Soybean Oil-based Non-Isocyanate Polyurethanes for Commercial Applications

Report - 2

(Jan 01, 2024 – June 30, 2024)

By

Dr. Ram Gupta

Pittsburg State University

Summary

This study examines the impact of blowing agent percentages (5,10,15, and 20%) and curing times (RT, 6, 12, 24, and 48 hours) on the microstructure and mechanical properties of non-isocyanate polyurethane (NIPU) foam. The NIPU foam was synthesized using carbonated soybean oil (CSBO) and ethylene diamine (EDA), with varying $NaHCO_3$ concentrations added as a blowing agent. After initial mixing, the foam was cured at room temperature for 24 hours, followed by further curing at 50°C for various durations. Scanning Electron Microscopy (SEM) analysis revealed that longer curing times resulted in more uniform and well-defined foam cells, indicative of improved polymerization and structural stability. Lower blowing agent percentages produced smaller and denser cells, leading to a more compact and mechanically robust structure. Conversely, higher blowing agent percentages resulted in larger and more irregularly shaped cells, reflecting increased foam expansion and reduced density. Compression strength testing showed a significant decrease in foam strength from approximately 0.6 MPa to 0.1 MPa as the blowing agent percentage increased from 5% to 20%. This reduction indicates that while higher blowing agent content can enhance foam insulation properties by reducing density, it also compromises the foam's mechanical strength. However, longer curing times from 24 to 48 hours were found to significantly improve compression strength across all blowing agent percentages, reaching up to 0.6 MPa, underscoring the importance of adequate curing for achieving enhanced foam stability and strength. Post-compression recovery tests demonstrated that longer curing times significantly improved the foam's ability to regain its original shape, highlighting the critical role of curing in maintaining foam integrity and performance after deformation. These findings emphasize the necessity of optimizing both blowing agent concentration and curing duration to tailor the foam's mechanical and structural properties for specific industrial applications, such as in construction and automotive industries, where a balance between lightweight, insulative properties.

Detailed Report of the Work Performed

In the synthesis of NIPU foam, 10 gm carbonated soybean oil (CSBO) was taken in a hard plastic cup followed by 10 wt.% surfactant B8404, 20 wt.% of ethylene diamine (EDA), and NaHCO3 (1 wt.%, 5 wt.%, 10 wt.%,15 wt.%, and 20 wt.%) (all weight percentage was taken in respect of CSBO) was added and vigorously stirred the mixture by using mechanical stirrer for 2 min. After the homogenous mixture, the reaction mixture was allowed to room temperature for 24 hours, for batter curing foam was cured in an oven for further (6 hr, 12 hr, 24 hr, and 48 hr) hours at ~50°C. Obtained foams with different weights (%) of $NaHCO_3$ are shown in **Figure 1**.

