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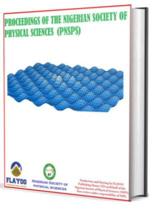

Tetrahydrofuran-based recycling of furan–lignin foams: comparative characterization of regenerated and virgin foams

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ABSTRACT

Foams are used widely in furniture, construction, automotive, and related industries because of their mechanical performance and durability. However, the poor recyclability of conventional polyurethane (PU) foams has raised environmental concerns because of their persistence and contribution to solid-waste accumulation. Bio-based furan–lignin foams (FLFs) offer a promising alternative because they are derived from renewable feedstocks and may provide improved end-of-life options. This study investigated the recycling of rigid FLFs through solvent-assisted dissolution. Virgin foams were prepared from furfuryl alcohol, used as a cross-linker, and lignin-based polyol obtained from corn husks and palm fruit shells, respectively. Glyoxal was used as a hardener and diethyl ether as a blowing agent. Eight solvents were evaluated for their dissolution indices and recyclability potential. Tetrahydrofuran (THF) gave the highest dissolution index (93.24%), followed by ethylene glycol and polyethylene glycol-400, whereas water gave the lowest value (1.10%). Four recycled furan–lignin foams (RFLFs) were then produced from the THF-derived dissolution product, which was used as recycled polyol. Comparative characterization showed that RFLF2 produced the highest char residue (24.45%), whereas RFLF4 showed the highest degradation temperature (556.80 °C), compressive stress, and density (0.876 g cm^{-3}). The regenerated foams retained, and in some cases improved, the mechanical and thermal performance of the virgin FLFs.

Keywords: Foam recyclability, Furan–lignin foam, Recycled polyol, Solvent-assisted dissolution.

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1. INTRODUCTION

Foams are well known for their functionality, adaptability, and durability. Polyurethane (PU) foams are widely used in buildings, automobiles, packaging, home appliances, children's toys, clothing, footwear, and aeronautical applications. However, the poor recyclability of PU foams and the accumulation of persistent foam waste create important environmental challenges during production, use, and disposal [1–4]. Conventional PU foams are produced from isocyanates and polyols, both of which can have environmental and health effects. Isocyanates are particularly hazardous because even low-level inhalation exposure can cause severe respiratory problems, including asthma and airway inflammation, and may be associated with other serious health risks [5].

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The main constituents of PU foams, especially isocyanates and polyols, are mostly obtained from petrochemical sources. PU foam production also involves petroleum-derived fillers and additives that may pose occupational and ecological concerns. In addition to not being environmentally sustainable, these feedstocks are subject to price volatility. These limitations have encouraged interest in bio-based substitutes for synthetic polyols, isocyanates, and conventional additives. The development of environmentally friendly and recyclable foam materials is also supported by tighter regulations and increasing environmental awareness [6, 7]. Recycling furan–lignin foams (FLFs), however, has received little attention. Furfuryl alcohol (FA) from corn husks and lignin-derived polyol from palm kernel shells were recently reported for the synthesis of rigid bio-foams [3, 8]. Related work on PU recycling, recycled polyols, lignin liquefaction, and polymer-polyol effects has provided useful background for foam recovery and reformulation [9–13]. Recent reviews of lignin extraction and application also support the use of lignin as a renewable feedstock in polymeric materials [14].

This research supports Sustainable Development Goal (SDG) 12, responsible consumption and production, by encouraging recycling, resource efficiency, and waste reduction through the development of furan–lignin materials. It also supports SDG 9, industry, innovation, and infrastructure, by promoting circular-economy technologies, and SDG 13, climate action, by reducing dependence on petrochemical polymers and their environmental impacts. Previous studies have provided detailed assessments of the thermal, mechanical, physical, and morphological properties of virgin furan–lignin foams [3, 8].

1.1. CURRENT KNOWLEDGE GAPS

The thermal, mechanical, physical, and morphological properties of synthesized virgin FLFs have been studied previously [3, 8]. Nevertheless, important gaps remain. Reports on the recycling of furan–lignin foams by tetrahydrofuran (THF)-assisted or other solvent-assisted dissolution, particularly for regenerating reusable foam materials, are limited. Comparative analyses of virgin and regenerated FLFs are also scarce, especially with respect to how recycling affects mechanical performance, thermal stability, and structural integrity. In addition, the feasibility of incorporating FLFs into a circular materials framework through solvent-based recycling has not been fully examined. This study addresses these gaps by investigating THF-assisted regeneration of FLFs and comparing the resulting regenerated foams (RFLFs) with their virgin counterparts.

1.2. NOVELTY OF THE STUDY

Although FLFs have recently been synthesized and characterized [3, 8], recycling of FLFs through a THF-assisted dissolution process has not been reported. This study presents the feasibility of reprocessing FLFs into RFLFs by THF-based dissolution and recycling. Unlike earlier studies that focused primarily on virgin material properties, this work provides a systematic comparison of virgin and regenerated foams in terms of thermal, mechanical, and morphological properties. It also provides a proof of concept for integrating FLFs into a circular materials framework and highlights their potential for reuse without substantial loss of performance.

1.3. OBJECTIVE OF THE PRESENT STUDY

The objective of this study was to investigate the feasibility of recycling FLFs using a THF-assisted dissolution process. The specific aims were to perform comparative dissolution studies of FLFs using eight solvents; characterize regenerated foams in comparison with virgin materials; and assess the thermal, morphological, and mechanical properties of the virgin and recycled foams using thermogravimetric analysis (TGA), scanning electron microscopy (SEM), compressive testing, and density measurements.

2. MATERIALS AND METHODS

Previously reported methods [3, 8, 14] were used to synthesize the lignin-derived polyol and FA for the development of RFLFs. Sulfuric acid (98%), glyoxal (40% aqueous solution), diethyl ether (99%), polyethylene glycol-400 (PEG-400), ethylene glycol (EG), THF, methanol, ethanol (96%), acetone, and isopropanol (propan-2-ol) were purchased from Sigma-Aldrich. The recycled polyol used to prepare the RFLFs was produced using the dissolution procedure described in Section 2.2.

2.1. FOAM PREPARATION

The RFLFs were prepared by replicating the virgin furan–lignin foam formulation, except that lignin-based polyol was replaced with recycled polyol while all other feedstocks were retained. This approach enabled direct comparison of the performance of the virgin and recycled foams. Table 1 summarizes the formulation procedure, which followed methods described in Refs. [3, 8]. Section 2.2 describes the dissolution of the virgin FLFs.

2.2. DISSOLUTION INDEX OF FURAN–LIGNIN FOAMS IN DIFFERENT SOLVENTS

The dissolution index is the extent to which a material quantitatively dissolves in a given solvent under specified conditions. It is expressed as the percentage ratio of the mass of dissolved material to the initial mass of the sample. Eight solvents were selected to provide a comparative basis for understanding the dissolution behavior of furan–lignin foam materials in highly polar to moderately polar solvents. To assess foam dissolution, a modified version of the procedure described in Ref. [16] was used. The solvents were isopropanol, distilled water, methanol, ethanol, THF, PEG-400, EG, and acetone.

The FLF samples were milled to a particle size of 0.2 mm using a laboratory mill. In each experiment, 5 mL of solvent was added to 0.5 g of foam material, and the mixture was stirred continuously for 30 min at 20 °C. The suspensions were centrifuged for 10 min at 4000 rpm using a laboratory centrifuge (Model SM 112). The resulting supernatant was collected using a vacuum filtration apparatus. Each test was performed in triplicate. The percentage of dry residue remaining after recovery of the filtrate was used to assess dissolution. The dissolution index was calculated using equation (1):

$$\mathrm{DI}(\%) = \frac{M_1}{M} \times 100, \tag{1}$$

where DI is the dissolution index, M_1 is the mass of dissolved material (g), and M is the initial sample mass (g).

Table 1. Formulations of virgin and recycled furan–lignin foams.

