Applied Chemical Engineering (2025) Volume 8 Issue 2 ISSN: 2578-2010 (O)
doi: 10.59429/ace.v8i2.5689

ORIGINAL RESEARCH ARTICLE

Analysis of reinforcement syntactic foam composite using K-15 microballoons

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ABSTRACT

Syntactic foam composites are used in the aerospace industry, one of which is for buoys because they are lightweight materials that have flexural strength and are resistant to corrosion. This study aims to determine the flexural properties and morphologies structure of syntactic foam composite with K-15 microballoons filler mixed by epoxy resin. Syntactic foam is a lightweight material used in the maritime sector for buoys to make them float easily in water. Variations of K-15 microballoons of 10%, 20%, 30%, 40% and 50% volume of syntactic foam composite. Flexural strength determinated using ASTM D790 and morphologie structure determined with scanning electron microscope (SEM). The addition of X-15 microballoons can reduce flexural strength compared to pure epoxy. Flexural strength in pure epoxy in the form of stress, elastic modulus, and strain each produces values of 71.28, 2928.47 MPa, 2.75 MPa. The highest impact is seen in the addition of 50% K-15 microballoons with stress, elastic modulus, strain and density values of 18.59, 1406.03 MPa, 1.43 MPa and 0.63 kgm-3 respectively. SEM analysis shows that plain epoxy has a smooth surface without voids, whereas with the addition of K-15 microballoons, voids appear on the surface of the syntactic foam composite.

Keywords: density; flexural; K-15 microballons; syntactic foam composite

ARTICLE INFO

Received: 26 June 2025
Accepted: 18 July 2025
Available online: 28 July 2025

1. Introduction

Syntactic foam composites are widely used in the maritime and aerospace sectors, one of which is for buoys. Syntactic foam composite materials have light buoyancy properties and low water absorption[1]. Syntactic foam composites exhibit superior resistance to extreme conditions, such as moisture and prolonged water exposure, when compared to bio-based composites. Bio-based composites, on the other hand, are more susceptible to biological degradation and the effects of moisture, which can lead to a decline in material strength or durability over time. Additionally, they are more prone to breakdown or damage caused by microbial activity, in contrast to synthetic materials[2]. Syntactic foam composites are materials consisting of a combination of matrix and filler. The matrix can be polymer, metal or ceramic. Fillers can be cenospheres, hollow spheres, solid spheres, hollow glass microspheres which in this study used K-15 microballons[3]. Syntactic foam composite layers are made by mixing epoxy resin with K-15 microballoons. The use of microballoons is due to their tensile strength, elastic modulus, strain, light weight and corrosion resistance. The utilization of microballoons that are lightweight yet strong can reduce the thickness and amount of resin required in the production of composites. This not only reduces the consumption of raw materials but also lowers energy consumption during the molding or forming process, as less energy is needed to shape lighter materials. Microspheres are uniformly dispersed in a resin system to create syntactic foams. The resin serves as the matrix material, while the microsphere serves as the filler. Syntactic foams are hence two-phase structures[4]. A certain amount of air is trapped during manufacturing. throughout the matrix and appears in the finished structure as voids. Syntactic foams are three-phase structures because of this air rapped, which functions as a third phase[4]. The schematics below illustrate this (**Figure 1**).

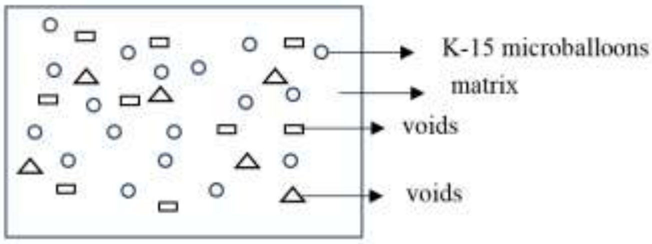

Figure 1. Syntactic foam composite structure.

