pre-mold gelatinisation’ (24-26).

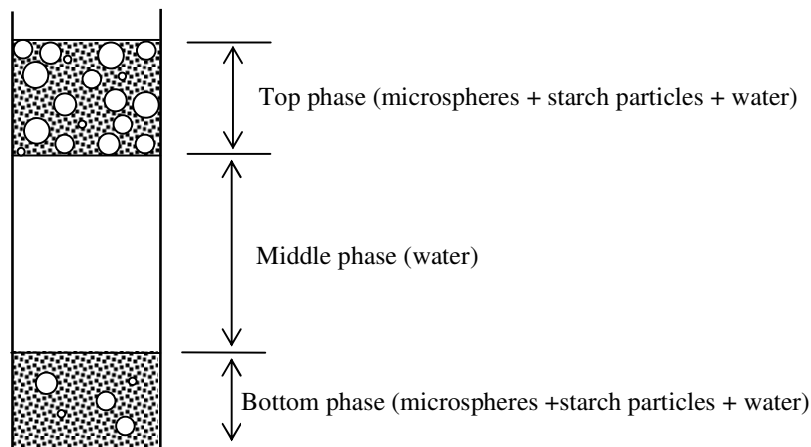


Figure 2 Schematic of phase separation

4. 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. (A plateau value for phase separation was found after 5 times).

5. Manufacturing details and shrinkage measurement for syntactic foams

Syntactic foam specimens for shrinkage measurement were prepared by varying starch content. Mixing was conducted in a container (120 mm in diameter 150 mm in height) for a microsphere amount of $115 \pm 35\text{g}$ by manually shaking up for at least 1.5 minutes after sealing. Subsequently, the container was left for 5 minutes to allow for phase separation. The top phase was scooped into a circular open mold (73mm in inside diameter and 15mm in height) placed on an aluminium plate covered with a sheet of paper and then the mold top was covered with another sheet of paper, and thereafter with an aluminium plate to keep sufficient moisture/water in the mixture for gelatinization. The molded mixture was placed in an oven at 80°C for one hour mainly for gelatinization. It was subsequently uncovered and left for further 5 hours mainly for drying, and then was demolded for final 2 hour drying. The shrinkage measurement was conducted right after the gelatinization of the first one hour. Two specimens were used for each measured value. The volume ratio of bulk microspheres to binder was approximately 1 to 3.

Syntactic foams for measurement of starch volume fraction in foam were manufactured in a similar manner described above but a measuring cylinder used for the phase volume measurement and split molds of cylindrical cavities (16mm in diameter and 24 mm high) were employed.

6. Results and discussion

The bulk volume expansion rate (VER) in water versus bulk volume in air of hollow microspheres and starch particles is given as part of characterisation in **Figure 3**. The VER is defined as bulk volume in water divided by bulk volume in air. The bulk volume of microspheres in water was measured from top phase volume (TPV) without starch and the bulk volume of starch particles in water from sediment volume without microspheres. The VER appears

approximately 1.2 and approximately independent of bulk volume variation in air. In addition, a long term VER of starch particles is given in **Figure 4** where VER is seen to be constant for the first three days and to slightly increase afterwards.

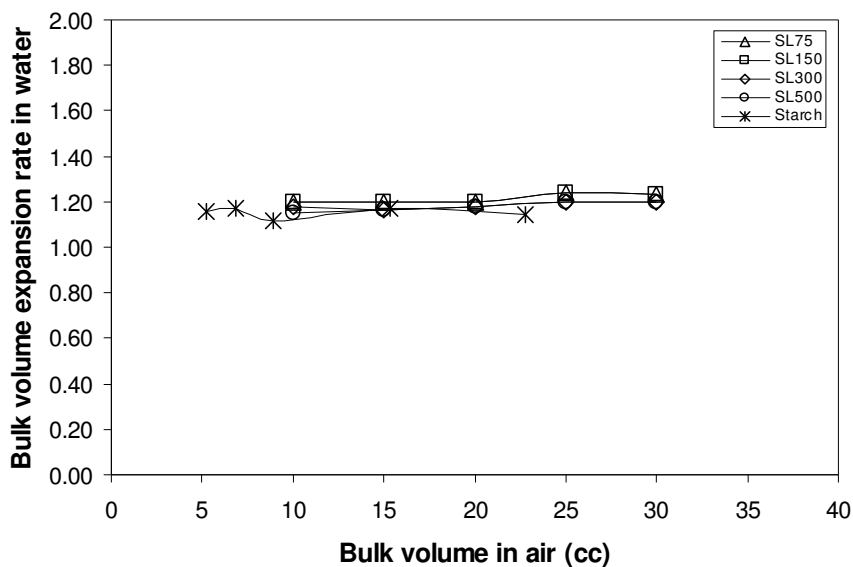


Figure 3 Bulk volume expansion rate (VER) (= bulk volume in water / bulk volume in air) versus bulk volume in air of hollow microspheres and starch particles.