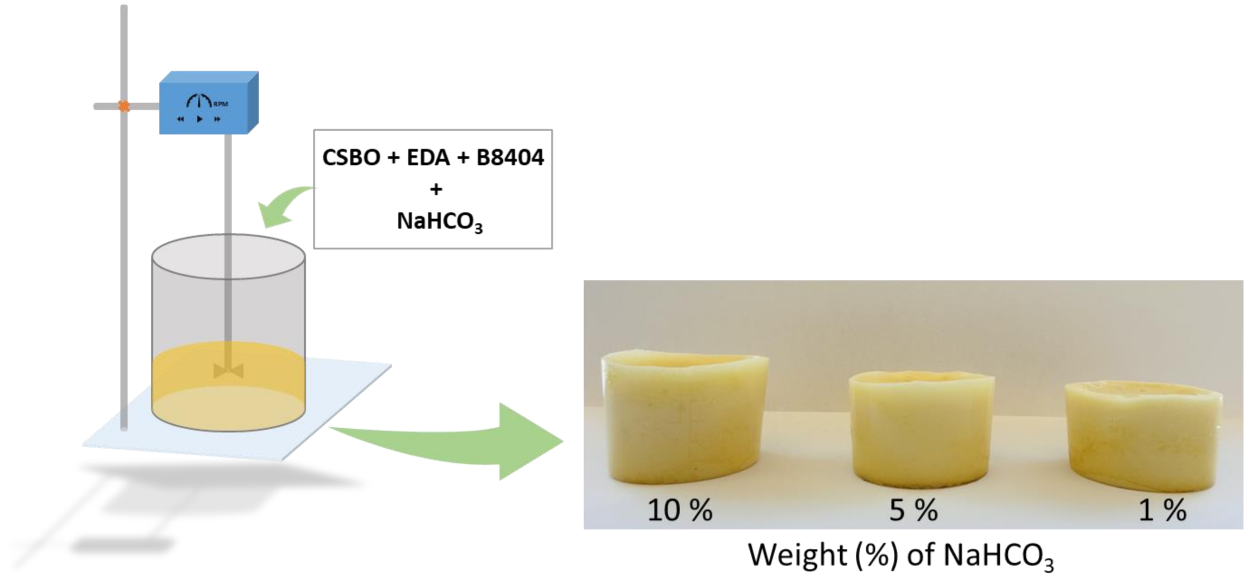


Figure 1. Schematic of preparation of NIPU foams.

In the FT-IR spectra shown in **Figure 2**, characteristic peaks indicative of specific chemical bonds are observed. The formation of amide bonds (-CONH) is confirmed by the presence of a peak at 1677 cm^{-1}, which signifies the reaction between CSBO and diamine (EDA). As the wt.% of $NaHCO_3$ increases, the carbonyl (-C=O) peak at 1800 cm^{-1} gradually decreases and eventually disappears at 10 wt.%, suggesting the successful reaction and incorporation of $NaHCO_3$ into the foam matrix. Notably, the N-H stretching peak around ~3350 cm^{-1} becomes sharper with higher $NaHCO_3$ content, specifically at 10 wt. %, compared to 1 wt. % and 5 wt. %, indicating enhanced hydrogen bonding. Additionally, other important peaks are present in the FT-IR spectra: C-N stretching at 1532 cm^{-1} and C-H stretching at 2925 cm^{-1} and 2850 cm^{-1}, further confirming the presence of these functional groups and the completion of the chemical reactions within the foam. These changes in the FT-IR spectra with varying $NaHCO_3$ content underscore the modifications in the chemical structure and composition of the foam.

