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Flammability and mechanical performance of fibreboards based on wool fibres and extracellular polymeric substances recovered from wastewater sludge

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ABSTRACT

This study investigates the influence of extracellular polymeric substances (EPS), recovered from wastewater sludge, on the flame-retardant and mechanical properties of wool-based fibreboards. The thermal properties of wool, resin, and EPS were analysed using thermogravimetric analysis and differential scanning calorimetry to determine manufacturing parameters and assess their impact on the thermal decomposition of the fibreboards. A specialised fibreboard manufacturing setup, incorporating a drum mixer, tube blender, and hot press, was developed to fabricate the composite boards. Results indicate that increasing the hot-pressing time enhances both flexural and internal bond strength. The incorporation of EPS significantly improves the internal bond strength compared to fibreboards without the biopolymer. Moreover, the combined effects of wool and EPS promote effective char formation and lead to a V-0 rating, showing self-extinguishing behaviour in vertical burn tests. Cone calorimeter analysis reveals that while EPS contributes to a reduction in the heat release rate, its effect reaches a saturation point. However, the fire growth index, along with barrier and protective effect values, demonstrates that EPS effectively mitigates fire spread and propagation. These findings highlight the potential of wastewater-derived EPS as a sustainable additive for enhancing the fire resistance and mechanical integrity of wool-based fibreboards.

1. Introduction

Fibreboard production process is constantly progressing, aiming to develop environmentally friendly, sustainable, and high-performance composite boards. By incorporating eco-friendly methods and utilising natural fibres or recyclable materials, the industry can effectively address sustainability issues and improve the functionality and performance of fibreboards. Currently, most research focuses on using plant-based fibres, such as linen, flax, and wood [1]. However, the drawback of these materials is their high flammability. In contrast, animal fibres such as wool and feathers exhibit inherent flame-retardant and sound insulation properties, making them promising candidates for fibreboard applications [2]. These characteristics can markedly improve the fire safety and acoustic performance of fibreboards, which are essential in building and transportation sectors where stringent fire and noise regulations are applied. Kurien et al. [3] have studied the incorporation of animal-based fibres, specifically chicken feather fibres into polyester and phenyl-ester composites and the resulting mechanical performance of the composites. Moreover, Rajkumar et al. [4] have conducted research on composites made of polypropylene, silk and wool fibres. The findings show that the addition of wool fibre enhances the thermal characteristics of the composites.

Wool fibres, the main product of the sheep breeding industry, are composed of keratin protein. Coarse wools are considered as the low-cost materials due to their inherent properties of low crimp, high bending rigidity and high proportion of medullated fibres [5]. Consequently, medium coarse wool (diameter of 25–40 μm) is mostly used for rugs production, while highly coarse wool (diameter >40 μm) remains limited in its applications [6]. According to Haugronning et al. [7], 80 % of wool production in the EU is categorised as waste, with an estimated

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160 thousand metric tons discarded annually. Therefore, research on repurposing this fibre is necessary for addressing wool and breeding industries.

Generally, a fibreboard can be manufactured by either a wet-forming or a dry-forming technique. Due to the absence of lignin in wool, manufacturing of wool fibreboards is better suited for dry forming involving the addition of adhesive components. Wet-strength resins significantly impact the mechanical and physical characteristics of a fibreboard. Typically, wood-based fibreboard production uses formaldehyde as an additive, which induces negative environmental consequences [8]. Lubos et al. [9] have stated that developing a non-toxic adhesive system is crucial for wood-based panel production due to formaldehyde's carcinogenic impact on the environment. PAE, a formaldehyde-free resin, can strongly interact with protein through a condensation reaction and covalent bonds [10]. During heating and drying, the azetidinium groups in the resin can react with the carboxyl groups of fibres, forming co-crosslinking between the resin and fibres [11,12]. Lee et al. [13] have already demonstrated that utilisation of PAE can improve the mechanical performance of wood fibreboards. Therefore, in this context, PAE resin is suitable for both wool fibreboard and environmental protection.

However, the application of PAE in fibreboard production has been limited due to its flammable nature. Therefore, adding a flame retardant is required to reduce the flammability of composites. Extracellular polymeric substances (EPS) are an eco-friendly flame retardant from municipal wastewater treatment plant sludge. Recently, our group has demonstrated that EPS have higher thermal properties than some bio flame-retardants, e.g. lignin and phytic acid, due to their high protein content [14]. Along with protein, EPS contain a lot of functional groups, which could contribute to the interaction between PAE and wool fibre. In addition, they have shown the enhancement of self-extinguishing properties of fabrics by effective char formation [15]. Therefore, using EPS in this context is expected to improve the mechanical and flammability of wool fibreboard. Although the fibreboard study has been intensive, the wool-based and wool-EPS fibreboards have not been studied yet.

The overarching aim of the current work was to investigate effects of EPS on flammability and mechanical properties of waste wool-fibreboards. The study presents a comprehensive set of experiments, including the characterisation of the raw materials, the manufacturing process of the fibreboards, mechanical tests for flexural properties and internal bonding strength, as well as fire performance measurements using a cone calorimeter and vertical burn test. This study will greatly improve our comprehension of the viability and future advancements in fire-resistant, eco-friendly composite fibreboards using low-grade wool and wastewater-derived biopolymers for building applications.

2. Experimental details

2.1. Materials

Coarse wool fibres were sourced from Bloch & Behren Ltd. (New Zealand), and Polyamideamine-epichlorohydrin (PAE) resin was supplied from Solenis, Delaware, USA. The general information of PAE resin includes a solid concentration of 20 %, a viscosity spanning from 90 to 140 cps, a density of 1.02 g/cm^3 at 20 °C, and a pH range of 2.6–3.