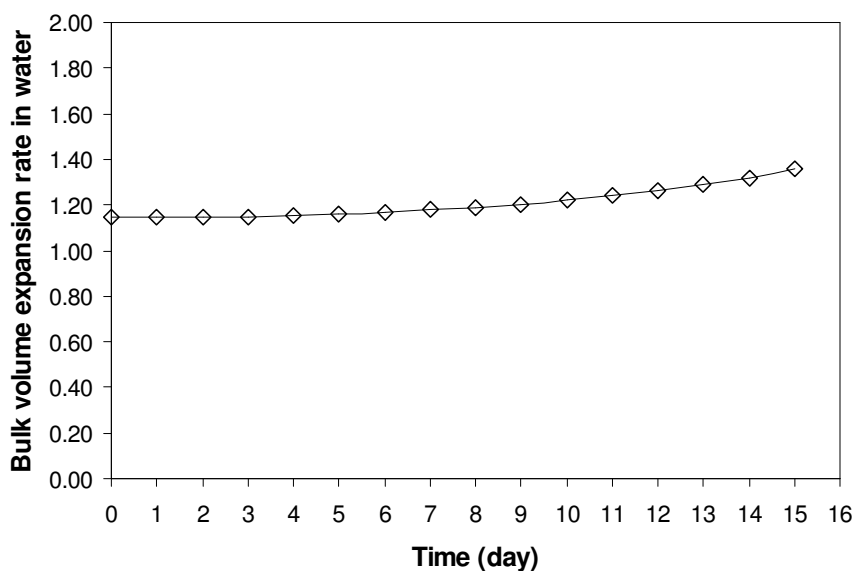
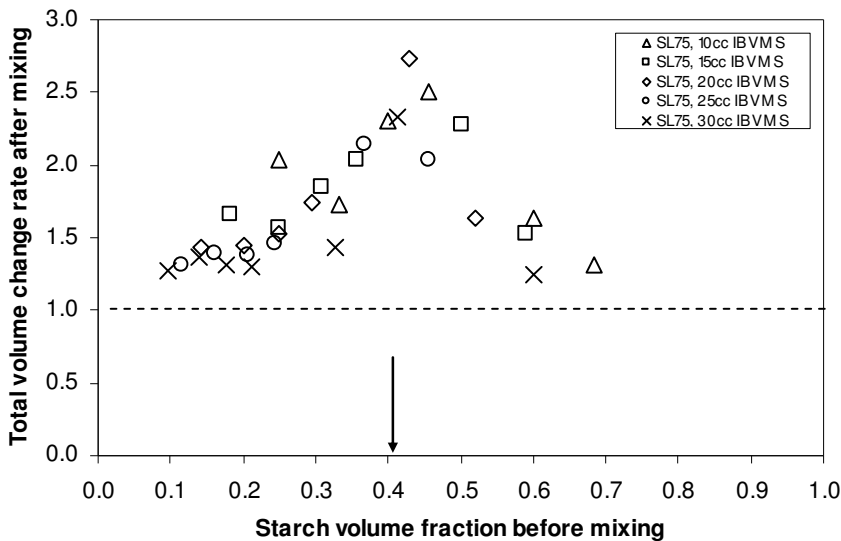
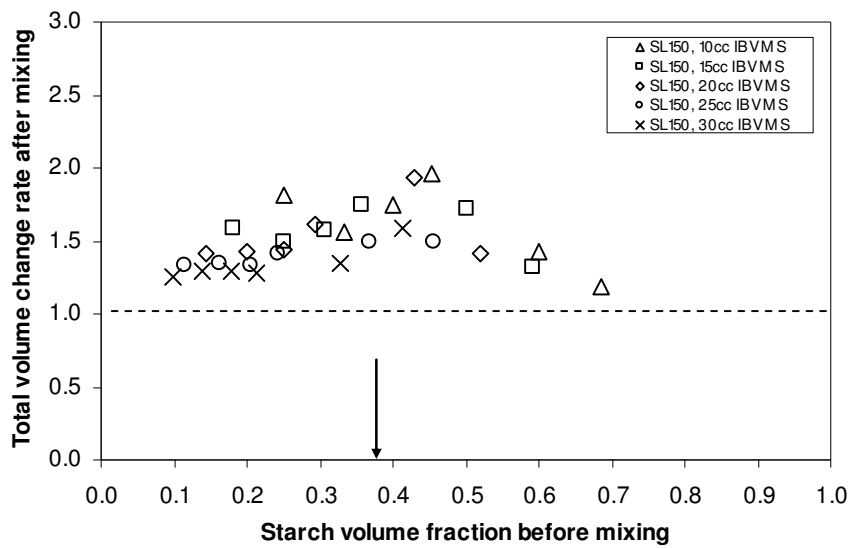


Figure 4 Long term bulk volume expansion rate (VER) (= bulk volume in water / bulk volume in air) of starch particles in water as a function of time (number of days) elapsed.

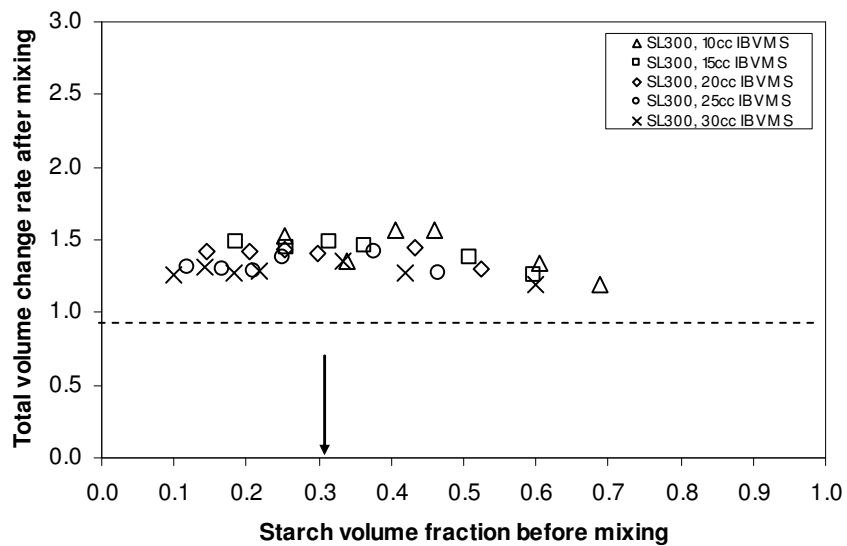
The total volume change rate after mixing in water (TVCRAM) (= top and bottom phase volumes after mixing / microsphere and starch bulk volumes in water before mixing) is given as a function of starch volume fraction before mixing (SVFBM) (= [starch bulk volume in water before mixing] / [microsphere and starch bulk volumes in water before mixing]) in **Figure 5**. 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. When microsphere sizes large, however, TVCRAM decreases towards 1 as seen in **Figure 5(d)**. Also, the maximum TVCRAM occurs at a starch volume fraction indicated with an arrow. (The position of the arrow will be further discussed quantitatively below.) Further, the TVCRAM does not appear to be affected by initial bulk volume of microspheres (IBVMS), indicating that buoyant force of microspheres may not be sufficiently high to compress the starch particles in top phase.



