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Tensile shear strength of wood adhesives with fire-retardants under elevated temperatures

Luana Silva[a,b], Matheus Alves[a,b], Djems Andrade[a], Débora Macanjo Ferreira[a], Paulo Piloto[a] and Luis Mesquita[a]

[a]GICoS, Instituto Politécnico de Bragança, Bragança, Portugal; [b]CERIS, Department of Civil Engineering, University of Aveiro, Aveiro, Portugal

ABSTRACT

The utilization of wood and wood derivatives is growing in building construction due to sustainability and circularity, despite the inherent flammability of wood. Woodworking often requires bonding elements with adhesives, which are prone to degradation under fire. A potential solution to improve the fire resistance of wood joints and their derivatives involves incorporating fire retardants into the adhesive compositions. This study explores the adhesion properties of bonded joints of medium-density fibreboard and pinewood panels using resins based on urea and polyurethanes derived from diphenylmethane diisocyanate (MDI). Various proportions of ammonium polyphosphate, borax, and expandable graphite were incorporated as fire retardants. Shear testing was conducted on lap joints under room and elevated temperatures to evaluate their adhesive performance. MDI-based resin demonstrated higher shear resistance at elevated temperatures than the urea-based resin. The addition of fire retardants to MDI revealed a complex mechanism: they decreased strength at room temperature but improved it at 100 and 200 °C through protective actions and a later reverse behaviour at 230 °C, suggesting the onset of catalytic degradation. The transition from panel tear to cohesive failure at higher temperatures confirmed that adhesive degradation became the limiting factor. The MDI formulations proved competitive with, and in some cases superior to, established formaldehyde-based structural adhesives at elevated temperatures. The study concludes that the optimal formulation is application-dependent: for fire-resistance applications, the MDI resin without fire retardant proved to be the most robust at 230 °C, whereas for high-temperature service, MDI with APP maximizes strength at 100–200 °C.

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CONTACT Mesquita, Luis lmesquita@ipb.pt GICoS, Instituto Politécnico de Bragança, Campus Santa Apolónia, 5300-253 Bragança, Portugal.

1. Introduction

The use of wood-based products in civil construction has been increasing, driven by ecological and environmental concerns that emphasize the importance of wood as a natural, ecologically sustainable, and renewable material. Alongside the growing use of wood in the construction industry, performance requirements have also heightened across its life cycle, encompassing aspects such as mechanical resistance and safety in case of fire. Indeed, ensuring fire safety remains a design concern, as the charring rate and residual bearing capacity of wood components are critical properties that can limit their application in buildings with demanding fire resistance requirements [1]. Therefore, thermal, physical, or chemical treatments are crucial to enhance the stability and durability of wood. Such modifications include thermal treatments designed to improve dimensional stability for structural use [2] and advanced chemo-physical processes, like delignification and densification, aimed to significantly improve the mechanical strength and thermal stability of lignocellulosic materials [3].

Adhesive bonding is widely utilized in the wood industry to manufacture sandwich composite structures or wood-based products, such as plywood and particleboard. Although the interest in natural adhesives has increased, synthetic adhesives remain prevalent due to their effectiveness and lower cost [4]. Principal synthetic resins often contain formaldehyde, a highly toxic volatile organic compound. Urea-formaldehyde (UF) resin, commonly used in wood-based panels, has advantages like low cost and fast curing but has limitations, including low water resistance and restricting indoor use [5]. For wet conditions, melamine-urea-formaldehyde (MUF) resin is preferred due to the percentage of melamine, enhancing moisture resistance [6]. Polyurethane (PU) adhesive, free of formaldehyde, offers environmental safety and features a short press time, good mechanical strength, and water resistance [7]. Moreover, resins based on polyurethanes derived from diphenylmethane diisocyanate (MDI) are excellent wood adhesives but are less common in the industry by being more expensive than the usual formaldehyde-based resins [8].

Wood flammability is one of the limitations of its use in construction. Therefore, the search for improving it is constantly increasing, as seen in recent review studies on treatments to improve the wood fire reaction [9–11]. There are different ways to improve it, such as by retardant coating surface treatment [12], adding fire-retardant to the adhesive or by chemical impregnation of wood [13]. Choosing an appropriate treatment that improves wood fire reaction without causing significant damage to its physical and mechanical properties is essential. The effectiveness of wood treatment relies not solely on its fire-retardant capabilities but also on how the retardant is distributed throughout the wood [14]. Flame and fire-retardants act by breaking the combustion reaction or creating a shield of surface coating, retarding heat release and fire propagation [9]. Different types of these retardants have been tested and studied to improve wood flammability. Ammonium polyphosphate (APP) is a widely used inorganic fire-retardant with a halogen-free composition [15,16], appreciated for its efficiency, cost-effectiveness and environmentally friendly attributes [9,16,17]. This efficiency is owing to the joint action between phosphorus and nitrogen present in it [10,15,18]. Boron compounds, including boric acid and borax, are popular wood fire-retardants [19,20], offering effective protection against wood-damaging organisms, minimal harm to mammals, and low volatility [21]. Studies have explored their combined application for enhanced flame retardancy [19,20,22–24]. Borax reduces flame spread, while boric acid suppresses smouldering, complementing each other. Researchers have also investigated combining boron compounds with APP for an increased flammability reduction effect [24–26]. Expandable graphite (EG) is frequently used as a nonhalogenated fire-retardant [27,28] due to its capacity to hinder or decelerate the propagation of flames [29]. The expansion of EG initiates around 200 °C through the decomposition of interlayer compounds [30–32], giving rise to a char-residue characterized by a worm-like structure [28,30,33,34], which serves as insulation and reduces the transfer of heat and the flow of flammable gases. During combustion, EG is known to manifest some effects like "popcorn," "candlewick," "labyrinth," and barrier [27,30,33,34]. Additionally, some studies use the EG combined with other additives to produce a synergistic fire-retardant effect [30,35].

