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RESEARCH ARTICLE

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Enhancing fire safety and thermal performance: Wood composites with bio-based phase change materials and fire retardants for building applications

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Abstract

This work explores the elaboration of wood particle-based composites incorporating bio-based phase change materials, with epoxidized linseed oil or clay as a binder. Fire performance evaluation of the novel composites includes an assessment to determine the impact of the addition of boric acid as a fire retardant, as well as the incorporation of recycled paper fibres containing boric acid, and the application of trimethoxymethylsilane coating. The study employs thermogravimetric analysis and cone calorimetry under uniform external irradiance, with a T-history method to analyse thermal behaviour. Results indicated that fire retardants do not compromise the energy functionality of bio-based phase change material composites, exhibiting a latent heat of approximately 50 J/g. The density ranges from 750 to 875 kg/m^3. The use of clay as a binder improves fire performance, leading to a 60% decrease in total heat release and 52% of the composite mass remaining after analysis. Although enhancing fire performance presents challenges, incorporating wood particles in clay demonstrates a promising potential approach for safe use in building applications, contributing to energy efficiency in indoor heating and cooling. The findings contribute valuable insights into these materials for creating safer and more efficient building solutions, particularly in terms of thermal regulation and fire safety.

KEYWORDS
cone calorimetry, energy efficiency, energy storage, engineered wood, ethyl palmitate

1 | INTRODUCTION

In the face of contemporary challenges in sustainable energy supply and the substantial portion of annual energy consumption attributed to residential buildings, there is a growing need for research on energy management and storage within the built environment with no additional operating expense.

Phase change materials (PCMs) are substances that can absorb and release thermal energy during the process of melting and freezing (phase changes). When a PCM melts, it absorbs a large amount of heat from the environment, which it releases when it solidifies. Incorporating PCMs into the building envelope is beneficial for managing, optimising energy consumption and reducing peak loads, improving overall energy savings in buildings. Unlike conventional materials that only store sensible heat, PCMs have superior thermal storage capacity, eliminating the need for voluminous and heavy materials.[1–4] The latent heat storage approach is adaptable to a variety of porous building materials.[5] Current research is exploring the application of PCMs within plasterboard,[6] brick,[7] cement-based composites[8,9] and wood.[10] PCMs in wood composites with working temperatures of 18–25°C can absorb extra heat and release it when the indoor temperature falls below a certain comfort point. The incorporation of PCMs into wood enhances the thermal mass whilst maintaining its lightweight properties, enabling the absorption and storage of thermal energy within the comfort temperature zone.[11,12] This, in turn, helps regulate temperature fluctuations and energy intermittency within buildings. The use of impregnated wood, whether in the form of solid wood or wood-based products containing PCMs, for interior wall cladding or flooring contributes to thermal storage within a building. Particleboards and fibreboards, cost-effective alternatives to massive wood, are now widely employed for internal building applications. These materials offer both economic advantages and aesthetic appearance, making them highly relevant in design contexts.[13] Moreover, utilising wood industry by-products and recycled materials, such as fibres and recycled papers, not only addresses industrial waste management but also adds value to less valuable by-products.[14] However, the moderate heat capacity of wood materials, along with challenges such as susceptibility to bio-deterioration, moisture adsorption, dimensional instability and fire safety concerns, limits their effectiveness in managing energy intermittency for indoor applications.[2,15] Previous studies have focused on incorporating bio-based PCMs (BPCMs) into solid wood and wood fibres, producing energy-smart bio-composites[15–21] wood fibres and particles producing bio-based energy smart fibreboards and particle boards[14,22–25] or adding microencapsulated PCM.[26] In those previous studies, the thermal behaviour, moisture interaction and susceptibility to biological attack were investigated. However, challenges related the flammability and fire performance remain underexplored.

This study is one of the first attempts to produce smart energy bio-based composites using sawmill by-products, recycled paper and bio-binders with added fire retardants to evaluate their fire performance. These bio-based composites could be incorporated into building elements as non-load-bearing elements for interior wall cladding or flooring, contributing to heat storage within a building to regulate indoor temperature. The effect of employing several techniques involving the use of flame retardants as fillers, the replacement of the binder, the application of the coating or the addition of fire-resistant fibres into the elaborated composites was investigated. The research utilises thermogravimetric analysis (TGA) and cone calorimetry to assess the fire performance, along with a T-history method to evaluate the thermal behaviour of the wood composites with BPCM. The results of this research should serve as a fundamental step in improving fire safety by understanding the flammability dynamics of wood-based composites incorporating BPCMs. In addition, they are expected to highlight the relevant challenges that need to be addressed prior to the practical implementation of these novel materials in building applications.

