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Failure Mechanism of Two-Phase Syntactic Foam Polymer Composites under Different Coating Conditions

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Abstract: In this study, the impact of coated hollow microsphere (HMs) on syntactic foams (SFs) failure analysis is performed. The HMs was made from polystyrene beads and were coated with $CaCO_3$ and cementite powder to produce the SFs by Vacuum Assisted Mould Fill Technique (VAMFT). The morphological analysis was performed using Scanning Electron Microscope (SEM) and the compressive test conducted using INSTRON 5982 machine model. From the results, the HMs failure mechanism showed the cementite surface coating enhanced the shell-rigidity by 65%. The physical and compressive properties of the SFs increased with increase in % vol. fraction of the HMs. The SFs compressive modulus, flexural strength and tensile strength for the $CaCO_3$ and cementite specimen are 470, 44, 36 MPa and 582, 48, 41 MPa, respectively. The energy absorption characteristic of the SFs was enhanced by 14% and 25% for the $CaCO_3$ and cementite HMs surface coating, respectively. But the optimal concentration for the SFs was 20% vol. weight for both HMs coating.

Keywords: Hollow microspheres, compressive stress, flexural stress, syntactic foam, and mechanical properties

1. Introduction

Here small spherical particles with diameter in the micrometer range (typically 1 to 1000 μm) are commonly referred to as microspheres. And in some cases, micro-particles [1-3]. Microspheres can be fabricated from different types of natural and synthetic materials or even inorganic materials. Cases of fabrication of microspheres from commercially available ceramics microspheres, glass microsphere, or polymers microsphere have been reported in literatures [2, 4-6]. Depending on the technique, solid or hollow microspheres are widely used in density and, therefore, are used for different or specific intended applications [7-9]. Hollow microspheres (HMs) offers superior properties and diverse range of application in engineering technology. And are typically used as additives to lower the density of material. There are numerous applications of microspheres depending on what material they are produced from and what size they are and there usage spawn across aerospace, marine underwater vessels, ground vehicles etc. The two very common types of polymeric microspheres are the polyethylene and polystyrene. Polystyrene microspheres present a flexible platform for applications and can be coated to enhance the shell-wall properties. Hollow microsphere from polystyrene have been studied widely for fabrication of syntactic foams (SFs) because of the advantage properties [10].

Studies on incorporation of microspheres into matrices to produce lightweight material (foams) have been reported in literatures [7, 11-13]. Composite materials produced by embedding porous particles in a matrix medium can be referred to SFs. Researchers studied SFs because of the low density, superior compressive strength, acoustic, thermal damping and electromechanical properties [14-16]. SFs have been used for buoyancy in subsurface and deep-sea applications, sandwich structures for aerospace, aviation and transportation, thermal insulation materials for oil and gas industries and other applications [17-23]. Also, matrix types, matrix formulation and SFs performance have been studied widely [24-26], while filler types [26-30], porous microsphere types [22, 31-33] and method of production [30,

34, 35]. SFs compressive properties, modulus elasticity, flexural strength, tensile strength, thermo-insulation, thermochemical and sound properties have been enhanced by manipulating various factors [36-38]. Although, there have been considerable progress achieved in the study of SFs application [18, 39, 40]. But there are few research endevoiur on the types and effect of microsphere surface coatings on the SFs [41]. Surface coating of microsphere considerable influence SFs performance [40, 42, 43]. However, more investigation on suitable coating materials - surface coating types, coating size, coating thickness, coating treatment, coating homogenousity and methods for the microsphere used inn fabrication of SFs in order to improve thermomechanical, sound acoustic and insulation properties. In view of the shortage in literature this study evaluates the effects of surface coating on SFs incorporated microsphere for acoustical applications.

2. Material and characterization

2.1 List of materials

All Expandable polystyrene beads and epoxy resin based diglycidyl ether of Bisphenol and epoxy hardener clear was purchased from Euro Chemo Pharma Sdn, Bhd, Pinang, Malaysia. The equivalent weight of the epoxy resin weight is 188 g/eq, and physical properties: viscosity 13456 mPa/s; density 1.16 g/ml; and weight 9.7 lbs. /gal). The chemical composition proportion is 5-11 Trimethylhexamethylene diamine (TMD), 30-42 Isophorone diamine, 29-41 benzyl alcohol. And the equivalent weight per H active is 95 g/eq, Amine value 278 mg KOH/g, viscosity 36, density 1.0 g/ml and gel time at 25˚C is 32 mins at ration 1:1.