The contact of glued wood composite structures or wood-based products with fire compromised their bond strength. Consequently, evaluating the mechanical properties of wood bonding under elevated temperatures. Many studies have conducted tensile tests to analyse the shear behaviour of these structures employing various types of wood, adhesives, and temperature ranges [36–46]. However, no studies of wood panels bonded with fire-retardant adhesives, with a focus on evaluating their shear strength, have been found.

Therefore, this research aims to assess the shear behaviour of wood panel joints bonded with fire-retardants under elevated temperatures. Two wood panels, comprising a combination of medium-density fibreboard (MDF) and pinewood, were bonded using two different adhesives—resins based on MDI and urea. The combination of MDF and solid pinewood for the bonded joints was deliberately chosen to represent a common and structurally significant application in construction. This assembly simulates the bonding of a panel or cladding sheet (represented by MDF) to a solid wood support frame (represented by pinewood). Such bonded connections can be used as an alternative to traditional mechanical fixings like nails or screws in various applications. Practical examples include the manufacturing of high-performance doors, including both standard and fire-doors, where a core frame is bonded to surface panels, and in Light-Wood Frame (LWF) construction for shear walls or diaphragms where panel-to-stud adhesion is critical. Additionally, three fire-retardants (APP, borax, and EG) were incorporated into the adhesive formulations. Subsequently, shear tests were conducted at room and elevated temperatures to evaluate the bound shear behaviour and resistance.

2. Materials and methods

2.1. Materials

This study investigated the adhesion of two wood panels, combining MDF and pinewood. The moisture content and bulk density of both materials were measured according to EN 322 [47] and EN 323 [48], respectively. The MDF panels, produced by Finsa, have a nominal thickness of 19 mm, a moisture content of 7.2 ± 1.1%, and a bulk density of 754 ± 9.6 kg/m^3. The pinewood, scientifically known as Pinus Pinaster Ait., was supplied by a local producer and has a nominal thickness of 20 mm, an oven dried moisture content of 11 ± 0.29%, and a bulk density of 660 ± 4.1 kg/m^3.

The experimental tests used two distinct adhesives to bond the wood panels: MDI-based resin and urea-based resin. FLEXPUR supplied the MDI-based resin, which has a brown aspect and is a clear liquid without any suspended particles. Its low viscosity facilitates efficient mixing and homogenization, making it widely utilized in granulated cork panel manufacturing. Operating within an ideal temperature range of 130 to 140 °C, this prepolymer boasts 1.18 g/cm^3 of density and 95–100% solid content. On the other hand, the urea-based resin, supplied by Wurth, is a hot-pressing glue in powder form that requires a 2:1 mixture ratio with water (resin: water) to form the adhesive. This resin has 0.5 g/cm^3 of density and demands a pressing temperature ranging from 80 to 100 °C.

In addition to the resins, three fire-retardants were introduced in varying percentages. Clariant supplied APP as Exolit AP 422, a fine-particle substance that is colourless, non-hygroscopic, non-flammable, and exhibits a powder-like appearance. The EG, supplied by Georg H. Luh GmbH, is identified as a sulphuric acid compound with graphite. It has an expansion rate of a minimum of 200 ml/g and a starting temperature of approximately 130-170 °C. Lastly, the utilized borax, also known as sodium borate or sodium tetraborate, is an alkaline mineral derived from the mixture of a hydrated salt and has a white powder appearance.

2.2. Methods

The experimental methodology was based on EN 205 [49], specifically designed to define the tensile shear strength of lap joints of wood and derived timber products. Specific parameters were adapted to suit the materials and objectives of this study, with the main adaptations being the wood type and panel thickness. Initially, the wood panels were cut to dimensions of 150 mm in length and 150 mm in width and then cleaned to ensure proper adhesion. Subsequently, the resins were prepared and applied according to the manufacturer's specifications, as outlined in Table 1. Throughout this process, the resins were combined with the fire-retardants, with varying percentages set at 2, 4, and 6%. In the case of MDI-based resin, the fire-retardant percentage was calculated based on the final adhesive weight spread per surface area. However, for the urea-based resin, this percentage was determined by the weight of the resin powder without water. The fire-retardants were

Table 1. Adhesive application and press conditions.

	Resin	
Adhesive application and press conditions	MDI	Urea
Adhesive spread (g/m^2)	150	150
Application on both sides	Yes	No
Pressing pressure (N/mm^2)	0.7	0.4
Pressing temperature (°C)	130	80
Pressing time (min)	120	8

incorporated to the resins and mixed for about 5 min, ensuring good adhesive homogeneity.

The adhesive application involved manual spreading using a spatula, after which the panels were bonded and subjected to the MonTech LP 3000 thermohydraulic press, under conditions outlined in Table 1. For the applications using the MDI-based resin, the pressing time was initiated with the press at 130 °C. In the cases of the urea-based resin, a thermocouple was placed between the panels, and the pressing time was only measured once the thermocouple reached the specified temperature of 80 °C at the resin layer. Following the pressing phase, the panels were at room temperature for at least seven days, and then the test samples were cut according to the design shown in Figure 1.

A tensile testing machine applied the load on the specimens, following the same procedure at both room and elevated temperatures. However, in the high-temperature cases, a furnace was affixed to the tensile testing machine with a PID system to control the internal temperature, as illustrated in Figure 2. The tensile load was applied until failure, using a displacement control velocity of 1 mm/min, initiated twenty minutes after reaching the desired temperature.

Figure 3 exemplifies the control of the internal temperature for the cases of 100 and 200 °C, which was measured with two thermocouples. While one measured the room temperature inside the furnace, the other was placed directly on the sample. Table 2 indicates all combinations of resin and fire-retardants used for the tests, with the reference cases denoted by the absence of the fire-retardant (–). For each combination, five specimens were tested at room temperature, 100 and 200 °C and three specimens were tested at 230 °C, as their results showed a residual shear resistance with a low coefficient of variance.

