Nondestructive Evaluation of Lignin-Foam Blocks Using Acoustic Velocity Method

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Abstract

Structural insulated panel foams are made primarily from petrochemicals. As a biopolymer, lignin is a promising raw material substitute for foam production. Nondestructive evaluation is routinely used for structural material strength and stiffness assessment. Herein, mechanical acoustic velocity (AV) was used to nondestructively evaluate lignin-foam blocks. Overall, foam block weight was determined, and flying time was measured for various foam thicknesses ranging from 7.26 to 8.89 cm (3.0 to 3.5 in.). Additionally, for each block, AV was measured at several different positions/locations. The AV was calculated by dividing the distance (mm) by flying time (μs). Data were analyzed using two-way analysis of covariance, and the Pearson correlation and regression were also determined for foam thickness. There was no significant interaction between position and thickness for AV. Also, no significant correlation or regression was found between foam thicknesses. However, 8.38 cm (3.3 in.) thick exhibited a higher AV compared with all other thickness treatments, and AV was the lowest for 8.38 (3.3 in.) and 7.87 cm (3.1) thick; 7.26 cm (3.0 in.) remained intermediate ($P < 0.0001$). Furthermore, the highest AV belonged to back positions, whereas the lowest AV was observed in the length position, and front and length positions were intermediate ($P < 0.0001$). These findings indicate that lignin-foam block uniformity may not be consistent at different positions and the appropriate foam thickness should perhaps not be >8.38 cm (3.3 in.).

Wood consists of three major components: cellulose, hemicellulose, and lignin. Cellulose and hemicellulose can be hydrolyzed into monosaccharides (de Vries et al. 2021). Lignin, however, is composed of *p*-hydroxyphenyl, guaiacyl, and syringyl units that are made from the polymerization of hydroxycinnamyl alcohols as well as other aromatic monomers. This polymerization results in a polyphenolic structure that has a variable subunit composition and complex interunit linkages (Vanholme et al. 2019). It is well documented that lignin is a readily available sustainable resource. The lignin portion of lignocellulosic biomass often ranges between 15 and 35 percent (Kang et al. 2014, Xu et al. 2014, Baeyens et al. 2015, Liu et al. 2015). Currently, commercially extracted lignin comes from the pulp and paper industry and is mainly used for producing fuels and chemicals (Hasan et al. 2023). Furthermore, lignin has a wide range of applications including water flocculation (Chen et al. 2020), ultraviolet aging resistance (Wang et al. 2020), epoxy asphalt (Song et al. 2023), and high-performance sustainable polymeric materials (Chiappero et al. 2021). However, there are limited applications for lignin in the construction industry.

Structural insulated panels (SIPs) are the high-performance building materials for residential and light commercial construction. SIPs have sandwich-type architecture in which an insulating foam core is located between two structural facers. Facers are usually oriented strand board (McDonald et al. 2014). Currently, expanded polystyrene, extruded polystyrene, or polyurethane are the most common foam insulation materials for commercial SIP production (McDonald et al. 2014). Although traditional foam-based SIP has shown promising results with respect to mechanical properties (Cox and Hamel 2021), energy efficiency, and thermal resistance (Aldrich et al. 2010), these materials do not break down in the environment. Instead, they break down into microplastics over time, particularly with exposure to sunlight. This structure is hardly recyclable, and its recycling process is associated with releasing toxic emissions and creating hazardous waste (Tan and Khoo 2005). Therefore, sustainable and recyclable alternatives that can be