Extracellular polymeric substances (EPS) were extracted from aerobic granular sludge, which was collected from a municipal wastewater treatment plant (WWTP) using Nereda® technology [15]. Briefly, 3 g of granular sludge was mixed with 100 mL of Na_2CO_3 1 %w/v (weight/-volume ratio), and the mixture was heated to 80 °C under a stirring rate of 6000 rpm and held for 30 min. Afterwards, the mixture was centrifuged for 20 min at 6000 rpm to remove the sludge pellet. The collected supernatant was acidified to a pH value of 2.2 by HCl 1 M and continuously centrifuged at 7000 rpm for 30 min to obtain acidic EPS. Then, the acidic EPS was dissolved in NaOH 1 M at pH = 8.5 to convert it into sodium form. The sodium EPS were put in a dialysis bag of 3.5 kDa for 24 h to remove the impurities and dried at 45 °C to obtain EPS in powder form. The extraction yield of EPS from aerobic granular sludge is 325 mg/g volatile solid content of sludge.

2.2. Manufacturing process

The manufacture of the wool-based fibreboards in this study involves several key steps: drying, mixing of wool fibres and EPS, curing with PAE resin and compression moulding, as shown in Fig. 1. First, wool fibres were dried in an oven at 80 °C for 24 h and then mixed with EPS powder in different ratios (0, 15, 30 wt%) using a blender (Petterson Kelly, USA) for 10 min. Subsequently, the wool/EPS mixture was coated with PAE resin using the tube blender for 1–2 min. The quantity of PAE incorporated into the fibre was determined by the solid content of the resin (16 wt%). The wool/EPS with the resin was dried in an oven at 80 °C for 7 min, during which the moisture content was monitored using a moisture analyser MA35 (Sartorius, Germany), reaching approximately 18 %. Afterwards, the dried materials with a final weight of 90 ± 5 g were put into a 130 x 130 × 5.9 mm mould for the compression moulding process. The pressing temperature and pressure were 180 °C and 2.61 ± 0.29 MPa, respectively. Three different types of samples were produced using the chosen pressing time (WP/0EPS representing a fibreboard without any EPS added, WP/15EPS with the addition of 15 wt% EPS and WP/30EPS with the addition of 30 wt% EPS).

2.3. Characterisations

2.3.1. Thermal properties

Thermal characterisation of the raw materials was conducted using thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). TGA tests were carried out via the TA Instruments Q500 model to understand the thermal decomposition of each material. Each sample was gradually heated from 30 °C to 850 °C at a rate of 10 °C/min under nitrogen with a flow rate of 25 mL/min. DSC tests were conducted to examine the thermal properties, such as glass transition temperature (Tg) of materials, using DSC 3500 Sirius (NETZSCH, Australia). The test condition was at a heat rate of 10 °C/min within the temperature range of room temperature and 270 °C. The gas employed was nitrogen, with a flow rate of 50 mL/min.

2.3.2. Morphology and chemical composition

A Leica Stereo Microscope Model IC90 E was used to examine the fibre diameters and lengths. The microscope-generated images were subjected to image analysis using ImageJ software. Furthermore, the functional groups of fibres, fibreboard, and EPS were determined by Fourier-transform infrared (FT-IR) spectrometer (Nicolet iS50) with wavenumber from 400 to 4000 cm^{-1}.

2.3.3. Mechanical properties

A universal testing machine (Instron 5567), equipped with a load cell with a maximum capacity of 30 kN, was used to conduct mechanical testing. Flexural properties were determined using ASTM D790. The sample size for flexural testing was contingent upon the thickness of the sample. The aspect ratio of the support span to thickness was 16:1, and the sample width did not exceed one-fourth of the support span [16]. Procedure A was employed, utilising a strain rate of 0.01 mm/mm/min. Internal bond strength of the fibreboards was obtained using ASTM D1037. Aluminium alloy blocks were attached to the top and bottom surfaces of the samples for pulling apart under a tensile load. Sample dimensions were 50 x 50 × 5.1 mm, and the crosshead speed was 0.8 mm/min [17].

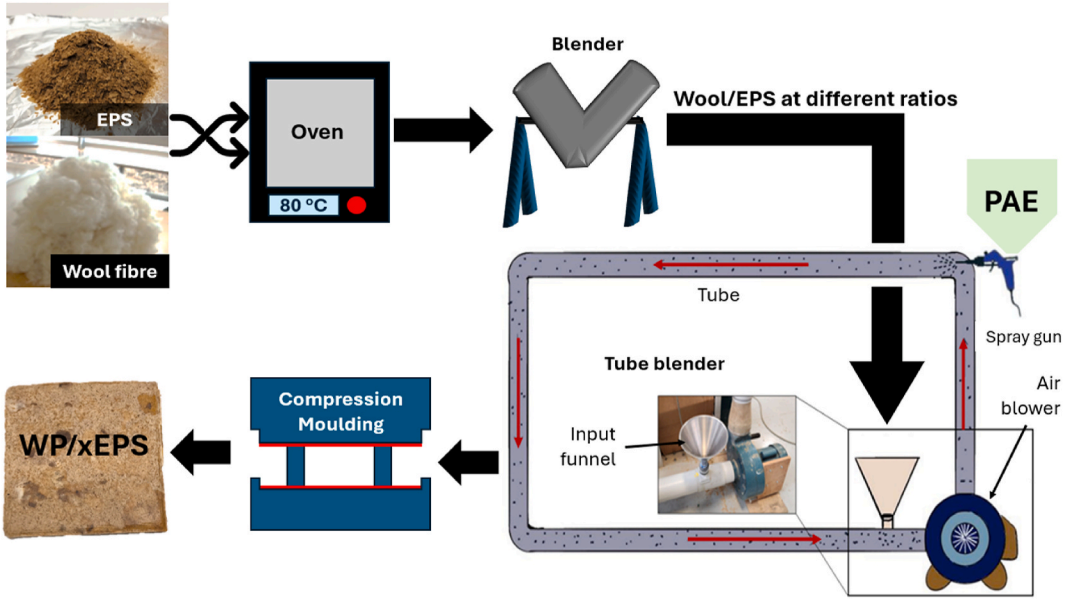

Fig. 1. Wool/EPS fibreboard manufacturing process.

