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Fabrication and Testing of Soy-Based Polyurethane Foam with Flame Retardant Properties

Gurjot S. Dhaliwal¹ · Dilpreet S. Bajwa² · Sreekala Bajwa³

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Abstract

Bio-based foams, particularly soy-based polyurethane, have gained a lot of traction for residential and commercial applications. Since the soy-based foam is widely used as insulation material in the construction industry, it must exhibit superior mechanical, insulation, and fire-retardant properties. In this study, soy-based PU foams were prepared with the addition of nanoclay (Cloisite Na+) in five different concentrations varying from 2 to 10%. The foam was fabricated using 100% soy-based polyol. The effects of adding nanoclay were investigated on the thermal, mechanical, and flame retardant properties of the foam. The fabricated foam samples were tested for differences in their morphology due to the addition of nanoclays using a scanning electron microscope. It was observed that with the addition of the nanoclays, the cell size of the foams was reduced and became more uniform. So, significant improvement was found in the mechanical strength of the foam samples. Also, it was observed that adding of nanoclays lead to the formation of a char layer during combustion. The char layer was effective in reducing the rate of burning of the foam by 38%. Overall, it was concluded that the addition of nanoclays resulted in the soy-based PU foam with superior mechanical and flame retardant properties.

Keywords Soy-based foam · Polyurethane foam · Flame retardent foam · Nanoclay

Introduction

Polyurethane (PU) is one of the most widely used polymeric materials in the world. Due to its easily tunable properties, PU is used in various forms, such as coatings, elastomers, sealants, rigid foams, and flexible foams [1]. Out of the total PU foam synthesized in 2016, 25% was the rigid PU foam, used in construction and insulation applications [2]. Rigid PU foams, compared to traditionally used insulation materials like extruded polystyrene, glass wool, and cellulose, have several advantages, including superior thermal insulation (R-Value), higher rigidity, and moisture barrier [3]. PU is synthesized by step-growth polymerization between isocyanate (di or poly), and hydroxyl-terminated oligomer

(polyol) with at least two reactive hydrogen atoms [4]. Precursors needed to make PU are traditionally obtained from petroleum-based non-renewable sources. Due to increasing concerns about global warming and uncertainty in the supply and price of petroleum, there is a push to develop polymeric materials from renewable vegetable-based resources [5].

For the purpose of making PU from plant-based oils, unsaturated vegetable oils, such as soybean oil, rapeseed oil, castor oil, etc. are modified to increase their hydroxyl content. The modified vegetable oil-based PU foams, though they have their challenges due to low reactivity, have shown promising results in terms of thermal stability and mechanical strength of the foams [6]. Various studies have been done by substituting petroleum-based polyol with soy-based polyols for the fabrication of PU foams. The results showed comparable R-Value to petroleum-based foams, but the compressive strength was lower [7–9]. Also, it was observed that the soy-based foams degraded faster as compared to petroleumbased foams [10]. An approach by Marle et al. to achieve better thermal and mechanical properties with vegetable-oil based using various surfactants resulted in the more uniform cell structure of the foam, leading to better mechanical

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Gurjot S. Dhaliwal gdhaliwal@uttc.edu

Intertribal Research and Resource Center, United Tribes Technical College, Bismarck, ND 58504, USA

Mechanical and Industrial Engineering Department, Montana State University, Bozeman, MT 59717, USA

Ollege of Agriculture, Montana State University, Bozeman, MT 59717, USA

strength [11]. Another approach to achieve better properties in bio-based foams is blending the vegetable-oil based polyol with petroleum-based polyol. Various vegetable-oil based polyols were blended with 50% petroleum-based polyol [12]. It was observed that blending petroleum-based polyol results in better mechanical and thermal properties [13]. With the advances in technology, the processing of vegetable oils has improved to yield polyols with better reactivity and higher hydroxyl content. In a recently reported study, the researchers were able to get both mechanical and thermal properties comparable to petroleum-based PU foams [14].

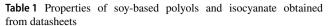
Rigid PU foam is widely used as an insulation material in both residential and commercial buildings, and flame retardancy is the desirable property. Researchers have studied the effects of various organohalogen and organophosphate based fire retardants on flame retardancy of foam materials [15–17]. Though these fire retardants are efficient in reducing the flammability, they also pose a threat to life due to their toxicity and deleterious effect on the environment. Various studies have been performed to investigate the use of safer flame retardants such as expandable graphite (EG) and nanoclays (NC) [18–20]. It was observed that EG and NC based fire retardants, though not as effective as organohalogen and organophosphate fire retardants, were still able to reduce the flammability of the polymeric materials.