Research related to syntactic foam composites has been conducted but there are still problems in maintaining strength so that the composite can withstand impacts and withstand loads[4]. So that research is still needed to analyze the performance of syntactic foam composites. Research conducted by Ullas et al. (2020) showed that the flexural strength and fracture toughness of syntactic foam composites with hollow glass microspheres can be reinforced with carbon nano fibers up to 1.5% volume[5]. A study of increasing flexural strength was also conducted by adding nano clay of 2% volume and 4% volume. Syntactic foam composites with mixed epoxy resin matrix and hollow glass microspheres showed an increase in flexural strength compared to plain epoxy[6]. Comparison of the use of hollow glass microspheres and nano carbon in syntactic foam composites conducted by Afolabi et al. (2020) showed that there was energy absorbed up to an increase in the volume fraction of 50-72.2% by volume when using hollow glass fillers. Other studies also stated that the addition of hollow glass microspheres had an impact on increasing the compressive and flexural properties of syntactic foam composites compared to plain syntactic foam composites[7]. From the existing research, the use of the right filler is very important but there has not been much research related to fillers in syntactic foam composites. This research, the use of fillers from hollow glass microspheres, especially K-15 microballoons, was analyzed. In the context of applying syntactic foam composites to ship floaters or submarine structures, achieving both high buoyancy and low density is crucial. Therefore, engineering the constituent components of the composite is essential to achieve the desired properties[8]. One significant parameter often overlooked in syntactic foam composite studies is flexural strength analysis. Flexural strength data determines the maximum strength of the composite before it undergoes failure (static deformation). Flexural analysis also provides critical information on material stress, flexibility, and elastic modulus when syntactic foam composite is used as a core in sandwich composites[9]. Based on the need to enhance structural strength and the importance of flexural analysis in understanding the material's flexibility properties, our focus will be on achieving a balance between lightweight properties and mechanical strength in composites. This endeavor has led to the development of various syntactic foam composites that are lightweight yet exhibit remarkable strength. The independent variable used will be the ratio of microsphere additions ranging from 10% to 50% v/v. Flexural analysis will evaluate material flexibility by applying a flexural load of 50 kN at the material's equilibrium point. The causes and points of fracture in flexural analysis can be observed qualitatively through surface morphology using SEM (Scanning Electron Microscopy). SEM analysis will reinforce data related to morphology, particle-matrix interactions, distribution of filler particles in the matrix, and their good dispersion within the composite.

2. Materials and methods

2.1. Material

This research utilized materials such as epoxy epocshon Bisphenol A Epychlorohydrin, Hardener EPH 555 Cycloaliphatic Amine, K-15 Microballoons, and acetone as the cleaning solvent. The instruments employed included Digital Scale, Stirrer, SEM JEOL JSM (Scanning Electron Microscope) - EDS, ASTM D790 Specimen Molding Tool, and a 50 kN UTM Machine.

2.2. Syntactic foam composite fabrication

The syntactic foam composite specimen is a small piece used for mechanical property testing. Flexural testing of syntactic foam composite follows the specimen size according to ASTM-D790 standard. The fabrication of syntactic foam composite specimens begins with weighing porous materials (K-15 microballons), epoxy resin epocshon A, and epoxy hardener EPH 555 using a digital scale, weighing the three materials according to the desired ratio. Mix K-15 microballoons with epoxy resin epocshon A at 100 rpm for 10 minutes. Add epoxy hardener, mix at 100 rpm for 4 minutes. Ensure the three materials are mixed homogeneously, then proceed with the molding process. Pour the mixture into the mold, avoid voids (air bubbles), and prevent spillage from the mold[10].

2.3. Curing and conditioning process

The curing process is carried out by allowing the composite mixture to sit in the mold for 24 hours for curing. The curing process aims to harden and bond the resin matrix with K-15 microballoons (polymerization reaction). In this study, curing occurs at room temperature, involving natural curing reaction. Conditioning process is performed by releasing the syntactic foam composite specimen from the mold and placing it into a conditioning chamber, conditioning process using operation (23°C ± 2°C, 50 %RH ± 5 %, ± 48 hour). The conditioning process is a process of environmental conditioning for stabilization (temperature and humidity control) of specimens, achieving thermal balance, moisture with the environment. During this process, specimens will absorb moisture, thereby enhancing the mechanical properties of syntactic foam composites[11].