2.3.4. Fire properties

2.3.4.1. Vertical burn test. The vertical burn test was conducted according to ASTM D3801. The dimensions of the specimens were: a length of 125 ± 5 mm, a width of 13 ± 0.5 mm, and a maximum thickness of 13 mm. Each sample required five tests. The after-flame time (t_1), which refers to the length of burning, was recorded following the initial 10-s application of flame. The durations of after-flame (t_2) and afterglow (t_3) were measured after the second 10-s exposure to the flame, where afterglow refers to the residual glow of the burned region after the flame has stopped. The test results were classified into different categories, specifically V-0, V-1, V-2, or no rating (NR), according to the criteria specified in the standard. Materials classed as V-0 exhibit superior fire performance with self-extinguishment and more details for other grades can be found in Ref. [18].

2.3.4.2. Cone calorimeter test. Cone calorimeter testing was carried out according to ASTM E1354 guidelines. An essential characteristic to determine from this test was the heat release rate. In addition, the cone calorimeter test allowed for the measurement of time of peak heat release rate (TPHRR), time to ignition (TTI), effective heat of combustion (EHC), total heat release (THR), total mass loss (TML) and smoke and CO production. During the test, the specimen was horizontally positioned above the load cell to determine its mass. The dimensions of the test specimen, as specified by the standard, were 100 × 100 mm with a maximum thickness of 50 mm. Before testing, the specimen underwent conditioning at temperature of 23 °C and a humidity of 50 % overnight. The heat flux imposed externally during testing was 50 kW/m^2 [19]. In order to further assess the fire resistance of materials, additional indicator values, such as the fire performance index (FPI), fire growth index (FGI), flame inhibition, charring effect value, and barrier and protective effect value, were calculated using the cone calorimeter parameters. Equations (1)–(5) were used to obtain the indicator values [20]:

$$\text{FPI} = \frac{\text{TTI (s)}}{\text{PHRR (kW/m}^2\text{)}} \tag{1}$$

$$\text{FGI} = \frac{\text{PHRR (kW/m}^2\text{)}}{\text{TPHRR (s)}} \tag{2}$$

$$\text{Flame inhibition} = 1 - \frac{\text{EHC}_{\text{FR-comp}}}{\text{EHC}_{\text{neat}}} \tag{3}$$

$$\text{Charring effect} = 1 - \frac{\text{TML}_{\text{FR-comp}}}{\text{TML}_{\text{neat}}} \tag{4}$$

$$\text{Barrier and protective effect} = 1 - \frac{\text{PHRR}_{\text{FR-comp}}/\text{PHRR}_{\text{neat}}}{\text{THR}_{\text{FR-comp}}/\text{THR}_{\text{neat}}} \tag{5}$$

3. Results and discussion

3.1. Raw materials characterisation

In this study, the average diameter and length of fibres were measured using a microscope. Fig. 2a shows microscopic images of short fibres and Fig. 2b shows the distribution of their lengths. The average diameter and length of wool fibres used in this research are 36.9 ± 4.2 μm and 1.8 ± 0.7 mm, respectively. Based on this measurement, the fibre can be classified as medium-coarse wool according to Babu et al. [21], who defined a range of 25–70 μm, and also meets the criteria for coarse wool as described by Allafi et al. [22], where fibres exceed 32.5 μm. EPS is a bright brown powder with different particle sizes (Fig. 1).

The DSC thermogram of wool, as shown in Fig. 2c, reveals that the initial endothermic peak occurs at 116.1 °C because of the release of absorbed moisture. The second endothermic peak at 229.1 °C can be associated with alpha-keratin crystallisation [23]. On the other hand, the PAE resin exhibits a distinct endothermic peak, indicating the resin's glass transition temperature (T_g) of around 78 °C. This temperature signifies the shift from a glass-like to a rubber-like state of the resin. EPS powder shows the endothermic peak at 130.25 °C due to water evaporation, but there are no other peaks, indicating no thermal degradation within this temperature range. The DSC data for EPS powder demonstrates that the initial thermal event occurs at a higher temperature than those of wool fibre and PAE resin.

3.2. Effects of EPS on the functional groups of wool fireboards

The physical appearance of Wool/EPS fibreboard is depicted in Fig. 3a and b. Mixing wool and PAE creates a yellow fibreboard due to the colour of PAE, while the presence of EPS can form some dark spots in the structure of fibreboards. When increasing the magnification, the