The effectiveness of NC is also investigated for reducing the surface tension of the mixture of foam reactants [21]. Reactants with low viscosity lead to foam with a homogenous cell structure, leading to better mechanical properties [22]. So this study aims to investigate the effectiveness of Cloisite Na+nanoclay on the fire retardancy, mechanical properties, and thermal properties of soy-based PU foam.

Materials and Methodology

Materials

The foam samples were manufactured using a two-part PU resin. Component A is Rubinate 1820, which is a blend of diphenylmethane diisocyanate (MDI) and polymeric methylene diphenyl diisocyanate (pMDI) procured from Huntsman International LLC (Auburn Hills MI). Component B is a blend of soy-based polyol HB230 and HB530 obtained from MCPU Polymer Engineering LLC (Pittsburg, KS). The properties of soy-based polyols and isocyanate are listed in Table 1. The surfactant used was Xiameter OFX-0193 obtained from Dow Corning (Auburn, MI) and dibutyltin dilaurate (DBTL) catalyst was purchased from Sigma-Aldrich (St. Louis, MO). Distilled water was used as a blowing agent. The fire retardant nano clay (NC) used was Cloisite Na+, which comprises of quartz (SiO₂), obtained from BYK Gardner (Columbia, MD). NC used had a particle



Properties	HB 230	HB 530	Rubinate 1820
Functionality	~2	~8	2.47
Hydroxyl number (mg KOH/gm)	220–240	520-540	-
Acid number (mg KOH/gm)	≤3.0	≤3.0	_
Viscosity at 25 °C (cps)	375	2500	70

size of < 25 μ m, and had optimized surface properties for exfoliation in aqueous systems. The NC was dried in an oven to remove any moisture before used in the fabrication of soy-based PU foams.

Preparation of Foam Samples

Soy-based PU foam samples were fabricated by mixing varying amounts of NC in soy-based foam. NC was added in the range of 2 g to 10 g with increment steps of 2 g. The amount of polyols used is 80 g and 20 g for HB230 and HB 530 respectively, the blowing agent is 1.50 g, the catalyst is 0.08 g and Isocyanate index (NCO) for all samples is 1.14. For comparison of the foam properties, the control sample was fabricated without NC, while keeping other composition of polyols, water, surfactant, catalyst, and NCO same. The detailed formulations of fabricated samples is mentioned in Table 2.

For fabrication of samples, measured amounts of polyol, blowing agent, surfactant, catalyst, and NC were mixed using a mixer at 1200 rpm for 5 min. The required amount of isocyanate was then added and mixed for 30 s at 1200 rpm. The resulting mixture was poured in a mold of size 245 mm×75 mm×75 mm (9.8 in.×3 in.×3 in.). Three samples of each formulation were fabricated for testing the samples for mechanical properties, thermal stability, and fire retardancy.

Characterization of Foam Samples

The fabricated foam samples were visually inspected for cracks and voids. The foam blocks free of defects were cut to the required size for further testing to analyze the mechanical and thermal properties. The prepared samples were conditioned at 23 °C and 50% relative humidity for 48 h prior to further testing, in accordance with respective ASTM standards.

Density and Cell Structure

The density of the prepared PU foam sample was tested using ASTM D1622. For this test, the sample size used was



Table 2 Formulation of foam samples

Sample code	HB230 (g)	HB530 (g)	Blowing Agent (g)	Surfactant (g)	Catalyst (g)	NCO	Nanoclay (g)
Control	80.00	20.00	1.50	5.00	0.08 g	1.14	0.00
I	80.00	20.00	1.50	5.00	0.08 g	1.14	2.00
II	80.00	20.00	1.50	5.00	0.08 g	1.14	4.00
III	80.00	20.00	1.50	5.00	0.08 g	1.14	6.00
IV	80.00	20.00	1.50	5.00	0.08 g	1.14	8.00
V	80.00	20.00	1.50	5.00	0.08 g	1.14	10.00

 $25.4 \text{ mm} \times 25.4 \text{ mm} \times 25.4 \text{ mm}$ (1 in. \times 1 in. \times 1 in.). Five specimens were cut from the core of the prepared samples and were weighed with a precision analytical balance. It has been observed in the previous studies that density of the foam can be changed with variation in the composition [10, 14]. In addition, density is a good indicator of the physical properties of the foam samples.