2.4. Flexural test

Flexural testing of syntactic foam composites is conducted according to ASTM D790 to assess the flexural strength of syntactic foam composites. Before conducting the flexural test, record the tool usage logbook, ensure proper calibration of the equipment, and read and understand the operation of the UTM Shimadzu 50 kN equipment. First, prepare the syntactic foam composite specimens, smooth the surface of the syntactic foam composite specimens, and measure the volume of each specimen using a caliper. Once the composite is ready, turn on the UTM Shimadzu 50 kN equipment. Prepare the flexural load and place it on two support points according to the span-to-depth ratio of 16:1[12]. The radius nose placement should not exceed 4 times the specimen depth. The load is applied at the mid-point of the specimen, as this is where the specimen experiences maximum deflection during bending, thus the flexural test will measure flexural properties including flexural strength, modulus of elasticity, and strain strength[13].

The length of the specimen is kept constant at 64 mm after measuring the sample volume (length, width, height). Insert each syntactic foam composite specimen one by one into the flexural testing machine, place it on the supports, and ensure that the flexural load is precisely centered on the syntactic foam composite specimen. Turn the knob, ensuring the load starts from 50 kN, and press start. The flexural load will then compress the composite until it fractures. Next, observe the graph and table on the operator's screen where the data represents the flexural strength, modulus of elasticity, and flexural strain test results. Repeat the same procedure for each specimen in each variation; in this study, each variation uses 5 specimens to ensure data validity. Then print the graph and table, and save the analysis results as primary research data[14].

2.5. SEM-EDS morphological analysis

Fracture analysis of syntactic foam composites is necessary to evaluate the microballoon structure and resin matrix. To understand the structure and distribution of the composite, surface analysis of samples with high resolution is conducted using a Scanning Electron Microscope (SEM)[15]. SEM Morphological Analysis provides qualitative images of filler particle distribution, pores, and particle-matrix interactions. The steps in conducting SEM morphology analysis begin with sample preparation: selecting the desired section, cleaning it from contaminants, and adjusting humidity[16].

The sample surface to be analyzed is thinly coated with Ag/Au metal using sputtering or vacuum coating techniques. Coating serves to reduce electrical charge on the sample surface and enhance electrical conductivity. Next, the sample is placed in the SEM chamber and subjected to an electrical voltage. Excited electrons are directed onto the sample surface and interact with it. The generated electron signals are used to produce high-resolution morphological images. Sample observation focuses on identifying surface areas representing resin matrix components, microballoons, and other constituents. Surface sample analysis is interpreted to understand surface structure and syntactic foam composite morphology. SEM morphological images provide data for analyzing filler particle distribution, particle-matrix interactions, pores, and material damage[17].

3. Result and discussion

Flexural composite analysis yields three data points: mechanical properties of the material, namely maximum flexural stress, strain, and elastic modulus at a specific point. Flexural property data are obtained from each specimen after the composite material fractures. Flexural testing of syntactic foam composites was conducted using ASTM D790 method to measure the strength and stiffness of a material under load that induces bending and deformation[18].

3.1. Stress

Stress is the highest stress experienced by a material before failure or reaching a certain limit, and serves to evaluate the material's ability to withstand applied loads before static failure. In **Figure 2**, the highest flexural strength is observed at 70 MPa for the 0% K-15 microballoons specimen, whereas the 50% K-15 microballoons specimen shows a flexural strength of 18.59. The graph above indicates that increasing the amount of K-15 microballons decreases the flexural strength value of the specimen. Data from the stress analysis can be graphed for each mechanical property.