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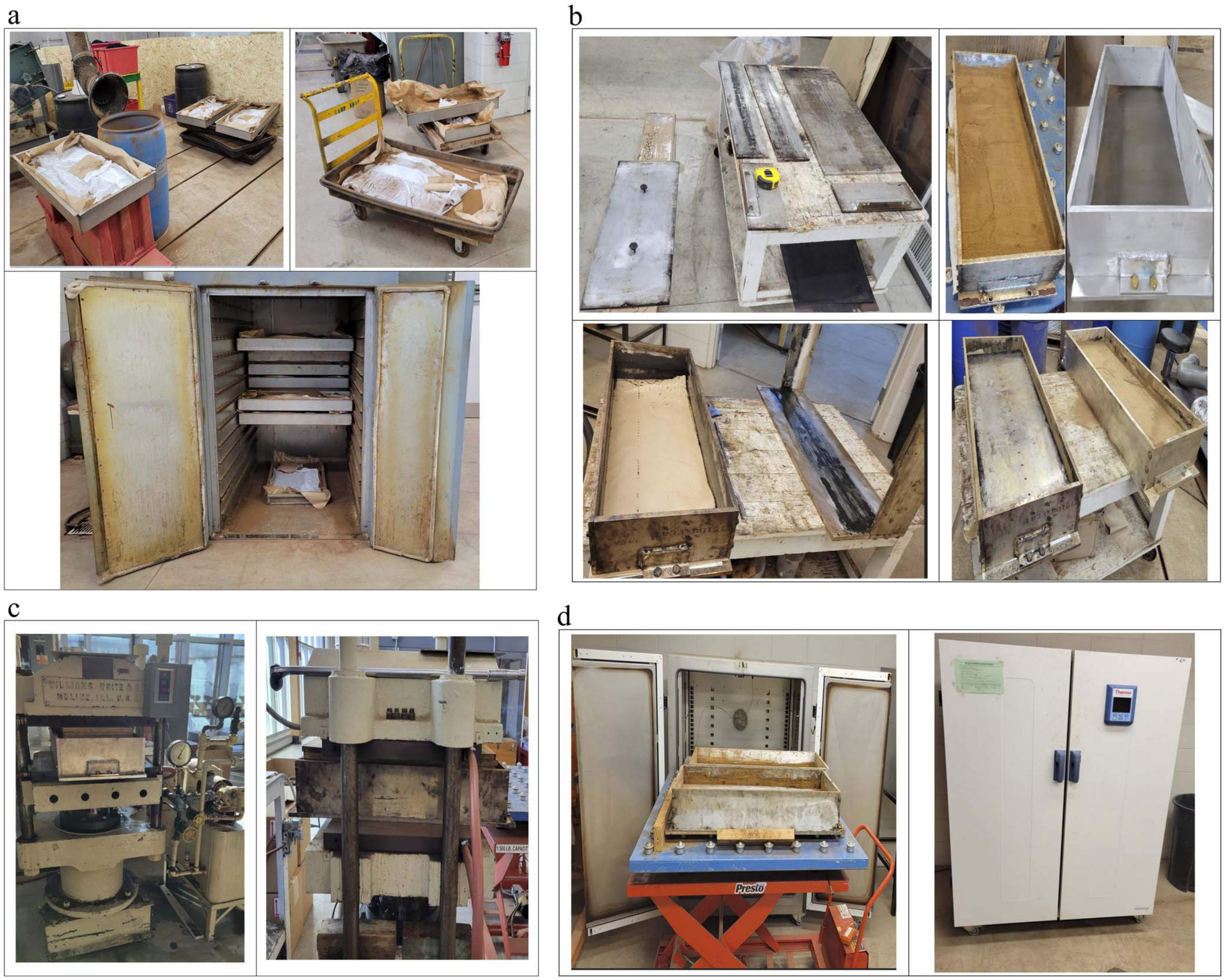

Figure 1.—(a) Drying lignin, (b) preparation and filling the lignin-foam molding frame, (c) pressing the molding frame, and (d) transferring lignin-foam blocks to the 240°C oven.

compatible with commercial foam core can be beneficial for environmental concerns.

