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Flammability of Plant-Based Loose-Fill Thermal Insulation: Insights from Wheat Straw, Corn Stalk, and Water Reed

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Abstract: This study investigates the fire resistance capabilities of newly developed loose-fill thermal insulation materials crafted from annual plants such as wheat straw, corn stalk, and water reed. Three processing methodologies were employed: mechanical crushing (raw, size $\leq$ 20 mm), chemi-mechanical pulping (CMP) using 4% sodium hydroxide, and steam explosion (SE). An admixture of boric acid (8%) and tetraborate (7%) was added to all treated materials to enhance fire retardancy. The fire reaction characteristics of the insulation materials were assessed using a cone calorimeter measuring the key parameters like time to ignition, total heat release, heat release rate, and total smoke production. The findings indicate that nearly all tested insulation samples, apart from the raw and SE water reed, demonstrated fire resistance comparable to commercial cellulose insulation, surpassing the fire performance of various synthetic foams and composite materials. Furthermore, the single-flame source fire tests indicated that the developed insulation materials achieved a fire classification E, except for the SE water reed sample. Thus, the fire performance results approve the suitability of developed plant-based insulation materials for competing materials in building constructions.

Keywords: annual plants; steam explosion; chemi-mechanical pulping; lignocellulosic biomass-based thermal insulation; cone calorimetry; single-flame source test; reaction to flame source; fire resistance

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1. Introduction

In recent years, there has been an increasing interest in investigating sustainable alternatives to traditional thermal insulation materials, motivated by the objective of reducing energy use for home heating and minimizing environmental effects [1–3]. The considerable role that buildings play in global energy consumption highlights the need for innovative approaches, especially in light of the European Union's goal to achieve climate neutrality by 2050 [4]. The traditional insulation materials are sourced 90% from petrochemicals, which not only require significant energy for their production but also emit greenhouse gases throughout their lifecycle [5]. This situation emphasizes the importance of developing new bio-based thermal insulation materials.

Using lignocellulosic biomass (LCB) from annual plants for insulation materials presents a sustainable option and a means to address challenges associated with deforestation and biodiversity decline [6–10]. Commercially available LCB-based insulation

materials primarily consist of virgin or recycled wood fibers and cellulose, whose production often depends on harvesting practices that can be detrimental to ecosystems, resulting in habitat loss and increased carbon emissions [11,12]. In contrast, utilizing renewable resources such as the LCB of annual plants can promote a circular economy [13]. The agricultural industry produces several hundred million tons of LCB each year, which, if utilized effectively, has the potential to be converted into valuable thermal insulation materials [14].

An examination of the current literature on LCB-based insulation materials reveals that numerous studies have emphasized the utilization of annual plants and agricultural by-products, such as the straw of maize and miscanthus [15], rapeseed [16], hemp [2,17], wheat straw [18], bamboo [19], bagasse, banana, cocoa, coffee, cork, corn, flax, jute, kenaf, rice straw, and others [20–22]. Each of these materials offers noteworthy possibilities for renewable insulation options.

Wheat straw (*Triticum aestivum*) is a widely available and cost effective agricultural by-product that has begun to be utilized in bales, offering effective insulation properties for green building construction globally. Efforts have been made to produce insulation fiberboards from refined wheat straw pulp, which have shown performance comparable to wood-based fiberboards [23–25]. In the absence of cereal crops, corn (*Zea mays*) stalk can serve as a significant alternative, yielding approximately 0.5 kg of biomass for each dry corn grain produced, though its applications remain limited [26]. Additionally, the wetland plant reed (*Phragmites australis*) is a highly productive and affordable local LCB source, capable of producing up to 30 tons per hectare per year, and has potential uses in thermal insulation [5,27]. However, there is a notable gap in information regarding processing these raw LCB materials into fibers for thermal insulation applications.

Some options for obtaining fibrous loose-fill thermal insulation materials from LCB include processing by chemi-mechanical pulping (CMP) or steam explosion (SE). SE causes the sudden expansion of water within the plant cell walls, leading to the rupture of the lignocellulosic matrix with resulting highly curled fiber mass. The SE fiber mass contains an opened, fibrillated structure with improved fiber accessibility. The high temperature in SE causes hydrolysis of hemicelluloses, leading to their partial degradation and solubilization, which can impact fire resistance since hemicelluloses begin degrading at lower temperatures (~220 °C). The heat and pressure of SE modify lignin, leading to partial depolymerization and redistribution across the fiber surface, enhancing fiber bonding in subsequent processes. At low residence time, SE-treated materials typically exhibit a lower bulk density and increased surface area, which can improve insulation properties but may also influence flammability by affecting heat transfer characteristics. The alkaline treatment in CMP selectively removes part of the lignin and hemicelluloses, increasing fiber flexibility and reducing inter-fiber bonding strength. Combining chemical softening and mechanical refining leads to more uniform and individual fibers with a refined, homogeneous structure. Since hemicelluloses are highly flammable, their partial removal may improve fire resistance. CMP-treated fibers are more hydrophilic due to the exposure of cellulose fibrils, which can influence their behavior in insulation applications.

Materials such as wheat straw, corn stalk, and reed are naturally flammable, which highlights the need for adequate fire retardants to improve their safety when used as insulation in residential construction [1,28]. Finding a balance between the use of renewable materials and maintaining fire safety standards is a primary concern for both researchers and industry professionals [29].