2 | MATERIALS AND METHODS

2.1 | Materials

Ethyl palmitate purchased from Sigma–Aldrich was used as BPCM throughout the study. Pine wood sawdust (*Pinus sylvestris* L.) was obtained from Sätra sawmill near Uppsala, Sweden. Prior to its use, the sawdust particles were dried in the oven set to 60°C for 24 h. After this drying, three 5 g samples of sawdust were further dried at 103°C to a constant weight to measure their moisture content. The achieved moisture content was 3.5%. Moreover, the sawdust was screened to obtain particle sizes between 0.425 and 2 mm. Epoxidized linseed oil (ELO) purchased from Traditem GmbH, Germany was used as a bio-binder. Boric acid purchased from Sigma–Aldrich (Radnor, USA) was used as a fire retardant. Recycled paper insulation was obtained from the company iCell®, Älvdalen, Sweden. Clay from a local shop in Uppsala (Sweden) and sodium silicate 32% (Fixor by Nitor, Bromma, Sweden) were employed as an alternative binder (instead of ELO). For the coating of the composites, trimethoxymethylsilane (Tokyo chemical industry, Toshima, Japan), hydrochloric acid 37% and ethanol 98% (VWR, Radnor, USA) were used.

2.2 | Wood sawdust impregnation

Ethyl palmitate (BPCM) was impregnated into wood particles by a vacuum pressure process in an autoclave. The temperature of the autoclave was set to 60°C to ensure a liquid state of the BPCM during the whole process. The wood particles (150 g per batch) were immersed in the melted BPCM, and a vacuum of 350 mbar was applied for 10 min, followed by a pressure of 6 bar for 1 h. After impregnation, the wood particles were placed in an oven for 24 h at 40°C to leak out the excess BPCM. The leaked particles were then dried with a paper towel to absorb the residual BPCM on the particle's surface.

2.3 | Composite preparation

The untreated control composite (without BPCM and fire retardant) thereafter designed C-Untreated and the control composite (with BPCM and without fire retardant materials) thereafter designed BPCM composites (BPCMC) were prepared as described by Nazari et al.[14] Briefly, for preparation of the BPCMC-C pre-weighted 40 g of impregnated wood particles with BPCM was mixed with 10 g of untreated wood fibres and pre-mixed ELO (30 g) with strong Lewis acid. After the mechanical mixing of all the components, they were placed in the mould and pressed at room temperature (1.2 N/mm^2) for 45 min the composite was then cured by placing the mould in an oven set at 50°C for 12 h. The target dimension was 9 × 90 × 90 mm. For the preparation of the untreated composite (C–untreated) instead of using 40 g of impregnated sawdust, 20 g of non-impregnated sawdust was used, to provide the same volume of

TABLE 1 Composite formulations, including flame retardants and average panel density.

Designation	Wood particles (g)	Impregnated wood particles (BPCM) (g)	Untreated wood fibres (g)	Bio-binder (g)	Fire-retardant additives (g)	Fire-retardant additives Description	Panel density (kg/m^3)
C-untreated	20	–	10	30	–	–	659.6
BPCMC–C	–	40 (20)	10	30	–	–	775.0
BPCMC–filler	–	40 (20)	10	30	8	Boric acid (powder)	860.3
BPCMC–fibres	–	40 (20)	–	30	10	Recycled paper fibres (iCell)	752.7
BPCMC–clay	–	40 (20)	10	–	48	Mixture of clay, water and sodium silicate	791.1
BPCMC–coating	–	40 (20)	10	30	16	Silane coating	875.5

Abbreviation: BPCMC, based phase change material composites.

the composite. Table 1 shows the amount and the materials used for the preparation of the various BPCMC with fire retardants. The BPCM composite with the filler (BPCMC–filler), boric acid as a fire retardant, was added during mixing. In the next formulation (BPCMC–fibres) the untreated wood fibres were replaced by fireproof recycled paper insulation (iCell®). For the surface coating (BPCMC–coating) a liquid solution was prepared out of triethoxymethylsilane (68.8%), water (20.7%), ethanol (10.4%) and hydrochloric acid 37% (0.1%). The particles' surfaces were covered with the prepared solution and airtight dried at 40°C for 6 h followed by drying without the airtight cover at 40°C for 18 h. The procedure of coating the composites was repeated three times. For the preparation of the clay-based composite (BPCMC–clay), first, clay was homogenously mixed with water in a ratio of 1:0.5 (clay:water) and then 25% of sodium silicate was added. The impregnated wood particles were mixed with the clay preparation and subsequently pressed in the same way as for the other composites.