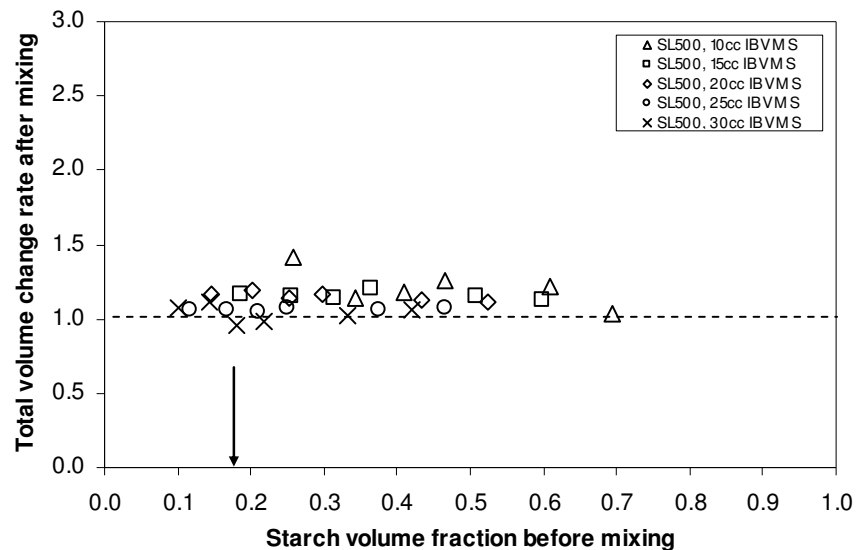
(a)



(b)



(c)

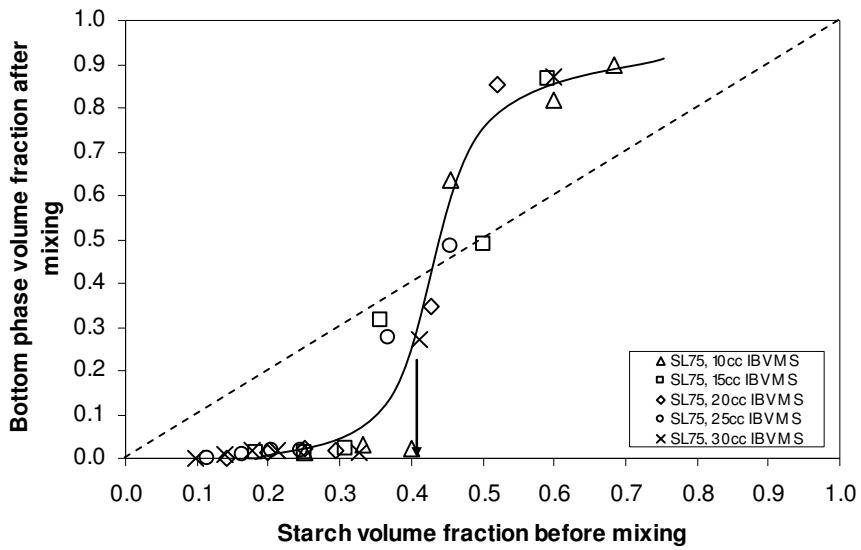


(d)

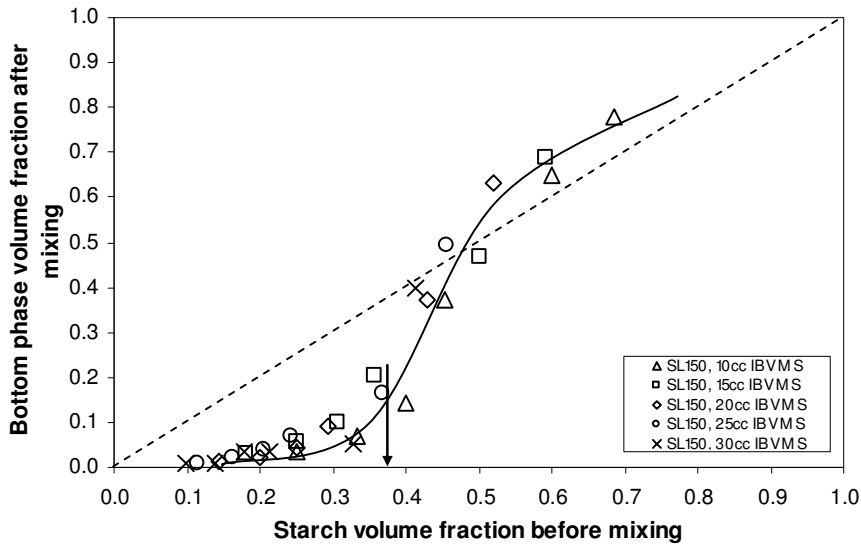
Figure 5 Total volume change rate after mixing (= [top and bottom phase volumes after mixing] / [microsphere and starch volumes in water before mixing]) as a function of starch volume fraction before mixing (SVFBM) (= [starch volume in water before mixing] / [microsphere and starch volumes in water before mixing]): (a) SL75, (b) SL150, (c) SL300, and (d) SL500.

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 **Figure 6**. 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 **Figure 6**. 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.