Lignin represents >25 percent of the dry constituent components of wood and is considered a renewable material that has been commercially used in various applications, for example, energy recovery (Sixta 2006, Ek et al. 2009) and as an additive for asphalt (Song et al. 2023). Spent pulping liquor, as a sustainable source, is the major source of lignin. It can be also used as a low-cost feedstock for the preparation of innovative materials. Foam from lignin can be denser than that from tannin (Tondi et al. 2016). Lignin-foam density can vary between 185 and 407 kg m^{-3}. Additionally, lignin foam has shown promising results for fire resistance and leaching tests (Tondi et al. 2016). These results indicate that lignin foam may serve as an environmentally friendly alternative for traditional insulation materials such as polystyrene and polyurethane. Although the above findings indicate that lignin foams can be environmentally friendly replacements for traditional foams, mechanical property evaluation is required.

Nondestructive evaluation (NDE) technology has been widely used to assess the quality and integrity of various wood properties (Ross 2015). Acoustic velocity (AV) is a NDE technique for predicting density and stiffness. Li et al. (2024) reported that approximately 35 percent of stiffness and density variation in bamboo materials was detected using AV technology. In addition, there is close relationship between AV and destructive evaluation. AV is often a suitable indicator for modulus of rupture, i.e., strength (Sato and Fushitani 1991). AV has been further used for tensile damage (Pierre 2000), micromechanisms of damage growth (Baensch et al. 2013), and internal damage (El-Hajjar and Qamhia 2013) in various wood species. However, the mechanical properties of lignin-foam blocks have not been nondestructively evaluated using AV. Therefore, the objective of this study is to measure and analyze the AV response in various lignin-foam block thicknesses at different foam block positions.

Materials and Methods

Lignin-foam block formation

Foam blocks were produced via a sequence of steps (Figs. 1a to 1d). Production occurred at the US Department of Agriculture (USDA) Forest Product Laboratory (FPL). Approximately 2 tons of fresh lignin were received. It had

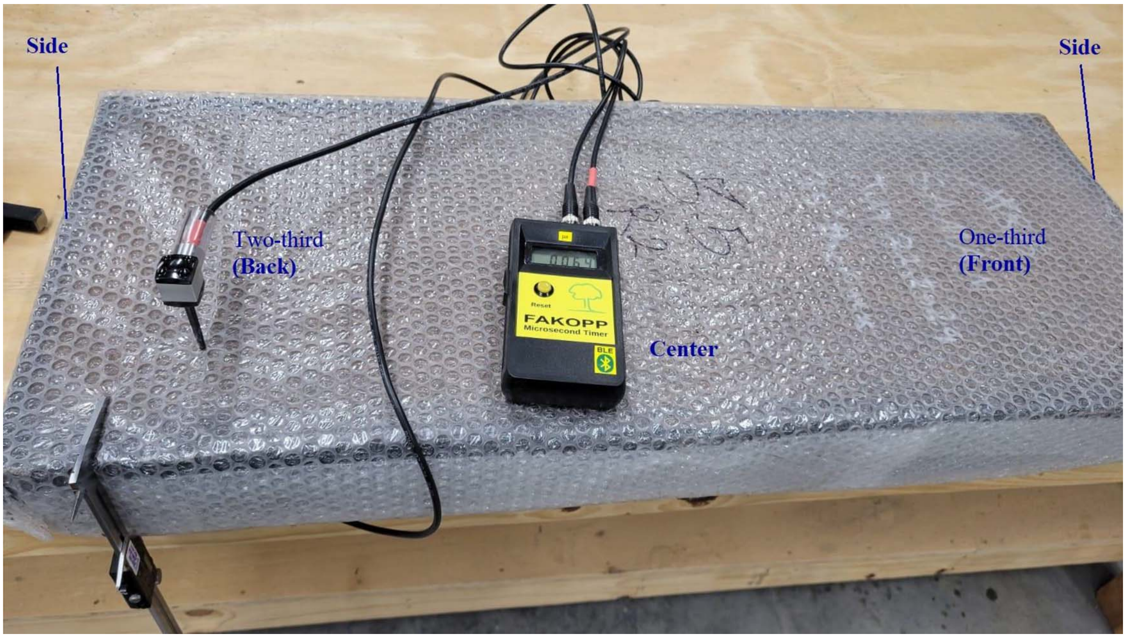

Figure 2.—Different positions that were assigned to measure flying time.