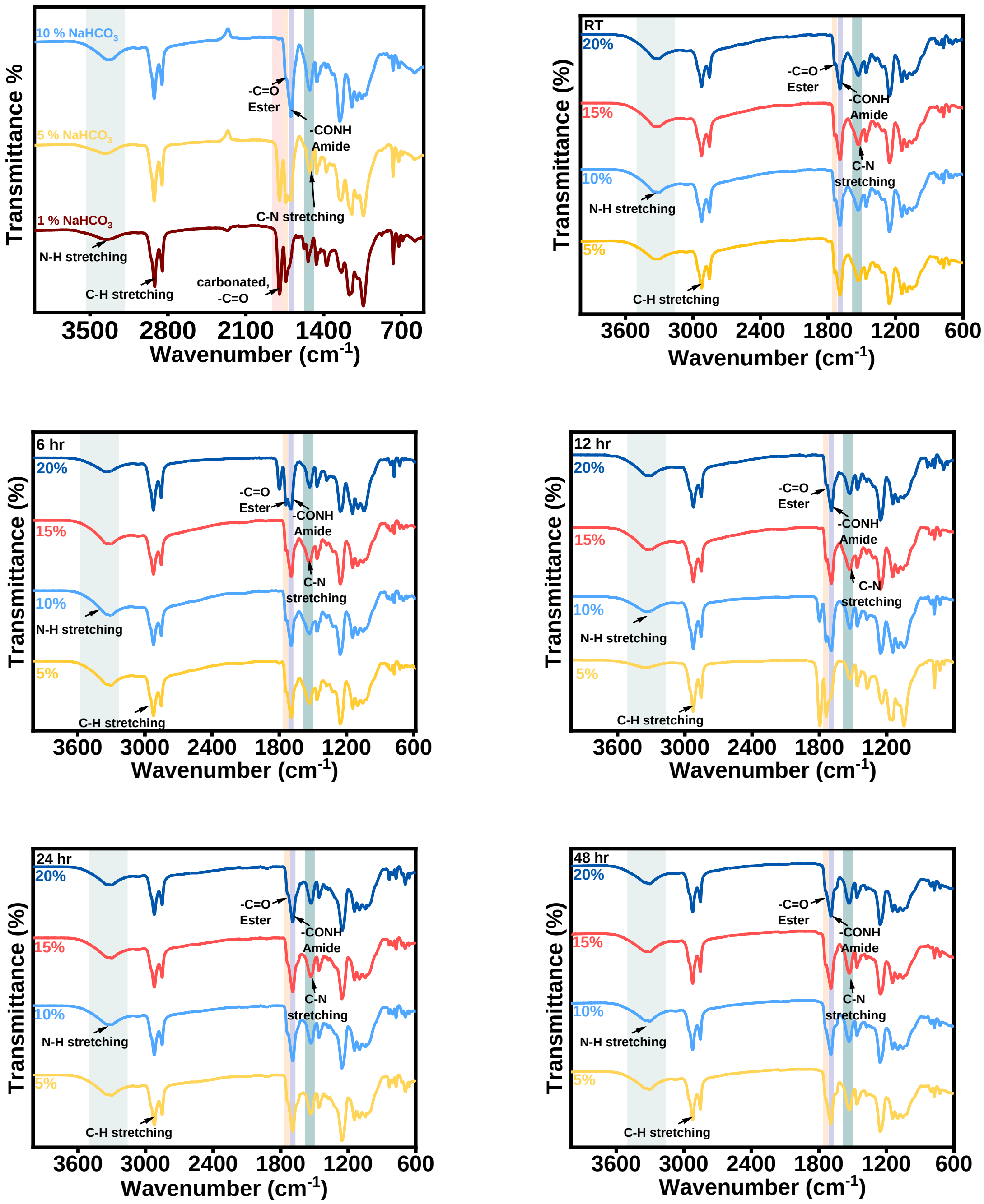
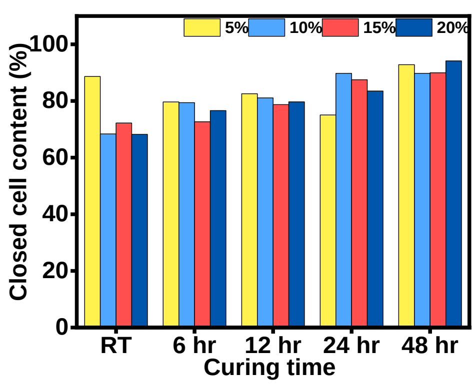
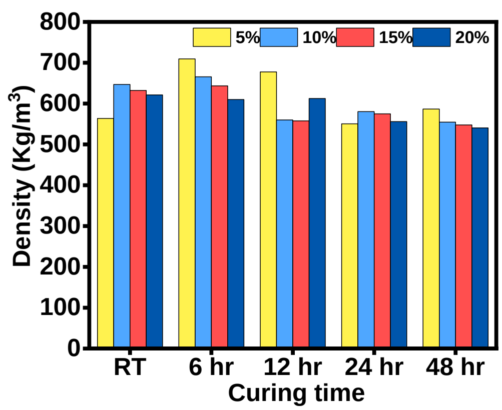


Figure 2. FT-IR data of different weight percentages of blowing agent and curing time.

The presented bar graphs analyze the impact of blowing agent percentage and curing time on the properties of NIPU foam, providing critical insights for optimizing its mechanical characteristics. **Figure 3(c)** illustrates that as the blowing agent percentage increases from 0% to 20%, the compression strength increases significantly, with values rising from approximately 0.1 MPa to 0.6 MPa. This indicates that higher blowing agent content results in a less dense, structurally weaker foam. **Figure 3(b)** further supports this observation, showing a reduction in foam density from about 100 kg/m^3 to 40 kg/m^3 as the blowing agent percentage increases from 0% to 20%. These findings suggest that while higher blowing agent percentages can improve insulation properties due to the lower density, they also compromise the mechanical strength of the foam. In contrast, the third graph highlights the positive correlation between curing time and compression strength, regardless of the blowing agent percentage. For example, extending the curing time from 24 hours to 48 hours results in an increase in compression strength from approximately 0.3 MPa to 0.6 MPa across all blowing agent percentages. This suggests that longer curing durations enable more complete polymerization and stabilization of the foam structure, leading to enhanced mechanical properties. Even with a blowing agent percentage ranging from 0% to 20%, the compression strength at 48 hours of curing time significantly surpasses that at 24 hours, underscoring the critical role of curing time in achieving the desired mechanical characteristics.