Foam ID	FA (g)	Blowing agent (g)	Catalyst (g)	Water (g)	Lignin (g)	Hardener (g)
FLF1	50	25	35	6	30	15
FLF2	60	25	35	6	20	15
FLF3	40	25	35	6	30	15
FLF4	60	25	35	6	40	15
Foam ID	**FA (g)**	**Blowing agent (g)**	**Catalyst (g)**	**Water (g)**	**Recycled polyol (g)**	**Hardener (g)**
RFLF1	50	25	35	6	30	15
RFLF2	60	25	35	6	20	15
RFLF3	40	25	35	6	30	15
RFLF4	60	25	35	6	40	15

Note: FLF denotes virgin furan–lignin foam, and RFLF denotes recycled furan–lignin foam.

2.3. MEASUREMENT OF THE HYDROXYL NUMBER OF POLYOLS

The hydroxyl numbers of the recycled polyols and virgin polyol were determined according to ASTM D4274 [12]. In this procedure, 1 g of polyol sample was esterified for 20 min at 110 °C using 25 mL of phthalation reagent composed of 150 g of phthalic anhydride, 24.2 g of imidazole, and 100 g of 1,4-dioxane. After esterification, the reaction mixture was diluted with 50 mL of 1,4-dioxane and 25 mL of distilled water. The equivalence point was determined using a pH meter, and the solution was titrated with 1 mol L^{-1} sodium hydroxide solution. The hydroxyl number, expressed in mg KOH g^{-1}, was calculated using equation (2):

$$X_n = \frac{(Y - Z)N \times 56.1}{W}, \tag{2}$$

where X_n is the hydroxyl number (mg KOH g^{-1}), Z is the volume of sodium hydroxide solution used to titrate the sample (mL), Y is the volume of sodium hydroxide solution used for the blank (mL), N is the normality of the sodium hydroxide solution (mol L^{-1}), and W is the mass of the recycled polyol sample (g).

2.4. DENSITIES OF THE VIRGIN AND RECYCLED FURAN–LIGNIN FOAMS

The densities of the virgin and recycled furan–lignin foams were calculated according to DIN EN 323 using equation (3):

$$\rho = \frac{w_t}{l \times b \times t}, \tag{3}$$

where w_t is the foam mass (g), and l, b, and t are the length, width, and thickness of the foam sample (cm), respectively. The dimensions and mass were measured to compute the density of each foam [14].

2.5. COMPRESSIVE STRENGTHS OF THE VIRGIN AND RECYCLED FURAN–LIGNIN FOAMS

Compression testing was performed using an Instron Universal Testing Machine (Model 3369) in accordance with ASTM D1621-04. The tests were carried out under ambient conditions using a flat-wise compression arrangement, in which the compressive force was applied parallel to the direction of foam rise [12]. The equipment included a computer system for accurate measurement of force and crosshead displacement. The crosshead speed was 2.0 mm min^{-1}. The foam specimens had dimensions of 10 cm × 10 cm × 2.5 cm. A compressive load of 10 kN was applied to reduce the foam to 10% of its initial thickness, following the approach described by Li *et al.* [15]. Each foam sample was tested in triplicate, and the average compressive stress was calculated.

2.6. FOURIER TRANSFORM INFRARED SPECTROSCOPY CHARACTERIZATION

Fourier transform infrared spectroscopy (FTIR) analysis was used to confirm whether the chemical structure of the foams was retained after THF dissolution and regeneration. FTIR allows structural changes, bond cleavage, and the possible formation of new functional groups after recycling to be evaluated by comparing the spectra of FLFs and RFLFs. FTIR spectra were recorded using a PerkinElmer Spectrum 2 FTIR spectrometer for both recycled and virgin furan–lignin foams. Potassium bromide (KBr) discs were used for sample preparation, and spectra were obtained over 600–4000 cm^{-1}. The KBr was oven-dried before use to minimize moisture interference.

2.7. THERMOGRAVIMETRIC ANALYSIS

TGA and derivative thermogravimetric analysis (DTG) were used to evaluate the thermal stability and thermal absorption characteristics of the virgin and recycled furan–lignin foams. All foam samples were oven-dried for 105 min at 125 °C before testing to determine their initial dry mass. Finely powdered dry foams were analyzed using a PerkinElmer TGA 4000 thermogravimetric analyzer. Approximately 5 mg of material was heated from 25 °C to 900 °C at a heating rate of 10 °C min^{-1} under a continuous nitrogen flow of 20 mL min^{-1}.

2.8. MORPHOLOGY OF THE VIRGIN AND RECYCLED FURAN–LIGNIN FOAMS

SEM was used to evaluate the structural characteristics and cell shapes of the virgin and recycled furan–lignin foams. The analysis was performed using a JEOL JSM-7600F SEM (Hitachi Co., Tokyo, Japan) at an accelerating voltage of 10 kV.

3. RESULTS AND DISCUSSION

The proposed reaction mechanism for producing recycled furan–lignin foam is shown in Figure 1. The mechanism highlights the function of hydrogen protons produced by the acid catalyst. These protons interact with lignin, FA, and glyoxal and initiate reactions that generate positively and negatively charged intermediates. Under acidic conditions, the three precursors undergo addition and condensation reactions to form a rigid bio-based foam. Glyoxal can react with both lignin and FA to form a three-dimensional structure containing ether linkages and methylene bridges. Furfuryl alcohol can also self-condense to form polyfuran structures. Lignin contributes to the foam structure and properties, and its aromatic content and complex chemical structure improve foam rigidity and stability [3, 8, 14].

Figure 2 shows images of the obtained foam products, while Table 1 lists the quantities of precursors and additives used in each formulation. The amount of FA relative to lignin-derived polyol was varied to develop the virgin FLFs (FLF1–FLF4). The recycled foams (RFLF1–RFLF4) were formulated by varying the proportion of FA to furan–lignin foam dissolved in THF, which was used as recycled polyol. The height, width, and length of each foam sample were measured to calculate volume. The foam masses ranged from 60.01 to 80.43 g, and the volumes ranged from 96.50 to 350.00 cm^3. FA concentration substantially affected foam volume; higher FA concentrations produced higher volumes and lower-density foams. The formulations with the highest FA content, FLF2 and RFLF2, showed the highest foam volumes. Other factors, including additives, fillers, and blowing-agent concentration, may also affect foam density and rise behavior. In contrast, the densest foams were produced by formulations with the highest recycled polyol content (RFLF4) and lignin-based polyol content (FLF4). These findings indicate that the proportions of FA and polyol components, whether lignin-derived or recycled, strongly influence foam volume and density.

3.1. DISSOLUTION INDEX OF THE FURAN–LIGNIN FOAMS IN EIGHT SOLVENTS

The dissolution behavior of virgin FLF1–FLF4 is shown in Table 2 and Figure 3. Eight solvents—distilled water, acetone, THF, EG, PEG-400, methanol, ethanol, and isopropanol—were used to assess dissolution. Figure 3 shows the percentage dissolution of the virgin foams, while Table 2 gives the corresponding hydroxyl values. The solvents showed different dissolution capacities. THF gave the highest average dissolution index (93.24%), followed by EG (82.33%) and PEG-400 (81.22%). Water gave the lowest dissolution index (1.10%).

Polymer dissolution has long been of interest to polymer scientists [16–22]. Compared with non-polymeric materials, polymers generally undergo more complex dissolution because mass transfer occurs at the solute–solvent boundary and is affected by the properties of both the solvent and the polymer. The general principle of "like dissolves like" suggests that polar solvents can dissolve polar polymers. All solvents used in this study were polar, whereas distilled water was the only inorganic solvent. THF, a polar cyclic solvent, produced the greatest dissolution, probably because of its favorable interaction with the polymer matrix of the foam. Polymer molecular weight, polydispersity, chain disentanglement, foam chemistry, additives, processing conditions, and environmental exposure may also influence dissolution efficiency [4, 16]. The overall dissolution efficacy followed the order THF ≥ EG ≥ PEG-400 ≥ methanol ≥ ethanol ≥ isopropanol ≥ acetone ≥ distilled water.

Table 2. Average dissolution indices of virgin furan–lignin foams in eight solvents and corresponding hydroxyl numbers.