2.4 | T-history

The T-history method, outlined by Nazari et al.,[14,15] was employed to evaluate the thermal characteristics of wood-based composites incorporating BPCM, alongside control specimens lacking BPCM. This method facilitated the assessment of various parameters, including melting and freezing points, latent heat of fusion and specific heat capacity.

Experimental procedures involved the utilisation of specimens measuring 9 × 90 × 90 mm, accompanied by a copper plate of identical dimensions serving as a reference. Temperature variations were tracked using K-type thermocouples. Both the specimens and the reference were insulated with 10 mm thick Armaflex material, and the thermocouples were placed at the centre of the specimens. Temperature measurements were conducted over a period of 200 min, within both cold (0°C) and warm (40°C) ambient conditions.

2.5 | Thermogravimetric analysis

TGA was conducted on the composites containing BPCM utilising a TGA Mettler-Toledo TGA 2 system in a controlled nitrogen environment. Each TGA trial involved a sample weighing approximately 25 mg from the specimens, which was then deposited and not sealed into a standard alumina crucible pan. The TGA experiments were performed over a temperature range spanning from 26 to 500°C, employing a heating rate set at 10°C/min. Additionally, 5-min isothermal intervals were incorporated at both the initial and final temperatures of the analysis.

2.6 | Cone calorimetry

Cone calorimetry tests were carried out according to the procedures indicated in the ISO 5660-1:2015.[27] The specimens, including both treated and control samples, were prepared deviating from standard dimensions of 90 × 90 × 10 mm and conditioned in a climate chamber at 23°C and 50% relative humidity until mass equilibrium was reached. The cone calorimeter was equipped with sensors and a load cell to measure parameters such as mass loss rate, smoke production and heat release rate (HRR), with the test being terminated after 625 s, using a time-based criterion. An electric spark igniter was employed. The specimens were irradiated with a heat flux of 50 kW/m^2 from an electrical heater with the distance between the heat source and the sample surface set at 25 mm. A standard horizontal sample holder frame, measuring 100 × 100 mm and made of stainless steel, without a grid, was used. The edges of the specimens were wrapped in aluminium foil to minimise edge effects, and no backing material was used during the tests. Throughout the test, flame ignition time, HRR, total heat release (THR), smoke production rate (SPR) and mass loss were recorded.

3 | RESULTS AND DISCUSSION

Table 1 reveals variations in density amongst the samples, particularly those with the filler and coating, attributed to the additional mass of the additives whilst maintaining the same volume as the reference samples. Moreover, the addition of fire retardant into the composites did not disrupt the polymerisation or hardening of the matrix as reported in the previous studies (Figure 1).[14]

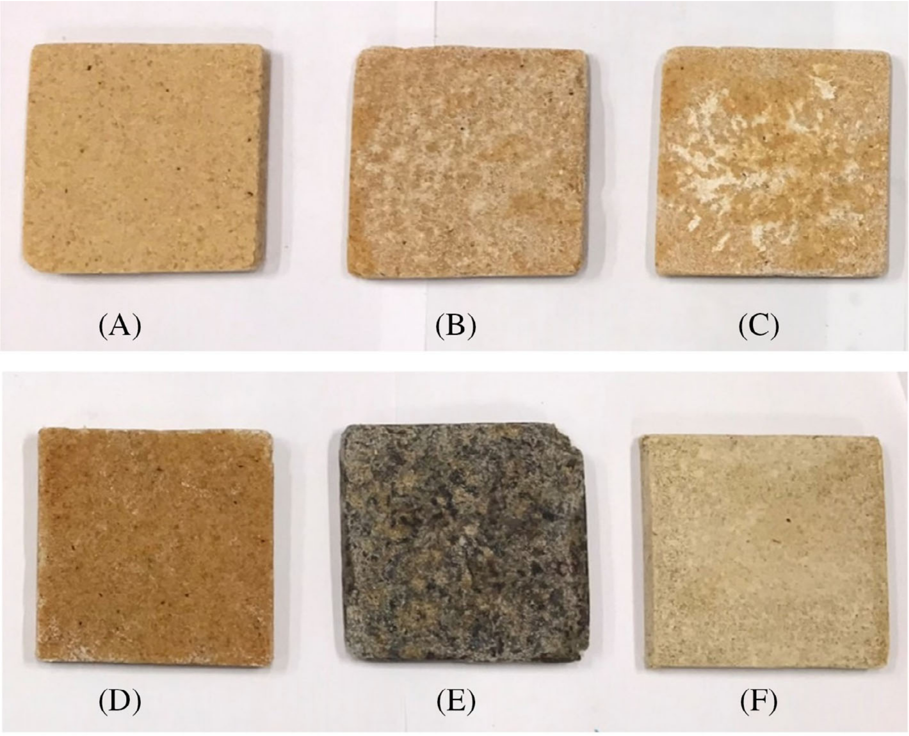

FIGURE 1 Wood composites used in the study. (A) C–untreated; (B) BPCMC–C; (C) BPCMC–filler, (D) BPCMC–coating; (E) BPCMC–fibres and (F) BPCMC–clay binder. BPCMC, based phase change material composites.