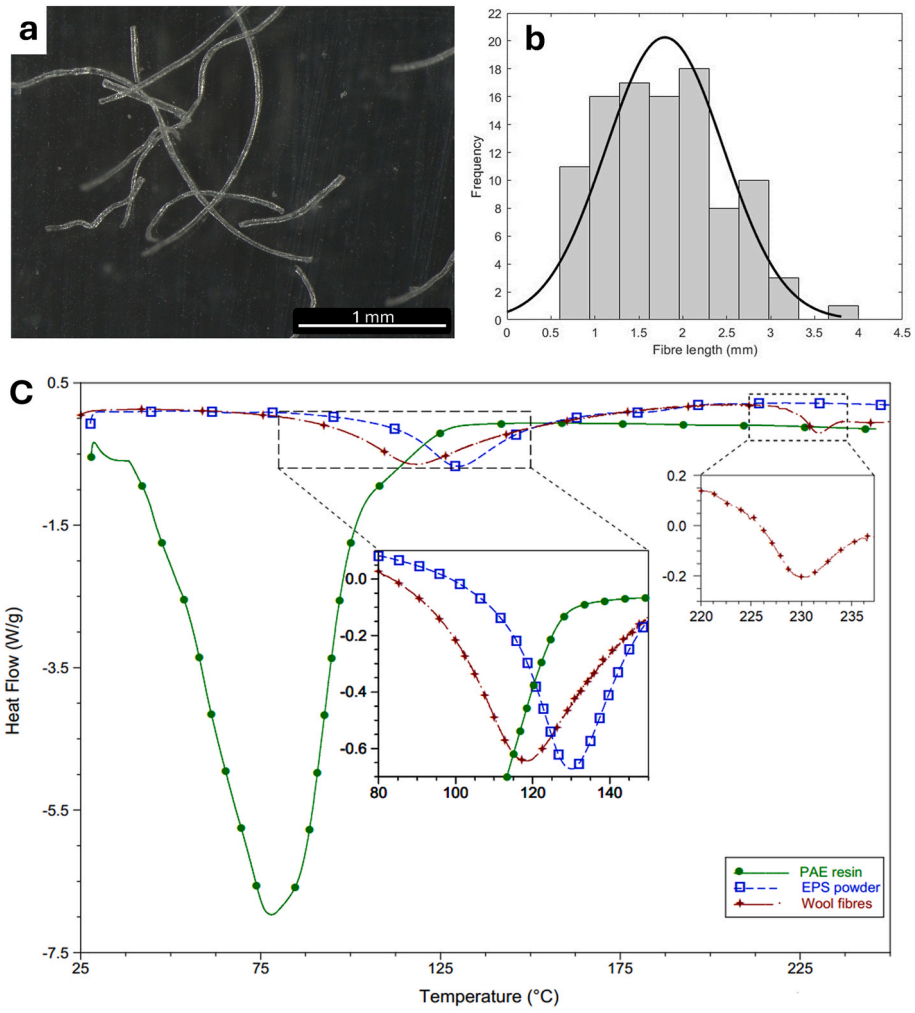

Fig. 2. The micrograph of short wool fibres (a) and length distribution of wool fibres (b); DSC curves for biopolymer (EPS) powder, PAE resin, and wool fibre.

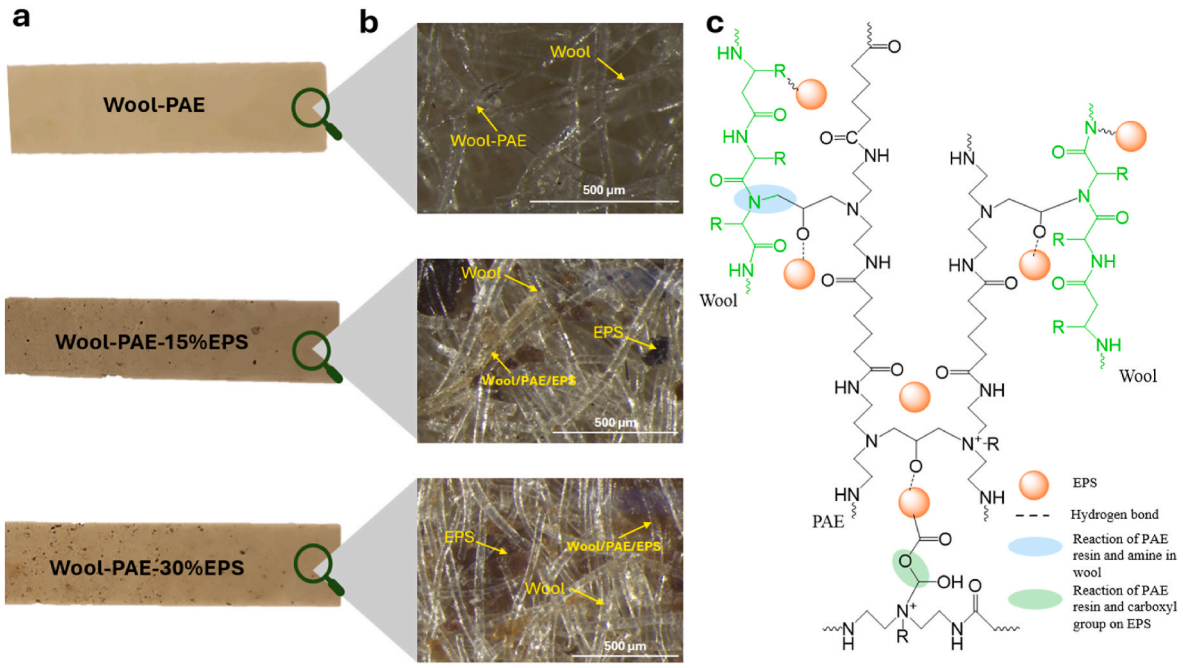

Fig. 3. The physical appearance and microscope images of Wool-EPS fibreboard (a, b) and proposed interaction between wool, PAE, and EPS.

wool fibre in WP/0EPS is quite smooth and has many pores and knots. The cross-linked between wool fibres (knots) indicates the effects of PAE as a binder. On the other hand, the density of wool fibres in WP/15EPS and WP/30EPS samples is denser than WP/0EPS. Both samples contain many EPS particles stuck on the surface of wool fibre due to EPS/wool and/or EPS/PAE/wool interactions. Surprisingly, the linkages of wool/PAE/wool in these samples are yellow compared to the white ones of WP/0EPS. This suggests that there are some reactions between PAE and EPS that could lead to changes in these linkages. In fact, EPS are a complex biopolymer containing various components, such as proteins, polysaccharides, and humic-like substances [14]. Therefore, EPS have amphiphilic properties due to their complicated structure [24], resulting in a part of EPS's functional groups dissolving in PAE liquids during spraying treatment. As a result, some functional groups of EPS, e.g. carboxyl groups, can react with azetidium groups of PAE at high temperatures to form a stronger interaction [10].

To confirm the interaction between wool, EPS, and PAE, the FTIR spectrum of all materials is measured and illustrated in Fig. 4. The absorption peak at 1635 cm^{-1} can be found in all spectra due to the presence of the N-H group in the amide I [25]. The combination of wool and PAE (in the WP/0EPS fibreboard sample) results in a reduced intensity of the peak at 1541 cm^{-1} (amide II), compared to the spectrum of pure wool fibres [6]. The spectra of all wool-EPS fibreboard samples contain most of the functional groups of PAE, wool, and EPS, indicating the well-blending of these raw materials. According to Zhang et al. [26], nitrogen atoms in the azetidinium groups of PAE can react with carboxyl or primary amine groups in wool/EPS in wool fibres, EPS, and PAE itself, resulting in the complicated matrix of fibreboard. Notably, the EPS spectrum has a significant peak at around 1050 cm^{-1}, which is attributed to the C-O-C stretching of ether linkages present in their structures [27]. However, the peak at 1050 cm^{-1} is significantly reduced in the WP/15EPS and WP/30EPS spectra (Fig. 4b), suggesting a reaction between EPS and PAE. Based on these findings, it can be concluded that the linkages in these wool-EPS fibreboards are formed by wool/EPS, EPS/PAE, EPS/PAE/wool, wool/PAE interactions and the reaction between EPS and PAE, as shown in Fig. 3b. Therefore, it can be expected that the addition of EPS can improve the mechanical properties of wool fibreboard.