Recycled polyol by solvent	Average dissolution (%)	Hydroxyl number (mg KOH g^{-1})
PEG-400	81.22	450
EG	82.33	445
Ethanol	61.01	275
THF	93.24	400
Methanol	63.21	270
Propanol	57.18	250
Acetone	44.53	150
Water	1.10	–
Lignin-based polyol (PEG-400)	–	450

Table 3 summarizes the dissolution performance of the solvents and the main characteristics associated with dissolution. THF was the best-performing solvent, with the highest dissolution efficiency for the virgin FLFs. Its effectiveness may be attributed to its moderate polarity, favorable hydrogen-bonding interactions, low viscosity, and compatibility with the polymer matrix. THF can enter the foam matrix readily because of its low viscosity and high diffusivity, and it provides favorable interactions with aromatic, oxygen-containing furan–lignin polymers. EG also showed high dissolution. As a polar protic solvent with strong hydrogen-bonding ability, EG interacts readily with polar functional groups such as hydroxyl and ether linkages in the furan–lignin foam matrix. PEG-400 was also effective, probably because of its polarity, hydrogen-bonding ability, and chemical compatibility with lignin-based materials, although its higher viscosity may slow diffusion.

Methanol and ethanol produced moderate dissolution. Methanol can penetrate the foam matrix because it is a small polar protic solvent with strong hydrogen-bonding capacity and low viscosity; however, its affinity for aromatic compounds appears lower than that of THF and EG. Ethanol behaved similarly but showed slightly lower dissolution, likely because of its larger molecular structure and weaker polarity. Propanol showed lower dissolution, which may be associated with greater viscosity, lower polarity, and weaker solvent–polymer interactions. Acetone, although moderately polar and low in viscosity, was less effective because of its limited hydrogen-bonding capacity and rapid evaporation. Water showed the lowest dissolution because it is poorly compatible with the non-ionizing aromatic and hydrophobic portions of furan–lignin polymers, despite its high polarity and strong hydrogen-bonding capacity. The chemical structure and arrangement of polymers influence dissolution behavior, and external variables such as temperature, solvent pressure, and agitation may also affect the rate of dissolution [20, 21].

One of the main goals of this study was to assess the hydroxyl values of recycled polyols for potential use in non-isocyanate foam production. The hydroxyl values of recycled polyols obtained by dissolving virgin FLFs in different solvents are summarized in Table 2. The acetone-derived polyol had the lowest hydroxyl value (150 mg KOH g^{-1}), whereas the PEG-400-derived polyol had the highest value (450 mg KOH g^{-1}). The higher hy-

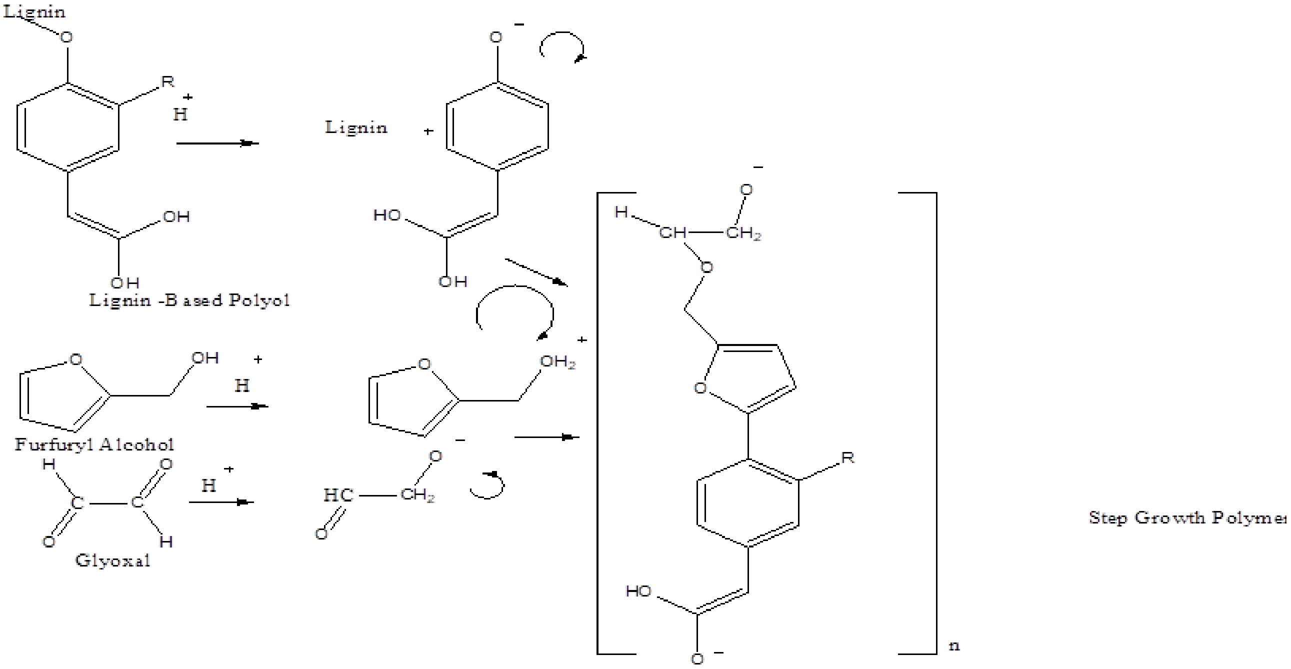

Figure 1. Proposed reaction mechanism for the phenolic part of lignin, glyoxal, and furfuryl alcohol under acidic conditions.

Table 3. Solvent dissolution performance of furan–lignin foams and important characteristics.

Solvent	Average dissolution (%)	Polarity	Hydrogen bonding	Viscosity	Solubility-parameter matching	Comment
THF	93	Good	Present	Low	Very good	Very good dissolution
EG	83	Very good	Strong	Medium	Good	Very good dissolution
PEG-400	80	Good	Strong	Medium	Good	Very good, but limited by viscosity
Methanol	65	Very good	Strong	Low	Moderate	Moderately high dissolution
Ethanol	62	Moderate	Moderate	Low	Moderate	Moderately high dissolution
Propanol	50	Moderate	Moderate	Medium	Less optimal	Average dissolution
Acetone	41	Moderate	Poor	Low	Poor H-bonding	Weak dissolution
Water	1.10	Very good	Strong	Low	Poor compatibility	Not soluble

droxyl value obtained for the PEG-400-derived polyol is probably associated with the polyhydroxyl functionality of PEG-400, which increases the number of reactive hydroxyl groups in the resulting polyol. This observation is consistent with previous lignin liquefaction studies in which PEG-400-liquefied lignin had a higher hydroxyl value than ethylene glycol-liquefied lignin [11]. The hydroxyl values obtained in this study fall within the recommended range of 350–830 mg KOH g^{-1} for bio-based polyols used in non-isocyanate foam production [17]. More broadly, solubility properties are used to assess polymer compatibility, chemical resistance, recyclability, and surface interactions in fillers, fibers, and colorants [17–19].

3.2. COMPARATIVE FTIR ANALYSIS OF RECYCLED AND VIRGIN FURAN–LIGNIN FOAMS

The primary components used to prepare the virgin furan–lignin bio-based foams were glyoxal, FA, and lignin-derived polyol. The FTIR spectra of the virgin (FLF1–FLF4) and recycled (RFLF1–RFLF4) foams are shown in Figure 4. The important functional groups and molecular bonds within the foam framework include C=C stretching in aromatic rings at 1450–1600 cm^{-1}, asymmetric and symmetric C–O–C ether linkages at 1050–1150 cm^{-1}, methylene C–H stretching at 2850–2925 cm^{-1}, and hydrogen-bonded hydroxyl groups at 3200–3600 cm^{-1}. These linkages are characteristic of furan–lignin foams [3, 14].