2.2 Fabrication coated microsphere and matrix formulation

The stoichiometry ratio used for the matrices was 1:1 epoxy resin clear and epoxy hardener clear. The expandable polystyrene beads size ranges between 1–5 mm. The epoxy resin was poured inside aluminium bowl, and polystyrene beads were added at apportioned quantity into it and mixed thoroughly till the outer surface of the beads are fully wetted. Thereafter, the wetted beads were poured onto a tray of dried $CaCO_3$ powder to form shell coating. The excess powders were removed from the bead to prevent agglomerating. The coated beads were cured inside oven at 60˚C for 30 minutes. Similar procedure was repeated for the cementite powder coatings.

2.3 Fabrication of microsphere

Post curing of the coated beads was done by putting the coated beads inside oven for another 90 mins at 120˚C to allow the shrinking process of the beads - creating intended inner hollow structure. Thereafter, the resultant cured coated beads – hollow microsphere (HM) were sieved to remove excess coatings on their surface. Table 1 presents the physical properties of the microsphere studied.

Table 1- Physical properties of microsphere

Types of microsphere /surface coating	Density (kg/m^3)	Exa Pressure (MPa)	Average diameter	Average thickness	Thickness to–radius-
Calcium carbonate coated	0.6431	3.67	1.8	1.15	0.042
Cementite coated microspheres	0.7234	4.12	2.0	1.23	0.038

2.4 Fabrication syntactic form

The SFs was produced using epoxy resin as binder and hollow microsphere as fillers. The fabrication was done by Vacuum Assisted Mould Filled Techniques (VAMFT). Teflon mould was filled up with cured HMs. Then after, the matrix mixture was gradually poured into the mould and simultaneously expelling the air inside the mould by vacuuming. The mould was air tight with silicon grease. Natural curing of the SFs occurred through 5 days, before the mould was opened and the SFs exposed. Figure 1 shows the photo-image of the cut-size SFs and the cured coated HMs.

2.5 Microstructure Analysis

Morphology of the epoxy HMs was viewed by Hitachi S-3000 N SEM imaging. The imaging captures the morphological alignment of the HMs in the SFs. Several images of the SFs were captured with varying magnifications and sizes, however, only the discussion was mainly focused on one magnification and concentration of HMs.