3.3. Thermal properties of wool fireboards

The effects of EPS on the thermal properties of wool fibreboard are evaluated using TGA, as shown in Fig. 5. The initial weight reduction is observed in the raw materials at approximately 100 °C due to the evaporation of moisture content. Notably, the PAE resin demonstrates a substantial weight decline, reaching 80 % of its original mass because of its water-based nature and a solid content of only 20 %. The main decomposition for PAE resin starts at around 250 °C with the maximum decomposition rate at about 370 °C. This suggests the lower thermal stability of PAE than those of EPS, and wool fibre. In the case of wool fibre, the major phase of thermal decomposition occurs within a temperature range between 230 and 400 °C. This phase encompasses the decomposition of the primary organic constituents within the wool fibre, specifically its protein form known as keratin. The initial decomposition that occurs within this phase range arises from the disruption of the matrix microfibril structure and the disulfide bonds present in wool [28].

EPS displays multiple endothermic phases. The secondary degradation takes place approximately within the temperature range of 190–420 °C. This subsequent degradation of EPS involves structural breakdown and the pyrolysis of polysaccharides or other organic elements. While wool fibre and PAE resin are completely decomposed, the char residues of EPS are 27.18 wt% at 800 °C. Adding EPS into the fibreboard significantly increases char residues compared to WP/0EPS. This suggests that the wool-EPS fibreboard can create a char layer against the fire in the flame-retardant application.

3.4. Mechanical performance of wool fibreboards

The addition of EPS to the fibreboard results in the enhancement of its flexural strength, as seen in Fig. 6. By including 15 wt% EPS, the flexural strength value can be increased by around 7 %. Furthermore, the incorporation of 30 wt% EPS to fibreboard leads to a substantial increase in flexural strength by more than 39 %. This enhancement signifies that EPS can improve the resistance of fibreboards to bending and fracture when subjected to external forces. Furthermore, the fibreboards containing 30 wt% EPS demonstrate an increase in flexural modulus by 24.6 % compared to those without EPS. It can be highlighted that the presence of EPS in the wool fibreboard enhanced the capacity of the composite to withstand deformation and retain its shape when

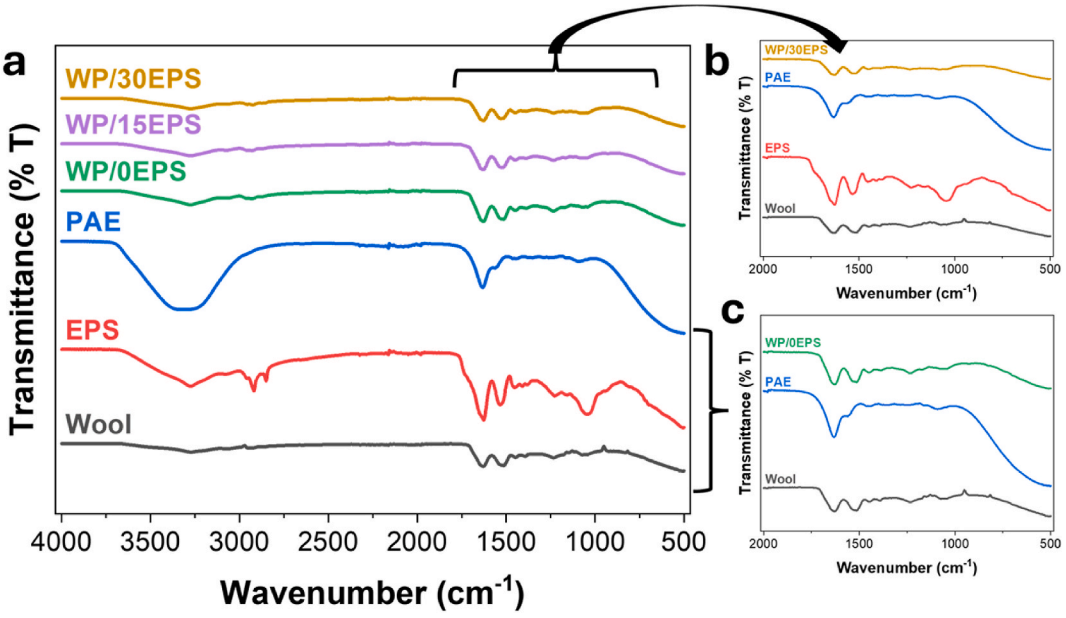

Fig. 4. The FTIR spectrum of wool, PAE, EPS, and fibreboards.