Furfuryl alcohol can undergo homopolymerization to produce polyfurfuryl alcohol, and lignin and FA can copolymerize. Glyoxal further contributes by reacting with lignin and FA. Therefore, the obtained foams are copolymers derived from these bio-based precursors. The proposed reaction mechanism between the components is shown in Figure 1. Tables 4 and 5 summarize the FTIR assignments for recycled and virgin foams, respectively. Figure 4 shows the similarity between the molecular functionalities of the two foam types. A broad absorption band from 3500 to 3200 cm^{-1} indicates O–H stretching vibra-

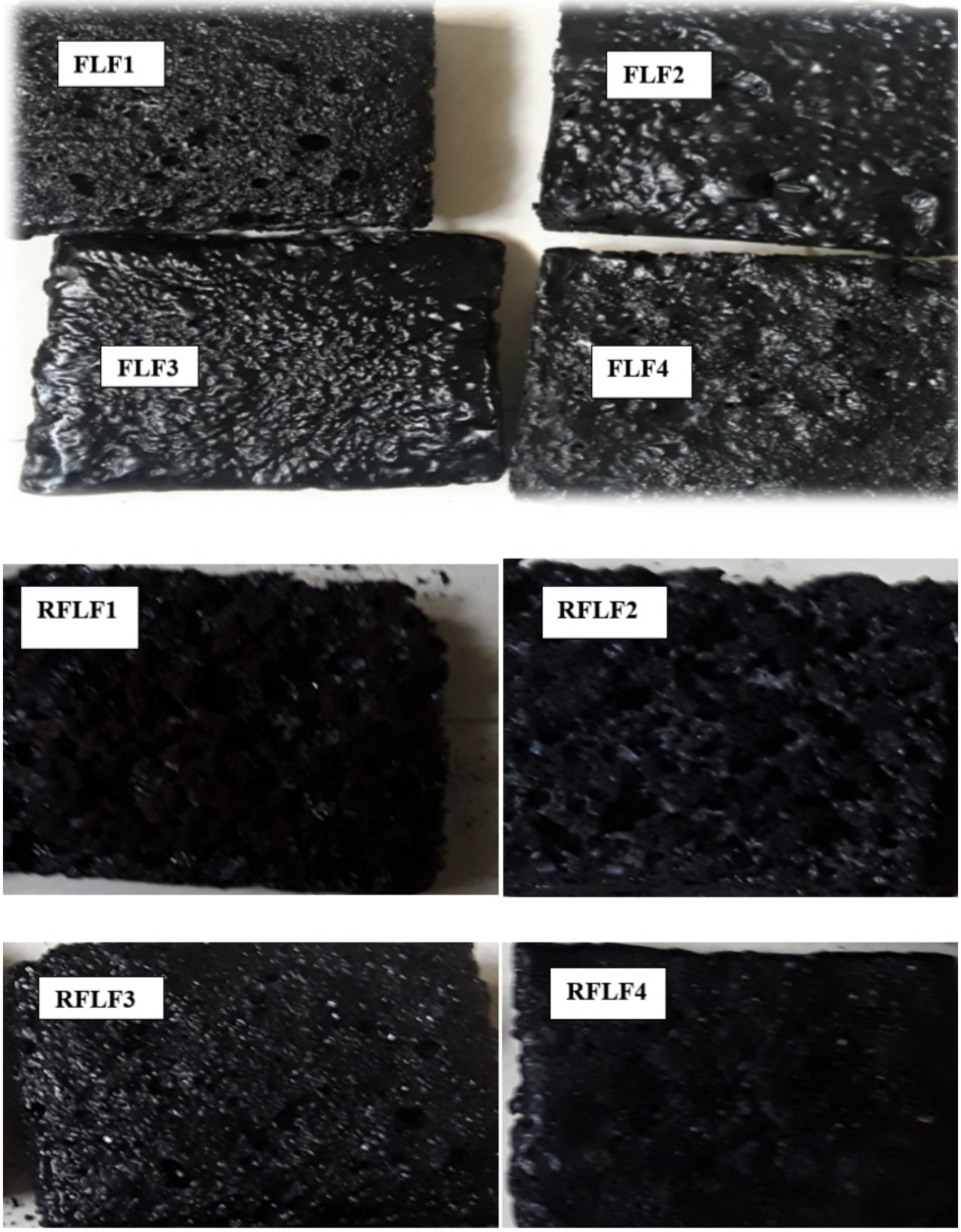

Figure 2. Image-based assessment of recycled furan–lignin foams and virgin foams prepared using the formulations in Table 1.

tions and hydroxyl groups from FA and lignin. A band at approximately 1610 cm^{-1}, assigned to C=C stretching, confirms furan or aromatic lignin units in the foam structure. All samples also show a strong peak around 1033 cm^{-1}, indicating the formation of ether bonds (C–O–C) during foam synthesis. Low-intensity bands near 2900 cm^{-1} correspond to C–H vibrations of CH_2 groups. A signal near 950 cm^{-1} suggests the presence of silica (SiO_2), probably from biomass sources such as corn stalk, which is known to contain silica [20]. Overall, the FTIR results indicate that the regenerated foams retained the main functional groups of the virgin foams, consistent with earlier furan–tannin foam data [21].

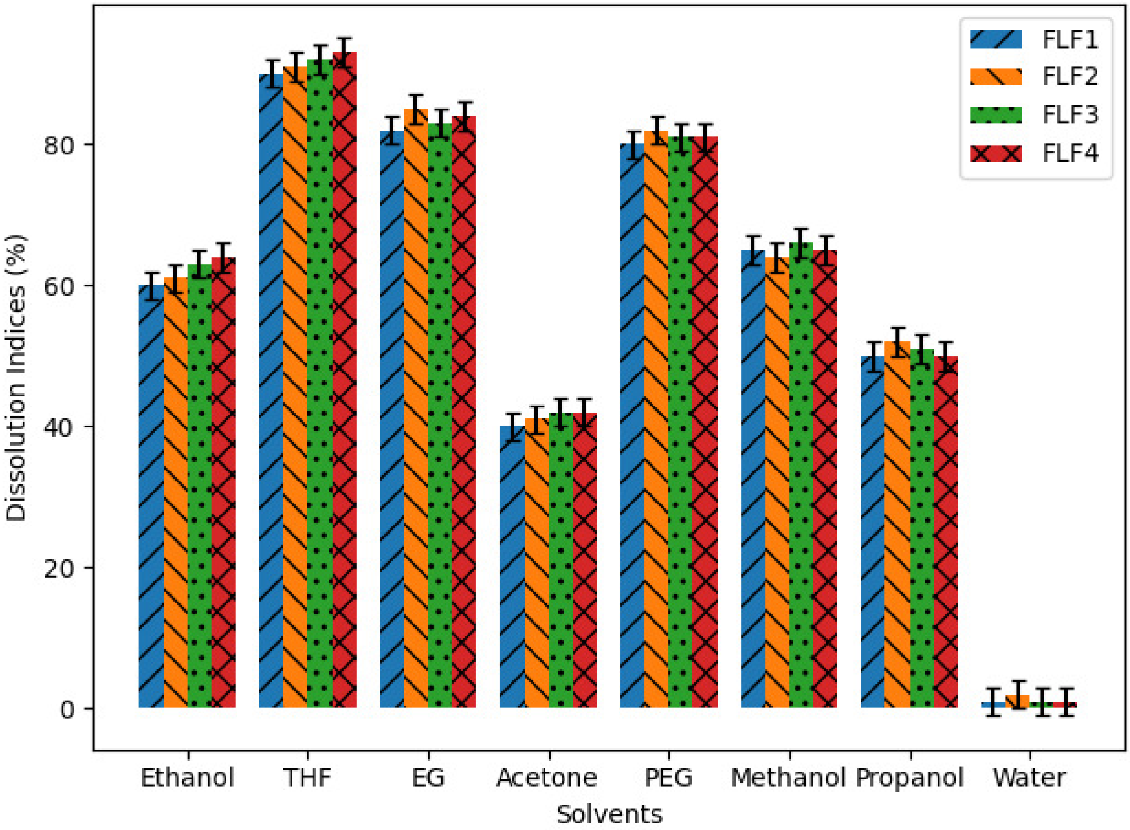

Figure 3. Dissolution index of the furan–lignin foams (FLF1–FLF4) in eight solvents.

Table 4. FTIR assignments for recycled foams (RFLF1–RFLF4).

Average wavenumber (cm^{-1})	Bond type	Functional group	Comment or assignment
1450–1600	C=C in aromatic rings	Aromatic rings	Aromatic ring stretching; moderate overtone bands may occur between 1660 and 2000 cm^{-1}.
3000–3100	=C–H stretching	Aromatic C–H	Narrow, weak to medium bands.
1050–1150	Asymmetric/symmetric C–O–C	Ether bonds	Strong, sharp peaks commonly observed in this range.
1650–1750	C=O in ketone, aldehyde, or ester groups	Carbonyl group	Strong, sharp band; exact position depends on the carbonyl type.
2850–2925	–CH$_2$– C–H stretching	Methylene linkages	Symmetric and asymmetric C–H stretching; medium to strong intensity.
1450	–CH$_2$– bending/scissoring	Methylene linkages	May overlap with aromatic C=C vibrations.
3200–3600	O–H stretching	Hydroxyl group	Strong, broad absorption due to hydrogen bonding.