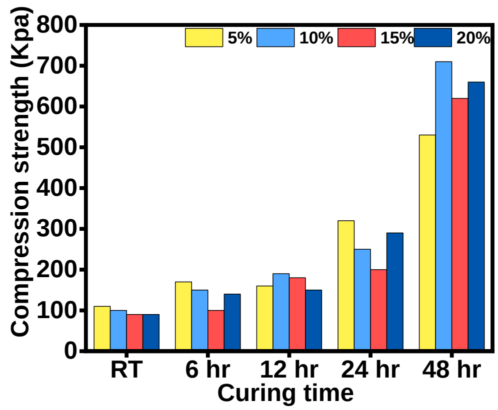


Figure 3. (a)closed-cell content data of different weight percentages of blowing agent and curing time, (b) density data of different weight percentages of blowing agent and curing time, (c) compression strength data of different weight percentages of blowing agent and curing time.

A comparative analysis of the graphs reveals that achieving an optimal balance between blowing agent concentration and curing duration is essential for tailoring NIPU foam properties to specific industrial applications. For instance, in applications where thermal insulation and lightweight properties are paramount, such as in the construction industry, a higher blowing agent percentage of up to 20% may be acceptable. This results in a reduced foam density, decreasing from approximately 100 kg/m^3 to 40 kg/m^3, while maintaining compression strength from 0.1 MPa to 0.6 MPa with adequate curing times. Conversely, for applications requiring robust mechanical support, such as in the automotive industry, a lower blowing agent percentage (e.g., 0% to 10%) combined with extended curing times of 48 hours can be necessary. This approach ensures compression strengths ranging from 0.3 MPa to 0.6 MPa , thereby maintaining the foam's structural integrity. The provided data emphasize that while higher blowing agent content reduces density and enhances insulation, ensuring sufficient curing time is crucial to achieving the necessary compression strength and mechanical stability for different applications.

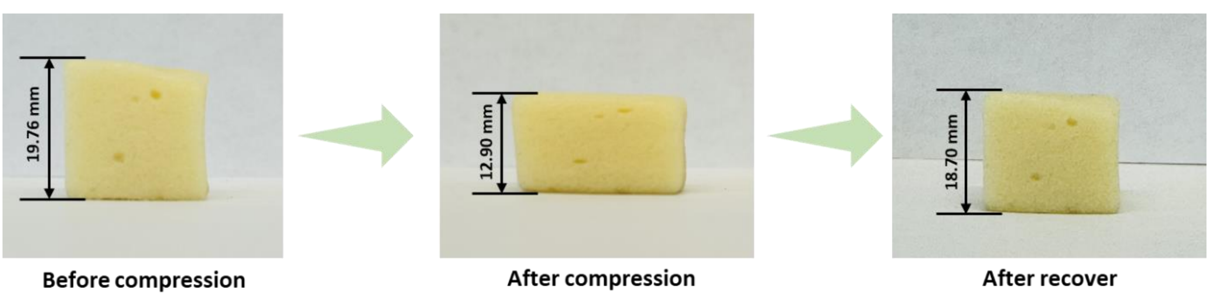


Figure 4. recovery of NIPU foam after compression.