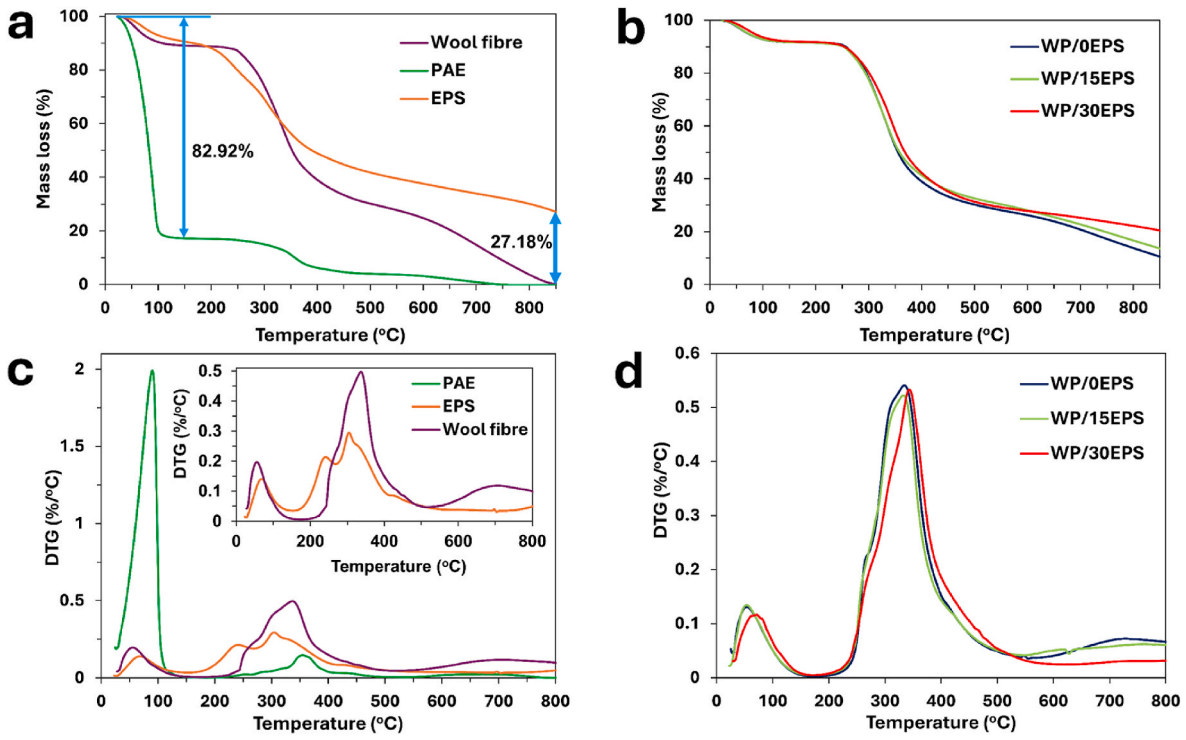

Fig. 5. Thermogravimetric curves (a, b) and derivative thermogram (c, d) of wool fibre, PAE, EPS, wool fibreboard, and wool-EPS fibreboard.

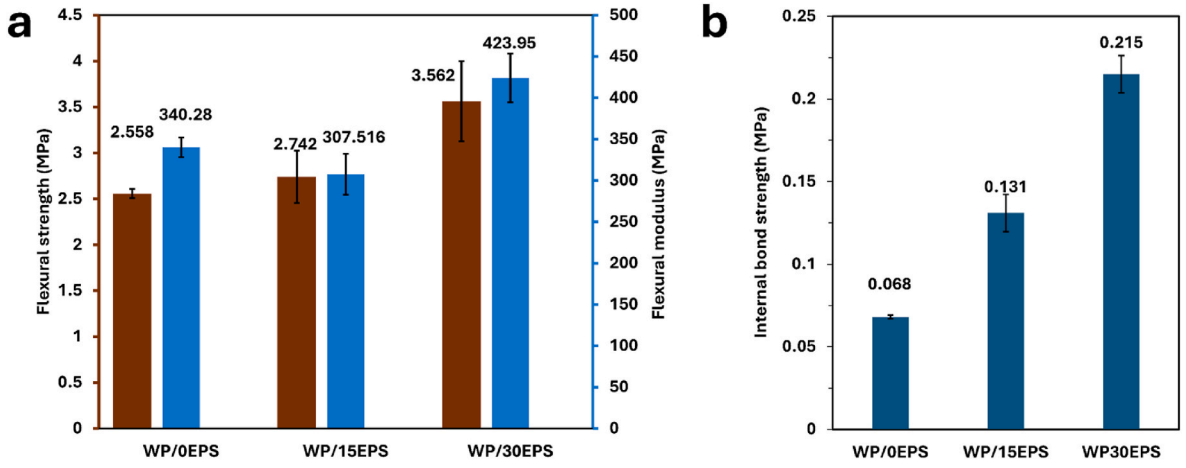

Fig. 6. Flexural strength and Flexural Modulus (a), and internal bond strength (b) of fibreboards.

subjected to stress.

The internal bond strength test is also essential since it is crucial for assessing the capacity of fibreboards to withstand delamination, splitting, or separating its layers when subjected to tension. As seen in Fig. 6, the addition of 15 wt% EPS results in a 92.65 % rise, whereas the incorporation of 30 wt% EPS achieves around 216 % increase in the internal bond strength of wool fibreboard compared to that of fibreboard without EPS. The improvement suggests that the application of EPS can facilitate the adhesion between wool fibres and PAE resin.

The improvement in both flexural strength and internal bond strength can be attributed to the role of EPS powder in promoting the dispersion of wool fibres. This process disrupts fibre agglomerates, leading to more homogeneous separation of fibres, which in turn enables a uniform distribution of the PAE resin during the spray application within the blender tube. The difference in fibre visualisation following the application of PAE before and after the addition of EPS can be observed in Fig. S1. In this case, the homogeneous distribution of fibres and binding agents can result in a more balanced and resilient structure, strengthening the internal bond strength by promoting better interlocking. Furthermore, EPS with a high concentration of functional groups can interact with wool and PAE through different types of bonding, including hydrogen bonding and covalent bonding, as shown in Fig. 3c.

3.5. *Fire performance of wool fibreboards*

3.5.1. *Cone calorimeter*

The effects of EPS on the flammability of fibreboards have been evaluated using a cone calorimeter. Table 1 demonstrates that addition of 30 % EPS into the fibreboard prolongs ignition time, reduces PHRR values and increases TPHRR values. The prolonged ignition time suggests the material necessitates higher temperatures before ignition

Table 1
Cone calorimeter results of wool and woo-EPS fibreboards.

Samples	TTI (s)	PHRR (kW/m^2)	TPHRR (s)	THR (MJ/m^2)	CO (kg/kg)	CO_2 (kg/kg)
Wool	4.5 ± 0.7	412.9 ± 51.2	25	19.6 ± 1.9	0.011 ± 0.001	1.21 ± 0.04
WP/0EPS	18.67 ± 3.79	250.04 ± 6.31	28.33 ± 5.77	69.43 ± 5.54	0.02 ± 0.001	1.16 ± 0.06
WP/15EPS	18.67 ± 1.53	267.99 ± 7.69	33.33 ± 2.89	71.30 ± 3.82	0.03 ± 0.005	1.19 ± 0.03
WP/30EPS	24.5 ± 0.71	260.85 ± 5.88	35 ± 0.00	68.3 ± 1.98	0.024 ± 0.001	1.19 ± 0.01

occurs. Such indications imply that the fibreboard integrated with EPS exhibits superior fire resistance at the outset of combustion compared to wool fibres.