approximately a 44 percent moisture content (MC). Once received it was kept in a walk-in cooler. To reduce MC for homogenization and production the fresh lignin was air dried at room temperature (18 to 20°C) for 40 to 60 days. The average daily moisture loss was approximately 0.5 percent. Homogenization was performed with a screen grinder. The ground lignin was then transferred to a dryer at 65°C for 12 hours (Thermo Fisher Scientific, Waltham, MA) to reach 2 to 4 percent MC. Then, the 2 to 4 percent MC lignin was mixed with homopolymer polypropylene (PP) at a 5.6:1.0 ratio, respectively. Essentially, 150 g of PP were added to 800 g of dried lignin and then mixed with an ultraheavy-duty HP blender (Ninja, Austin, TX) for 35 seconds. Once prepared, up to 2 tons of this mixture were maintained at room temperature for subsequent lignin-foam block production.

After mixture preparation, the lignin-foam molding frame (88.9 cm [35 in.] length, 35.6 cm [14 in.] width, and 14.7 cm [5.8 in.] depth) was assembled. Synthetic grease as a lubricator (release agent) was brushed onto the interior surfaces of the molding frame to minimize bonding between the lignin foam and said frame. Then, each molding frame was fully filled with 12.5 kg (27.5 lb) of the mixture. Later, the lignin mixture within each molding frame was pressed at 900 psi for 100 to 120 seconds. After pressing the foam was transferred using a manual lift table (National Presto Industries, Inc., La Crosse, WI) to an oven (Thermo Fisher Scientific) for heating at 240°C for 1 hour. The sample and oven temperatures were continuously monitored during this step. After the heating step, the 7.1- to 8.89-cm (2.8- to 3.5-in.)-thick, 35-cm (13.8-in.)-wide, and 86.4-cm (34-in.)-long lignin-foam block was complete. This entire process required

Figure 3.—Exemplar of measuring the distance and flying time on the lignin-foam block.

Table 1.—Experimental layout indicating replicates used in this study and summary statistics of acoustic velocity in five different lignin-foam thicknesses and four block positions.

Treatment combination		Mean	Median	SD	Coefficient of variation (%)	Maximum	Minimum	Replicates
Thickness × Position								
3.0	Length	1.098	1.104	0.0482	4.391	1.177	1.016	12
	Front	1.318	1.311	0.1283	9.737	1.624	1.163	12
	Center	1.351	1.323	0.1580	11.697	1.739	1.173	12
	Back	1.346	1.371	0.1634	12.138	1.721	1.153	12
3.1	Length	1.052	1.056	0.0412	3.911	1.115	0.968	12
	Front	1.265	1.277	0.1001	7.915	1.436	1.109	12
	Center	1.299	1.298	0.1120	8.619	1.459	1.106	12
	Back	1.321	1.322	0.1166	8.830	1.572	1.148	12
3.2	Length	1.071	1.070	0.0580	5.411	1.195	0.988	12
	Front	1.291	1.294	0.1137	8.810	1.434	1.115	12
	Center	1.269	1.243	0.1070	8.431	1.466	1.143	12
	Back	1.333	1.326	0.1486	11.148	1.633	1.105	12
3.3	Length	1.060	1.064	0.0653	6.162	1.158	0.900	12
	Front	1.298	1.299	0.0676	5.209	1.435	1.150	12
	Center	1.230	1.264	0.1491	12.121	1.382	0.927	12
	Back	1.341	1.311	0.1212	9.035	1.602	1.166	12
3.5	Length	1.113	1.122	0.0612	5.495	1.202	1.031	9
	Front	1.395	1.357	0.0785	5.627	1.557	1.329	9
	Center	1.396	1.357	0.1036	7.423	1.582	1.262	9
	Back	1.441	1.469	0.1119	7.763	1.582	1.254	9

approximately 15 to 17 hours for two molding frames, which produced two lignin-foam blocks. Given the relatively small production sizes, there is inherent variability introduced from block to block. To optimize homogeneity and minimize variability one can envision either a continuous production process or a batch process of sufficient size and scale wherein fewer batches are required to produce the same volume of foam blocks. Once produced, the lignin-foam blocks were transported to Department of Sustainable Bioproducts at Mississippi State University for subsequent evaluation. The lignin foam formation procedure is presented in Figures 1a to 1d.