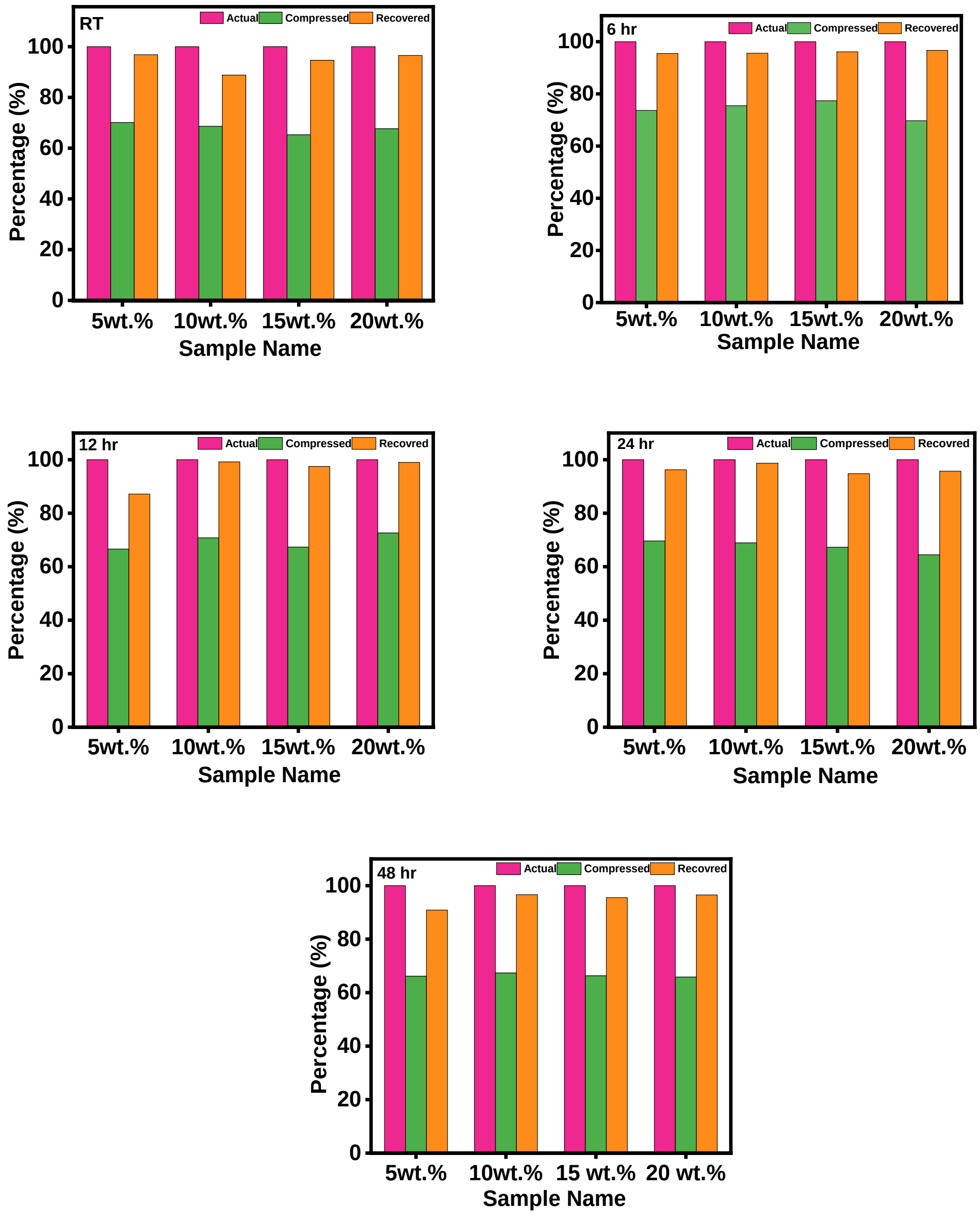


Figure 5. comparison data of actual, after-compression, and recovered foam.

The provided bar graphs display data on the recovery of NIPU foam after compression at various percentages of blowing agents and curing times. Each graph represents a distinct curing time, ranging from RT to 48 hours, and assesses the foam's ability to regain its original structure post-compression. The data indicate that recovery performance improves with extended curing times. For example, foam with a moderate blowing agent percentage shows substantial improvement in recovery as curing time increases, highlighting the importance of adequate curing for enhanced resilience. Similarly, foams with higher blowing agent percentages exhibit marked increases in recovery rates with longer curing times, indicating that sufficient curing time is crucial for maintaining foam integrity and performance after compression. This trend suggests that while higher blowing agent content initially compromises foam density and strength, sufficient curing time facilitates better post-compression recovery, highlighting the importance of optimizing both factors to achieve desired mechanical properties and durability in industrial applications.