The PHRR value of 260.9 kW/m^2 for WP/30EPS is smaller than that of wool fibre, indicating that the fibreboard consumes less oxygen and emits less energy curing combustion. This trend, along with the higher TPHRR value, suggests that the addition of EPS can effectively reduce fire growth. However, there is a threshold beyond which increasing the flame retardant (FR) content no longer improves fire reaction properties. At this saturation point, the fire reaction characteristics reach a stable state, indicating that the additional FR does not further enhance flame resistance. Research by Levchik et al. [29] on PP and Nylon (6 and 66) blends demonstrates that the trend in flame-retardant properties with increasing FR concentration does not follow a linear pattern but instead exhibit an 'S'-shaped curve. Moreover, Fig. 7a displays a noticeable secondary peak, which is especially prominent in the fibreboard specimens combined with EPS. According to Gan et al. [30] this distinct peak signifies the emergence of fissures in the charcoal layer, facilitating the passage of flammable gas within the sample.

A notable increase in the THR WP/0EPS value was recorded at 69.43 MJ/m^2, much exceeding that of pure wool at 19.6 MJ/m^2. This increase is attributed to the incorporation of PAE resin, which enhances fuel supply to the system according to its organic characteristics. PAE comprises functional groups, including amine and epichlorohydrin, that undergo exothermic decomposition and generate increased volatiles during combustion, leading to a greater overall heat release compared to wool, which often forms a protective char layer. These findings align with the TGA results, indicating that PAE exhibits lower thermal stability compared to wool fibre.

Table 2 demonstrates that there are no substantial variations in the values of the EHC, fire performance index, and flame inhibition. The relationship between EPS concentration and fire growth index is inverse, meaning that as the EPS concentration increases, the fire growth index value decreases. This suggests that the inclusion of EPS aids in mitigating fire risk by reducing both the likelihood of ignition and the potential spread of fire. The incorporating EPS also impacts the barrier and protective effects. The value of the WP/30 EPS sample is higher than that of the WP/15 EPS sample. Greater values for barrier and protective effect suggest that the sample is more efficient in inhibiting fire propagation. This is further confirmed through visual observation in Fig. 8.

3.5.2. *Vertical burn test*

The vertical burn test results indicate favourable outcomes, warranting V-0 qualifications according to the UL-94 standard. In all three types of samples, the time taken for the flame to extinguish after the removal of the fire source has an average of approximately 1–2 s. There are no instances of burning drips or material holes detected during the combustion process, as depicted in Fig. 8a. Optical zoom results on samples that have been burned, Fig. 8b, show that the samples without EPS have a darker colour, and get lighter with the increasing percentage of EPS added. Furthermore, Fig. 8c demonstrates a direct correlation between the quantity of EPS added and the thickness of the char formation from the cone calorimeter tests. This suggests that the inclusion of EPS facilitates the creation of a char layer, which serves as a protective barrier that can shield the underlying layer from heat and flame exposure. The increase in char yield with EPS addition is noteworthy; however, it is essential to also consider the contribution of PAE in enhancing char formation. PAE, being a nitrogen-containing resin, has the ability to catalyse charring reactions and improve thermal residue. Nonetheless, because of its organic nature, PAE also leads to a greater flammable content, which in turn results in a higher THR when contrasted with pure wool.

Upon visual examination, it is evident that the extent of char coverage varies among the three samples after the vertical burn test as seen on Fig. S2. Fibreboard without EPS produces char that nearly covers the entire surface area as seen on Fig. S2a, while the fibreboard integrated with EPS displays that char only formed edges of the sample as seen on Fig. S2b–c. This observation indicates an enhancement in the fire performance of the fibreboard due to the inclusion of EPS which plays an active role as a localising barrier, thus preventing fire from spreading easily. Combustion on fibreboard containing EPS is less prone to propagation, making it more challenging for the fire to advance than fibreboard without EPS. Consequently, the top sections of the vertically mounted samples do not exhibit signs of burning. Furthermore, an increase in the EPS content correlates with a decrease in the reach of the flame towards the top of the sample, signifying that the material comprising EPS is less susceptible to the spread of fire.

The findings from the vertical burn test align with the cone calorimeter results, demonstrating that the fibreboard, when combined with EPS, demonstrates more excellent resistance to initial sparks during combustion, denoted by an elevated TTI value. Fibreboard without EPS displays commendable char coverage, implying its capability to

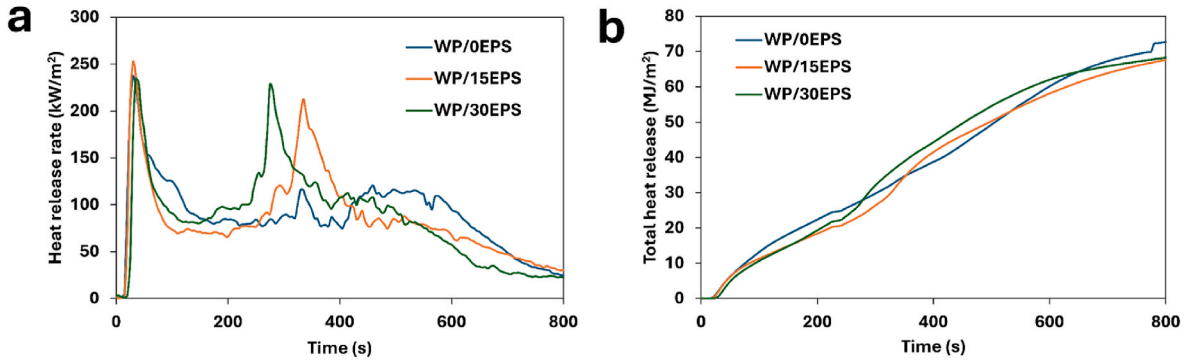

Fig. 7. Heat release rates (a) and total heat release rates (b) of wool and wool-EPS fibreboards.

Table 2
Fire index parameters calculated from the cone calorimeter results of wool and wool-EPS fibreboards.