AV and its reciprocal, time of flight (TOF), have been widely used to measure wood quality and soundness. A review of this technology is provided by Pellerin and Ross (2002). In principle, denser and more mechanically sound solid materials conduct sound more quickly. Wood, and in this case lignin foam, are each comprised of solid materials and air. It has been widely demonstrated that denser wood types, with more woody substance and less air per unit volume, transmit sound waves faster. Here it was hypothesized that lignin-based foam would behave according to the same principle.

Experimental layout

In total, 57 blocks were individually weighed and measured. Twelve replicates of each thickness 7.62, 7.87, 8.13, and 8.38 cm (3.0, 3.1, 3.2, and 3.3 in.), respectively and nine replicates for the 8.89-cm (3.5-in.) thickness were measured at one-third (front), central (center), two-thirds (back), and sides (length) regions. The 8.63-cm (3.4-in.) thickness was excluded for statistical comparison because of inadequate number of replications. These different block positions are illustrated in Figure 2. Thus, five different thicknesses and four positions were examined with NDE.

Nondestructive evaluation

AV was determined with a Fakopp microsecond timer (Fakopp Enterprise, Agfalva, Fenyo u, Hungary). The flight time (μs) was measured for each of front, central, back, and side positions for each of the 57 lignin foam blocks. Figure 3 shows the block lengths as well as TOFs for one lignin-foam block. The corresponding probes for measuring TOF were inserted at approximately 2.54-cm (1-in.) depth at various locations. The AV was calculated using the following formula:

$$\text{AV or speed of sound} = \frac{\text{Distance (mm)}}{\text{Time of flight } (\mu\text{s})} \tag{1}$$

$$\text{TOF} = \frac{\text{Time of flight } (\mu\text{s})}{\text{Distance (mm)}}$$

Statistical analysis

The experimental design was a completely randomized design where each lignin-foam block was considered an experimental

Table 2.—Difference on lignin-foam block weights.

	Treatment				
	7.62 cm (3.0 in.)	7.87 cm (3.1 in.)	8.13 cm (3.2 in.)	8.38 cm (3.3 in.)	8.89 cm (3.5 in.)
Weight (kg) Weight (Ib)	11.64 (25.66)	11.74 (25.88)	11.99 (26.43)	11.70 (25.79)	11.77 (25.95)
SEM (kg)	0.107				
SEM (Ib)	0.236				
P value	0.130				

Table 3.—Pearson correlation of different lignin-foam blocks.

	Thickness 3.0	Thickness 3.1	Thickness 3.2	Thickness 3.3	Thickness 3.5
Thickness 3.0	1	0.22378	−0.07692	0.55245	0.03333
		0.485	*0.812*	*0.063*	*0.932*
		12	12	12	9
Thickness 3.1		1	0.3986	−0.04895	0.15
			0.199	*0.880*	*0.700*
			12	12	9
Thickness 3.2			1	0.11189	−0.41667
				0.729	*0.265*
				12	9
Thickness 3.3				1	0.48333
					0.188
					9
Thickness 3.5					1

P values for each comparison are italicized. There were 12 replicates per each comparison between 7.62 cm (3.0 in.), 7.87 cm (3.1 in.), 8.13 cm (3.2 in.), and 8.38 cm (3.3 in.), but those comparisons with 8.89 cm (3.5 in.) had 9 replications.