The fabricated samples were analyzed using a JEOL JSM-6490LV scanning electron microscope for the cellular structure. Five samples of each formulation were sputter-coated with Au/Pd to avoid electrostatic charging during the examination. Samples were analyzed using the accelerating voltage of 15.0 kV. SEM images were obtained to determine the average cell size of the foam samples. The different cellular structure of the foam is the result of variable amounts of nanoclay used in the fabrication of the samples. The SEM images were analyzed with ImageJ software to calculate the average cell size of the samples.

Thermal Analysis

Thermogravimetric analysis (TGA) was performed on the fabricated samples using Q50 (TA Instruments, New Castle DE) to evaluate the thermal stability of PU foam. Samples were heated from room temperature (22 °C) to 600 °C, at a heating rate of 10 °C min $^{-1}$, in an air atmosphere. The air atmosphere was chosen to allow the degradation of foam due to oxidation at elevated temperatures.

The horizontal burning characteristics test was performed on the foam samples to determine the rate of burning of the foam samples, according to ASTM D4986-18. The samples were cut to size 150 mm×50 mm×13 mm. Twenty samples of each configuration were tested for this test. The samples were exposed to a burning gas flame for 60 s. After removal of the flame, the time duration sample takes to extinguish (t), and the length of sample burnt (L) is noted. The rate of burning (V) is calculated using the following equation:

$$V = 600 L/t \tag{1}$$

FTIR Test

Fourier transform infrared spectroscopy (FTIR) was performed to investigate the interaction between different functional groups of surfactants and other components of the PU foams and nano-clay. The transmission spectra were recorded and analyzed within the range of 3800–400 cm⁻¹ using Nicolet 6700 (Thermo Scientific, USA) with Omnic software for data collection.

Mechanical Properties

The mechanical properties of the foam, namely tensile strength, compression force deflection (CFD), and constant deflection compression, were evaluated. The CFD test determines the ability of the foam to withstand compressive stresses and its agility after compression. This test was performed according to ASTM 3574-Test C using TestResources (Shakopee, MN, USA) universal testing machine. For this test, preconditioned samples were cut into 50.8 mm×50.8 mm×25.4 mm (2 in.×2 in.×1 in.) and preflexed twice by compressing to 50% of the thickness at the rate of 50 mm/min. After 6 min of final preflex, the samples were compressed at the rate of 50 mm/min until 50% deflection (12.5 mm) was achieved. The load reading after compressing for 60 s was recorded.

The constant deflection compression set test (C_t) consists of deflecting the foam specimen to a specified deflection, exposing it to specified conditions of time and temperature, and measuring the change in the thickness of the specimen after a specified recovery period. This test was conducted according to ASTM D3574-Test D using the TestResource universal testing machine. The specimens were cut to 50.8 mm \times 50.8 mm \times 25.4 mm (2 in. \times 2 in. \times 1 in.), with parallel top and bottom surfaces and perpendicular sides. For each foam sample formulation, three specimens were tested. In this test, the initial thickness of the preconditioned specimen was noted and then compressed to 50% of the thickness. Within 15 min, the deflected specimen was placed in a convection oven at 23 °C for 22 h and 50% relative humidity. The sample thickness was measured after the recovery time.



The constant deflection compression set (C_t) is expressed as a percentage of the original thickness, as shown in Eq. 2:

$$C_t = (t_o - t_f)/t_o \times 100 \tag{2}$$

where t_o is the original thickness of the specimen and t_f is the final thickness of the sample.

The tensile and tensile adhesion properties of foam were evaluated using tensile tests. This test was performed according to ASTM D1623-Type C, using a universal testing machine. For this test, preconditioned samples were cut into $50.8 \text{ mm} \times 50.8 \text{ mm} \times 25.4 \text{ mm}$ (2 in. $\times 2 \text{ in.} \times 1 \text{ in.}$) and were joined with $50.8 \text{ mm} \times 50.8 \text{ mm}$ (2 in. $\times 2 \text{ in.}$) cross-section aluminum blocks with hot melt adhesive. The prepared samples were subjected to tensile loading at a rate of 1.3 mm/min.