Samples	EHC (MJ/kg)	Fire performance index	Fire growth index	Flame inhibition	Charring effect	Barrier and protective effect
WP/0EPS	18.23 ± 0.81	0.08 ± 0.02	9.08 ± 1.85	–	–	–
WP/15 EPS	17.67 ± 0.28	0.07 ± 0.01	7.68 ± 1.25	0.031	−0.062	0.012
WP/30 EPS	17.76 ± 0.10	0.11 ± 0.02	6.68 ± 0.93	0.026	0.029	0.05

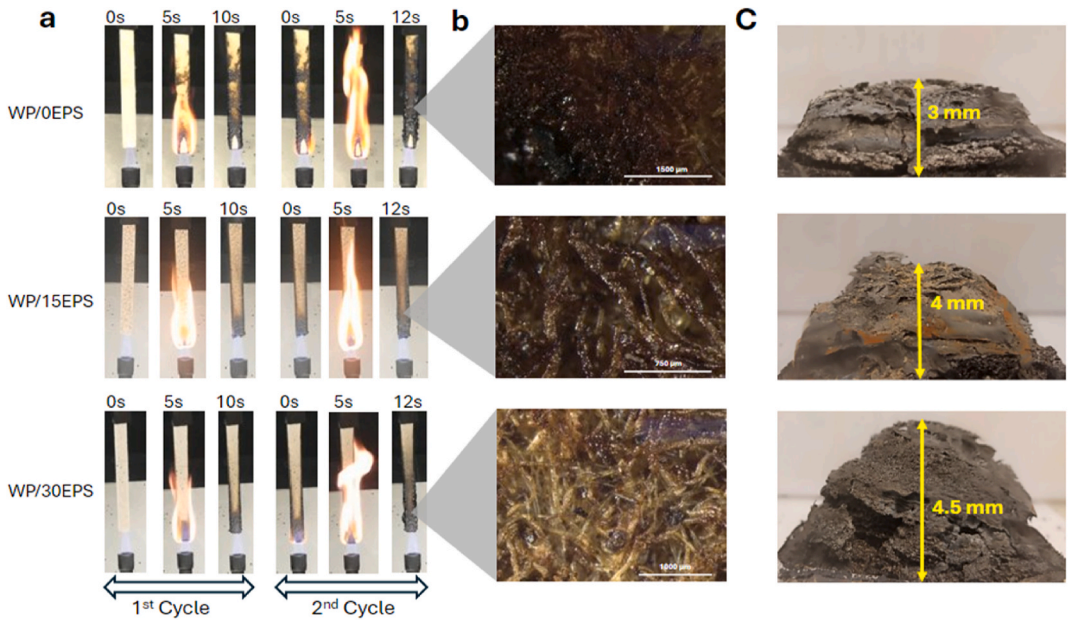

Fig. 8. Vertical burn test processes on wool fibreboards (a), optical images of the burned samples (b) and char formation from cone calorimeter tests (c).

establish a carbonaceous layer, acting as a barrier or safeguard against the material's flammability when ignited. Conversely, the fibreboard with incorporated EPS exhibits improved fire resistance at the initial ignition phase, indicating that the EPS addition enhances the board's resilience to combustion when exposed to elevated temperatures, effectively reducing the likelihood of ignition or the formation of an initial spark.

3.6. Comparison of wool-EPS fibreboards with medium-density wood fibreboards

The self-extinguishing properties and fire reaction characteristics of wool-EPS fibreboards highlight their potential as interior building panels, particularly in applications where flame-retardant performance is critical. In the medium-density fibreboard (MDF) industry, wood fibres have predominantly been utilised, and significant research has focused on enhancing the fire-resistant properties of fibreboards. Conventional MDF panels composed of wood fibres and melamine-formaldehyde resin typically exhibit a heat release rate (HRR) of approximately 400 kW/m^2 [31]. Recent studies by Lee et al. [32] have investigated the influence of protein-based flame retardants (FR) on the HRR of medium-density wood fibreboards, revealing a 25.2 % reduction in HRR (321.9 kW/m^2) when compared to untreated panels (430.3 kW/m^2). A comparison of these findings with the HRR of wool-EPS fibreboards demonstrates that the incorporation of low-grade wool and EPS achieves superior flame-retardant performance compared to conventional fibreboards. Regarding mechanical properties, while an increase in FR content improved bond strength, further optimisation is necessary to achieve mechanical properties comparable to those of wood fibreboards.

4. Conclusions

Thermogravimetric analysis (TGA) revealed that wool fibres, EPS powder, and fibreboards produce a residue at 900 °C, indicating their effectiveness in char formation. Incorporating 15 wt% or 30 wt% EPS into a fibreboard significantly improve mechanical properties, particularly flexural and internal bond strength. The vertical burn test indicated that all samples (WP/0EPS, WP/15EPS, and WP/30EPS) achieve V-0 quality for non-flammability. Visual observation showed that EPS effectively reduce the fire spread. Cone calorimeter data demonstrated that samples containing 30 wt% EPS have higher time-to-ignite values, demonstrating enhanced thermal endurance. The fire growth index value and the barrier and protective effect values further confirmed that EPS incorporation reduces fire growth and improves fire resistance. Overall, this study highlights the potential of wool and EPS to enhance both fire retardancy and mechanical strength, making these of fibreboards suitable for building applications.

CRediT authorship contribution statement

Nur Mufidatul Ula: Writing – original draft, Visualization, Methodology, Investigation, Formal analysis, Conceptualization. **Tan Minh Le:** Writing – review & editing, Writing – original draft, Visualization, Investigation, Formal analysis. **Yuemei Lin:** Writing – review & editing, Supervision, Methodology, Investigation, Funding acquisition, Conceptualization. **Mark C.M. van Loosdrecht:** Writing – review & editing, Methodology, Investigation, Funding acquisition. **Debes Bhattacharyya:** Writing – review & editing, Methodology, Investigation, Conceptualization. **Krishnan Jayaraman:** Writing – review & editing, Supervision, Methodology, Investigation, Conceptualization. **Nam Kyeun Kim:** Writing – review & editing, Supervision, Project administration, Methodology, Investigation, Funding acquisition,

Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.mtsust.2025.101210.

Data availability

Data will be made available on request.

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