unit. All data were tested and confirmed to be normally distributed. Minimum replication number per each treatment was determined to 6 using power analysis. One-way analysis of variance was used for block weight and AV data were analyzed using two-way analysis of covariance with five by four factorial arrangement of treatments to test for the interaction and main effects of the five lignin-foam block thickness treatments (3.0, 3.1, 3.2, 3.3, and 3.5 in.) and four positions (front, center, back, and length). Block weight was considered as covariate for AV data. The following statistical model was used for AV data:

$$Y_{ij} = \mu + T_i + P_j + (TP)_{ij} + \beta(x_{ij} - \bar{x}_{ij}) + E_{ij} \qquad (2)$$

where μ is the population mean; T_i is the effect of lignin-foam block thickness treatments ($i = 1$ to 5); P_j is the effect of position treatments ($j = 1$ to 4); $(TP)_{ij}$ is the interaction of each lignin-foam block thickness treatment with position treatment; $\beta(x_{ij} - \bar{x}_{ij})$ is a covariate coefficient multiplied by the difference between any individual variable (x_{ij}) and the average of that variable ($\bar{x}_{ij}$); and E_{ijk} is the residual error.

The lignin weight and AV data were analyzed using a general linear mixed model (PROC GLIMMIX) of SAS 9.4© (SAS Institute, 2013), and differences between means were classified by pairwise comparisons, and unless otherwise noted, differences were considered significant at $P \leq 0.05$. In addition, Fisher's protected least significant difference was used for determination of mean separation (Steel and Torrie 1980). Pearson correlation coefficients were analyzed for the different thicknesses and regression was performed between lignin weight and AVs of different thicknesses using the CORR and REG procedures, respectively of SAS 9.4© (SAS Institute Inc, 2013).

Results and Discussion

The summary statistics of AV at different block thicknesses combined with various positions are presented in Table 1. Lignin-foam block weights did not differ significantly among all five thicknesses (Table 2). No significant positive or negative correlations among block thicknesses and AV were observed (Table 3). There was also no meaningful linear regression relationship in AV among thicknesses ($P = 0.920$; Fig. 4). Additionally, no linear ($P = 0.228$), quadradic ($P = 0.678$), or cubic ($P = 0.271$) regression was observed between AV and block weight. There was no interaction effect detected between thickness and position on AV; however, there were significant main effects for thickness and position ($P < 0.0001$; Table 4). The 8.89-cm (3.5-in.) thickness had a higher AV compared with all other thicknesses and 8.38 cm (3.3 in.) exhibited lower AV compared with 7.26 cm (3.0 in.), 7.87 cm (3.1 in.), and 8.89 cm (3.5 in.; Fig. 5). In addition, AV was the highest in the back compared with other positions and it was the lowest in the length; front and center were intermediate (Fig. 6).

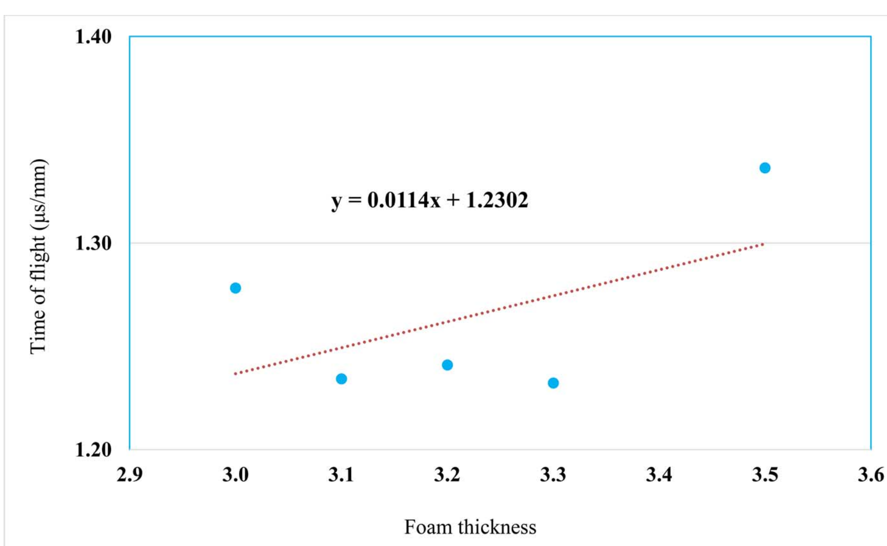

Figure 4.—Regression among lignin-foam block thickness. $P = 0.9195$; $R^2 = 0.1743$.