Results and Discussion

The results of various tests conducted to analyze the samples are mentioned in Tables 3 and 4.

Density

The prepared samples were tested for the density, and the range of the density of the samples was found to vary from 62.05 to 76.41 kg/m³. It was observed that as the amount of NC in the formulation of the foam sample increased, the density of the foam decreased. The control sample had

 $79.60 \pm 2.09 \text{ kg/m}^3$ density. The decrease in density of the foam with increasing amounts of NC is attributed to change in the morphology of the cell structure of foam samples. It was observed that with higher amounts of NC, the resulting cell walls had lower thickness, leading to lower density.

The change in the density of the foam samples is also correlated with the size of cells of the foam samples. It was observed in the SEM images that as the amount of NC is increased, the cell size is reduced, and due to the thining of cell walls, the cell structure is more damaged or opened. Similar findings were observed in another study as well [21]. The SEM images are shown in Fig. 1.

Thermal Analysis

TGA test was performed to compare the thermo-oxidative degradation behavior of the fabricated samples. The weight loss and weight-loss derivative (DTG) curves are shown in Fig. 2 and Fig. 3 respectively. It was observed that all the samples started to degrade at the same temperature, i.e., 250 °C (Fig. 2). The different amounts of residue remaining at the end of the test correspond to the amount of NC added in the samples. It was observed that the degradation of the samples took place in 3 steps (as shown in Fig. 3). The degradation started at 250 °C due to the oxidation of urethane bonds. The difference in the degradation was observed in the 2nd step, where the control sample showed degradation at 300 °C, whereas samples with NC showed degradation at 325 °C. This difference in the degradation behavior of the control sample and samples with NC is due to the action of

Table 3 Testing results of foam samples

Sample Code	Density		Cell Size		Flame Test	
	kg/m ³	S.D	$\overline{\text{mm}^2}$	S.D	cm/min	S.D
Control	79.60	2.09	0.160	0.006	17.49	0.19
I	76.41	1.65	0.055	0.007	16.06	0.24
II	72.45	1.55	0.047	0.002	14.78	0.18
III	68.91	2.02	0.043	0.006	12.51	0.17
IV	64.04	2.46	0.039	0.005	11.42	0.26
V	62.05	1.07	0.035	0.002	10.75	0.27

Table 4 Mechanical testing results of foam samples

Sample code	Density		Tensile St	Tensile Strength		CFD		C _t	
	kg/m ³	S.D	kPa	S.D	kPa	S.D	%	S.D	
Control	79.60	2.09	89.45	5.53	115.75	8.53	1.45	0.13	
I	76.41	1.65	96.87	7.01	119.83	3.55	3.33	0.78	
II	72.45	1.55	132.85	8.47	141.40	1.07	3.46	0.09	
III	68.91	2.02	153.79	9.82	158.87	5.20	3.37	0.05	
IV	64.04	2.46	160.75	9.05	169.43	6.44	4.06	0.94	
V	62.05	1.07	161.52	6.45	173.93	5.06	3.30	0.39	

CFD compression force deflection, C_t constant deflection compression set test



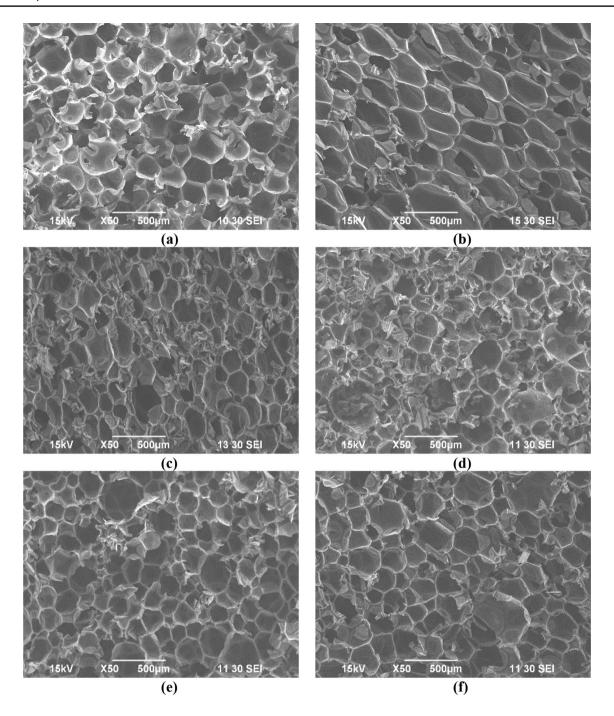


Fig. 1 SEM images showing the differences in the cell structures of foam samples with different amounts of nanoclay. a Control sample, b Sample I, c Sample II, d Sample III, e Sample IV, f Sample V

NC clay as a thermal barrier that thwarts the thermal degradation of the foam [23].