The tensile tests provided data on the maximum load applied, denoted as F_{max} in N. Then, the shear strength (τ) in MPa is determined according to Equation (1), in which L_2 represents the length in mm of the bonded test surface, and b is the width in mm of the bonded test surface, see Figure 1.

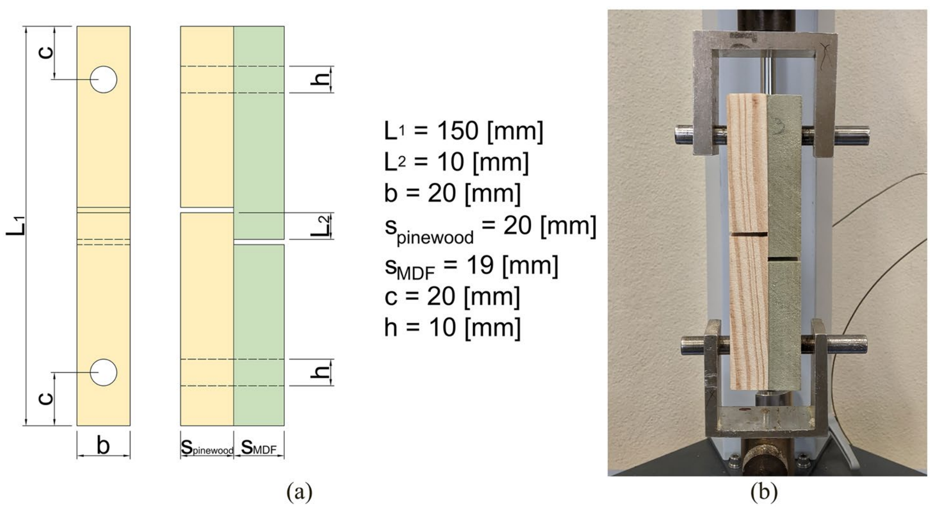

Figure 1. (a) Test specimen dimensions in mm; (b) Experimental test specimen.

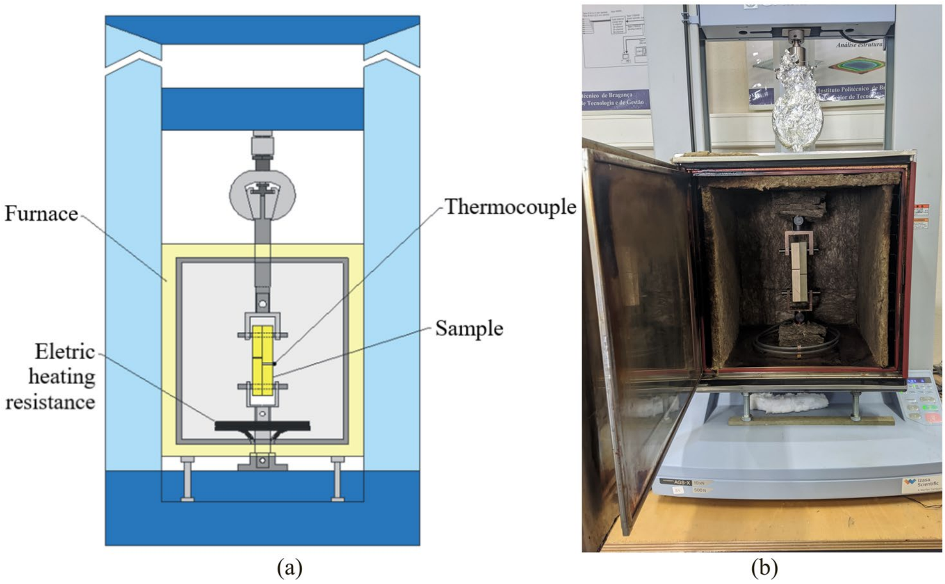

Figure 2. (a) Layout of the tensile testing machine; (b) Experimental tensile testing machine and furnace.

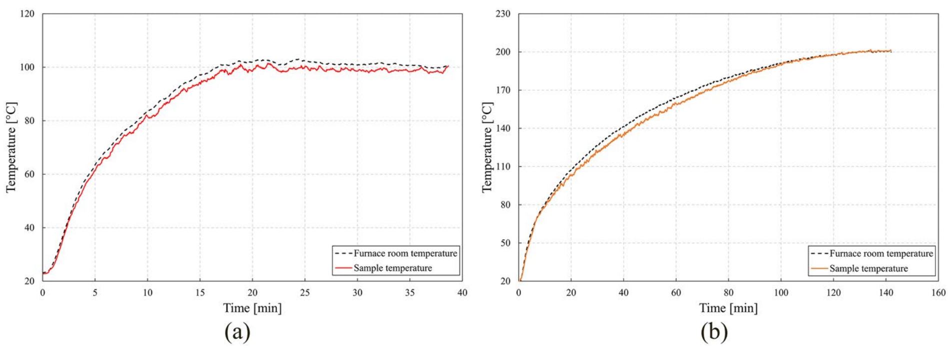

Figure 3. Examples of temperature control: (a) 100 °C; (b) 200 °C.

Table 2. Combinations of resins and fire-retardants for each temperature.

Resin	Fire-retardant	Fire-retardant percentage (%)	Temperature (°C)
MDI	–	–	Room, 100, 200, 230
	APP	2,4,6	Room, 100, 200, 230
	Borax	2,4,6	Room, 100, 200, 230
	EG	2,4,6	Room, 100, 200, 230
Urea	–	–	Room, 100, 200
	APP	2,4,6	Room, 100
	Borax	2,4,6	Room, 100
	EG	2,4,6	Room, 100

$$\tau = \frac{F_{max}}{L_2 \times b} \quad (1)$$

The standard EN 14869-2 [50] outlines a procedure to ascertain the shear characteristics of an adhesive in a single lap joint bonded assembly when subjected to a tensile force. Figure 4 illustrates the shearing of an adhesive joint in the specimen. The shear modulus (G) of the adhesive, calculated by $G = \Delta\tau / \Delta\gamma$, corresponds to the gradient of the low-strain linear region when the shear stress (τ) is plotted against the shear strain (γ). Linear regression analysis, recommended in EN 789 [51], was employed in the section between $0.1F_{max}$ and $0.4F_{max}$.