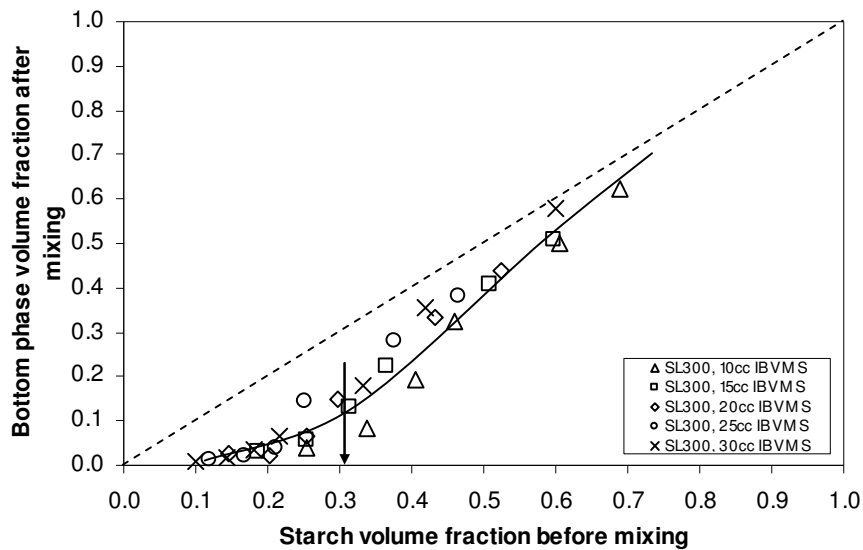
Thus, those data points close to the dashed line provide only a necessary condition (not sufficient condition) for the case where no interaction between microspheres and starch particles exists. Further, a physical transition (not graphical) in each plot is seen to occur at the similar starch volume fraction already indicated with an arrow in **Figure 5**.



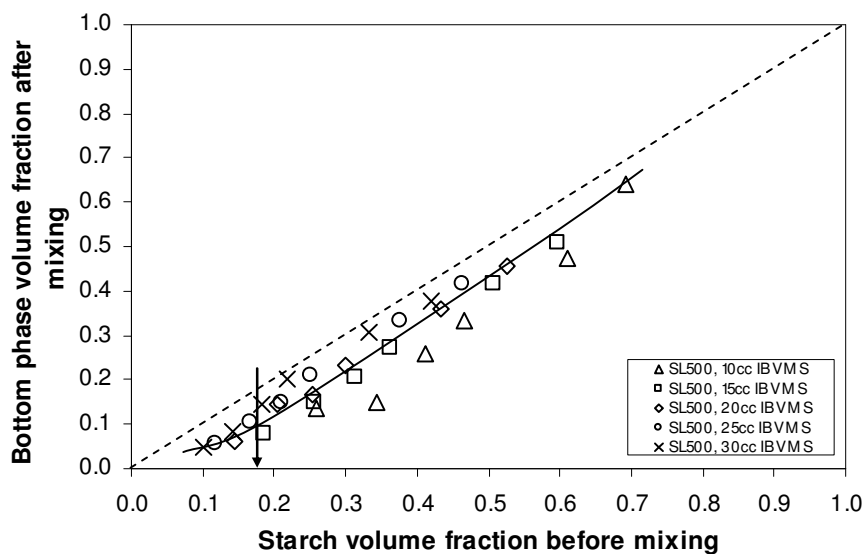
(a)



(b)



(c)



(d)

Figure 6 Bottom phase volume fraction after mixing (= [bottom phase volume after mixing] / [top and bottom phase volumes after mixing]) as a function of starch volume fraction before mixing (SVFBM) (= [starch volume in water before mixing] / [microsphere and starch volumes in water before mixing]): (a) SL75, (b) SL150, (c) SL300, and (d) SL500.

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 motion of the microsphere changes from translation (**Figure 7(a)**) to rotation (**Figure 7(b)**) as illustrated in **Figure 7**. 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. Numbers, and volume fraction, of starch particles on a microsphere making a relative density of 1.0 (NSMRD & VFMSMRD) were calculated and listed in **Table 3**. The maximum number of starch particles covering one microsphere (MNSSF) was also calculated (**Table 3**). 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 VFMSMRD correspond to the points indicated by arrows in **Figure 5** and **Figure 6**. Thus, the VFMSMRD appears a good indicator for both the maximum TVCRAM and the transitional points.

Table 3 Starch particle numbers on one microsphere making a relative density of 1.0 for an agglomeration and corresponding volume fractions (NSMRD & VFMSMRD). The maximum number of starch particles covering the whole surface of one microsphere (MNSSF).

Hollow microspheres	NSMRD	VFMSMRD	MNSSF
SL75	1	0.41	590
SL150	8	0.38	2803
SL300	24	0.31	8095
SL500	99	0.18	47564

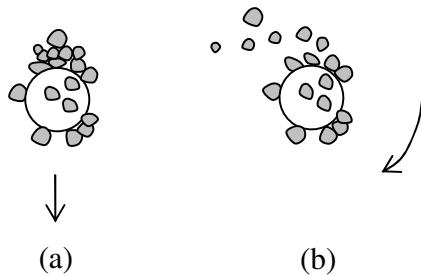
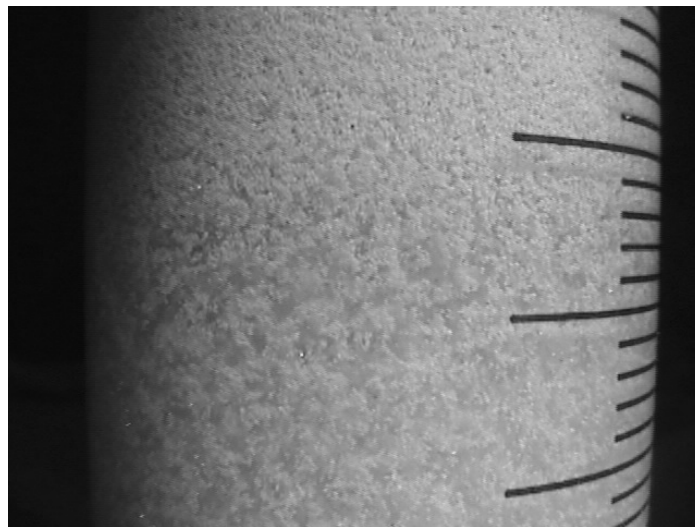


Figure 7 Schematic representation of observation for starch particles on a microsphere in water: (a) translational motion; and (b) rotational motion.