3.3. THERMAL BEHAVIOR OF RECYCLED AND VIRGIN FURAN–LIGNIN FOAMS

The thermal behavior of the RFLFs was assessed using TGA and DTG, and the results were compared with previously described virgin FLFs [3, 8]. The TGA thermogram in Figure 5 indicates that RFLF4 showed similar or slightly better thermal performance than the corresponding virgin foam, FLF4. Table 6 lists the onset decomposition temperature (T_o), temperature at 50% mass loss ($T_{50\%}$), maximum degradation temperature (T_{max}), and char residue at 800 °C. Compared with the virgin foams, the regenerated foams generally showed improved thermal stability. This improvement may be associated with the aromatic structure of lignin, which contributes to thermal stability and makes lignin useful for applications that require thermal insulation or

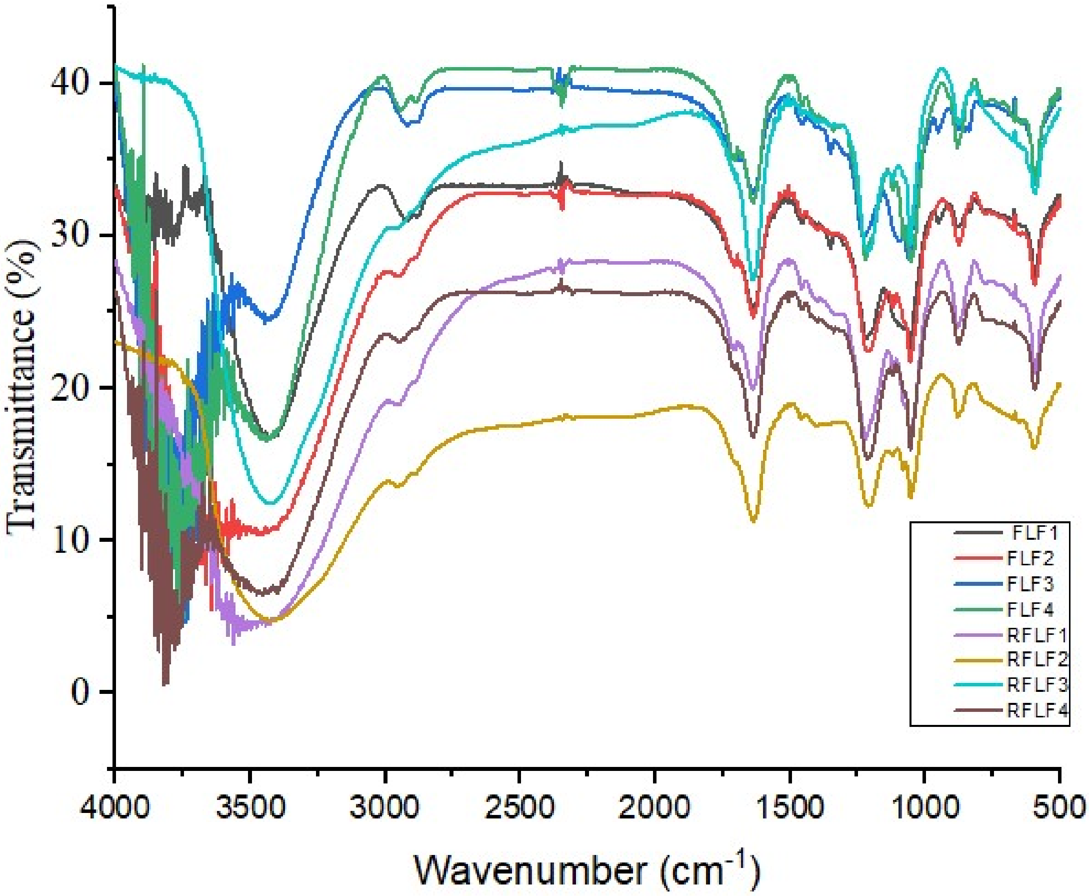

Figure 4. Comparative FTIR spectra of virgin furan–lignin foams and recycled furan–lignin foams.

Table 5. FTIR assignments for virgin foams (FLF1–FLF4).

Average wavenumber (cm^{-1})	Bond type	Functional group	Comment or assignment
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1050–1150	Asymmetric/symmetric C–O–C	Ether bonds	Strong, sharp peaks commonly observed in this range.
1650–1750	C=O in ketone, aldehyde, or ester groups	Carbonyl group	Strong, sharp band; exact position depends on the carbonyl type.
2850–2925	–CH$_2$– C–H stretching	Methylene linkages	Symmetric and asymmetric C–H stretching; medium to strong intensity.
1450	–CH$_2$– bending/scissoring	Methylene linkages	May overlap with aromatic C=C vibrations.
3200–3600	O–H stretching	Hydroxyl group	Strong, broad absorption due to hydrogen bonding.

fire resistance [23–30]. Lignin is therefore often considered as a flame-retardant or thermal-stability enhancer in foams and polymeric materials.

Thermal degradation usually occurs in multiple stages. The

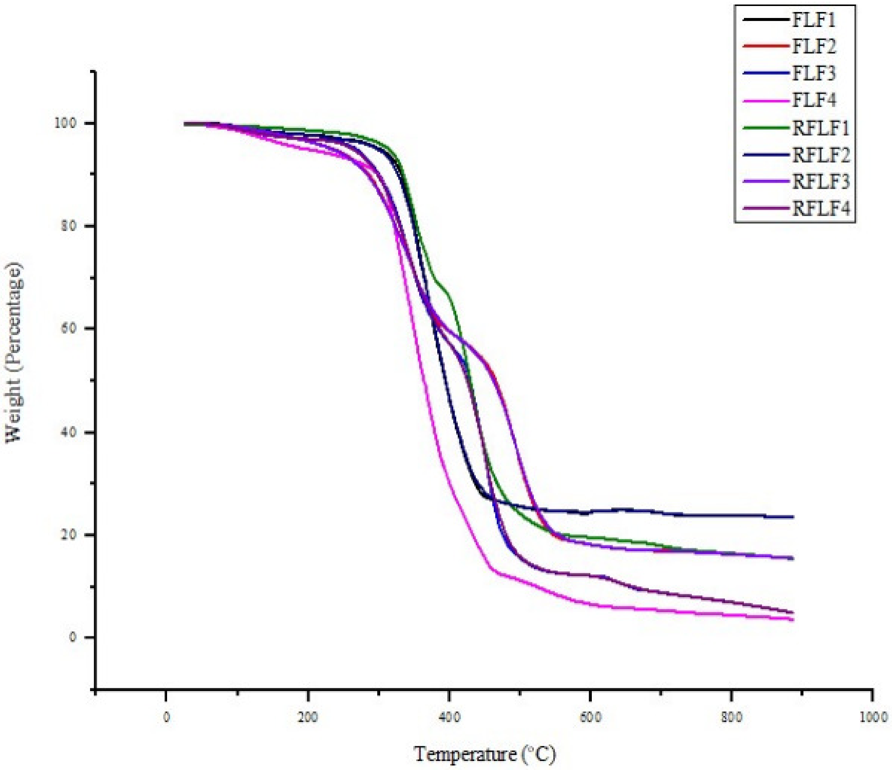

Figure 5. TGA thermogram comparing the thermal stability of recycled furan–lignin foams with their virgin counterparts.