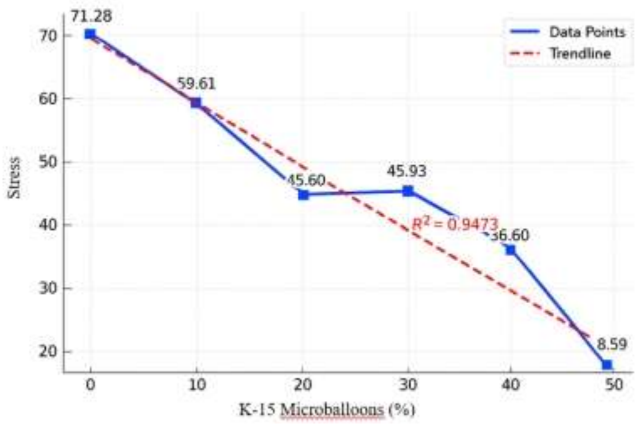

Figure 2. K-15 Microballoons vs Stress.

Based on **Figure 2**, Factors such as specimen preparation, microsphere strength, and the presence of voids contribute to the decrease in strength values of syntactic foam composites. Imperfect material mixing introduces voids (empty spaces between matrix and resin), which become points of composite fracture during flexural testing. This phenomen indicates that adding K-15 microballoons foam results in uneven filler distribution within the composite matrix. This uneven distribution can create areas with low filler concentration, thereby reducing the flexural strength of the composite[19]. The graph illustrates the relationship between the variation in K-15 microballoons percentage and the maximum stress (MPa). The data shows a general downward trend, indicating that increasing the K-15 microballoons percentage reduces the maximum stress. A linear regression analysis was performed, yielding an R^2-value of 0.9473, which signifies a strong correlation between the variables. This result suggests that approximately 94.73% of the variation in maximum stress can be explained by changes in the K-15 microballoons percentage. The trendline confirms the consistent decrease in material strength as the K-15 microballoons content increases, potentially due to the weakening of the material's structural integrity. This result is consistent with the findings of Swetha et al. (2022), where a 69% increase in flexural strength was observed at a 5% microballoon volume fraction. However, when the filler volume reached 25%, a significant decrease in flexural strength was noted[20].

3.2. Strain

Strain analysis aims to evaluate the composite's ability to withstand maximum deformation under load. In flexural testing of syntactic foam composites, max strain refers to the maximum strain occurring before the sample reaches its elastic limit, where strain is measured as the relative elongation from its original length. The graph illustrating the influence of microballoons addition on max strain can be seen in **Figure 3**.

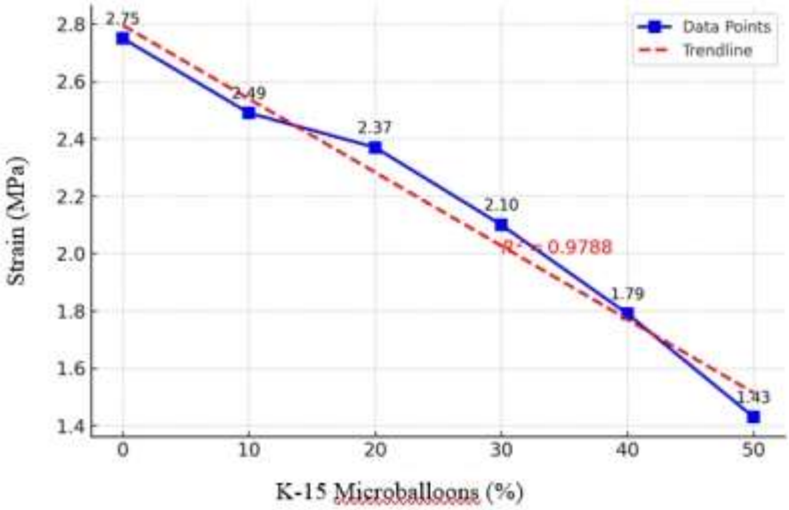

Figure 3. Microballoons K-15% vs Strain.