The horizontal burning characteristics test shows the time taken to burn 125 mm of foam. As shown in Fig. 4, it is observed that the rate of burning is reduced as the amount of NC is increased in the samples. However, as the amount of NC is increased from 8 to 10 g, a small decrease is observed in the rate of burning.

Fourier Transform Infrared Spectroscopy

The FTIR spectra of the fabricated samples showed absorbance bands corresponding to the chemical bonds in the foam samples. Collected spectra for the samples of different formulations were very similar, as shown in Fig. 5. In the spectra, a band was observed between 3250 and 3400 cm⁻¹ for the foam samples, which represents the stretching of N–H



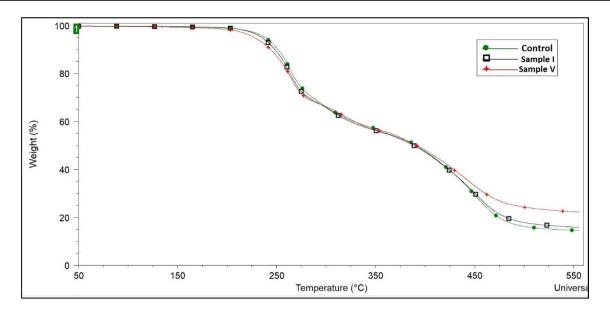


Fig. 2 Weight-loss behavior of foam samples during TGA test

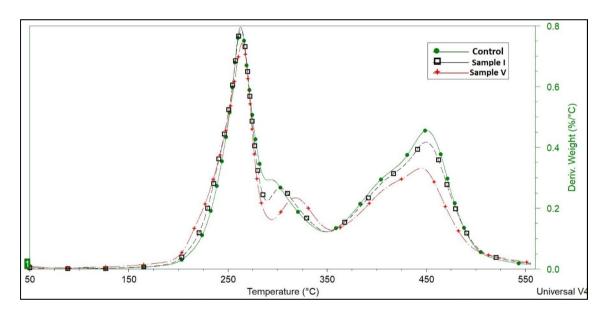


Fig. 3 Derivative weight loss behavior of foam samples during TGA test

bond in urethane and urea groups and stretching vibrations in O–H groups. Also, a sharp peak was observed for the NC at 3622 cm⁻¹, which is due to the presence of a free O–H group. The absence of this peak in foam samples means the absence of free O–H, leading to a nearly complete chemical reaction between polyol and isocyanate. The peaks at 2925 cm⁻¹ and 2850 cm⁻¹ in the foam samples are attributed to asymmetric and symmetric stretching vibrations of C-H bonds, respectively.

The strong peaks at 1740 and 1720 cm⁻¹ correspond to the stretching of the C=O bond in aldehyde bond in the

urethane. The NC spectra show another peak at 1632 cm⁻¹ that represents the presence of C=C bonds. The small peak in the foam samples at 2275 cm⁻¹ is due to the presence of an unreacted isocyanate group. The peak at 1470 is attributed to free C=O stretching, whereas the peak at 1710 cm⁻¹ is due to hydrogen-bonded C=O stretching. The peaks at 1599 cm⁻¹ and 1512 cm⁻¹ are attributed to stretching and bending vibrations of C-N and N-H bonds, respectively, in urethane. The peaks at 1215 cm⁻¹ show the stretching vibrations of the C-O bond. The peak at 1070 cm⁻¹ is attributed to the vibration of the C-O bond.