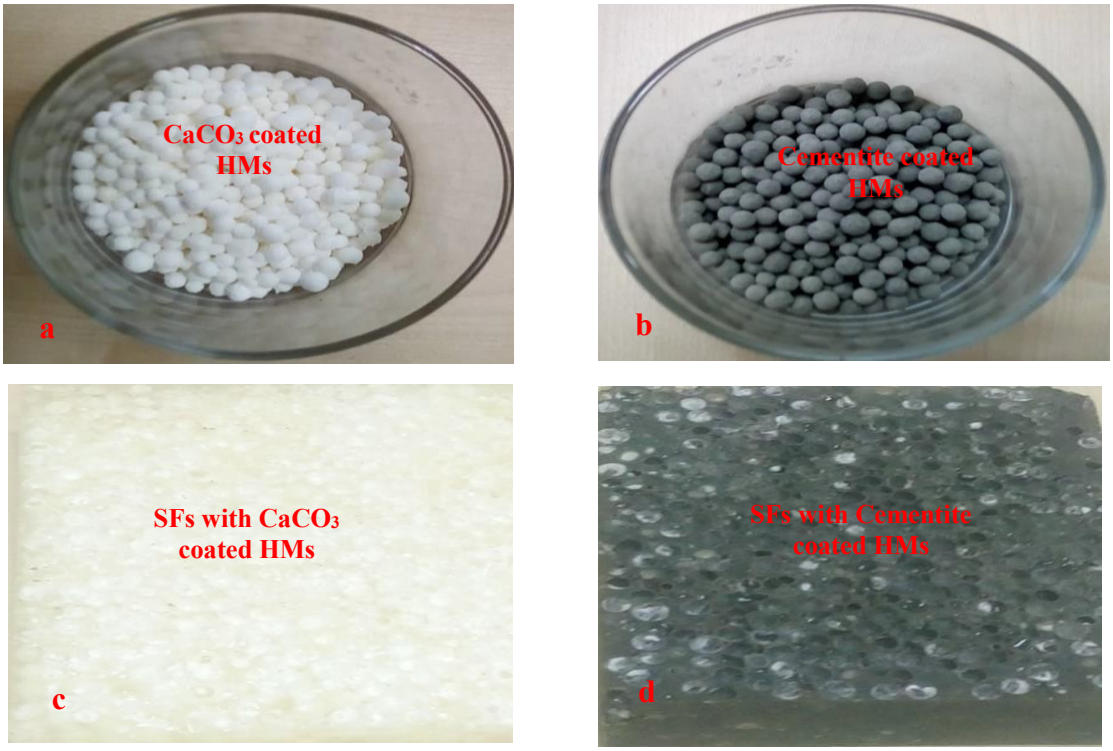

Fig. 1- Photo-image of different hollow microsphere (HMs) surface coating and Syntactic foams (SFs) (a) $CaCO_3$ coated HMs; (b) Cementite coated HMs; (c) SFs with $CaCO_3$ HMs; (d) SFs with Cementite HMs

2.5 Compression test

ASTM D3575 standard was adopted for the compression test. The specimen dimension is 50x50x25 mm. The test was conducted in triplicate every batch of the epoxy SFs for accuracy of results. The test was conducted at room temperature using a universal testing machine model INSTRON 5982 at constant cross-head speed of 5 mm/mins at 60% strain between two parallel flat plates.

3. Result and discussion

3.1 Microsphere and crushing mechanism analysis

The compressive stress – strain curve is shown in Figure 2. The Matrix formulation is known to impact the macromolecular structure of the microspheres thus influencing the microspheres performance [40, 44].

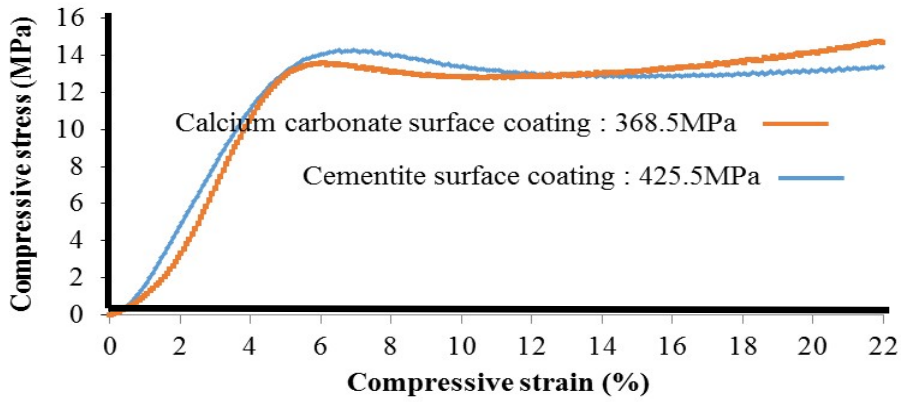

Fig. 2 - Stress-strain curve for hollow microsphere with different surfaces coating: (a) Calcium carbonate coating and (b) Cementite coatings

Analysis of crushing mechanism on HMs shows resistance towards compression load because both the matrix and HMs strength was combined in yielding the foam's compressive deformation response. The coated HMs with cementite exhibited higher compressive modulus (425.5 MPa) than the epoxy HMs coated with CaCO3 (368.5 MPa). Their curves pattern is linear-elastic which is hypothetical of stress-strain curve analysis. Characteristic behavior of the epoxy HMs for both coating surfaces under compressive testing was visualized using the Stereo Zoom microscope.

Figure 3 and 4 describes the fracture propagation of the CaCO3 and cementite coated HMs, respectively. The failure propagation stages of the microsphere is captured on image camera and presented in Figure 3. The arrow shows the captured images as the crush is been impacted. The microsphere crushing strength impacts the compressive strength of foam. However, have less contribution when in the deformation was in flexural mode, [19, 45-47]. For example, in a resin rich matrix, the excess number of epoxy resin monomer that is unreactive avoids a complete crosslink thereby causing a weak polymer structure formation.