In **Figure 3**, the highest strain value is observed in the syntactic foam composite with 0% K-15 microballoons variation, having an elastic modulus of 2.75 MPa, and the lowest is in the 50% microsphere variation at 1.43. In the 20% microsphere variation, there is a slight decrease in flexural elastic modulus with a value of 2.37. **Figure 3** shows a good linear trend with a regression value of 0.97. The addition of K-15 microballons to syntactic foam composites can decrease max strain due to the thin-walled and hollow structure K-15 microballoons, which results in lower strength and elastic deformation compared to the resin matrix. Loading on syntactic foam composites is distributed between the resin matrix and K-15 microballoons, causing a decrease in the elastic deformation value of syntactic foam composites[21]. The results of the maximum strain decrease trend graph are in line with the research conducted by Wang et al. (2021), which showed an increase in the volume fraction of K-15 microballoons from 30% to 60%. The linear regression of 0.9788 indicates that

97.88% of K-15 microballoons are able to withstand strain effectively. This is due to the reduction in cohesion in the matrix[22].

3.3. Elastic modulus

Elastic modulus analysis measures the elongation of the composite during elastic deformation (ability to return to its original shape) and is quantified as a relative change in length from the initial material length. The value of 0.05 N/mm² refers to the load pressure per unit, while 0.25% indicates the percentage of elastic strain relative to the initial sample length. The graph illustrating the influence of K-15 microballoons addition on flexural elastic modulus can be seen in **Figure 4**.

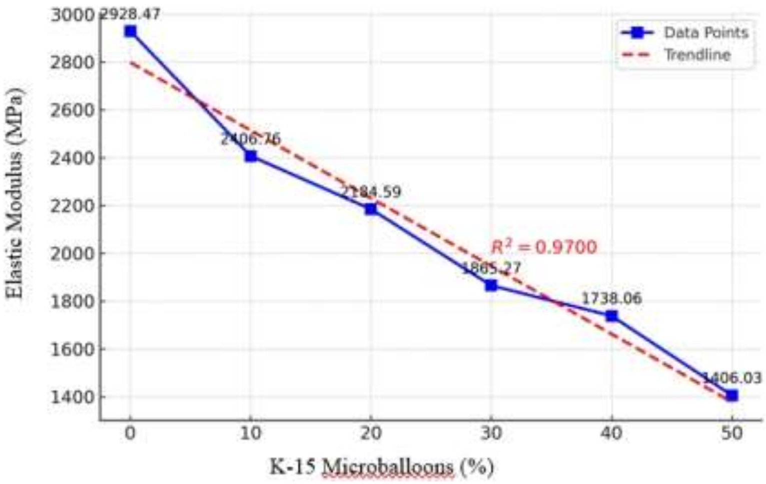

Figure 4. Microballoons K-15 (%) vs Elastic modulus.

Figure 4 illustrated that elastic modulus testing of 0.05 N/mm² - 0.25% on syntactic foam composites was conducted to understand the deformability (shape change) of the test specimen under flexural load. The graph above shows that increasing the addition of K-15 microballoons can decrease the value of the elastic modulus. The decrease in elastic modulus is attributed to the characteristics of microspheres, including geometric structure, stiffness, density, and low strength. According to Hooke's law, the decrease in stress in syntactic foam composites results in a reduction in elastic deformation[23]. The elastic modulus vastly decreases with increasing amount of K-15 microballoon. This is supported by a strong linear regression that yields an R^2 of 0.9700, meaning that changes in the K-15 microballoons share contribute to 97% of the varying elastic deformation. This trend suggests that the addition of K-15 microballoons affects the deforming resistance of the composite, probably due to the variations of the microstructures and stress distributions of the material[24]. This result is consistent with the findings of Patil et al. (2020), where an increase of 27.16% in specific flexural modulus and 27.7% in flexural modulus was observed with 50% filler content[25].