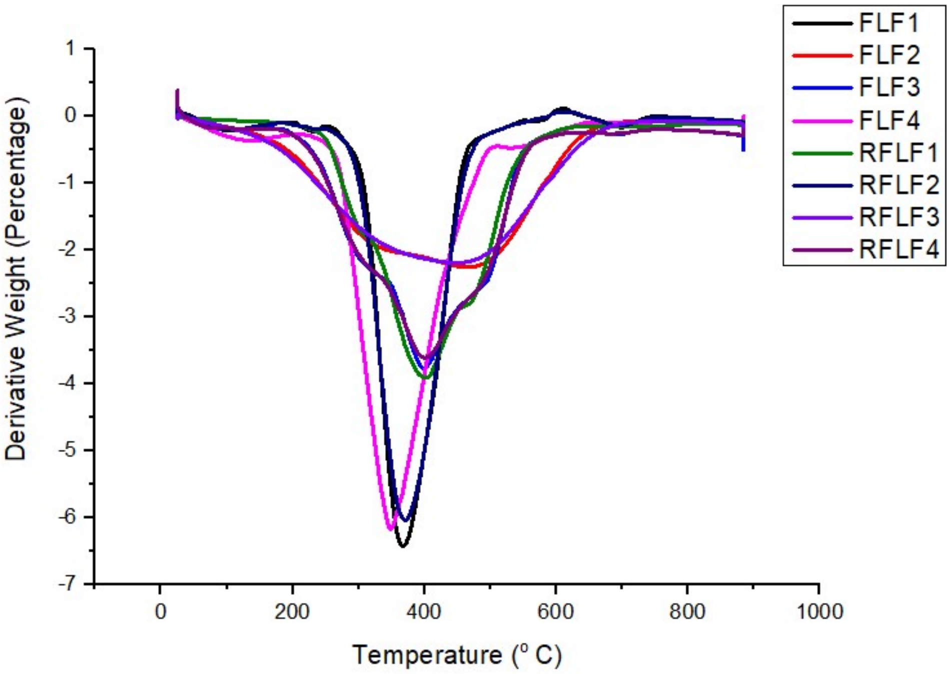

Figure 6. DTG curves of recycled furan–lignin foams and virgin counterparts.

first mass loss, between 20 °C and 125 °C, is associated with moisture and low-molecular-mass volatile evaporation. A second degradation stage, between 200 °C and 500 °C, corresponds to the breakdown of polymer chains into smaller molecules. At temperatures above 450 °C, pyrolysis of tannin and lignin becomes dominant. These degradation stages are reflected in Figure 5. The first stage of mass loss between 120 °C and 160 °C is associated with the evaporation of fluids and volatile organics. A

Table 6. Comparative thermal behavior of recycled furan–lignin foams and virgin counterparts.

Sample	T_o (°C)	T_{max} (°C)	$T_{50\%}$ (°C)	Char at 800 °C (%)
FLF1	171.28	459.98	390.50	21.45
FLF2	151.34	441.85	461.42	14.62
FLF3	162.60	456.44	399.82	5.95
FLF4	131.13	499.21	470.52	4.72
RFLF1	121.64	541.10	429.13	16.55
RFLF2	161.59	453.44	395.24	24.45
RFLF3	144.09	504.06	458.58	16.62
RFLF4	156.35	556.80	493.93	5.96

major second decomposition stage between 350 °C and 500 °C is associated with the breakdown of ester linkages, consistent with furan–tannin foam behavior reported previously [22]. The slightly higher thermal stability of the recycled foams may result from higher lignin and aromatic contents, which are associated with better thermal resistance [3, 8, 27].

The third degradation stage, between 400 °C and 600 °C, is attributed to thermal cleavage of the supporting chains of FA, lignin, and regenerated polyol. The fourth stage, between 600 °C and 800 °C, is characterized by degradation of organic structures, especially cleavage of C–O–C ether bonds. The TGA and DTG curves in Figures 5 and 6 support these observations. Table 6 summarizes the thermal degradation characteristics. Better thermal stability was associated with higher polyol concentrations. For example, FLF2 had the lowest T_{max} (441.85 °C), whereas RFLF4 had the highest T_{max} (556.80 °C). A higher FA proportion also led to greater residual mass after degradation. These results show that polyol concentration influences the thermal stability of both virgin and recycled furan–lignin foams.

According to earlier reports, hydrogen bonding promotes ether-bond formation, while acid-catalyzed self-condensation reactions generate both ether bonds and methylene bridges [24]. Similar processes are likely involved in the present system. Previous polyurethane foams reinforced with bamboo fiber showed lower residual contents and lower T_{max} values than those observed here [25]. The T_{max} range in the present work is more consistent with earlier studies of furan–glyoxal foams. The DTG thermograms show one main maximum at the negative end for both virgin and recycled furan–lignin foams, suggesting endothermic behavior and notable heat absorption. RFLF4, RFLF2, and FLF4 showed greater thermal absorption, whereas the remaining foams showed medium to weak absorption. This behavior may be related to the higher polyol concentration and the aromatic structure of lignin [21–23]. The results support the thermal stability and potential applications of the regenerated furan–lignin foams [26].

3.4. MECHANICAL PROPERTIES OF RECYCLED AND VIRGIN FURAN–LIGNIN FOAMS

The mechanical properties of the virgin and recycled foams are shown in Figures 7 and 8. Figure 7 shows the mechanical performance of four virgin foams (FLF1–FLF4) prepared with different precursor compositions. The recycled foams showed mechanical performance comparable to, and in some cases better than, their virgin counterparts (Figure 8). Earlier studies showed that increasing the proportion of phenolic polyol precursors can improve the tensile strength and structural integrity of lignin-based foams [26]. The regenerated foams generally showed moderate compressive strength comparable to that of the virgin foams. RFLF2 showed noticeably lower compressive strength, probably because it contained less recycled polyol. These results emphasize the importance of polyol content in determining the mechanical strength of furan–lignin foams; higher recycled polyol contents generally improved mechanical performance.

The recycled foams behaved mechanically in a manner similar to the virgin foams. The response to compressive loading is an important feature that distinguishes rigid foams from noncellular materials. In the closed-cell foams studied here, the initial elastic response is mainly due to compression of gas within the cells and bending and stretching of the foam cell walls and struts [1, 28, 29]. Rigid foams generally show three stages during compression: an initial linear elastic region at low stress, a plateau-like region in which deformation occurs at nearly constant stress, and a final densification region characterized by a sharp increase in stress [27].

3.5. DENSITIES OF VIRGIN AND RECYCLED FURAN–LIGNIN FOAMS

A clear relationship was observed between the densities of virgin and recycled furan–lignin foams, which ranged from 0.427 to 0.876 g cm^{-3}. The highest density was obtained for RFLF4, which was prepared using 40 g of recycled polyol and 60 g of FA. Variations in precursor composition affected foam density, as shown in Figures 9 and 10. Previous studies have highlighted the importance of polyol content in determining the properties of flexible foam composites [17, 18]. However, increased lignin molecular weight has been associated with decreased reactivity when lignin reacts with cross-linkers such as isocyanates [16].

For example, a formulation containing 50 g of FA and 30 g of lignin-based polyol produced low-density foams (0.427 g cm^{-3}). Recycled foams showed a similar trend (Figure 10). The density of virgin foams decreased to 0.495 g cm^{-3} when the lignin-based polyol concentration was increased to 40 g. The rapid depletion of free radicals required for effective cross-linking may explain why lignin-based polyol contents above the optimum reduced foam density. Consequently, density decreases when formation of a well-structured polymer network is hindered [31–33]. In contrast, regenerated furan–lignin foams showed an almost linear relationship between density and precursor variation. THF used during dissolution may increase the availability of furan monomers, thereby improving cross-linking efficiency and producing higher-density foams.