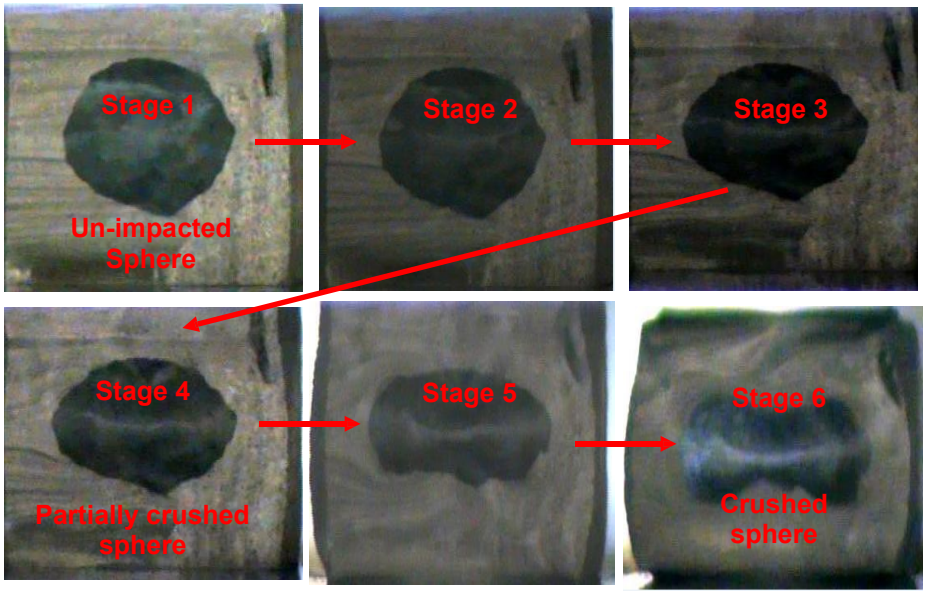

Fig. 3 - Failure propagation image captured during compression testing of CaCO3 coated HMs

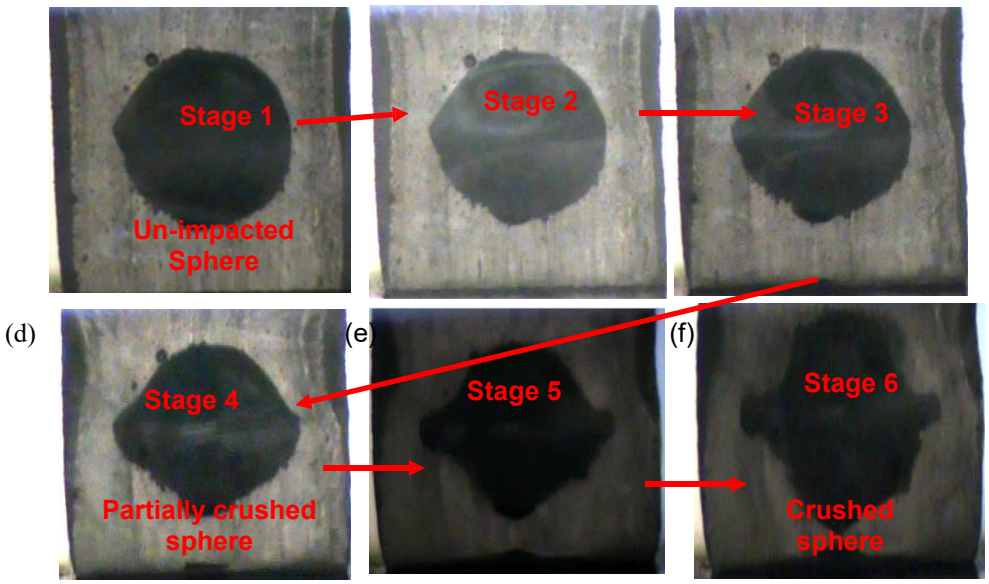

Fig. 4 - Failure propagation Image captured during compression testing of Cementite coated HMs

At weak structure of epoxy resin, cracks initiates throughout the system and primarily point of cracks occurs at excess epoxy spot identified as voids thus contributes to the low deformation capacity of overall system. The crushing behaviour of the $CaCO_3$ coated HMs was brittle and protrudes during compression. Noticeable crack formation which are failure concentration points are observed as shown in Figure 3. This implies that the surface coating initiated

minimized crack initiation does enhancing the compressive strength of the epoxy HMs. The Figure 4 presents the capture images showing the failure mechanics for the cementite coated surface microsphere. The cementite coating coated HMs was more effective and unbroken because of the presence of substantial number of active amine group which exhibit stronger shell, structural rigidity and stronger interfacial bond between HMs and epoxy matrix. Whereas, the $CaCO_3$ coated epoxy HMs, the surface exhibited brittleness because of formation of rigid macromolecular structure. During compressive testing, the HMs cracked and creates void which leads to stress concentration points. Overall compressive strength rely on contribution strength of the individual HMs. Microspheres of averagely even sizes, and coated surfaces can contribute to elongation of the sphere.