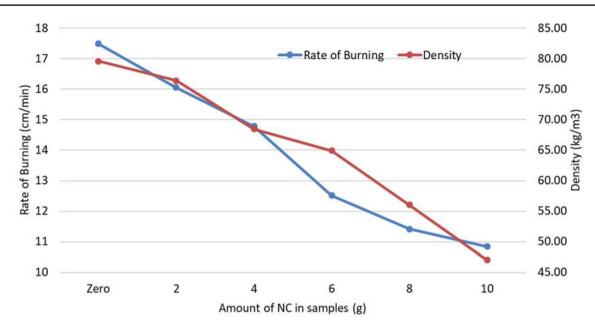


Fig. 4 Relationship between the rate of flame and density as a function of different amounts of nanoclay

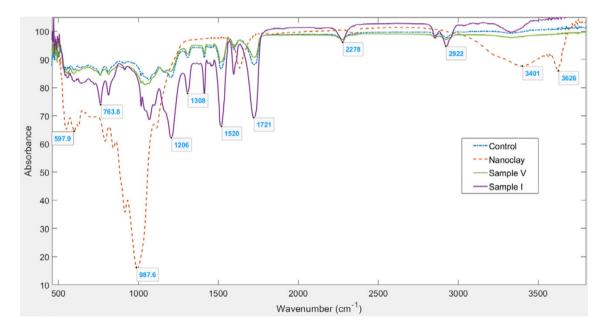


Fig. 5 FTIR spectra for the fabricated samples and nano-clay fire retardant

Mechanical Testing

CFD, C_t, and the tensile tests were performed on the fabricated samples to evaluate the effect of adding NC on the mechanical properties of the foam. The results of the testing are mentioned in Table 4.

The foam samples tested for CFD exhibited the range of CFD from 119.833 to 173.933 kPa, while the control sample had CFD of 115.754 kPa, as shown in Fig. 6. It was observed

that the CFD of the samples increased with an increase in the amount of NC.

The tensile strength of the foam samples varied from 96.867 to 161.520 kPa, while the control sample had a tensile strength of 89.451 kPa. A similar trend was observed in the CFD and tensile test results, where samples with higher amounts of NC had higher tensile. The increase in CFD and tensile strength in the samples with a higher percentage of NC can be explained by the change in the surface tension of



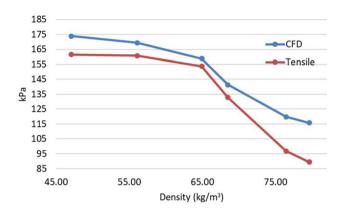


Fig. 6 Change in CFD and tensile strength with respect to the density of foam samples

the reactants mixture of the foam. It was observed that as the percentage of NC was increased in the reactants mixture, the surface tension of the mixture reduced. The reduced surface tension resulted in fewer damages and a more homogenous cell structure, leading to better load distribution while testing the samples [24, 25].

The C_t values of the fabricated foam samples ranged from 3.298 to 4.060%, whereas the control samples had C_t of 1.45%. It was observed that with the addition of NC in the foam samples, the C_t values increased twofold. The increase in the C_t can be explained by the thinning of the cell walls that resulted due to the addition of NC. The thin walls of the foam fractured due to the compression of foam while testing, leading to poor retention of the original dimensions of the samples [25]. Also, NC also leads to different cell morphologies in the samples, and it had been observed in another study that C_t of the foam is dependent on the cell structure of the foam [26].

Conclusions

PU foam was fabricated with soy-based polyol with varying amounts of NC varying from 2 to 10% by weight of polyol. The prepared foam samples were tested for thermal stability, rate of burning, mechanical strength, and dimensional stability. It was observed that as the amount of NC is increased in the foam samples, the density of the samples was reduced. Also, changes were observed in the cell structure of the samples with different amounts of NC. The rate of burning was reduced by about 38%, from 17.5 to 10.85 cm/min with the addition of NC. The mechanical strength of the foam samples was also improved due to the addition of NC. The CFD of the foam samples improved by 66.5%, and tensile strength improved by 55.4% with the addition of NC. However, the dimensional stability of the samples was reduced by 37% with the addition of NC. Also, it was observed that as the

amount of NC was increased beyond 8%, a little improvement was noted in both the thermal and mechanical properties of the foam.

Overall, the addition of NC resulted in achieving soybased PU foam with better thermal and mechanical properties. However, further studies need to be conducted to achieve a lower rate of burning of the foams.

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Compliance with Ethical Standards

Conflict of interest The authors declare that they have no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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