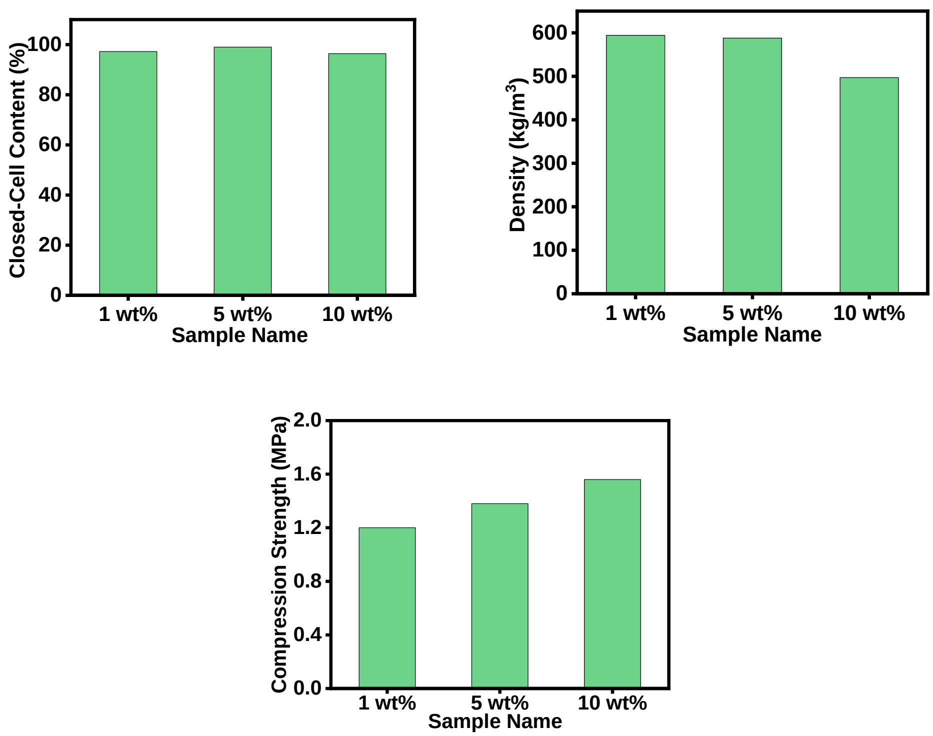


Figure 6. (a) Closed-cell content data of three different weight percentages of blowing agent, (b) density data of three different weight percentages of blowing agent, (c) compression strength data of three different weight percentages of blowing agent.