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 in a mixture 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 (**Figure 6**) 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 as shown in **Figure 8**. 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 **Figure 6**. It is also possible that, when the agglomeration size increases, the TVCRAM increases as well because of irregularities of agglomeration shape. Therefore, this explains that the VFSMRD can be a good indicator for the maximum TVCRAM as well.



(a)



(b)

Figure 8 Agglomerations in measuring cylinders in 30 seconds after tumbling/stirring for a SVFBM of 0.4 and a IBVMS of 10cc: (a) SL75; and (b) SL300. It is seen that the top phases are partially formed. The smallest division on the scale represents 1.8 mm.

One could expect some effect of IBVMS on the transitional behaviour because starch-microsphere distance can be affected by it since starch-microsphere inter-distance is relatively small for a large IBVMS. The inter-particle distance (d) can be readily estimated using a Simple Cubic unit cell model with an initial distance (d_0) (**Figure 9**). The distance is from surface to surface but in this case it can be approximated to the distance from center to center because

particle size is much small compared to inter-particle distance in a large volume of liquid. When IBVMS increases from 10cc to 30cc (extreme 300% increase) for example, particle numbers 8 become 24 ($= 3 \times 8$) in a given space and $d = 0.69 d_0$ (31% decrease). However, when a SL150 microsphere is replaced with an equivalent volume of SL75 microspheres (only moderate 48% decrease in mean size), particle numbers 8 become approximately 74 and $d = 0.48 d_0$ (52% decrease). Therefore the IBVMS effect on the inter-particle distance appears not significant as seen in **Figure 6** compared to the microsphere size effect (SL75 and SL150) which can be seen in **Figure 6** (a) and (b). Further, a follow-up experiment was conducted to see the effect of water volume in the mixture (using a starch volume fraction of 0.4 and a water volume range of 90 – 400cc in the same measuring cylinder) given that the more water volume the longer inter-particle distances. However, no noticeable effect was found on the scales in **Figure 5**. The water volume effect seems be offset by the effect of particle travelling distance - the longer the travelling distances the higher the chance of collision between starch particles and, hence, the higher chance for forming agglomerations.

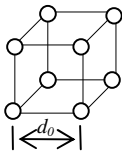


Figure 9 Simple Cubic cell model with an initial distance d_0 .

Starch particle volume fractions ($=$ starch particle volume / TPV) in the top phase (SVFTP) for a constant IBVMS of 30cc were estimated using manufactured foams (see **Appendix** for formula used) and showed in **Figure 10**. In general, the SVFTP tends to be high for small microspheres, indicating small microspheres carry more starch particles perhaps due to small inter-particle distances as discussed above.

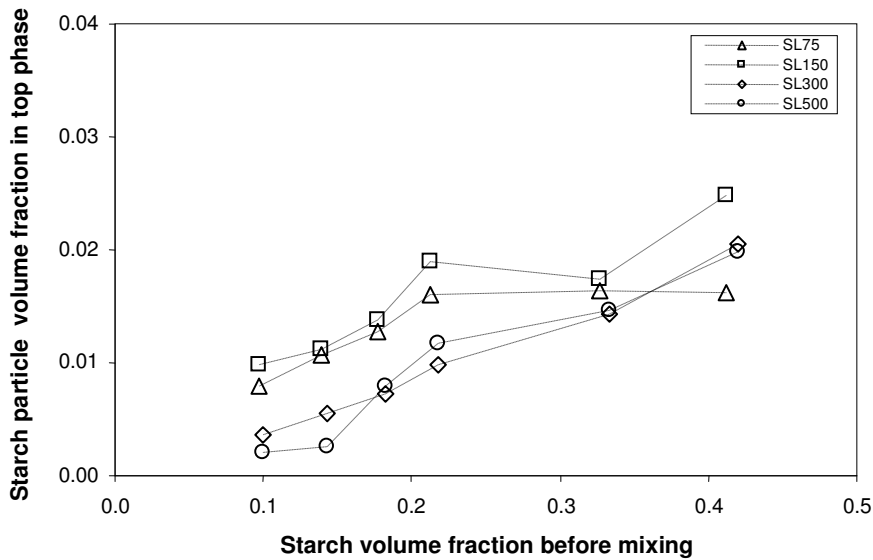


Figure 10 Starch particle volume fraction in the top phase (SVFTP) after phase separation versus starch volume fraction before mixing (SVFBM) for an IBVMS of 30cc.

Starch volume fraction in foam (SVFF) manufactured for various microsphere size groups but a constant IBVMS of 30cc are given as a function of SVFBM in **Figure 11**. They increase in the given range of SVFBM's linearly with increasing SVFBM with high correlation coefficients, 0.994, 0.966, 0.997, and 0.982 for SL75, SL150, SL300, and SL500 respectively. Both SVFTP and SVFF would be expected to be affected by the transitional point (or VFMSRD point) but the linearity of the foam density (**Figure 11**) in particular does not appear to be much affected. Probable reasons are that (a) the transitions of SL300 and SL500 are relatively smooth (**Figure 6**) and (b) the transitional points of SL75 and SL150 are around the high ends of the range of SVFBM (**Figure 11**).