Fire retardants fall into several categories: halogens, phosphates, minerals, nitrogen-based compounds, silicon-based compounds, nanometric compounds, and innovative bio-based solutions, each with unique mechanisms for flame suppression [30]. Phosphorus-

based retardants, like ammonium polyphosphate, promote char formation, creating a protective layer that slows heat and flame penetration while lowering environmental risks [31]. Mineral-based options, such as magnesium hydroxide and calcium carbonate, are non-toxic and integrate well with bio-based materials, enhancing fire resistance [32]. When exposed to high temperatures, silicate-based retardants form a glassy layer, blocking heat transfer and reducing flammability [33]. Bio-based fire retardants, sourced from biomass like lignin, tannin, and chitosan, are being explored for their eco-friendly properties. Other natural polysaccharides, like starch and alginate, are also under investigation for developing composite materials with improved fire resistance [34]. Nanotechnology is advancing the field, with nanoclays and metal nanoparticles incorporated into insulation materials to enhance thermal stability, acting as heat sinks or barriers to improve fire safety [35].

Borates, specifically borax and boric acid, have emerged as promising options among the various fire retardants. These naturally occurring compounds are effective in reducing flammability by promoting charring and forming a protective layer that shields the underlying material from flames and heat [36,37]. Their effectiveness is complemented by their low toxicity and non-hazardous nature, making them suitable for use in environments that prioritize health and safety [38]. In addition to their fire-retardant capabilities, borates offer additional benefits for LCB-based fiber insulation materials. They can enhance mold and insect resistance, which contributes to the longevity and performance of insulation products [39].

The authors of this paper have previously performed investigations into the thermal conductivity, chemical composition, structural characteristics [40], and fungal resistance [41] of the same LCB-based thermal insulation materials from annual plants—wheat straw, corn stalk, and reed, which are among the most studied species in the works of other authors [21]. Recent publications have compiled and shared earlier findings, providing a foundation for the current research examining the fire resistance of developed loose-fill thermal insulation materials. This study aims to explore the fire resistance properties of these materials when treated with fire retardant, addressing fire safety considerations for sustainable purposes.

2. Materials and Methods

2.1. Raw LCB

Locally (Latvia) sourced raw materials were utilized as LCB for heat insulation: Wheat straws (WS, *Triticum aestivum*) were collected from the Limbaži district; water reeds (WR, *Phragmites australis*) were harvested in winter from Puzes Lake in the Ventspils district; and corn stalks (CS, *Zea mays*) were obtained fresh, along with their ears/grains, from the farm "Pauri" in Blome. The raw materials were processed by chopping using a knife mill (CM4000, LAARMANN, Roermond, The Netherlands) to pass through a sieve with a Ø 20 mm. The chopped LCB materials were then prepared for subsequent processing steps. A commercial cellulose thermal insulation material produced by LTD BalticFloc in Latvia, Cesis, was used as a reference sample containing up to 14% borax and boric acid as flame retardants.

2.2. Processing of Raw LCB

2.2.1. Steam Explosion (SE) Pulping

According to earlier research [42,43], the chopped LCB was soaked in water for 24 h to achieve an 80% moisture content. The moisturized LCB was drained and processed in a custom-built SE device featuring a 0.5 L batch reactor. This treatment was conducted under controlled conditions, specifically at 230 °C, a residence time of 30 s, and a pressure

of 30 bar. The resulting wet SE pulp was collected and manually pressed using a juice-like press to extract the liquid portion.

2.2.2. Chemi-Mechanical Pulping (CMP)

The CMP of chopped and soda-treated LCB was conducted using a Regmed MD-300 single-disc refiner (Osasco, Brazil) at 1450 rpm. The soda treatment was performed by adding 8% NaOH for WR and 2% NaOH for WS and CS based on the dry weight of LCB and cooking it in water at a ratio of 1:28 for 30 min. The soda-treated LCB was drained and processed in the refiner with water at 20 °C. The duration of defibration was fixed at 10 min for all samples, maintaining a gap between the plates of 0.25 mm. The resulting fiber solution was drained through a 2 mm sieve and manually pressed in a juice-like press to extract the water.

2.2.3. Mechanical Foaming of Processed LCB

The SE and CMP materials were mechanically foamed using a custom device with two rotating cylinders (900 rpm) accomplished with stainless steel wires, as outlined in [40]. This process was repeated thrice to separate and homogenize the fiber mass, creating a fluffy texture suitable for loose-fill thermal insulation material.

2.2.4. Admixture of Fire Retardant and Fungicide

Based on the dry LCB, 7% of sodium tetraborate ($Na_2B_4O_7 \cdot 10H_2O$, CAS: 1303-96-3; Chempur, Piekary Śląskie, Poland) and 8% of boric acid (H_3BO_3, CAS: 10043-35-3; Chempur, Piekary Śląskie, Poland) were incorporated into the prepared materials (raw, SE, and CMP) as flame retardants, simultaneously protecting against fungi and insects. The chemical powders were gradually added to the LCB by mixing the components in a blowing device (Model 575, KRENDL, Delphos, OH, USA) and blown into a container. Before further testing, the prepared loose-fill insulation materials were conditioned at 20 °C and 60% relative humidity.