Table 4.—*Effects of lignin-foam block thicknesses and positions on acoustic velocity.*

Treatment	Acoustic velocity (mm/μs)[a]
Thickness	
7.62 cm (3.0 in.)	1.281b
7.87 cm (3.1 in.)	1.234c
8.13 cm (3.2 in.)	1.237bc
8.38 cm (3.3 in.)	1.234c
8.89 cm (3.5 in.)	1.336a
SEM	0.0207
Position	
Length[b]	1.079c
Front[b]	1.313b
Center[b]	1.309b
Back[b]	1.356a
SEM	0.0207
P value	
Thickness	<0.0001
Position	<0.0001
Thickness × Position	0.846

[a] Treatment means within the same column within effect without common lowercase letters are significantly different ($P \leq 0.05$).
[b] One-third (front) and two-thirds (back), central (center), and two sides (length) areas of lignin-foam block, represented in Figure 2.

The aim of current study was to evaluate the AV response as a representative of NDE technology for lignin-foam blocks when different thicknesses and positions were investigated. The results of this study showed that density and AV are not uniform through the lignin-foam block. If AV is calculated by dividing distance by time, the higher AV response seemed to be associated with lower specimen density. AV has been used as an indicator for stiffness and strength, in which a linear increase in AV response was shown to be likely linked be a higher stiffness (Li et al. 2024). Subsequent testing will help indicate the extent to which AV may be an indicator of strength or stiffness in these foam blocks. Although promising results were reported for characteristics such as being fire retardant and leaching (Tondi et al. 2016), the mechanical characteristics of lignin foam destructively and nondestructively have not been previously evaluated. The findings of current study suggested that there is minimal variation among blocks (Table 1) regardless of thickness or position; however, 8.89-cm (3.5-in.) thick blocks may not be recommended because of higher AV response considering minimal differences were observed for 7.87- to 8.38-cm (3.1- to 3.3-in.)-thick blocks. Therefore, on the basis of the production equipment used herein the lignin-foam blocks should perhaps be in the range of 7.87 to 8.38 cm (3.1 to 3.3 in). Given that each foam block contained the same mass of material, it is logical that thicker blocks were necessarily less dense. TOF did not exactly correlate to thickness or density. A possible source for this variation may be that the pressing conditions varied from panel to panel and that some panels had more of a density gradient than others, which would influence the overall TOF or AV response.

Moreover, AV response was varied throughout the block without following a consistent pattern, indicating that foam blocks are not necessarily internally homogenized. Lignin-foam block formation can also be enhanced by better homogenization and distribution of the lignin and PP mixture.

It is intended that this foam be developed for use in SIPs. Greater homogeneity of the foam during production will likely lead to better homogeneity of SIP mechanical properties. Greater homogeneity of material strength facilitates greater allowable design value. As such, the importance of improving homogeneity has a direct and positive impact on utility and economic value.

There are several sources of potential error in this process. These include but are not necessarily limited to foam density and its interaction with stress wave pin coupling, pin coupling driving force, pin coupling depth across block density gradients from surface to core, and within block density gradients from surface to core.