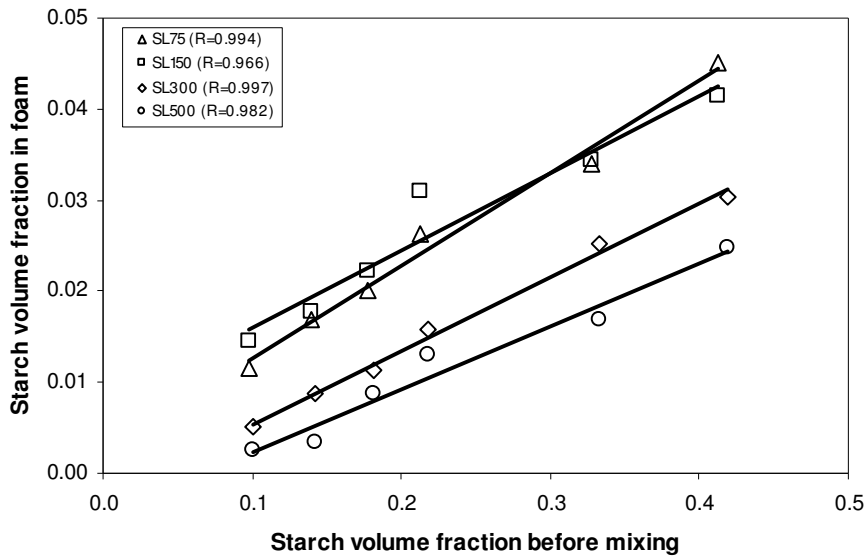
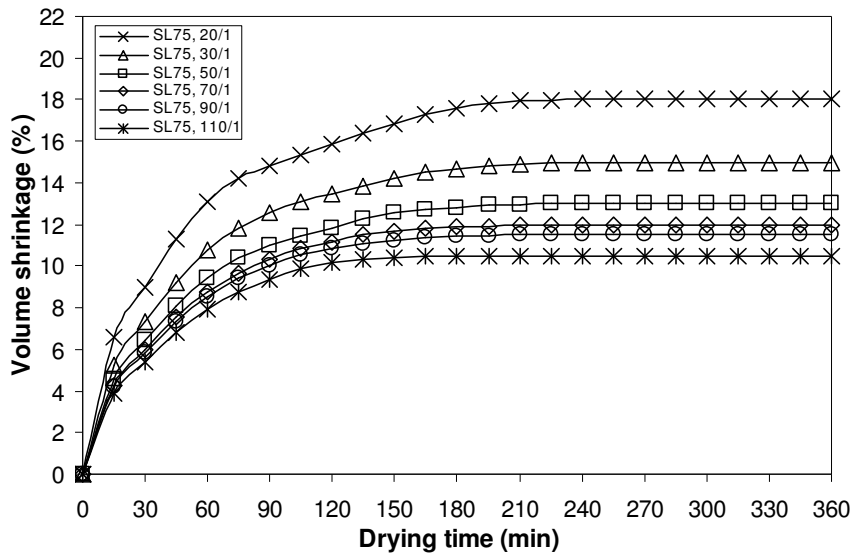


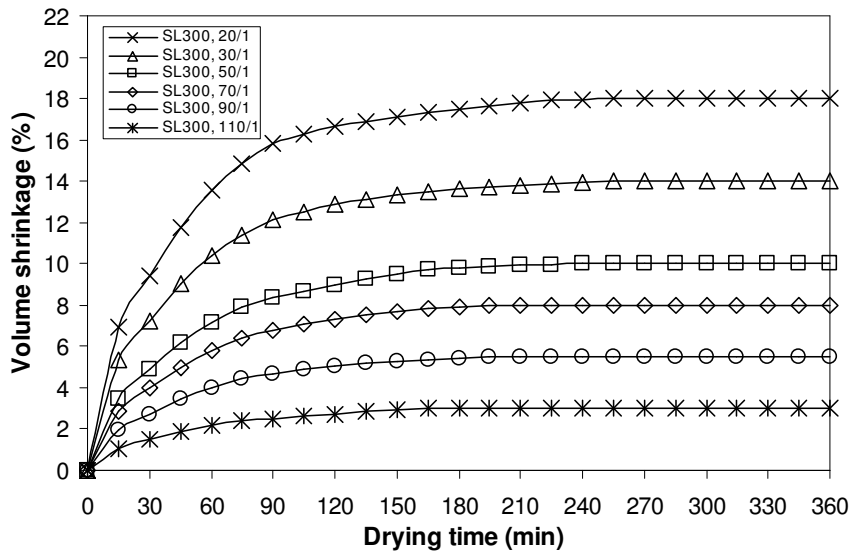
Figure 11 Starch volume fraction in foam (SVFF) versus starch volume fraction before mixing (SVFBM) for an IBVMS of 30cc. Correlation coefficient (R) is given for each microsphere size group.

Volume shrinkage ($= \frac{\text{[progressive volume]}}{\text{[initial volume]}}$) of microsphere-binder mixture in percentage for various mass ratios of water/starch in binder is given in **Figure 12** as a function of drying time prior to completion of manufacturing syntactic foams for various microsphere size groups: (a) SL75; (b) SL300; and (c) SL500. It is found that small microspheres tend to be high in shrinkage. A possible reason is that relative inter-microsphere distances ($= \frac{\text{[mean inter-microsphere distance]}}{\text{[mean microsphere diameter]}}$) are likely large for small microspheres due to the presence of starch particles between microspheres. It is also found that high starch particle content ($=$ low water/starch ratio) in a given water volume tends to be high in shrinkage. The similar reasoning would be applied to this case because the more starch particles between microspheres the higher relative inter-microsphere distance.