2.3. Determination of Flammability

2.3.1. Sample Designations and Incorporation Density

Table 1 summarizes information on LCB samples prepared for further flammability analysis. The indicated density was used in the flammability tests and is based on previously performed settlement results of developed loose-fill LCB materials. The density emphasizes the effect of SE and CMP processing, indicating the efficiency of material consumption compared to the chopping process.

Table 1. Summary of prepared LCB samples.

LCB	Treatment	Designation	Density, kg·m^{-3}
Wheat straw	Chopped	WS_raw	115
	Steam explosion	WS_SE	60
	Chemi-mechanical pulping	WS_CMP	60
Water reed	Chopped	WR_raw	145
	Steam explosion	WR_SE	85
	Chemi-mechanical pulping	WR_CMP	60
Corn stalks	Chopped	CS_raw	135
	Steam explosion	CS_SE	85
	Chemi-mechanical pulping	CS_CMP	60
Commercial cellulose insulation material		Cel_insul	60

2.3.2. Cone Calorimetry

Cone calorimeter testing was used to predict the behavior of an individual material during a fire performance. The response to a heat flux of 35 kW/m^2 for 600 s was evaluated using an FTT Dual Cone calorimeter from Fire Testing Technology Ltd. (East Grinstead, UK). The dimensions of the test LCB samples were 100 × 100 × 50 mm. Key metrics, including time to ignition (TTI, s), total heat release (THR, MJ·m^{-2}), heat release rate (HRR, kW·m^{-2}), and total smoke release (TSR, m^2·m^{-2}), were measured following the standard ISO 5660-1:2015 [44].

2.3.3. Determination of Fire Resistance Class

The single-flame source test using an ignitability apparatus was conducted by Fire Testing Technology (East Grinstead, UK), following the standard ISO 11925-2:2020 [45]. A small propane flame was directed at a 45-degree angle from the bottom upward of each sample and applied for 15 s. After removing the flame source, the sample was allowed to burn independently for 5 s. Several observations were recorded during the test. These included whether (1) the sample ignited, (2) the flame tip reached a distance of 150 mm, (3) the flame was outside the burning sample, and (4) the flaming droplets were produced. Based on the testing results and the standard requirements, a reaction to fire classification is assigned for LCB-based materials, either class E or potentially class F.

2.4. Statistical Analysis

Before statistical analysis, data normality and homogeneity of variances were assessed using Levene's test. Statistical analyses were conducted using IBM SPSS Statistics Version 20.0 (IBM Corp., Armonk, NY, USA). Mean values (MV) and standard error of the mean (SEM) were calculated from three independent measurements (n = 3). The data underwent one-way ANOVA analysis, followed by a post-hoc Tukey test. The significance level for all statistical analyses was established at $\alpha = 0.05$.

3. Results and Discussion

3.1. Visual Characterization of LCB Samples

Figure 1 illustrates the photo visualization of the developed and analyzed samples. On the left side of the image (a,d,g), chopped raw LCB samples are depicted, revealing large, irregular particles of the material. The central section (b,e,h) displays LCB samples following the SE treatment, characterized by a cotton-like appearance with visible fibrillation. This alteration in color indicates that chemical transformations and partial degradation of the samples have occurred due to the SE treatment. The samples subjected to CMP treatment are presented on the right side (c,f,i). In this case, the flakes exhibit a refined and homogeneous structure.

3.2. Fire Resistance Analysis

3.2.1. Results of Cone Calorimetry

Figures 2–6 present the results from the cone calorimetry tests conducted on the LCB samples, including fire performance metrics such as time to ignition (TTI), total heat release (THR), heat release rate (HRR), maximum average heat emission rate (MAHRE), and total smoke release (TSR). Additionally, the fire properties of commercially available cellulose insulation material have been compared.

TTI is defined as the duration between the onset of heat exposure and the moment sustained flaming occurs. The TTI of the investigated LCB samples varied from 7 to 9 s, comparable to commercial samples (9 s). It is established that the density of the sample significantly influences its fire resistance. However, as illustrated in Figure 2, there is no

statistically significant difference in TTI among the investigated samples. Their apparent density and surface texture influence the flammability of samples. Generally, materials with a higher density have a smoother surface and exhibit a longer ignition time compared to light-weight materials [46,47].