The shear strain in the adhesive is approximately given by $\tan\gamma = d/t$, in which t is the adhesive thickness. Although [50] recommends measuring the adhesive thickness and calculating the approximate shear displacement, an extensometer measurement was unworkable due to the adhesive's relatively thin thickness. Consequently, d is the measured displacement of the specimen during the tensile test, and the shear modulus per thickness of the adhesive, in Pa/m, is calculated using Equation (2).

$$G/t = \frac{\tau_2 - \tau_1}{d_2 - d_1} \quad (2)$$

where $\tau_2 - \tau_1$ is the increment of shear strength corresponding to the load between $0.1F_{max}$ and $0.4F_{max}$, and $d_2 - d_1$ is the increment of the measured displacement between $0.1F_{max}$ and $0.4F_{max}$.

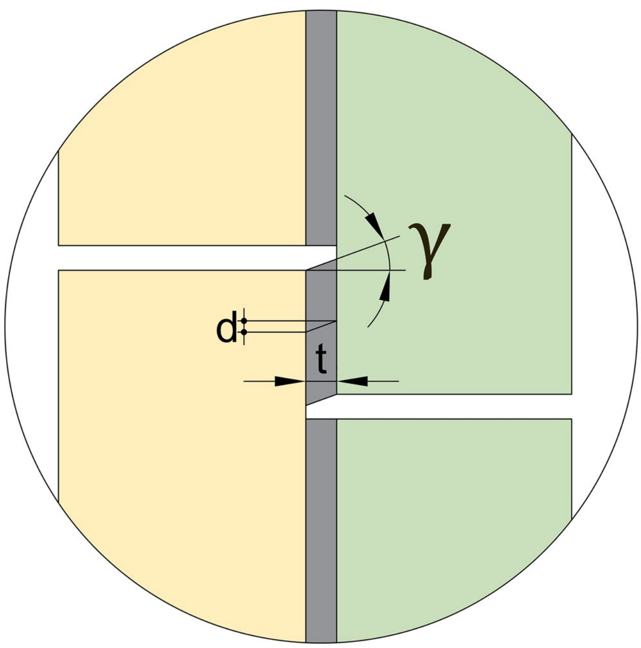

Figure 4. Shearing of an adhesive joint in the specimen.

3. Results

3.1. Failure modes

While the expectation is that a structurally bonded joint should outperform the connected structural elements, there are different expected failure modes. The standard D 5573 [52] covers the classification, identification, and characterization of failure modes in adhesively bonded fibre-reinforced plastic. Figure 5 shows the main failure modes considered in this study, classified solely through visual observation without microscopic magnification.

Two primary failure modes involve the detachment of the adhesive joint. Adhesive failure arises from separation at the adhesive-adherend interface, while cohesive (C) failure involves separation within the adhesive itself. Panel tear (PT) failure entails the tearing of the adherent near the adhesion surface. When the bond strength is greater than the element's strength, the final mode, known as stock-break failure, takes place beyond the area of the adhesive bonded joint.

The failure modes observed for all cases at each tested temperature are presented in Table 3 (for MDI-based adhesives) and Table 4 (for urea-based adhesives). A clear trend can be observed from these tables. At room temperature and 100 °C, the predominant failure mode for nearly all variants was panel tear. This failure mode indicates that, at lower temperatures, the adhesive bond strength was greater than the substrate's own strength.

When the temperature increases to 200 and 230 °C, the failure mode changes to cohesive failure. This transition physically demonstrates that the thermal degradation of the adhesive became the limiting factor for the joint's integrity at elevated temperatures. Figure 6 provides a visual example of this transition for a representative case (MDI with 4% borax).

3.2. Shear strength

The tensile test results demonstrated that as the temperature increased, the maximum force applied and the axial displacement of the specimen decreased for all the cases evaluated. The force-displacement behaviour of each specimen is exemplified in Figure 7, considering the case of MDI resin with 2% of borax at different temperatures. In addition, the standard deviation of the maximum applied force is greater

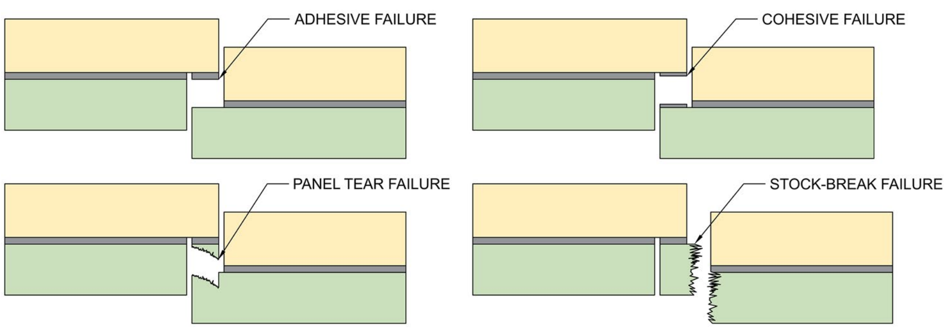

Figure 5. Failure modes.

Table 3. Failures modes for MDI-based resin.

MDI resin											
		Failure mode									
Temperature	Sample	No fire retardant	APP 2 %	APP 4 %	APP 6 %	Borax 2 %	Borax 4 %	Borax 6 %	EG 2 %	EG 4 %	EG 6 %
Room temperature	1 to 5	PT	PT	PT	PT	PT	PT	PT	PT	PT	PT
T = 100 °C	1 to 4	PT	PT	PT	PT	PT	PT	PT	PT	PT	PT
	5	PT	PT	PT	PT	PT	C	PT	PT	PT	PT
T = 200 °C	1	PT	C	C	C	PT	C	PT	C	PT	C
	2	C	PT	C	C	C	C	PT	PT	PT	PT
	3	C	PT	C	PT	PT	C	PT	C	C	C
	4	PT	PT	C	PT	PT	C	PT	C	C	PT
	5	C	C	C	PT	PT	PT	PT	PT	C	C
T = 230 °C	1 to 3	C	C	C	C	C	C	C	C	C	C

Cohesive failure (C), Panel Tear (PT).