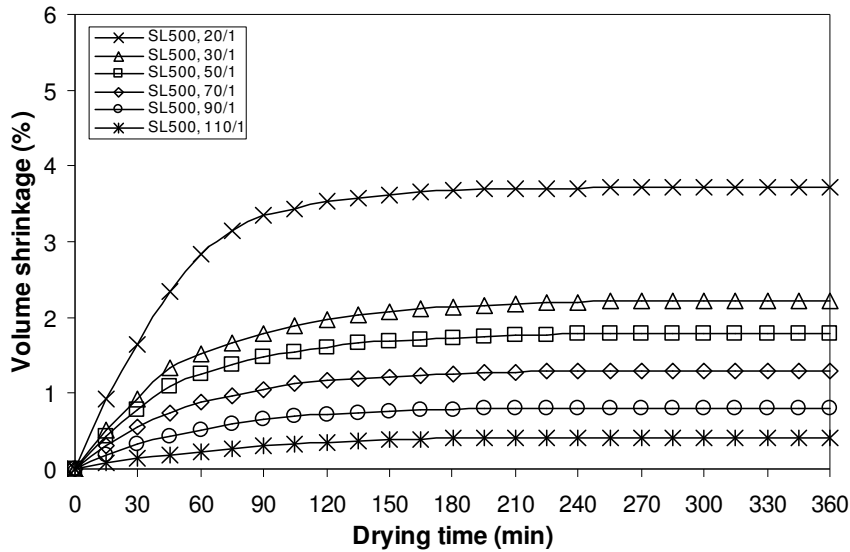
Other characteristics of manufactured foams such as volume fractions of voids, volume ratios of foam/bulk microspheres, and densities are listed in **Table 4**.



(a)



(b)



(c)

Figure 12 Shrinkage of microsphere-binder mixture versus drying time prior to completion of manufacturing of syntactic foams for various mass ratios of water/starch in binder and various microsphere size groups: (a) SL75; (b) SL300; and (c) SL500.

7. Conclusions

Post-mold processing for manufacturing syntactic foams using starch as binder has been studied and the following conclusions are drawn:

- A transition in carrying starch particles by microspheres during phase separation has been found and explained using a calculated relative density value of 1 for an agglomeration consisting of multiple starch particles and one microsphere.
- It has been found for a microsphere to attract starch particles that (a) hollow microsphere size effect is relatively high, (b) initial bulk volume of hollow microspheres (IBVMS) effect is not relatively significant, and (c) water volume effect for a given diameter of cylindrical mixing container is not noticeable.

- A Simple Cubic cell model for the starch-microsphere inter-distance has been adopted to quantitatively explain various effects on starch content in agglomeration such as hollow microsphere size, IBVMS, and water volume.
- It has been found for manufactured syntactic foams that (a) volume fraction of starch in foam is of linear relation with starch content before mixing for a given experimental data range and (b) shrinkage is relatively high for small hollow microspheres and high starch content.

Table 4 Syntactic foams manufactured.

Microsphere grade	Water/starch mass ratio	Fraction of starch in binder		Fraction of starch in foam		Fraction of microspheres in foam		Volume fraction of void in foam (v_v)	Volume ratio of foam / bulk microspheres (r_v)	Foam density (g/cc)
		Mass (m_b)	Volume (v_b)	Mass (m_s)	Volume (v_s)	Mass (m_m)	Volume (v_m)			
SL75	20/1	0.0476	0.0323	0.1639	0.0398	0.836	0.448	0.51	1.28	0.36
	30/1	0.0323	0.0217	0.1197	0.0283	0.880	0.460	0.51	1.25	0.36
	50/1	0.0196	0.0132	0.0809	0.0187	0.919	0.468	0.51	1.23	0.35
	70/1	0.0141	0.0094	0.0602	0.0137	0.940	0.471	0.52	1.22	0.34
	90/1	0.0110	0.0074	0.0421	0.0094	0.958	0.471	0.52	1.22	0.33
	110/1	0.0090	0.0060	0.0272	0.0060	0.973	0.474	0.52	1.21	0.33
SL150	20/1	0.0476	0.0323	0.1166	0.0310	0.883	0.482	0.49	1.19	0.40
	30/1	0.0323	0.0217	0.0909	0.0236	0.909	0.486	0.49	1.19	0.39
	50/1	0.0196	0.0132	0.0706	0.0181	0.929	0.489	0.49	1.18	0.38
	70/1	0.0141	0.0094	0.0530	0.0134	0.947	0.493	0.49	1.17	0.38
	90/1	0.0110	0.0074	0.0310	0.0077	0.969	0.496	0.50	1.16	0.37
	110/1	0.0090	0.0060	0.0234	0.0058	0.977	0.496	0.50	1.16	0.37
SL300	20/1	0.0476	0.0323	0.1007	0.0281	0.899	0.470	0.50	1.15	0.42
	30/1	0.0323	0.0217	0.0775	0.0211	0.923	0.472	0.51	1.14	0.41
	50/1	0.0196	0.0132	0.0421	0.0111	0.958	0.474	0.52	1.14	0.40
	70/1	0.0141	0.0094	0.0272	0.0071	0.973	0.474	0.52	1.14	0.39
	90/1	0.0110	0.0074	0.0196	0.0051	0.980	0.476	0.52	1.13	0.39
	110/1	0.0090	0.0060	0.0119	0.0031	0.988	0.477	0.52	1.13	0.39
SL500	20/1	0.0476	0.0323	0.0975	0.0233	0.903	0.364	0.61	1.10	0.36
	30/1	0.0323	0.0217	0.0775	0.0182	0.923	0.364	0.62	1.10	0.35
	50/1	0.0196	0.0132	0.0385	0.0087	0.962	0.365	0.63	1.09	0.34
	70/1	0.0141	0.0094	0.0196	0.0043	0.980	0.365	0.63	1.09	0.33
	90/1	0.0110	0.0074	0.0119	0.0026	0.988	0.366	0.63	1.09	0.33
	110/1	0.0090	0.0060	0.0119	0.0026	0.988	0.366	0.63	1.09	0.33

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Appendix

Starch particle volume fraction in top phase

$$= [\text{IBVMS}/\text{TPV}] \times [\text{Foam volume}/\text{IBVMS}] \times [\text{Starch volume in foam}/\text{Foam volume}]$$

Starch volume in foam

$$= [\text{Starch mass in foam}] / [\text{Starch particle density}]$$

$$= [\text{Foam mass} - \text{Microsphere mass}] / [\text{Starch particle density}]$$

$$= [\text{Foam mass} - \text{Microsphere bulk volume} \times \text{Microsphere bulk density}] / [\text{Starch particle density}]$$

$$= [\text{Foam mass} - \{ \text{Foam volume} / (\text{Foam volume} / \text{Microsphere bulk volume}) \} \times \text{Microsphere bulk density}] / [\text{Starch particle density}]$$

where values for (Foam volume / Microsphere bulk volume) are in a range of 1.2 - 1.3 (**Table 4**) for SL75 and SL150, and 1.1 for SL300 and SL500.