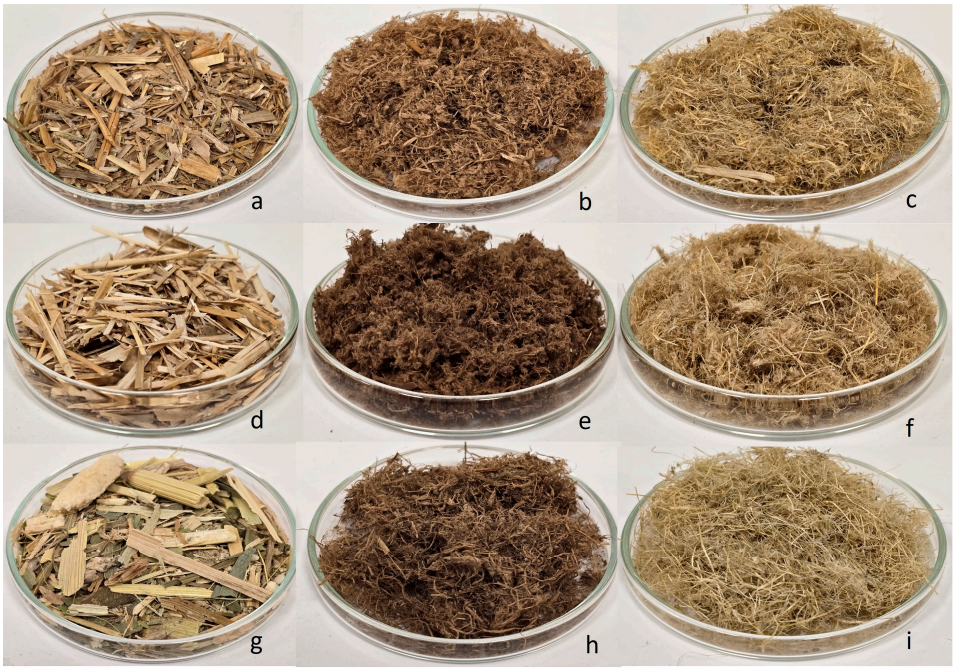


Figure 1. Visual characterization of LCB samples. (**a**–**c**)—wheat straw; (**d**–**f**)—water reed; (**g**–**i**)—corn stalk. (**a**,**d**,**g**)—raw chopped materials; (**b**,**e**,**h**)—steam explosion treatment; (**c**,**f**,**i**)—chemi-mechanical pulping.

According to other authors' studies on insulation materials from hemp, wheat straw, and wood fiber, the ignition time was 9, 12, and 11 s, respectively [48]. Additionally, mycelium bio-composite materials showed TTI values of 7 and 18 s [49,50]. These findings align closely with the results of our research. In comparison, synthetic foam like extruded polystyrene ignites in 5 s, and expanded polystyrene takes 29 s to ignite [51]. In contrast, oriented strand board and plywood, due to their higher density and composition, presented longer ignition times, occurring in 24 to 25 s [52,53].

In studies analyzing natural fiber insulation materials using cone calorimetry with a heat flux of 50 kW·m^{-2}, shorter ignition times were observed. For example, wood-fiber insulation boards with various adhesives ignited in just 1 s [31], while treated hemp fiber insulation materials ignited in 1 to 3 s [2], attributed to their low density and uneven surfaces. Other materials, such as wood fiberboard, recycled wood fiberboard, flexible wood fiber batt, and flexible hemp batt, exhibited TTI values between 2 and 5 s [54]. These studies do not mention the use of retardants, which also significantly affects the fire reaction properties. Although these results are not directly comparable, as a higher heat flux was used, it can be assumed that the results for natural fiber materials are relatively similar.

Figure 3 illustrates that the THR for LCB samples ranges from 10 to 20 MJ·m^{-2}.

The WS and CS sample groups exhibit no statistically significant difference in THR values with each other or the commercial cellulose sample (Figure 3). Conversely, the WR_SE sample demonstrates twice as high THR value as the commercial sample. It is statistically distinct from other LCB samples. The WR_raw sample also shows a significantly higher THR than for the commercial insulation. THR of the WR_CMP sample aligns closely with those of other LCB samples. These findings indicate that the LCB species and the treatment methods influence the THR. Chemical analyses of LCB samples determined and

published in our previous study revealed that WR possesses a notably higher hemicellulose content (30% compared with 24–25% to other tested LCB materials) [40], which may diminish fire resistance since hemicelluloses begin to decompose first of all components at 220 °C [55].

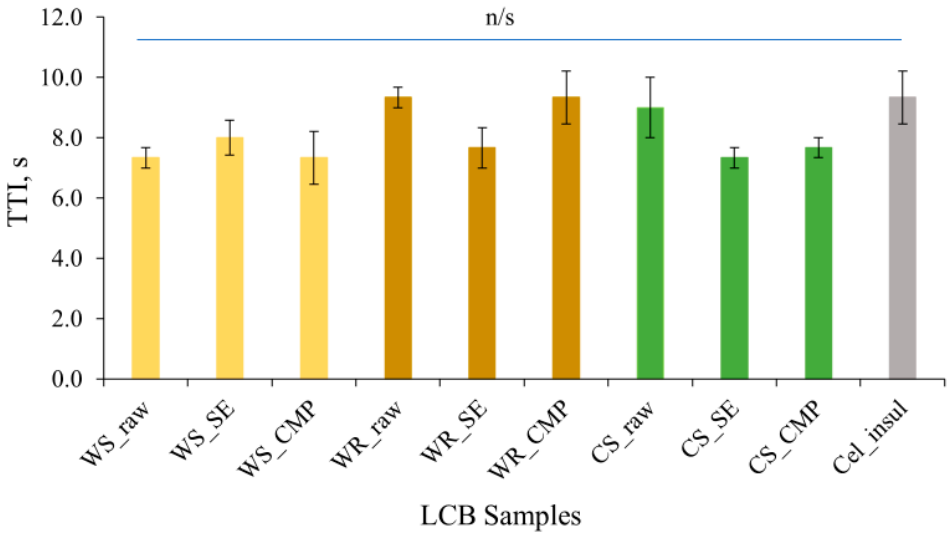


Figure 2. Time to ignition (TTI) of LCB samples compared to commercial cellulose sample. n/s—not significant, $n = 3$.