Table 4. Failures modes for urea-based resin.

Urea resin											
		Failure mode									
Temperature	Sample	No fire retardant	APP 2 %	APP 4 %	APP 6 %	Borax 2 %	Borax 4 %	Borax 6 %	EG 2 %	EG 4 %	EG 6 %
Room temperature	1 to 5	PT	PT	PT	PT	PT	PT	PT	PT	PT	PT
T = 100 °C	1 to 5	PT	PT	PT	PT	PT	PT	PT	PT	PT	PT
T = 200 °C	1	C									
	2	PT									
	3	C									
	4	C									
	5	C									

Cohesive failure (C), Panel Tear (PT).

for the cases considering room temperature and 100 °C, decreasing with increasing temperature. This behaviour can be related to the results of the failure modes, since at room temperature and 100 °C, the failure is panel tear and at 200 and 230 °C is cohesive.

The shear strength's average value, calculated using Equation (1), was determined for all experimental cases with the same temperature, resin type, and fire-retardant percentage. Table 5 presents these results for the MDI-based resin, and Table 6 compares the average value results of the reference case ($\tilde{\ }_{[-]}$) with the other cases using fire-retardant (τ_i).

Figure 8 graphically presents the results for the specimens where APP was added to MDI-based resin, along with standard deviation bars for comparison. At room temperature, the shear strength of the reference case without fire retardant was about 30% higher than that of the other cases. However, at 100 and 200 °C, the shear strength increased with the addition of APP. The most favourable outcomes were achieved at 100 °C with a 4% APP content, demonstrating a 30% improvement over the reference example. Specimens containing 2% APP were particularly noteworthy at 200 °C, displaying a shear strength 16% greater than the reference example. However, at 230 °C, the tests behaved similarly to room temperature, resulting in a higher shear strength in the case with no fire-retardant.

Figure 9 illustrates the results of the cases of MDI-based resin with borax. Similar to samples with APP cases, the results for cases with borax addition at room

Figure 6. Failure modes results with temperature variation for the case of MDI with 4% of borax.

temperature and 230 °C were, around 30 and 40% lower than the reference case, respectively. Conversely borax enhances shear strength at 100 and 200 °C. At 100 °C a 4% borax concentration yielded the highest shear strength among the borax samples, showing a 22% improvement compared to the reference case. At 200 °C the 6% concentration showed to be the most effective, resulting in a 12% increase over the reference.

Results for MDI-based resin cases with EG are depicted in Figure 10, exhibiting similar behaviour to the other fire-retardant cases. The addition of EG improved shear strength by up to 19 and 9% for cases with 2% EG at 100 °C and 4% EG at 200 °C, respectively. Moreover, the reference case showed the highest shear strength at 230 °C and room temperature.

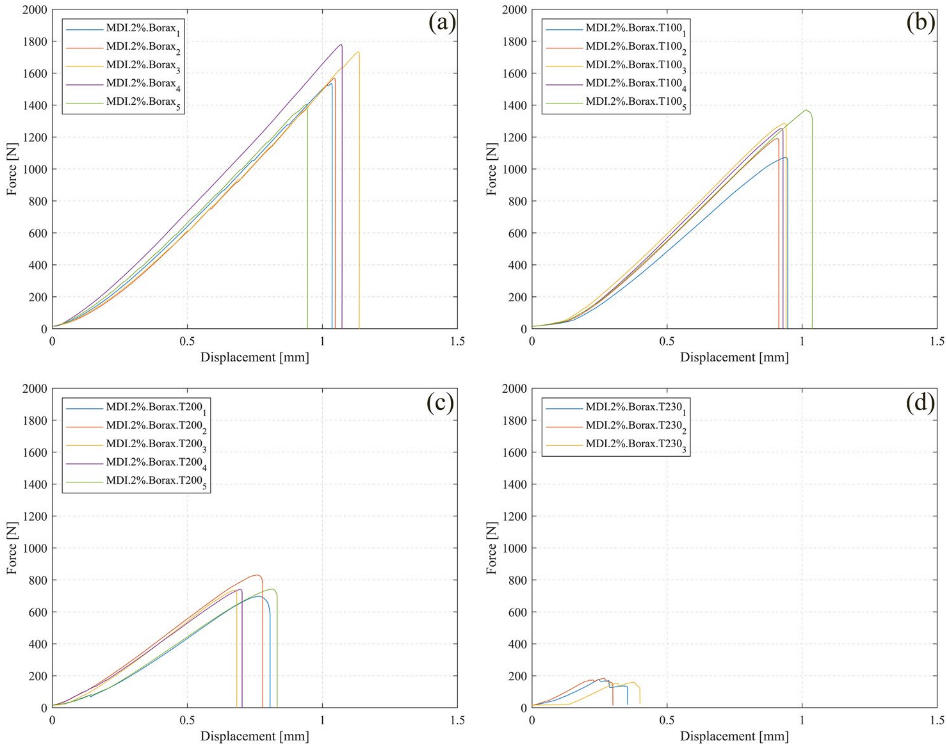

Figure 7. Example of the force-displacement behaviour at: (a) room temperature; (b) 100 °C; (c) 200 °C; (d) 230 °C.

Table 5. Shear strength's average value in MPa, for MDI-based resin.