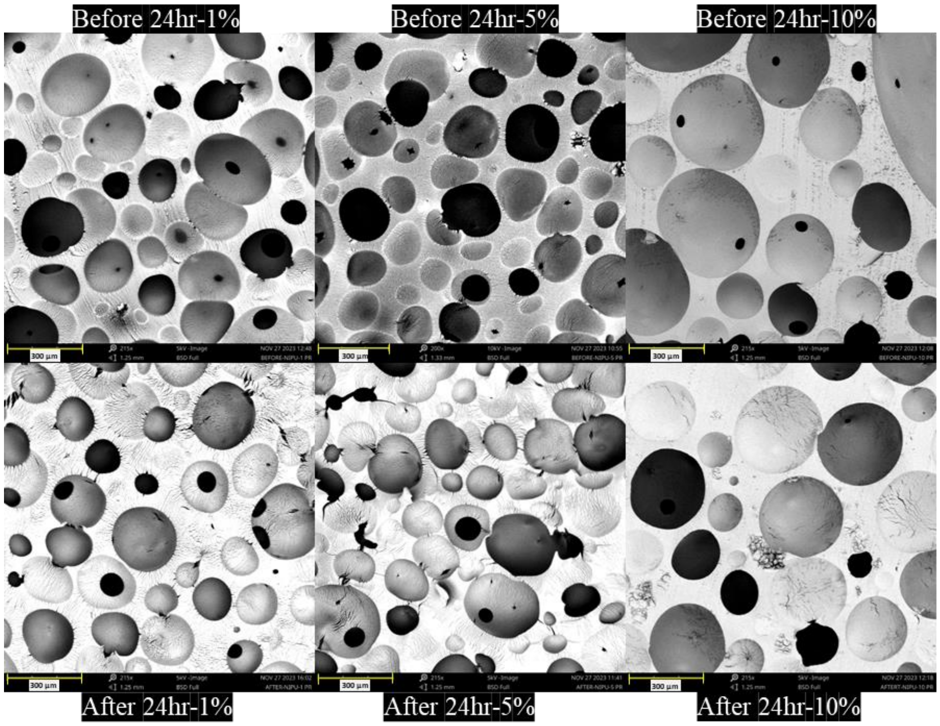


Figure 7. SEM image of after and before compression strength with three different weight percentages of blowing agent.

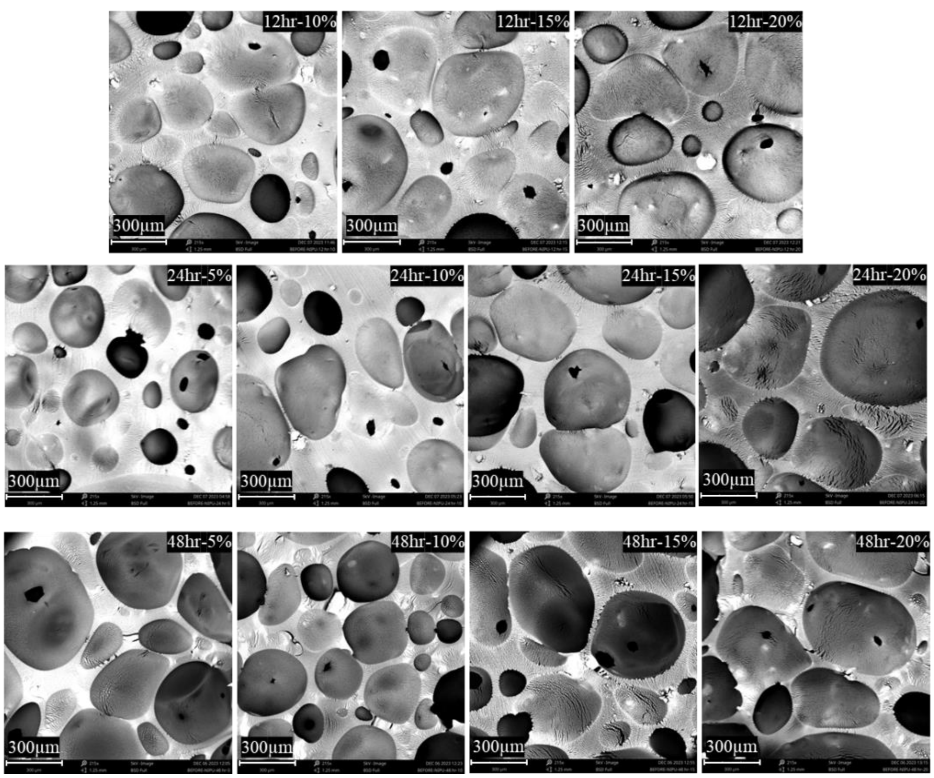


Figure 8. SEM image of before compression strength with different weight percentages of blowing agent and curing time.

The SEM images illustrate the microstructure of NIPU foam under varying curing times (12, 24, and 48 hours) and blowing agent percentages (5%, 10%, 15%, and 20%). As curing time increases, the foam cells become more uniform and well-defined, indicating improved polymerization and stability. At lower blowing agent percentages, the cells are smaller and more compact, leading to a denser structure. Conversely, higher blowing agent percentages result in larger and more irregularly shaped cells, reflecting increased foam expansion and reduced density. The images highlight how longer curing times enhance the structural integrity and uniformity of the foam, while higher blowing agent percentages lead to more pronounced cell formation, affecting the foam's mechanical properties and density.