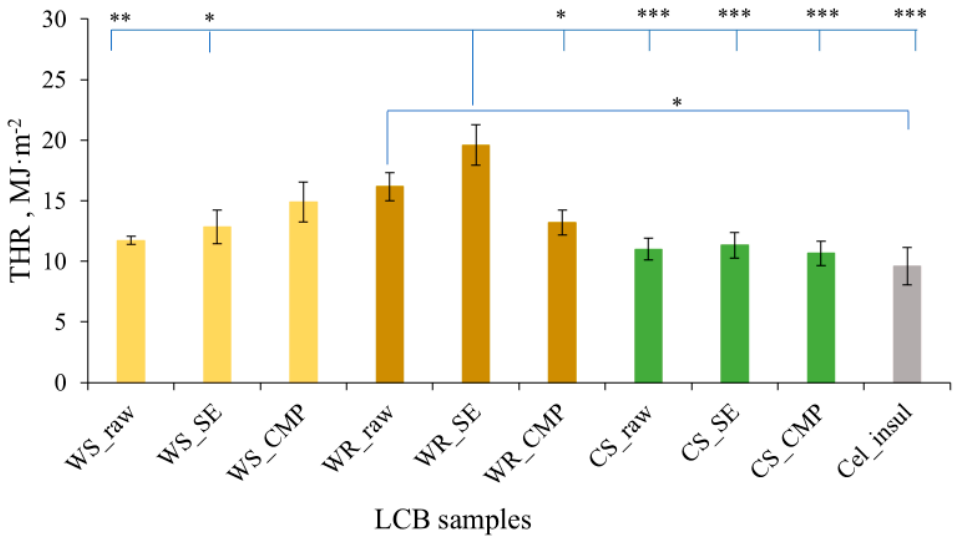


Figure 3. The total heat release (THR) of LCB samples compared to commercial cellulose sample. The significance level α * < 0.05, ** < 0.01, and *** < 0.001.

A study on thermal insulation materials made from hemp, straw, and wood fibers yielded THR results of 35, 79, and 109 MJ·m^{-2}, respectively [48]. The THR for the mycelium bio-composite demonstrated a broad range, spanning from 6 to 62 MJ·m^{-2} [50]. The THR values were found to be significantly correlated with the apparent density of the samples, indicating that a higher density corresponds to increased THR. Consequently, wood composites, including oriented strand board and pine plywood, displayed markedly elevated THR values, ranging from 98 to 127 MJ·m^{-2} [52,53]. At the same time, extruded polystyrene and polystyrene show THR values of 26 and 11 MJ·m^{-2} [51], respectively. These findings highlight the satisfactory fire performance characteristics of LCB materials in this study compared to other insulation materials, indicating a favorably developed technology and sufficient level of added fire retardants.

The assessment of fire hazards significantly depends on the HRR, with elevated HRR values enhancing the risk of flame propagation. Moreover, the peak heat release rate (pHRR) plays a crucial role in evaluating fire performance, as it directly impacts the design of fire safety measures in buildings. The shape of the HRR curve reveals the speed, intensity, and duration of a sample burning.

The HRR results of developed LCB insulations are summarized in Figure 4, showing that all the samples ignite within 7–10 s, with pHRR noted around 30 s. The pHRR values vary within 120–150 kW·m^{-2} for the WS and CS sample groups and 140–190 kW·m^{-2} for the WR group. The commercial cellulose sample exhibits a pHRR of 115 kW·m^{-2} at 30 s. While all samples display a higher pHRR than the commercial cellulose, the pHRR values of WS_CMP and CS_CMP are comparable. Figure 4a,c shows that LCB raw materials (WS_raw and CS_raw) ignite quickly, reaching peak combustion in 30 s and completely burning out in 60 s. In contrast, the sample WR_raw (Figure 4b) exhibits the highest pHRR and the most extended burn duration. This is due to WR's distinct chemical composition, determined and published in a previous study [40], featuring higher levels of hemicelluloses (30%) and lignin (24%).

The combustion of hemicelluloses contributes to a higher pHRR, while lignin combustion generates char, which slows down the burning process. Lignin degrades gradually within a temperature range of 200 to 900 °C, making it the most stable component in fiber [55]. Considering a low apparent density, all LCB samples had completely burned after 120–125 s (Figure 4). The chopped LCB samples exhibit varying pHRR values: 195 kW·m^{-2} for WR_raw (Figure 4b), attributed to its high hemicellulose content, while WS_raw reaches 150 kW·m^{-2} (Figure 4a), and CS_raw reached the lowest peak at 135 kW·m^{-2} (Figure 4c), likely due to over twice the ash or mineral content (8%) determined and published in a previous study [40], which lowers flammability. Different treatments of produced LCB materials result in varying effects on HRR and pHRR. SE and CMP treatments notably lowered pHRR for the WR group, while for the CS group, SE treatment raised pHRR. This discrepancy can be attributed to the CS_raw sample's higher ash content (8%), which was determined and published in a previous study [40] and initially resulted in a lower pHRR. Acids formed during SE autohydrolysis helped dissolve minerals, reducing the ash content (5.5%) [40] and consequently increasing pHRR (Figure 4).