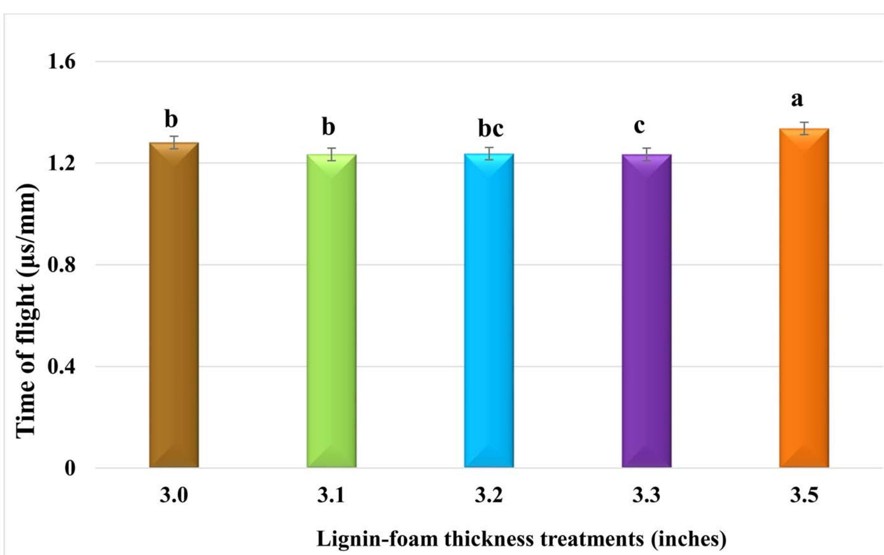

Figure 5.—*Main effect of 3.0- to 3.5-in. thickness on the acoustic velocity of lignin foam blocks. Treatment means within the same column within effect without common lowercase letters are significantly different. Number of replications per treatment combination is 12 or 9; $P < 0.001$; SEM = 0.0246.*

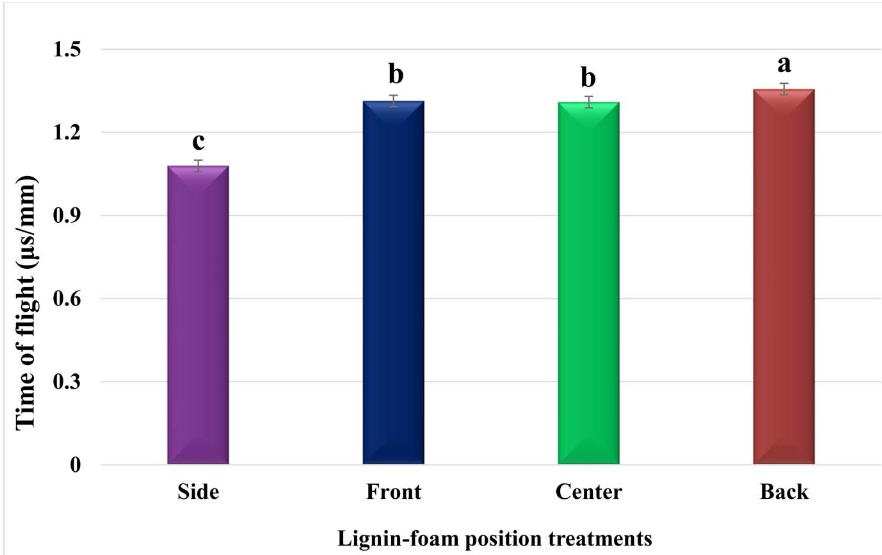

Figure 6.—Main effect of different positions on the acoustic velocity of lignin foam blocks. Treatment means within the same column within effect without common lowercase letters are significantly different. Number of replications per treatment combination is 12 or 9; $P < 0.001$; SEM = 0.0207).

Conclusions

The impact of various lignin-foam thicknesses at different block positions on the AV response were investigated. Findings revealed that there was no significant interactive effect between thickness and position but their main effects were significant. The AV response was higher for 8.89-cm (3.5-in.)-thick blocks as compared with other thicknesses. There was no significant difference in AV from blocks in the 7.87- to 8.38-cm (3.1- to 3.3-in.)-thickness range. No consistent pattern was observed for AV in the different block positions. Generally, the longitude (side) position resulted in the lowest AV, whereas the highest AV was observed in the back position. Further research is aimed at determining the extent to which these AV responses correlate with destructive test results. It is anticipated that the results of the current bench/pilot-scale study will ultimately be adaptable to commercial conditions.

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