	MDI									
	No fire-retardant	APP			Borax			EG		
Temperature (°C)	(–)	2%	4%	6%	2%	4%	6%	2%	4%	6%
Room temperature	11.43	8.17	8.64	8.17	8.03	8.97	8.05	8.49	6.14	8.13
100	5.46	6.46	7.10	6.14	6.17	6.64	6.53	6.48	5.41	5.77
200	3.63	4.23	3.91	3.22	3.75	3.98	4.05	3.70	3.96	3.90
230	1.98	1.08	1.02	1.20	0.87	1.50	1.16	0.47	1.18	0.80

Table 6. Comparison of the shear strength's average value results for MDI-based resin between the reference case with no fire-retardant ($\tau_{[-]}$) and the other cases (τ_i).

	$\tau_i / \tau_{[-]}$								
	MDI								
	APP			Borax			EG		
Temperature (°C)	2%	4%	6%	2%	4%	6%	2%	4%	6%
Room temperature	0.71	0.76	0.71	0.70	0.78	0.70	0.74	0.54	0.71
100	1.18	1.30	1.12	1.13	1.22	1.20	1.19	0.99	1.06
200	1.16	1.08	0.89	1.03	1.09	1.12	1.02	1.09	1.07
230	0.55	0.51	0.61	0.44	0.76	0.58	0.24	0.60	0.40

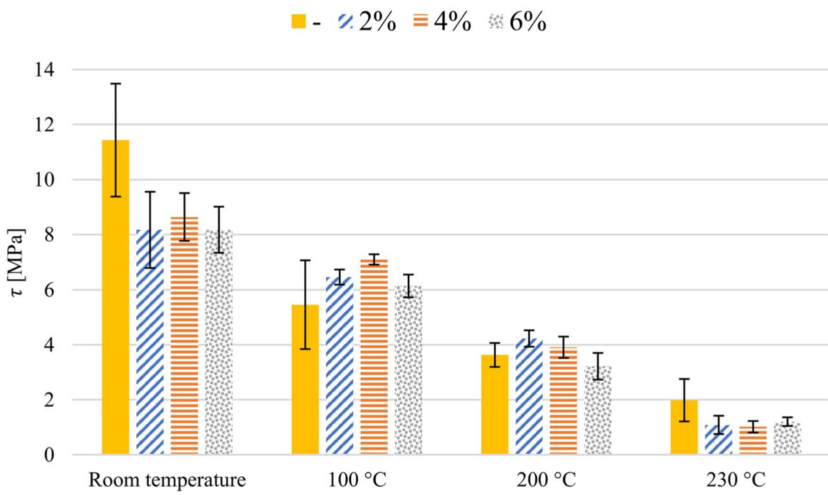

Figure 8. Shear strength variation with temperature for MDI-based resin with different APP percentage.

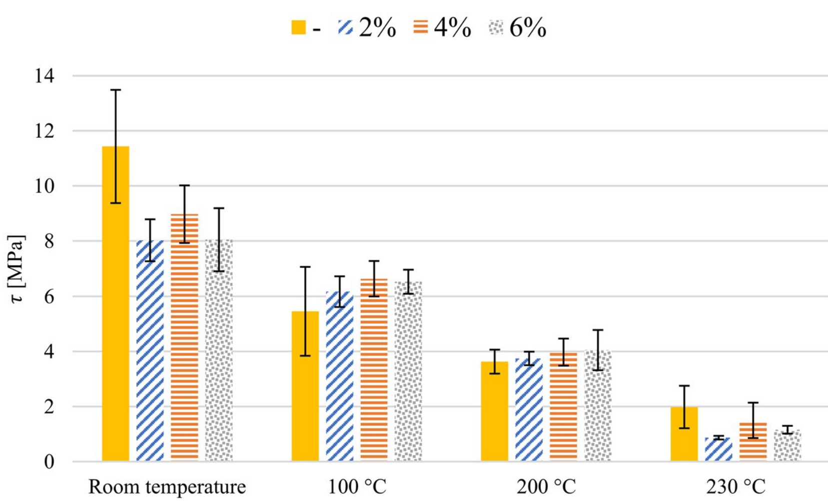

Figure 9. Shear strength variation with temperature for MDI-based resin with different borax percentage.

In summary, among all the experimental MDI-based resin cases, the 4% APP concentration demonstrated the best performance at 100 °C, and the case of 2% APP performed best at 200 °C. Moreover, cases considering 6% and 2% of EG have the lowest shear strength value at 100 and 200 °C, respectively.

Table 7 presents these results of urea-based resin, and Table 8 compares the average value of the reference case ($\tau_{[-]}$) with the other cases using fire-retardant (τ_i). The fire-retardant cases were not tested at 200 °C for the urea-based resin due to its poor adhesion in the reference case, that resulted in an average shear stress of only 0.18 MPa.

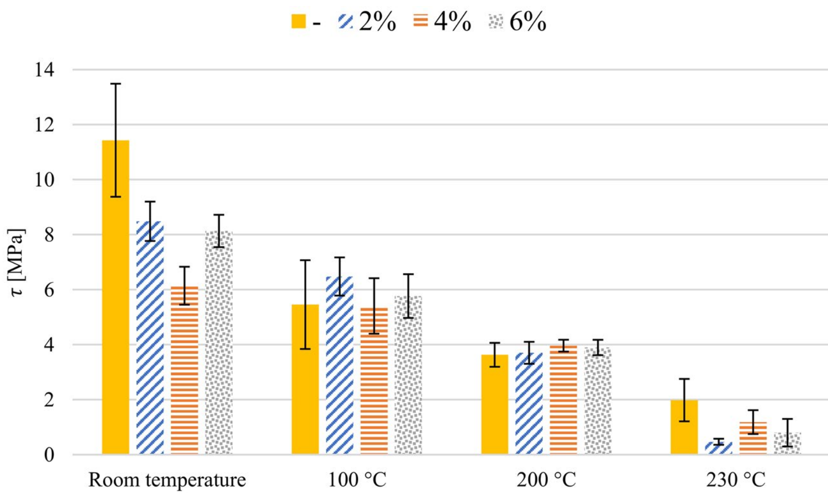

Figure 10. Shear strength variation with temperature for MDI-based resin with different EG percentage.