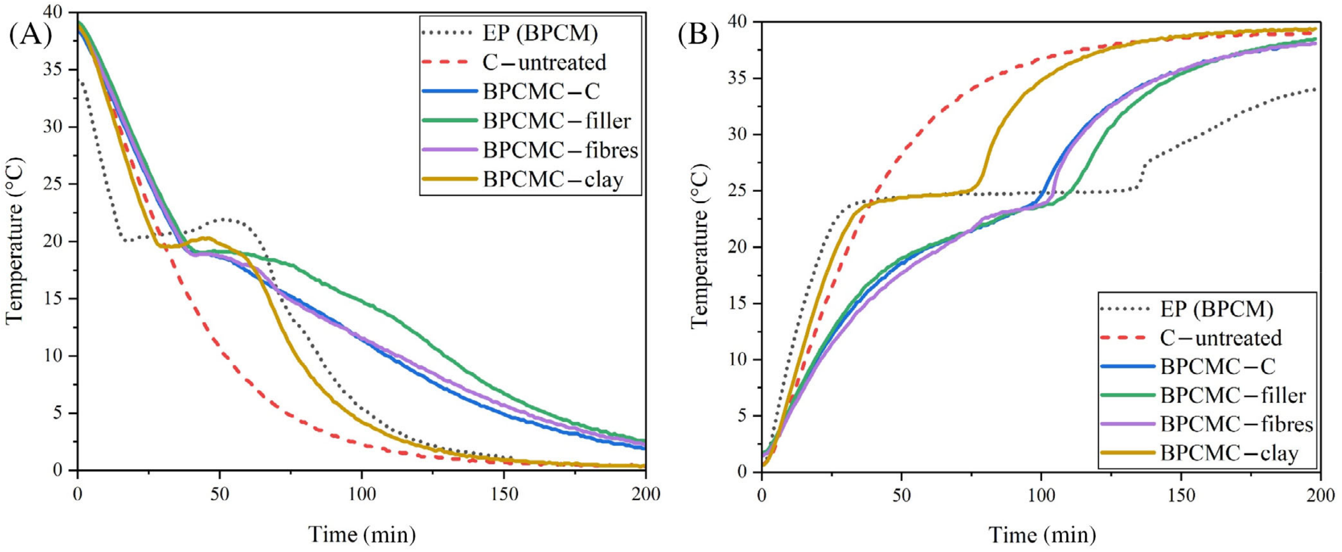

FIGURE 2 T-history temperature measurements inside the specimens with and without based phase change material (BPCM) during cooling (A) and heating (B) cycle. BPCMC, based phase change material composites.

3.1 | Thermal properties

A T-history test was conducted to assess the thermal energy storage capacity of the wood-based composites, both with and without BCPM. This analysis investigated the panels' ability to absorb and release thermal energy, thus maintaining the indoor temperature profile constant for a longer period compared to controls without BPCM. The temperature profiles versus time for pure BPCM, composites with and without BPCM are presented in Figure 2. The figure shows both the profiles during the cooling process (Figure 2A) and during the heating process (Figure 2B). All samples were first preheated to 40°C and then placed in a climate chamber with a temperature set at 0°C. During the cooling process (Figure 2A), it is observed that all samples with BPCM start to cool down until they reach the phase transition temperature (solidification temperature), at which the samples start to change phase from liquid to solid. It is observed that the pure BPCM starts to solidify at 20°C, and fully solidifies at 21.7°C, due to the supercooling effect. The samples without BPCM achieved the