3.4. Fractures of syntactic foam composites

In every K-15 microballoons addition variation, the flexural study revealed syntactic foam composite cracks. The previous graph shows that when the amount of K-15 microballoons increases, the strain reduces. This illustration shows that as stress is applied, the material's ability to undergo deformation decreases. The reduction is probably the result of adding stiff filler particles, which make the matrix less flexible and more rigid. Maximum stress data showed a similar pattern, with a higher filler content decreasing the material's capacity to support heavier loads. Weaker interfaces between the filler and matrix are thought to be the cause of this reduction, which causes stress concentration and early failure. The material characteristics exhibit notable deterioration at higher percentages (40% (4) and 50% (5)), indicating that an excessive amount of filler compromises the matrix's structural cohesiveness. The observed mechanical weakness is most likely due to voids or poor bonds between the filler and matrix. Higher filler content impairs mechanical qualities even if it may lower material density and total weight. When creating materials for applications that demand particular strength-to-weight ratios, this trade-off needs to be carefully taken into account. More particle surfaces are introduced into the matrix as the filler % rises, which concentrates stress and makes it harder to distribute loads uniformly. This explains why strain and stress capability consistently decrease as filler quantity rises. **Figure 5** shows the syntactic foam composite fractures from flexural testing.

Figure 5. Fracture analysis of syntactic foam composite flexural testing.

The developments in material properties highlight the significance of optimizing composite design. Although the maximum stress and elasticity are reduced by adding more filler, there may be benefits like as weight or cost savings. For practical applications, it is crucial to find the optimal filler-to-matrix ratio that balances mechanical performance with other desired attributes, including thermal resistance or lightweight design. These findings are in line with material science studies that emphasize the importance of composite structure, filler dispersion, and interface bonding in achieving the desired mechanical properties[26].

3.5. Morphology structure

The morphology analysis of the syntactic foam composite fracture using SEM-EDS at 100x magnification is displayed in the **Figure 6**.

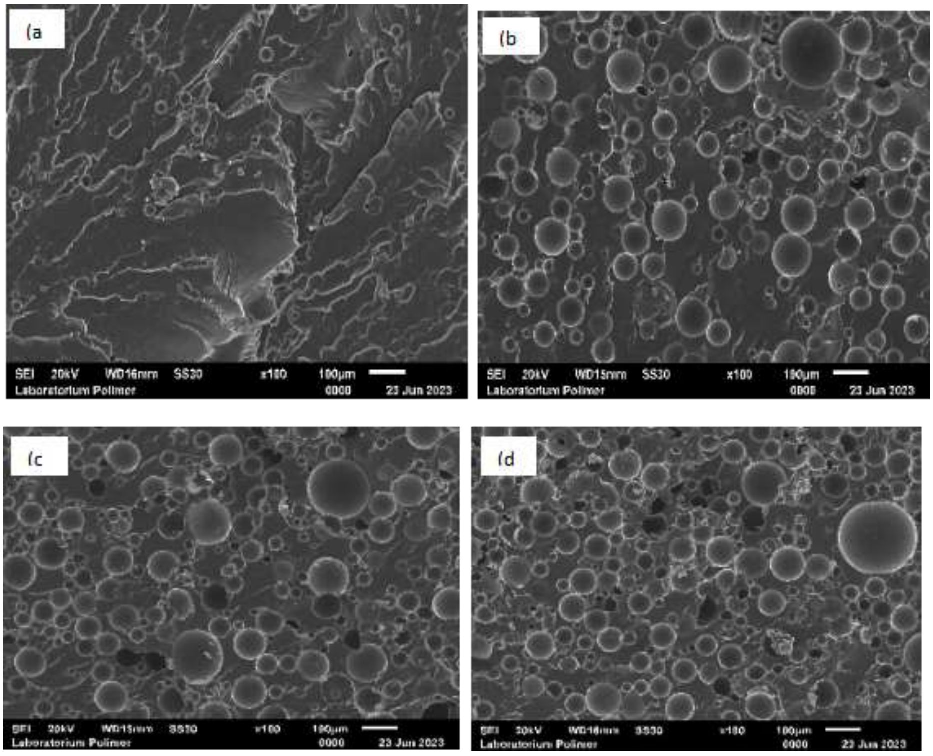

Figure 6. Surface Morphology of Syntactic Foam Composite (a) plain epoxy (b) epoxy + 10% K-15 microballoons (c) epoxy + 30% K-15 microballoons (d) epoxy + 50% K-15 microballoons.