There is limited research explicitly examining the effects of FA, lignin-based polyol, and recycled polyol ratios on foam density [3, 8]. Previous studies have shown that increasing the concentration of hardening additives such as glyoxal can increase foam density [34–36]. Foam density and compressive properties are important and closely related parameters for applications requiring energy absorption, such as packaging and sports footwear [24, 25]. Feedstock composition also strongly influences the mechanical properties of the final foam [24]. The present results show that variations in precursor quantities affected both density and compressive strength (Figures 9 and 10). Lignin-

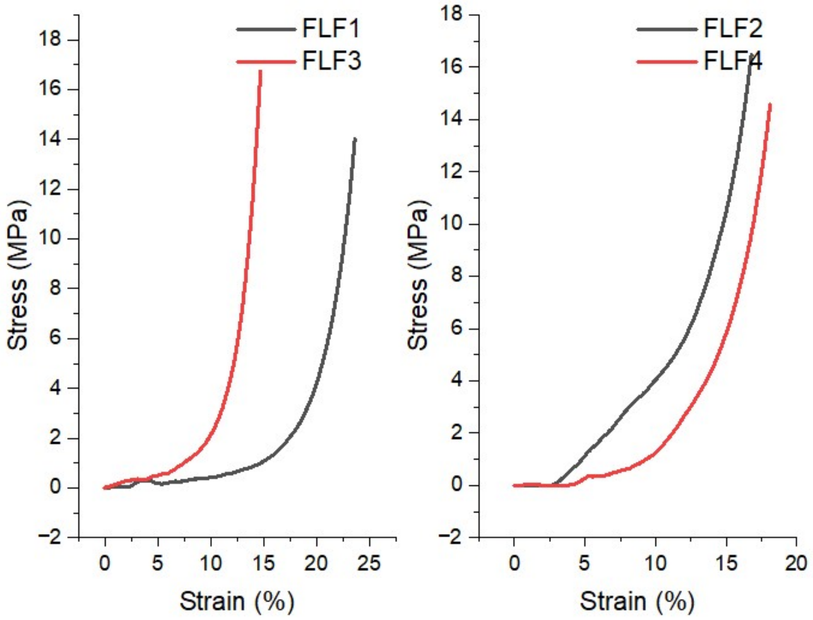

Figure 7. Compressive strengths of virgin furan–lignin foams.

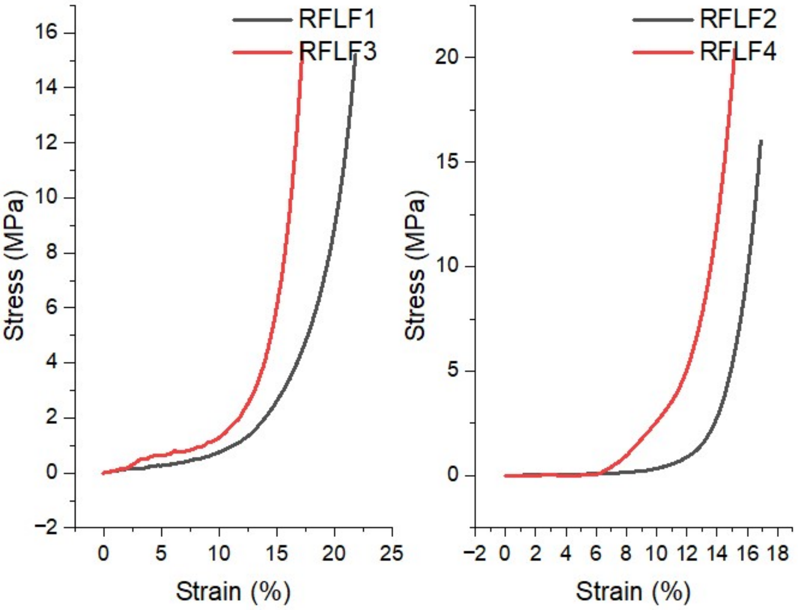

Figure 8. Compressive strengths of recycled furan–lignin foams.

containing rigid foams have been reported to improve biodegradability, compressive strength, and tensile strength [19, 32]. These improvements are probably associated with enhanced curing, gelling, and foaming conditions. Foam density is also influenced by the type and source of lignin [19].

The densities of the furan–lignin foams in this study (0.427–0.876 g cm^{-3}) are higher than the density ranges reported for tannin-based non-isocyanate foams (0.12–0.26 g cm^{-3}) [25, 36]. Lignin-reinforced tannin/furan foams with densities of 0.051 and 0.46 g cm^{-3} have also been reported, which are lower than those obtained in this work. These results suggest that the foams produced in this study, particularly the regenerated foams, may be suitable for impact-absorption applications because of their higher density and improved mechanical performance [19, 25].

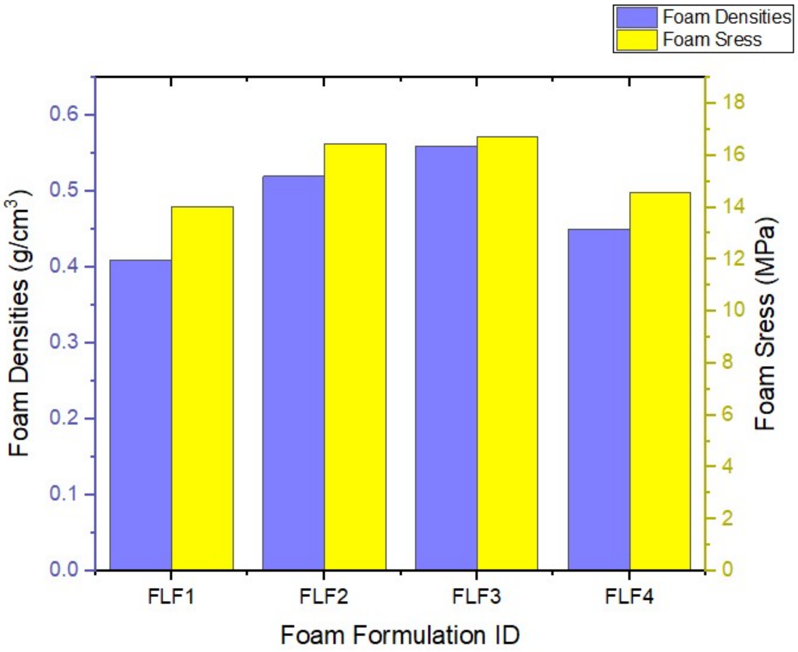

Figure 9. Densities and compressive strengths of virgin furan–lignin foams.

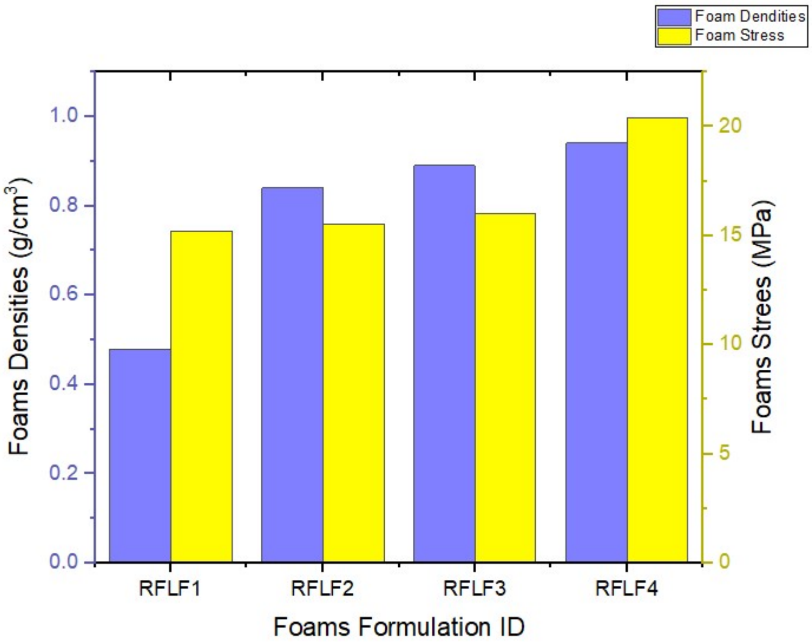

Figure 10. Densities and compressive strengths of recycled furan–lignin foams.

3.6. SEM MORPHOLOGY OF VIRGIN AND RECYCLED FURAN–LIGNIN FOAMS

SEM images of the virgin and recycled furan–lignin foams are shown in Figures 11 and 12. The images show that all foams have a predominantly closed-cell architecture with some interconnecting windows, which makes them potentially suitable for load-bearing applications and for thermal and acoustic insulation. The cell sizes range from approximately 20 to 200 μm and are affected by precursor concentrations. The similar and homogeneous cell morphologies of FLF1 and FLF2 indicate a relatively uniform structure. In contrast, imbalances in the proportions of polyol, either virgin or recycled lignin-based polyol, and cross-linker (FA) can disrupt foaming, cause premature or incomplete cell formation, and lead to collapsed or irregular cells [2, 3, 20].