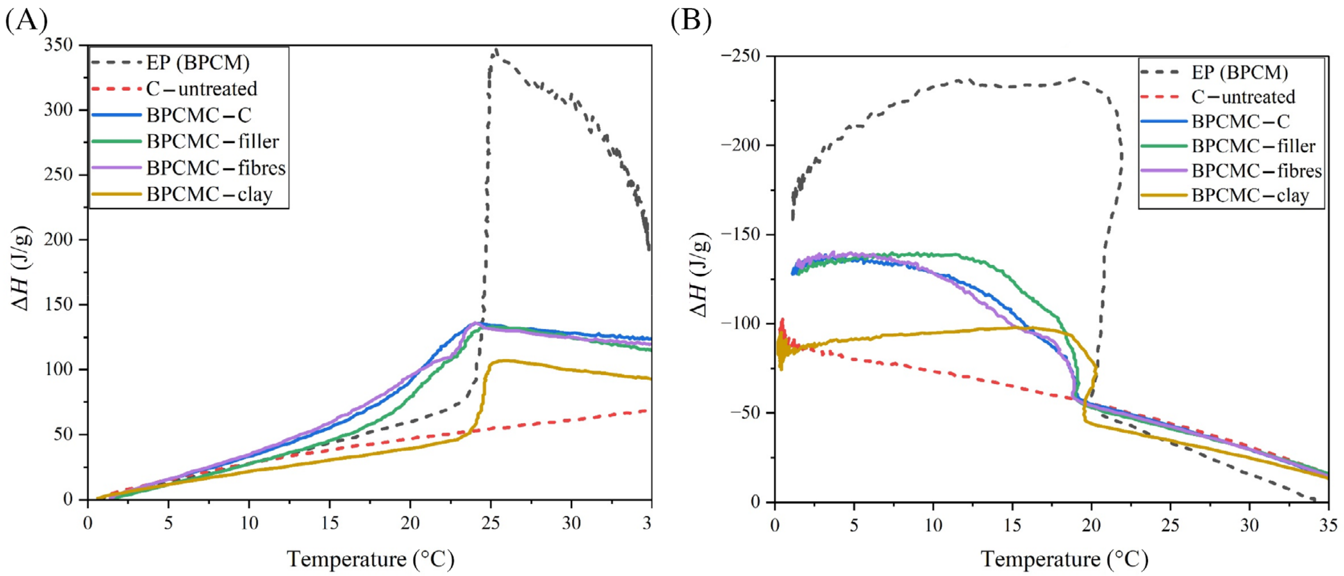

FIGURE 3 Melting (A) and freezing (B) enthalpy of pure based phase change material (BPCM) and wood composites with BPCM, calculated from the T-history measurements. BPCMC, based phase change material composites.

equilibrium temperature faster than the samples with BPCM. All the composites with BPCM experienced shifts to lower transient temperature and smaller supercooling compared to pure BPCM.[14]

During the heating process, as depicted in Figure 2B, the samples initially at 0°C were subjected to heating up to 40°C. Notably, the composite with clay (BPCMC–clay) as a bio binder exhibited a uniform transition pattern, whereas the remaining composites with BPCM displayed varying degrees of non-uniformity. In addition, the measurements showed that the addition of fire retardant to the composites did not affect the thermal behaviour of BPCM in the composites. Indeed, it was observed that the composites with ELO undergo certain non-uniformity whilst the pattern for composites with clay as a bio binder showed a similar pattern as pure BPCM. The analysis confirmed that modification of the formulation of the composites by incorporating boric acid (acidic medium) or sodium silicate (alkali medium) has no negative effect on the performance of BPCM. The phase transition temperature of the material is maintained, indicating that the modified composites retain their desired properties.

The enthalpy of the pure BPCM (Figure 3A,B) shows that the latent heat measured with T-history is around 200 J/g. The results for composites with BPCM, both with unmodified fibres and those containing recycled paper fibres, are at around 54 J/g. It is observed that clay composite shows a more uniform performance with 58 J/g storing energy. This observation suggests a correlation between the latent heat of composites and the amount of BPCM incorporated into the composite. The results are consistent with the findings of other authors when solid wood was impregnated with BPCM.[15]

The thermal stability of pure BPCM and the composites with BPCM was investigated using TGA (Figure 4). The analysis shows that pure BPCM exhibits stability up to 150°C, with no observable thermal degradation. However, as the temperature exceeds 150°C, degradation begins, leading to a weight loss of approximately 24 wt.% due to the decomposition of ester moieties.[14] This weight loss can be attributed to the degradation of the ethyl part of the ester. A gradual degradation continues up to 250°C, followed by a sharp degradation process that concludes at 325°C, resulting in complete degradation of the material.