Figure 5 and 6 describes the failure propagation under compressive test for both coated surfaces using the edgewise and flatwise orientation. From Figure 5, It can be observed that the edgewise compression is more impacted upon than the flatwise orientation for the calcium carbonate coated HMs. The modulus for the flatwise and edgewise was 42 MPa and 26 MPa, respectively.

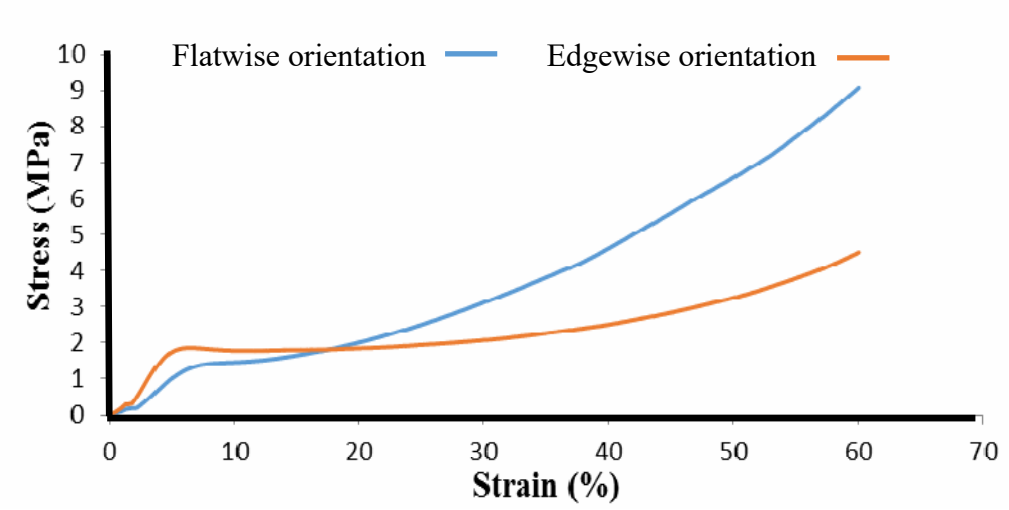

Fig. 5 - Stress-strain curve for calcium carbonate coated HMs under different failure propagation orientation

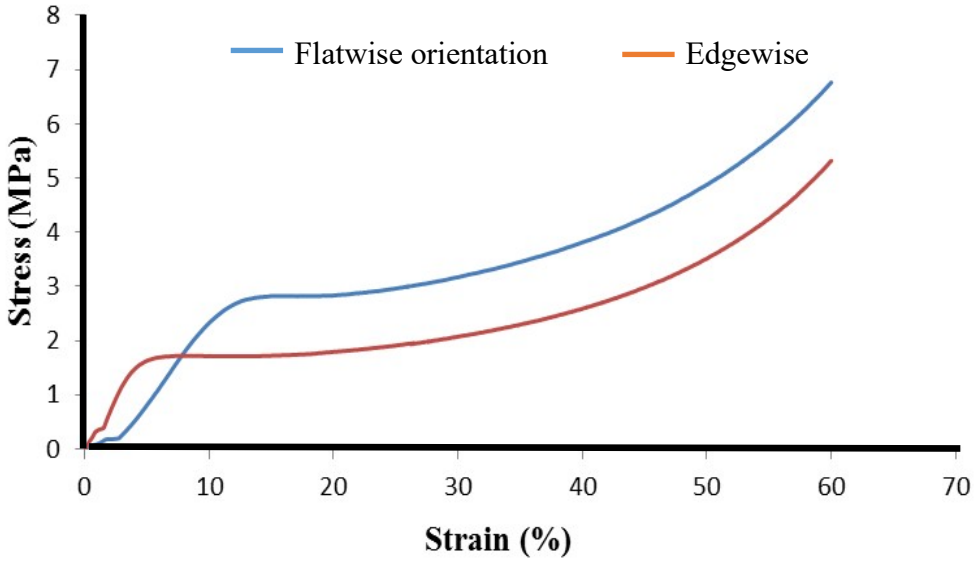

Fig. 6 - Stress-strain curve for cementite coated HMs under different failure propagation orientation

From Figure 6 the cementite coated HMs, the modulus for the edgewise and flatwise was 46 MPa and 29 MPa, respectively. The difference in their modulus can be traced to the barrier the coating provided to the HMs. whereas the cementite coated surface with relatively higher modulus from both failure propagation orientation is because of the yield elasticity provided to the microsphere shell.