Table 7. Shear strength's average value in MPa, for urea-based resin.

	Urea									
	No fire-retardant	APP			Borax			EG		
Temperature [°C]	(–)	2%	4%	6%	2%	4%	6%	2%	4%	6%
Room temperature	8.03	7.19	8.76	7.18	7.53	7.25	7.50	8.19	7.01	7.66
100	5.58	5.23	5.02	5.30	4.96	5.25	4.77	4.48	4.73	5.05

Table 8. Comparison of the shear strength's average value results for urea-based resin between the reference case with no fire-retardant ($\tau_{[-]}$) and the other cases (τ_i).

	$\tau_i / \tau_{[-]}$								
	Urea								
	APP			Borax			EG		
Temperature (°C)	2%	4%	6%	2%	4%	6%	2%	4%	6%
Room temperature	0.90	1.09	0.89	0.94	0.90	0.93	1.02	0.87	0.95
100 °C	0.94	0.90	0.95	0.89	0.94	0.86	0.80	0.85	0.90

Results for specimens with APP added to urea-based resin are graphically shown in Figure 11, with standard deviation bars for comparison. The 4% APP concentration exhibited the best results at room temperature, surpassing the reference case by 9%. However, APP did not enhance shear strength at 100 °C, where the reference case demonstrated the highest value.

In Figure 12, the graph illustrates the results for urea-based resin cases with borax. In contrast to APP cases, the reference case of urea exhibited the highest shear strength at room temperature and 100 °C. Thus, borax did not improve shear strength, resulting in differences of up to 10% for the 4% borax case at room temperature and up to 14% for the 6% borax case at 100 °C.

The results for urea-based resin cases with EG are represented in Figure 13. Although the 2% EG resulted in the highest shear strength at room temperature, the EG did not improve the shear strength at 100 °C either. At room temperature, the 2% EG case displayed a shear strength that was 2% greater than the case without a fire retardant; however, when the same case was heated to 100 °C, the shear strength decreased by as much as 20% in comparison to the reference case.

When comparing all the experimental cases involving urea-based resin, the 4% APP concentration showed the best performance at room temperature, and the reference case performed the best at 100 °C. Moreover, cases considering 4 and 2% of EG exhibited the lowest shear strength values at room temperature and 100 °C, respectively.

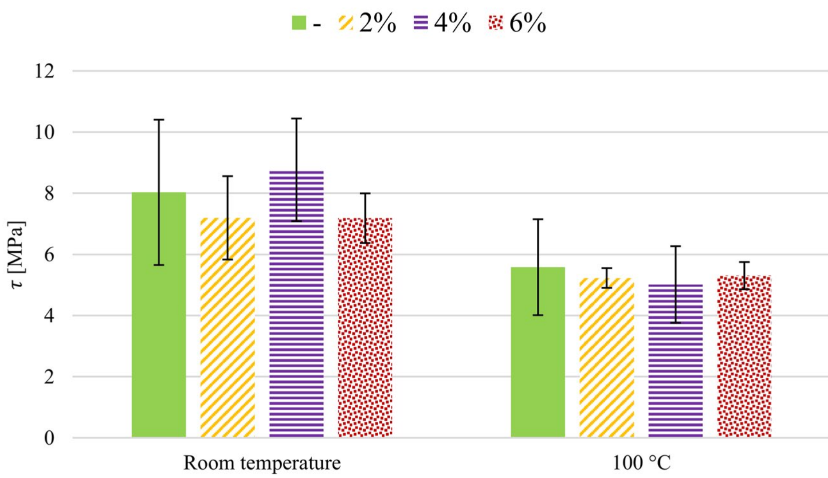

Figure 11. Shear strength variation with temperature for urea-based resin with different APP percentage.

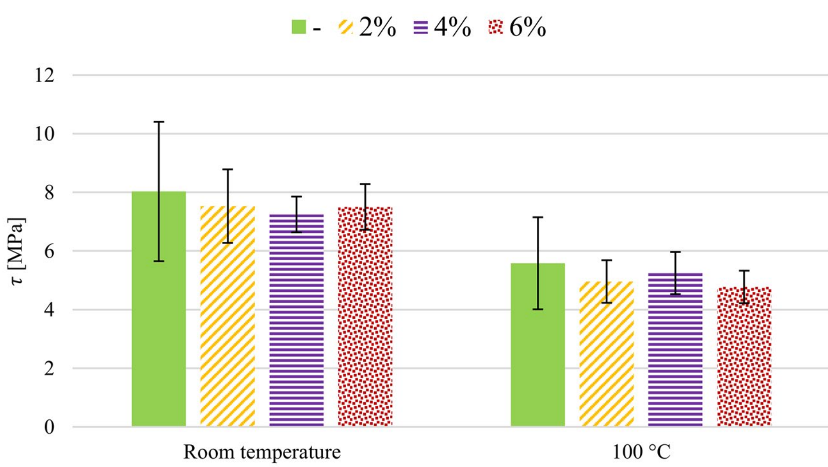

Figure 12. Shear strength variation with temperature for urea-based resin with different borax percentage.

3.3. Shear modulus

The average shear modulus per metre, calculated using Equation (2), was determined for all experimental cases with the same temperature, resin type, and fire-retardant percentage. Table 9 presents the results for the MDI-based resin. Overall, the adhesive rigidity exhibited a decreasing trend with rising temperature. Specifically, at room temperature and 230 °C, the reference MDI case, without a fire-retardant, demonstrated the highest shear modulus of 8.76 and 3.58 Pa/m, respectively. At 100 °C, the 4% borax yielded the maximum shear modulus per metre of 7.82 Pa/m, while the 6% borax case reached the highest shear modulus of 6.22 Pa/m at 200 °C.