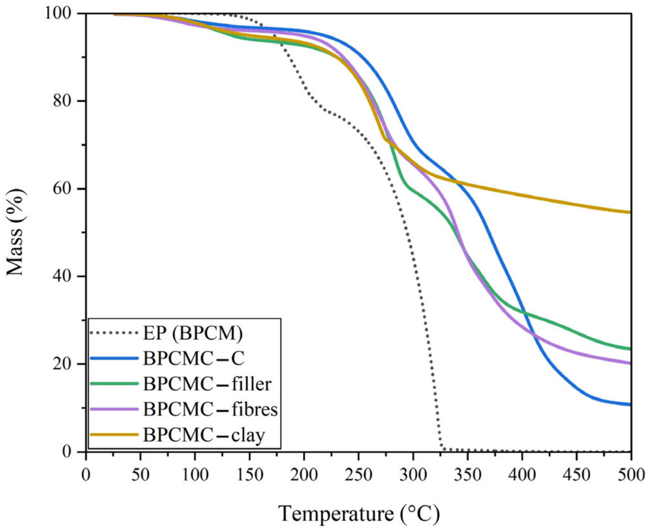

FIGURE 4 Thermogravimetric analysis curves for pure EP based phase change material (BPCM) (ethyl palmitate) and the composites with BPCM. BPCMC, based phase change material composites.

Similarly, the composites demonstrate comparable patterns. The degradation initiates around 100°C, primarily due to moisture evaporation, leading to a weight loss of around 4 wt.%. The subsequent stage predominantly involves the degradation of BPCM, starting at approximately 210°C and continuing until 320°C, resulting in the near-complete degradation of BPCM and an overall weight loss of

TABLE 2 Cone calorimetry results for composite materials: time to ignition, peak heat release rate (HRR), total heat release (THR) and maximum average rate of heat emission (MARHE).

Designation	Time to ignition (s)	Peak HRR (kW/m^2)	THR (MJ/m^2)	MARHE (kW/m^2)
C-untreated	13	488.7	147.4	292.3
BPCMC–C	6	547.7	210.1	430.3
BPCMC–filler	6	412.3	178.1	309.5
BPCMC–fibres	4	526.2	188.0	353.7
BPCMC–clay	6	347.8	82.2	217.5
BPCMC–coating	6	517.9	213.4	379.4

Abbreviation: BPCMC, based phase change material composites.

30 wt.%. The final stage corresponds to the degradation of the matrix (ELO polymer), and the lignocellulosic materials constituting the composites, occurring between 325 and 450°C. During this stage, the composite experiences a weight loss of approximately 70–90 wt.% of its initial weight. As expected, the BPCMC–filler and BPCM–fibres exhibit higher residual weights compared to the BPCMC–C, as the fire retardant (boric acid) is thermally stable under the studied range of temperature.[28] Moreover, the TGA results for the BPCMC–clay indicates that, like other composites, BPCM degrades up to 315°C, whilst the clay component and probably partial wood fibres enclosed in the clay matrix demonstrate thermal stability beyond this temperature range, exhibiting no further changes in weight loss.

3.2 | Fire performance characterisation

The cone calorimetry method is bench scale equipment to compare the fire response of materials reducing heat release, especially the flammability of materials.[29] This method allows a wide range of outputs to be obtained, reflecting variations in material behaviour under fire exposure.

The samples without BPCM experienced a longer initial ignition time of 13 s, whilst for the composites containing BPCM the ignition started after 6 s, apart from the BPCMC–fibres, which started igniting after 4 s (Table 2). Similar findings were reported by Hartig and Haller[30] showing that impregnated wood with paraffin PCM starts to burn faster than wood without PCM. The faster ignition times observed in the composites with BPCM can be attributed to the presence of BPCM on the surfaces of the composites, even in cases where the coating was applied. Since it is challenging to achieve completely leakproof surfaces, some BPCM still remain on the composite surfaces. As a result, ignition started from the burning of the BPCM in all cases, leading to faster ignition times for the composites with BPCM than the samples without BPCM.

Furthermore, the HRR is a crucial parameter measured with cone calorimetry tests that quantify the rate at which thermal energy is released during a fire event for a given material. This information is vital for assessing the fire performance of materials and understanding their flammability characteristic. This is even more important for wooden materials knowing that the wood-based products without fire retardants contribute to fire growth. HRR and the THR are shown in Figure 5.