Research on LCB-based thermal insulation materials conducted by other authors has claimed similar findings. For instance, materials like hemp, wheat straw, and wood fiber exhibited pHRR values of 195, 130, and 156 kW·m^{-2}, with corresponding TTP values of 27, 25, and 30 s, respectively [48]. The pHRR for the treated and raw hemp thermal insulation materials ranged from 70 to 170 kW·m^{-2}, with TTP values between 10 and 40 s [2]. For the comparison, the sheep wool exhibited four distinct peaks in the HRR graph, achieving a maximum pHRR of 200 kW·m^{-2} and a TTP value of 20 s [55]. In another investigation focusing on mycelium bio-composite materials, the pHRR ranged between 130 and 190 kW·m^{-2}, with one specific instance recorded at 240 kW·m^{-2} and TTP values ranging from 19 to 31 s [50]. A study on extruded polystyrene and polystyrene, common thermal insulation materials in construction, revealed pHRR values of 423 and 277 kW·m^{-2}, with TTP values of 33 and 32 s, respectively. Again, the mentioned examples indicate that the developed LCB materials might offer a competitive fire performance.

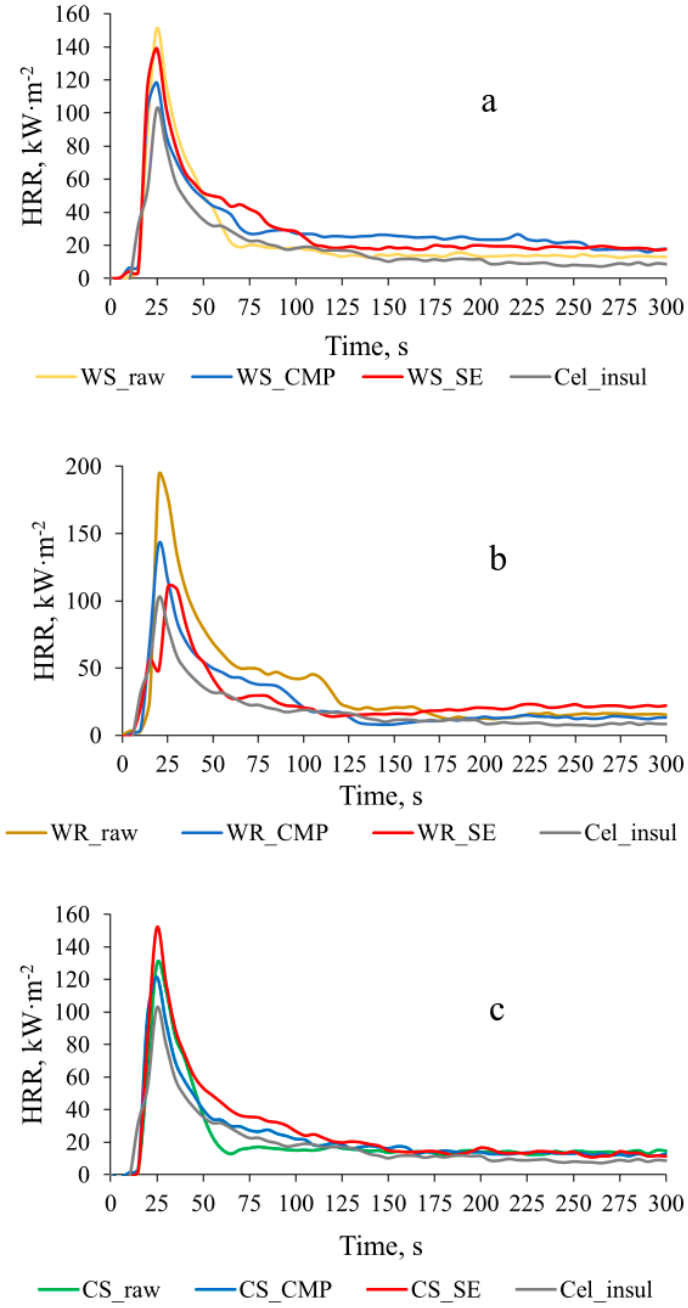


Figure 4. Heat release rate (HRR) of LCB samples composed of (**a**) wheat straw, (**b**) water reed, and (**c**) corn stalk, compared by commercial cellulose sample.

TSR is a crucial indicator for evaluating smoke hazards. It plays a key role in assessing the fire safety of building materials, given that smoke inhalation is the leading cause of fatalities in fires. TSR values of developed LCB insulations are summarized in Figure 5, revealing that CS materials have the lowest TSR (165–190 $m^2 \cdot m^{-2}$), while WR has the highest TSR (225–270 $m^2 \cdot m^{-2}$). This is attributed to the higher ash content of CS samples (8%), which reduces smoke release, while the higher hemicellulose content of WRs (30%) leads to more smoke at lower temperatures (Figure 5). Chemical analyses of LCB samples were determined and published in a previous study [40]. WR_raw and WR_SE exhibit significantly higher smoke formation than other LCB samples, while CS_raw and WS_raw show significantly lower TSR than WR_CMP. The WS and CS group samples demonstrate similar TSR results to commercial cellulose insulation. Although no significant TSR dif-

ferences were found between treatment methods, SE treatment tends to increase smoke formation due to the released sugars and decomposition products on the fiber surface [40].