References

1. Kim, H.S. and Plubrai, P. Manufacturing and failure mechanisms of syntactic foam under compression. *Composites Part A: Applied Science and Manufacturing* 2004, 35(9), 1009-15.
2. Jize, N. N., Hiel, C. and Ishai, O. Mechanical Performance of composite sandwich beams with syntactic foam cores, R. B. Deo and C. R. Saff, (Eds), *ASTM STP 1274*, 1996, pp.125-138.
3. English, L. K. Lighter weight and lower cost with foam-core composites. *Materials Engineering*, 1987, 4, 51-54.

4. Young, K. S. Value-adding syntactic foams gain in composites applications. *Modern Plastics*, April 1985, pp.92-97.
5. Jackson, D. and Clay, P. Syntactic foam sphere improves oceanographic mooring performance. *Sea Technology*, Sep 1983, 24, 29-31.
6. Harruff , P. W. and Sandman, B. E. Carbon/epoxy composite structures for underwater pressure hull applications”, In 28th National SAMPE Symposium, Anaheim CA, April 12-14, pp.40-49,1983
7. Watkins, L. Syntactic foam buoyancy for production risers. In the Seventh International Conference on Offshore Mechanical and Artic Engineering, Houston, Texas, Feb 7-12, pp.403-410, 1988.
8. Seamark, M. J. Use of syntactic foam for subsea buoyancy. *Cellular Polymers* 1991, 10, 308-321.
9. Hinvies, J. B. and Douglas, C. D. The development of a hybrid advanced composite-syntactic foam structural component for use in undersea vehicles. *IEEE* 1993, pp.III-468 – 472.
10. Narkis M, Gerchcovich M, Puterman M, and Kenig S. Syntactic foams III. Three-phase materials produced from resin coated microballoons. *Journal of Cellular Plastics*, July/August, 1982, 230-232.

11. Narkis, M., Puterman, M. and Boneh, H. Rotational molding of thermosetting three-phase syntactic foams. *Polymer Engineering and Science* 1982, 22, 417-421.
12. Lawrence, E., Wulfsohn, D, and Pyrz, R. Microstructural characterisation of a syntactic foam. *Polymers and Polymer Composites* 2001, 9(7), 449-457.
13. Lawrence E, Pyrz R. Viscoelastic properties of polyethylene syntactic foam with polymer microballoons. *Polymers and Polymer Composites* 2001, 9(4), 227-237.
14. Verweij, H., De With, G. and Veeneman, D. Hollow glass microsphere composites : preparation and properties. *Journal of Materials Science* 1985, 20, 1069-1078.
15. Narkis, M., Puterman, M. and Kenig, S. Syntactic foams II. Preparation, and characterization of three-phase systems. *Journal of Cellular Plastics*, Nov/Dec, 1980, 326-330.
16. Puterman, M. and Narkis, M. Syntactic foams I. Preparation, structure and properties. *Journal of Cellular Plastics* July/August, 1980, 223-229.
17. Kenig, S., Raiter, I. and Narkis, M. Three-phase silicone based syntactic foams. *Journal of Cellular Plastics* Nov/Dec, 1984, 423-429.
18. Meter, C. Syntactic foam core material for composite structures, International patent classification : B29C, 65/00, B29D 9/00, B32B 3/26, 5/18.

19. Kim, H. S. and Oh, H.H. Impact behaviour of syntactic foam, In The first ACUN International Composites Meeting on Composites : Innovation and Structural Applications, 23-25 February, University of New South Wales, Sydney, Australia, 1999:83-86.
20. Kim, H. S. and Oh, H. H. Manufacturing and Impact Behavior of Syntactic Foam. Journal of Applied Polymer Science 2000, 76, 1324-1328.
21. te Nijenhuis, K., Addink, R. and van der Vegt, A. K. A study on composites of nylon-6 with hollow glass microspheres. Polymer Bulletin 1989, 21, 467-474.
22. Kim, H. S. "Syntactic foam", International Publication Number: WO 03/074598 A1, International Patent Classification: C08J 9/32, International Patent Application Number: PCT/AU03/00250, International Publication Date: 12 Sept 2003.
23. Kim, H. S. "Cenosphere composite and method for preparing same", Application date: 9 July 2004, Australian patent application No: 2004903795. PCT Patent Application No. PCT/AU2005/001009 (Title: Method of forming syntactic foams).
24. Islam, Md M. and Kim, H. S. Manufacture of syntactic foams: pre-mold processing", Materials and Manufacturing processes 2007, 22, 28-36.
25. Islam, Md M. and Kim, H. S. Novel syntactic foams made of ceramic hollow micro-spheres and starch – theory, structure and properties, In Proc ACUN-5 "Developments in composites: Advanced, Infrastructural, Natural, and Nano-composites", UNSW, Sydney, Australia, 11-14 July 2006, pp. 13-18.

26. Islam, Md M. and Kim, H. S. Novel syntactic foams made of ceramic hollow micro-spheres and starch – theory, structure and properties. *Journal of Materials Science*, 2007, 42, 6123-6132.

27. Thompson, W. CSR Building Products Limited, NSW, Australia, Private communication, 2005.

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