The C–untreated sample displays behaviour consistent with the combustion characteristics observed in burning wood and wood-based materials.[31] Combustion generates heat that sustains wood pyrolysis, resulting in the release of more volatile compounds. A dip in the curve corresponds to the formation of an insulating char layer, which hampers heat transfer and slows down the pyrolysis process.[32] The second peak in the curve is likely caused by sample burn-through and char cracking, facilitating the release of additional volatile compounds.[33] Flaming combustion ends after volatile compounds are depleted, and the HRR returns to a steady baseline.[34] The BPCMC–coating behaves similarly to the sample without the BPCM, but the dip in the curve remains at a higher level, likely due to the contribution of the BPCM to the burning process.[35] The char layer and the coating do not completely prevent the escape of BPCM from leaking out of the composite. The BPCMC–fibres and BPCMC–filler show a less distinct second peak, suggesting a thinner char layer, and the continuous contribution of BPCM and the composite elements to the fire. The difference between the BPCMC–C, the BPCMC–filler and BPCMC–fibres can be observed in the THR, where these samples release 11.1% and 15.2% less heat than the reference composite with BPCM. The BPCMC–clay sample exhibits behaviour typical of materials where a thick char layer is formed. The THR of the composite with clay binder shows a 60.9% reduction in heat generation compared to the BPCMC–C. The results indicate that, in terms of THR, all the fire retardancy techniques employed in particular using clay binder led to a reduction in the THR value, except for the coating, whose results showed a higher THR value.

The SPR curves of the composites are presented in the Figure 6. The BPCMC–coating displayed the highest SPR amongst the tested samples. The SPR of BPCMC–clay was the lowest, likely due to the presence of clay, a non-combustible material and probably complete combustion of the BPCM, resulting in minimal smoke formation. Remarkably, during the test, this sample reached negative values for the SPR, which can be attributed to measurement drift and is within the noise level of the system.

The other samples exhibited a continuous high SPR value above 0.03 m^2/s. Furthermore, it can be deduced that the high SPR can be assigned to the presence of ELO. The SPR values for the composites

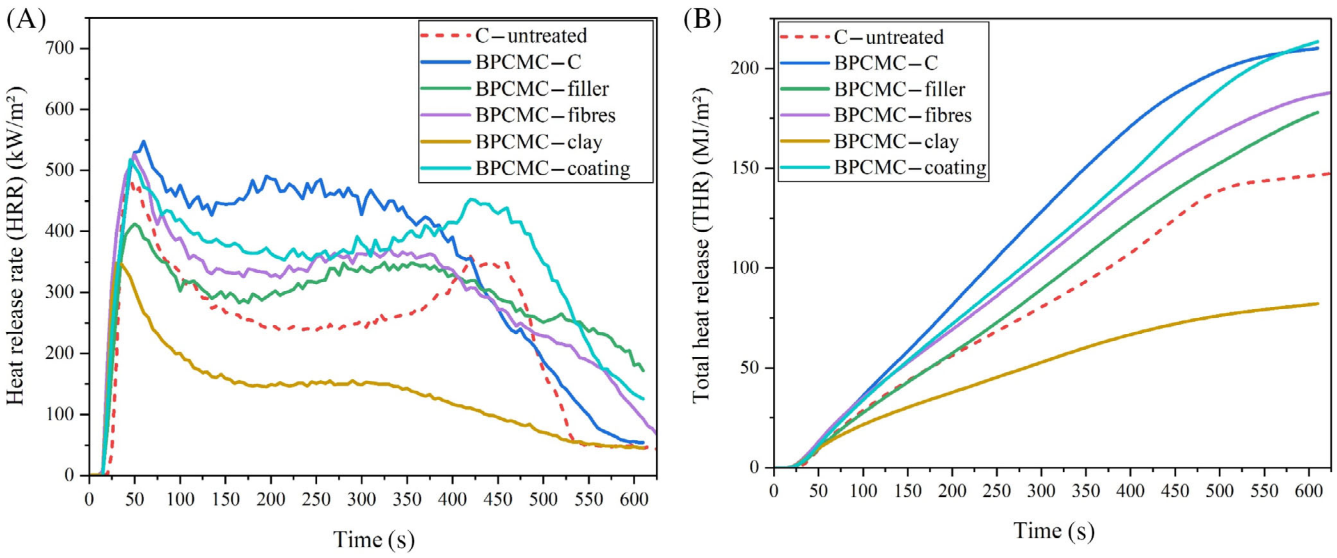

FIGURE 5 (A) Heat release rate and (B) total heat release of the composites as a function of time. BPCMC, based phase change material composites.