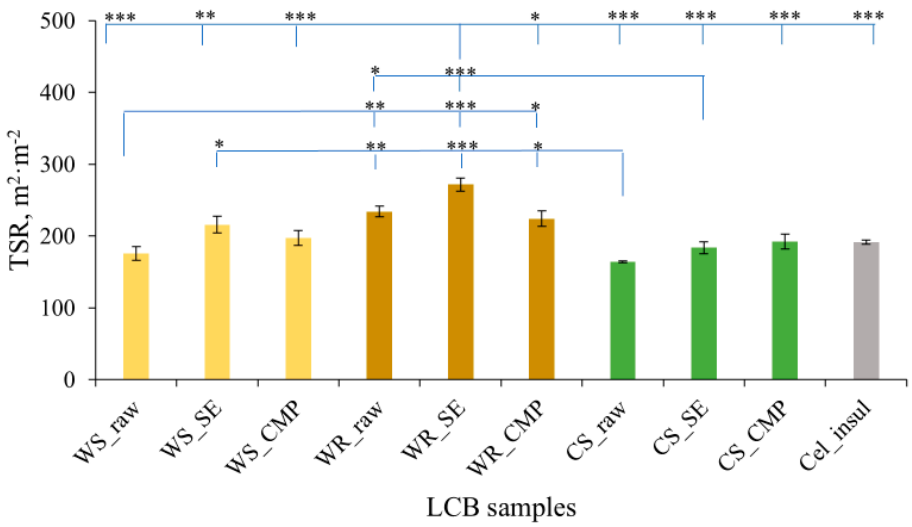


Figure 5. Total smoke release (TSR) of LCB samples compared by commercial cellulose sample. The significance level α * < 0.05, ** < 0.01, and *** < 0.001.

Various options are being considered to improve the flame retardancy of WR samples. These include increasing the duration of NaOH treatment and/or raising the concentration of NaOH in the CMP process. SE samples could also be subjected to additional rinsing with water or treated with NaOH. However, these additional steps contribute to a greater environmental impact, which is unwelcome. Alternatively, increasing the amount of flame retardants could be more effective; commercial natural fiber materials typically contain up to 20%, compared to our study's 15%. Using different flame retardants might also offer benefits; this could be a focus for future research [56].

A study of impregnated wood scab-based loose-fill insulation showed TSR values of 25–60 $m^2 \cdot m^{-2}$ [57], while a study on mycelium bio-composite reported values between 10 and 280 $m^2 \cdot m^{-2}$ [50]. In contrast, rigid polyurethane foam has TSR values from 400 to 990 $m^2 \cdot m^{-2}$ [58], and polyisocyanurate modified with rapeseed oil has TSR values from 204 to 560 $m^2 \cdot m^{-2}$ [59]. The results indicate that the developed LCB thermal insulation materials are a promising alternative to current options, offering comparable performance to natural fiber materials and significantly outperforming synthetic ones. Natural fibers mainly consist of cellulose, hemicellulose, and lignin, which contain oxygen, hydrogen, and carbon. When burned, they typically generate carbon dioxide, water vapor, and minimal soot, often forming char instead of completely volatilizing, which reduces smoke. As a key component, lignin helps control oxidation by creating insulating char and generating fewer volatiles; it produces more char than cellulose or hemicellulose [60]. In contrast, synthetic polymers containing hydrocarbons and sometimes halogens release more volatile organic compounds and incomplete combustion products, increasing smoke.

3.2.2. Reaction to Flame Source

Single-flame source tests were conducted to evaluate the performance of the developed LCB loose-fill thermal insulation materials when exposed to direct flames. Figures 6 and 7 display the results of the samples subjected to an open flame for 15 s. The results indicate whether the material meets the fire resistance class E, achieving the flame-damaged height on a sample lower than 150 mm.

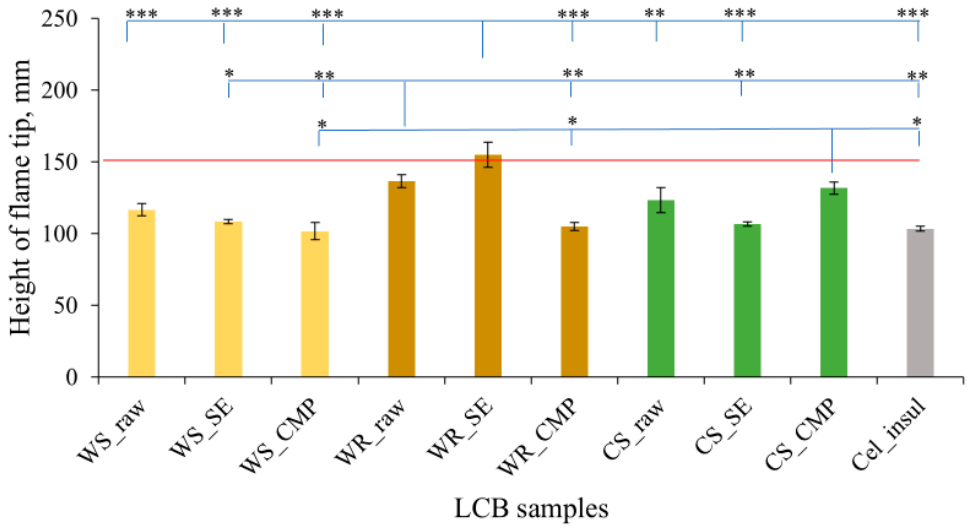


Figure 6. Flame-damaged height after 15 s during single-flame source test of LCB samples, compared by commercial cellulose. The red line indicates the requirement height of class E. The significance level α * < 0.05, ** < 0.01, and *** < 0.001.