Figures 11 and 12 also show how polyol content affects cell size. Higher recycled polyol levels in RFLF3 and RFLF4 produced smaller cell widths of approximately 50 μm and 20 μm, respectively. In contrast, RFLF1 and RFLF2, which were synthesized with lower polyol contents, had larger cell diameters of

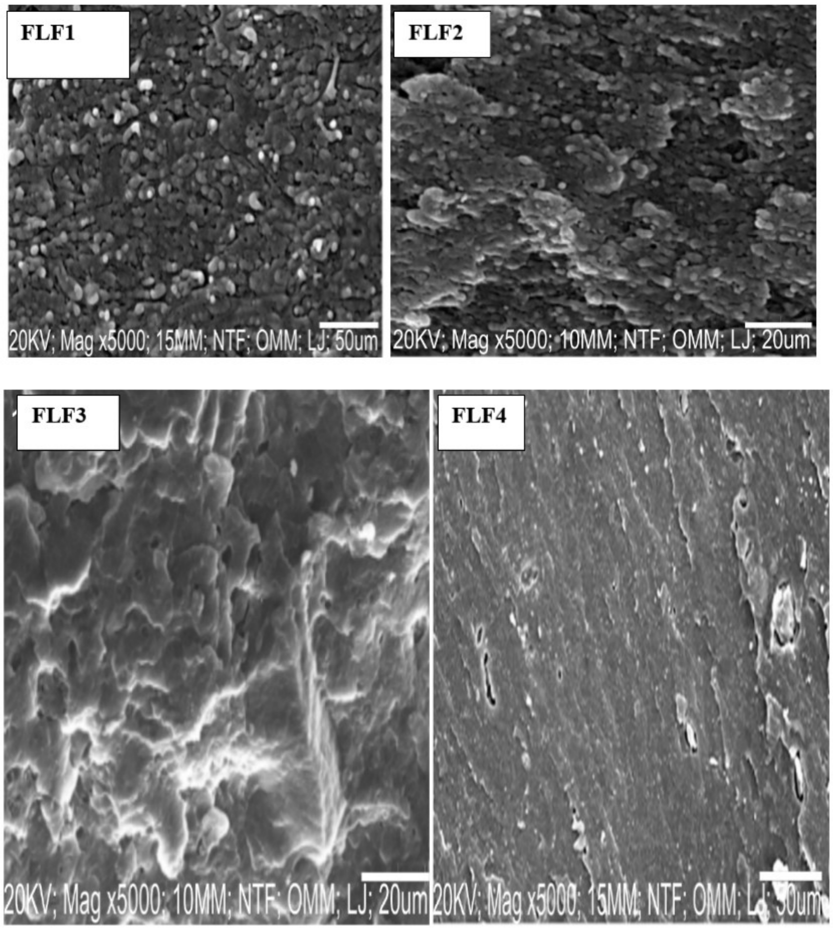

Figure 11. SEM morphology of virgin furan–lignin foams.

up to 200 μm. The virgin foams FLF1, FLF2, FLF3, and FLF4 retained smaller and more uniform cell sizes between approximately 20 and 50 μm. These results suggest that recycled polyols have a stronger influence on foam-cell morphology than FA concentration or virgin lignin-derived polyol. The recycled components may introduce differences in chemical structure or reactivity that alter foaming behavior and cell formation. The effects of lignin-based polyols, FA, and regenerated polyols on the SEM microstructure of lignin-based foams remain relatively underexplored [3, 11]. Gama *et al.* found that the addition of bamboo fiber to a rigid bio-based foam containing biodegradable polyol increased the cell diameter from 821.3 to 1101.5 μm [16]. Lignin addition has also been shown to improve flame retardancy, thermal insulation, cell irregularity, and stability in rigid foam formulations [26, 27].

The developed FLFs and RFLFs compare favorably with previously reported lignin-based and furanic foam systems. Lignin–furanic rigid foams have been reported to exhibit recyclability, flame retardancy, and variable density–strength relationships [31]. Tannin-based and furanic foam systems also show a wide range of physical and mechanical properties that depend on formulation and curing conditions [32–39]. Recent reviews and studies of bio-based foams, lignin-based rigid polyurethane foam nanocomposites, lignosulfonate flame-retardant systems, alkali-treated wood polyurethane foams, and self-blowing lignin-based foams further emphasize the importance of precursor chemistry, additives, and processing conditions for mechanical and thermal performance [40–44]. Similar density–strength relationships were observed in this study, but the RFLFs showed improved mechanical strength at relatively higher densities, suggesting better structural consolidation after recycling.

The aromatic character of lignin has previously been associated with good thermal stability and high char residue in lignin-based foams [43]. Compared with the virgin FLFs, the RFLFs prepared here showed improved thermal stability, indicating that THF-assisted dissolution and regeneration may promote structural rearrangement or higher cross-link density, thereby improving resistance to thermal decomposition. In addition, these foams achieved improved mechanical properties without high-temperature post-treatment, which is an advantage in terms of energy conservation and process simplicity compared with some lignin–tannin–furanic foams that require post-treatment such as carbonization to improve mechanical strength [24]. This study therefore demonstrates a closed-loop recycling approach, whereas many previous studies have focused mainly on the synthesis and characterization of virgin lignin-based foams. The re-

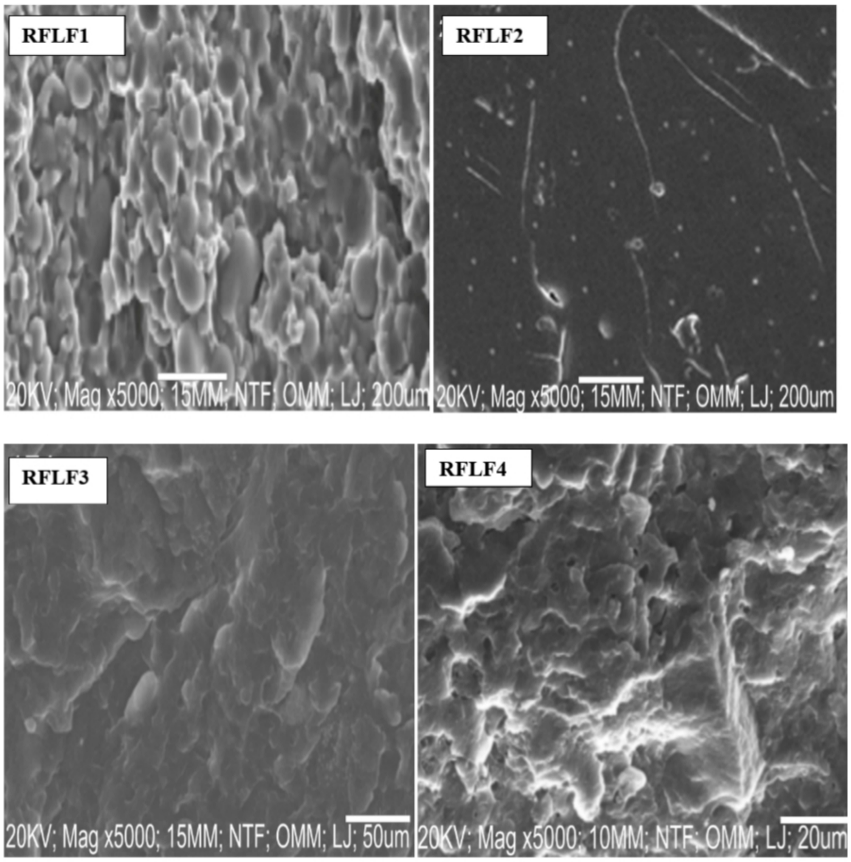

Figure 12. SEM morphology of recycled furan–lignin foams.

generated foams retained or improved key properties, advancing the practical relevance of lignin-derived foams within a circular-materials framework.

4. CONCLUSION

This study demonstrates that furan–lignin foams can be recycled using THF-assisted dissolution to produce regenerated foams that perform comparably to, or better than, the virgin materials. The THF-derived polyol showed hydroxyl values suitable for use in bio-based polymer systems. In regenerated foams, increased density was positively associated with improved mechanical strength and thermal stability. Morphological analysis also revealed structural differences that may contribute to the improved properties. Overall, the results demonstrate the potential of furan–lignin foams for circular-materials applications, particularly in load-bearing and thermally demanding uses. This work supports ongoing efforts toward resource efficiency and environmentally friendly materials development and provides a basis for further development of sustainable reuse and recycling strategies for bio-based foams.

DATA AVAILABILITY

The data will be available on request from the corresponding author.

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