3.2 Syntactic foams (SFs)

Figure 7 describe the SFs compressive curve. The position of the curves exhibits identical cross-linking because of the microspheres. However, the slight variation can be traced to the coating effects. The coated SFs compressive modulus was enhanced by 45% compared to the uncoated epoxy SFs. Similar findings have been reported in the works of many researchers [46, 47]. Thus implies that the mechanical properties of SFs had obvious enhancement by adding external surface coatings to the microspheres and the matrix. However, the ESF coated with cementite showed higher modulus (582 MPa) than the epoxy SFs coated calcium carbonate with modulus (470 MPa). This can be traced to the effect of the surface coating material. The two coating have identical microsphere size and thickness.

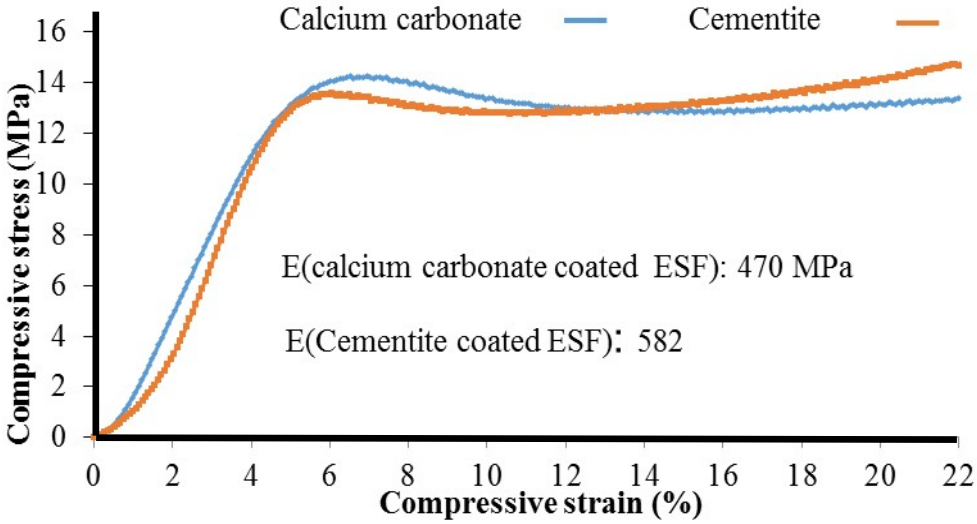

Fig. 7 Compressive Stress-strain curve comparison of calcium carbonate coated SFs and cementite coated SFs

Table 2 shows the specific tensile strength and specific flexural strength against percentage volume concentration. Literature reports shows that the flexural strength, tensile strength and elastic modulus are enhanced by the filler [48, 49]. In the case of the calcium carbonate shell coating, the specific tensile strength increases and the specific flexural strength decreases with increase in volume percentage (%) of the epoxy HMs. Whereas, in the case of the cementite shell coating, the specific tensile strength increased and specific flexural strength decrease with increase in the epoxy HMs volume percentage (%). For both shell coatings at 20% volume concentration of epoxy HMs demonstrated the highest specific tensile strength of 44 MPa and 48 MPa for calcium carbonate and cementite epoxy SFs respectively. Whilst the lowest specific flexural strength was recorded at 50% volume percentage of epoxy HMS for both shell coating on the epoxy SFs.