The WR_SE sample significantly differs from other LCB samples, including commercial cellulose thermal insulation, which failed the single-flame test by overpassing the flame-damaged height of 150 mm (Figures 6 and 7). The WR_raw sample had a significantly higher flame tip (137 mm) than WS_SE, WS_CMP, WR_CMP, and CS_SE, as well as the commercial insulation. CS_CMP also had a significantly higher flame tip (132 mm) than WS_CMP, WR_CMP, and commercial cellulose. The remaining samples had flame tips ranging from 105 to 120 mm and were statistically similar to each other and the commercial cellulose insulation.

Figure 7 illustrates the post-experiment of the single-flame source test. It is also noted that no flames extended beyond the burning sample, and no flaming droplets were generated during the test.

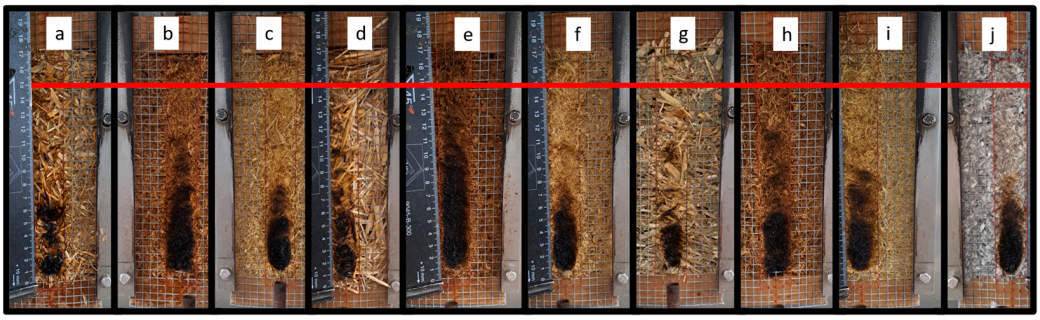


Figure 7. Images of LCB samples after 15 s of single-flame test: (**a**–**c**)—wheat straw; (**d**–**f**)—water reed; (**g**–**i**)—corn stalk; (**a**,**d**,**g**)—raw chopped samples; (**b**,**e**,**h**)—SE samples; (**c**,**f**,**i**)—CMP samples, and (**j**)—commercial cellulose insulation material. The red line indicates the required height of class E (150 mm).

Other studies on natural loose-fill insulation materials from wheat straw [61] and wood scobs [57], have yielded similar findings approving the conformity of fire resistance class E. According to the study results, all developed LCB loose-fill samples, except WR_SE, comply with fire resistance class E.

While the developed materials demonstrate commendable fire resistance, along with satisfactory thermal insulation [40] and fungal resistance [41], as corroborated by previous studies, there remains considerable scope for further enhancement of their fire resistance

properties, particularly for water-resistant species like WR. One approach could involve increasing the concentration of fire-retardant agents, aligning with industry practices. Furthermore, exploring alternative or supplemental fire retardants beyond the current options of boric acid and tetraborate warrants investigation. Phosphorus-based fire retardants, such as ammonium polyphosphate, have shown promise in promoting char formation, which can significantly improve material fire resistance [31]. Moreover, delving into innovative solutions such as bio-based fire retardants or nanotechnology-enhanced options is essential [30]. For instance, employing silica-based coatings, nano-clays, or flame retardants derived from lignin may enhance fire resistance while potentially mitigating environmental impact [62]. These approaches aim to bolster the materials' performance and contribute to sustainability, positioning them favorably within contemporary ecological frameworks. Further research in these areas could pave the way for advancements in material development addressing fire safety and environmental concerns. Previous studies on LCB-based thermal insulation from SE hemp shives and black alder have resulted the commercialized products with demonstrated favorable scalability and competitive production costs (https://www.rignocell.com/). This suggests that producing thermal insulation materials from LCB-based resources may also be scalable and commercially feasible. To effectively commercialize the developed insulation materials, further research is essential to assess their long-term stability under real application conditions. Investigating factors such as resistance to humidity, biodegradation, and the potential erosion of fire-retardant properties over time will provide critical insights for practical application.

4. Conclusions

This study on LCB thermal insulation materials derived from wheat straw, water reed, and corn stalk proves that annual plants could be a viable alternative to conventional insulation products. Both WS and CS insulation materials, whether in their raw state or treated by SE or CMP, exhibited fire reaction properties statistically comparable to those of commercially available cellulose-based insulation. In addition, WR treated with CMP demonstrated fire resistance characteristics on par with commercial cellulose insulation, whereas WR in its raw form and treated with SE showed significantly poorer performance, failing to meet established construction fire reaction standards. The developed LCB insulation materials outperformed various synthetic foams and composite materials in terms of fire resistance, exhibiting lower pHRR (120–190 $kW \cdot m^{-2}$) and TSR (165–270 $m^2 \cdot m^{-2}$), as well as lower THR (10–20 $MJ \cdot m^{-2}$) than of certain wood composites. Results from the single-flame source test approved the conformity of fire resistance class E for almost all developed LCB insulation materials except the WR_SE sample. Finally, the performed study approves the suitability of developed LCB loose-fill materials for building construction to increase its sustainability and energy efficiency.

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