Figure 6 (a) illustrated the surface of the syntactic foam composite with plain epoxy matrix without K-15 microballoons filler has a smooth and uniform surface, and no cracks, pores, or foreign particles are visible. The surface structure of the syntactic foam composite without filler shows a high level of density and homogeneity of pure epoxy, which is usually positively correlated with the elastic modulus and tensile strength. However, without the K-15 microballoons filler, this material becomes denser and more brittle, making it ineffective especially for applications that require lightweight and damage-resistant structures. **Figure 6 (b)** shows the surface of syntactic foam composite with epoxy matrix and addition of 10% volume of K-15 microballoons. The composite surface is rough and uneven, has voids. This occurs because the addition of K-15 microballoons causes the surface to become non-uniform so that there is a decrease in mechanical properties which will be the initial location of cracks when the load is received. **Figure 6 (c)** shows the syntactic foam composite with the addition of 30% K-15 microballoons evenly distributed in the epoxy matrix with a reasonable amount of voids. The microscopic surface remains homogeneous and no cracks are found. This indicates that the interaction between phases is stable and maintained even though there is a load. The epoxy formulation plus 30% volume of K-15 microballoons is the best because it can produce a lightweight material but still has high mechanical resistance. With the ability of microspheres to spread stress and delay crack propagation, the formed structure shows a crack deflection effect. In **Figure 6 (d)** of syntactic foam composite with 50% volume addition of K-15 microballoons, there is significant agglomeration of microspheres and larger void and debonding areas. K-15 microballoons do not bond well with the epoxy matrix, and microcracks appearing along the micropaths indicate that there is stress concentration in the interface area. The structural quality of the material decreases due to the change of surface morphology. The addition of 50% causes microstructural inhomogeneity, weakening of interphase bonds, and increased risk of structural damage. Syntactic foam composites with 50% volume of K-15 microballoons experienced imperfect mixing, resulting in obvious particle agglomeration in some areas. This agglomeration indicates that K-15 microballoons particles clump together and are not evenly distributed in the epoxy matrix, which can cause structural inhomogeneity and decrease local mechanical strength. In addition, there are microcracks around the agglomeration site, indicating residual stress or incompatibility of interphase bonding. By increasing stress concentration in certain areas, agglomeration can accelerate material failure[27].

4. Conclusion

The microscopic observation of the surface of the syntactic foam composite revealed a lack of interfacial bonding between the K15 microballoon particles and the epoxy resin, which limits the load transfer at the interface. This is evidenced by the presence of voids on the surface of the syntactic foam composite. The hypothesis and analytical results demonstrated significant agreement, confirming that the incorporation of microballoons into syntactic foam composites results in material lightening accompanied by a reduction in flexural strength. At a microballoon content of 30%, the flexural strength remains high at 45.93 MPa, while the material maintains its lightweight properties, even underwater. This balance between high flexural strength and low density makes K15 microballoon-filled syntactic foam composites an ideal solution for lightweight materials. Future research should focus on determining the required flexural strength for syntactic foam composites to identify the optimal composite variation for specific applications. Further studies could explore the improvement of the interfacial bonding between the microballoons and the resin matrix, potentially enhancing the overall mechanical properties of the composite.

Akcnowledgment

Thank to Faculty of Engineering Universitas Negeri Semarang for research funding with Contract Number 5.15.4/UN37/PPK.05/2025.

Author contributions

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Conflict of interest

The authors declare no conflict of interest.

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