Table 2 - Comparison of the compressive strength properties of SFs against weight volume (%)

Epoxy Syntactic Foam Shell Coatings	Specific Tensile Strength (MPa)	Specific Flexural Strength	Epoxy Hollow Microsphe
Calcium carbonate	2	7	10
	4	5	20
	3	4	30
	3	4	40
	3	3	50
Cementite	2	7	10
	4	6	20
	4	5	30
	3	5	40
	3	4	50

However, 41% and 49% reduction in the flexural strength property of the epoxy SFs was recorded for the calcium carbonate and cementite shell coating. Thus, external surface coatings can enhance the strength of composite, however,

the enhancement in the cementite coating was more because of the crosslink formed with the matrix-resin and the increased content of the epoxy HMs. Overall, the epoxy HMs shell coating contributes to enhancement of compressive properties of epoxy SFs. However, the respectful contribution is based on the shell coating material type, coating thickness, and load concentration in the epoxy SFs. The cementite surface coating improved the compressive properties of the epoxy SFs more than the calcium carbonate coating in most retrospect examined.

3.3 Microstructure Analysis

Figure 8 presents the SEM images for both the epoxy SFs from calcium carbonate and cementite shell coating surfaces. From Figure 8a, the epoxy HMs are closely-packed with noticeable deformation in the epoxy HMs from the original spherical shape, though in some cases. Also, the particle concentration increases and volume of matrix resin decreases with increasing epoxy HMS loading, though as expected. The microstructure suggests the rigidity and toughness of the coating affect the external surfaces of the epoxy HMs unlike the shape and size that evolves during the composite fabrication process.

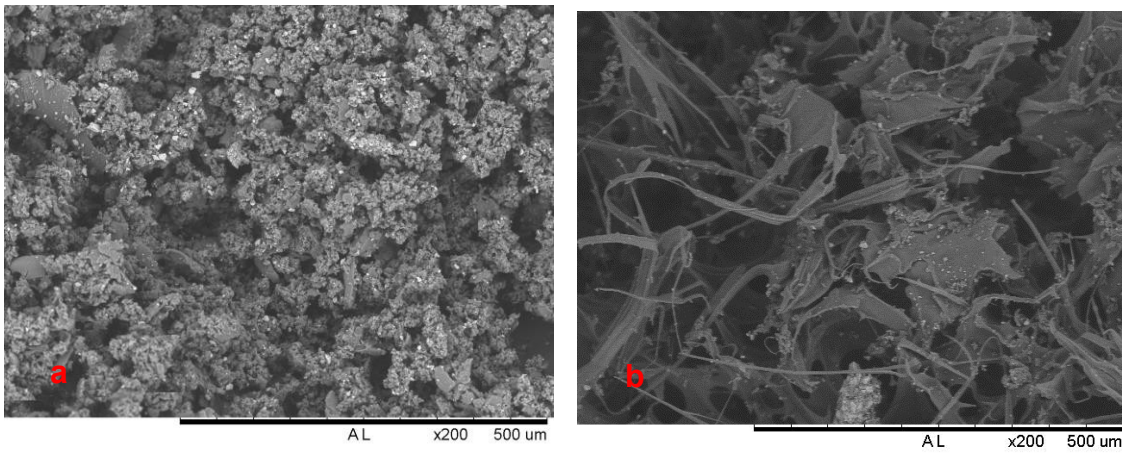

Figure 8: SEM microstructural images for epoxy syntactic foam (SFs).
(a) Calcium carbonated coated surface and (b) Cementite coated surface.

The moduli and fracture properties often improve with increased solid filler content, given an intrinsically brittle matrix system and good interfacial bonding between the filler and the matrix [50, 51]. Herein, both surface coatings provided enhanced interfacial bonding between the matrix and the filler, but the calcium carbonate coated surfaces was much smoother and homogenous than the cementite because of the fine particle size and density. Average of 50 samples of the epoxy HMS were used to determine the density of the syntactic foam.

4. Conclusion

The theme of the study focuses on the evaluations of the contribution of different HMs surface coatings on SFs physical and mechanical properties. And the SFs failure mechanism analysis from edgewise and flatwise orientation upon loading systems. The experimental characterization of the HMs and SFs was carried out using scanning electron microscopy (SEM) for the morphology of the SFs and HMs internal structure and the compressive strength was carried out with compression test machine. From the results the HMs wall coating were observe to enhance the structural rigidity, toughness (malleability) and compressive strength of the SFs. However, the cementite wall coating SFs exhibited the high modulus but low density when compared with $CaCO_3$ wall coating SFs and plain SFs. This light-weight SFs can be used in various acoustical and mechanical engineering applications especially where impact assessment and light weight applications are highly required. And where materials are exposed to repetitive compressive and impact events because of the advantageous and tailored properties.

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