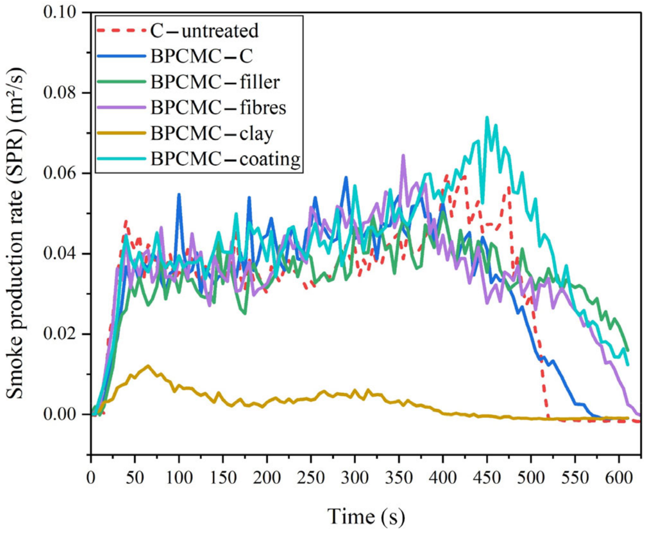

FIGURE 6 Smoke production rate of the composites as a function of time. BPCMC, based phase change material composites.

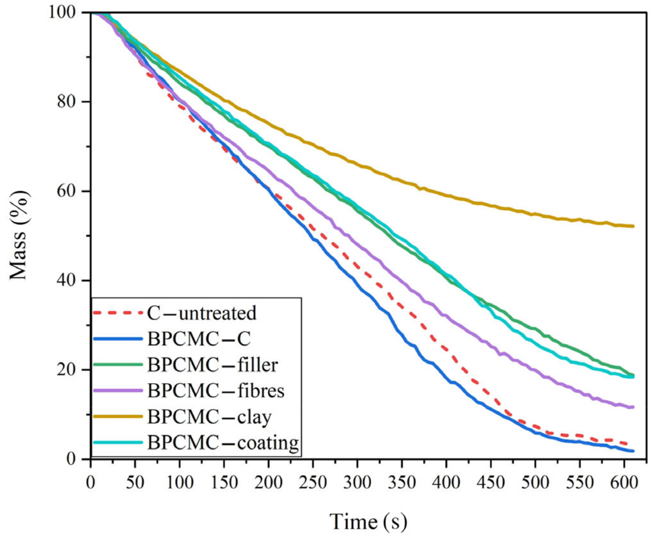

FIGURE 7 Mass loss of the composites as a function of time. BPCMC, based phase change material composites.

are higher than those reported by other authors for comparable wood-based products such as particle board, medium-density fibreboard, high-density fibreboard and plywood, where SPR values typically range around 0.01 m^2/s, with the single peak reaching a maximum of 0.04 m^2/s for high-density fibreboard.[36]

The total mass loss corresponds to the difference between the initial specimen mass and the residual mass (Figure 7). The results show that the composites could be categorised into three groups. The first group comprises composites without fire retardants, both with and without BPCM, with final weights of only 2% and 3%, respectively, at the end of the analysis. The second group includes composites with fire retardants, showing residual weights in the range of 12%–18%. In the third category, a sample of the BPCMC–clay is presented, with residual weights of 52%.

The higher mass observed at the end of the analysis in samples with fire retardants can be attributed to the presence of thermally stable fire retardants, such as boric acid. On the other hand, the BPCMC–clay had a residual mass of over 50%, indicating that clay could play a protective role in preventing complete combustion of the wood.

4 | CONCLUSION

In this study, the thermal properties and fire performance of wood-based composites containing BPCM were investigated. Various approaches were explored to improve the fire performance of the elaborated composites, including the addition of fire retardant (boric acid), application of silane coating, the inclusion of recycled paper containing boric acid or using clay as a binder. The results demonstrate that the inclusion of fire retardants did not affect the preparation process, and composite panels were successfully manufactured. The composites containing BPCM demonstrated their capacity to absorb and release thermal energy, with an average value of latent heat of 50 J/g.

The thermal and fire performance of the composites were evaluated using TGA and cone calorimetry, revealing a reduction in the ignition time for all samples containing BPCM due to its presence on the surface The leakage of the PCM during the service life should be included in the scope of further investigations as it might alter the flammability behaviour and favour the ignition of the fire. However, the findings indicate that the use of boric acid, recycled paper or silane coating alone did not improve the fire performance, suggesting that the heat release and smoke production may not solely be attributed to the BPCM but also to the binder, ELO. Interestingly, the samples with clay binder exhibited limited smoke production and retained around 52% of their mass after the cone calorimetry analysis.

These results not only confirm the feasibility of incorporating BPCM materials into wood composites but also highlight the challenge of increasing fire retardancy, which requires further exploration in the future.

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CONFLICT OF INTEREST STATEMENT

The authors declare no conflict of interest.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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