Table 10 compares the average shear modulus per meter of the MDI case at room temperature, $(G/t)_{room}$, with the other cases at elevated temperatures, $(G/t)_i$. At 100 °C, the reference case showed a 23% reduction, while cases with fire retardants exhibited only up to an 8% decrease. At 200 °C, the overall rigidity is reduced by around 30%. At 230 °C, the 2 and 6% EG cases, on the other hand, showed a more notable drop of up to 95 and 84%, respectively. In contrast, decreases of almost 60% were observed in the reference instances of borax and APP.

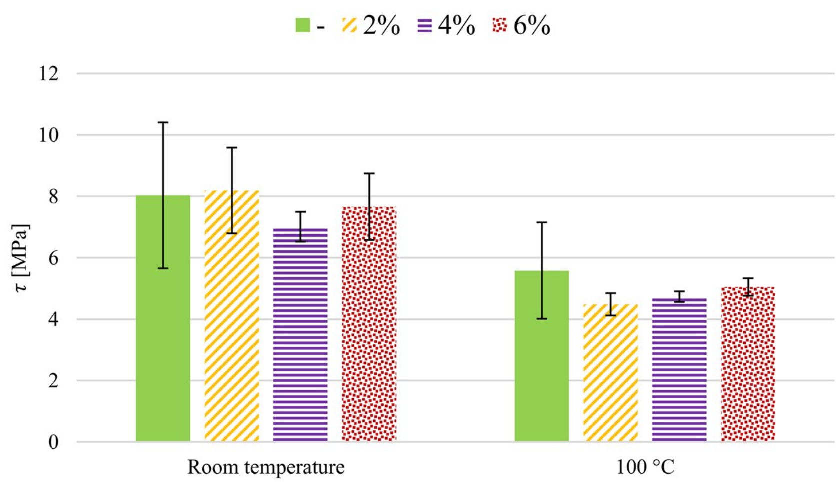

Figure 13. Shear strength variation with temperature for urea-based resin with different EG percentage.

Table 9. Average shear modulus per meter values for MDI-based resin, in Pa/m.

	G / t (Pa/m)									
	MDI									
	No fire-retardant	APP			Borax			EG		
Temperature (°C)	(–)	2%	4%	6%	2%	4%	6%	2%	4%	6%
Room temperature	8.76	7.12	8.04	7.09	7.53	7.80	7.80	8.27	6.29	7.73
100	7.15	6.94	7.53	6.88	7.17	7.82	7.42	7.59	6.43	7.58
200	6.20	5.36	5.04	4.45	5.07	5.10	6.22	5.09	5.28	5.00
230	3.58	2.86	2.80	3.49	2.32	3.36	3.26	0.39	2.33	1.21

Table 10. Comparison of the shear modulus per metre average values for MDI-based resin between the same case at room temperature $(G/t)_{room}$ and the other temperatures $(G/t)_i$

	$(G/t)_i / (G/t)_{room}$									
	MDI									
	No fire-retardant	APP			Borax			EG		
Temperature (°C)	(–)	2%	4%	6%	2%	4%	6%	2%	4%	6%
100	0.82	0.98	0.94	0.97	0.95	1.00	0.95	0.92	1.02	0.98
200	0.71	0.75	0.63	0.63	0.67	0.65	0.80	0.62	0.84	0.65
230	0.41	0.40	0.35	0.49	0.31	0.43	0.42	0.05	0.37	0.16

Table 11. Average shear modulus per meter values for urea-based resin, in Pa/m.

	G/t (Pa/m)									
	Urea									
	No fire-retardant	APP			Borax			EG		
Temperature (°C)	(–)	2%	4%	6%	2%	4%	6%	2%	4%	6%
Room temperature	9.49	7.48	7.79	7.49	7.99	7.58	7.58	7.41	7.20	7.17
100	2.23	6.68	6.42	6.41	6.60	5.50	5.04	5.78	4.87	6.22

Table 12. Comparison of the shear modulus per metre average values for urea-based resin between the same case at room temperature $(G/t)_{room}$ and the other temperatures $(G/t)_i$

	$(G/t)_i / (G/t)_{room}$									
	Urea									
	No fire-retardant	APP			Borax			EG		
Temperature (°C)	(–)	2%	4%	6%	2%	4%	6%	2%	4%	6%
100	0.24	0.89	0.82	0.86	0.83	0.73	0.66	0.78	0.68	0.87

Analysing the shear modulus per metre results shown in Table 11 for urea-based resin, adhesive rigidity decreased with increasing temperature. At room temperature, the reference urea case had the highest shear modulus of 9.49 Pa/m, and at 100 °C, the 2% APP case resulted in the highest shear modulus per metre of 7.48 Pa/m. In this situation, the shear modulus of the reference case is merely 0.03 Pa/m at 200 °C.

Table 12 compares the average shear modulus per meter of the urea case at room temperature, $(G/t)_{room}$, with the other cases at elevated temperatures, $(G/t)_i$. Differently from the MDI reference case, the adhesive rigidity of the urea reference case reduced by 76% at 100 °C and nearly 100% at 200 °C. For the fire-retardant cases at 100 °C, the 6% borax case showed the highest rigidity reduction of 34%, while the 2% APP case exhibited the lowest reduction equal to 11%.

4. Discussion

The results from the previous section, showed a variation of shear strength with the type of resin and the fire retardants used, in function of the temperature. Liu et al. [43] and Yue et al. [53], also performed a set of shear tests in different temperature conditions, in a nitrogen-filled chamber to simulate a hypoxia environment and prevent degradation due to oxidation, evaluating the strength of the adhesives of phenol-resorcinol-formaldehyde (PRF) and melamine-urea-formaldehyde (MUF), from room-temperature until 280 °C. Their results show a shear strength at room-temperature equal to 9.62 and 9.07 MPa, for MUF and PRF, respectively. These values decrease to 6.56 and 6.48 MPa, for 110 °C, to 3.61 and 3.67 MPa for 200 °C, and to 1.11 and 1.18 MPa for 250 °C, respectively. The shear strength of the unmodified MDI and urea resins was normalized with respect to the room-temperature values, as shown in Figure 14. For comparison data from Liu et al. [43] and Yue et al. [53], are also shown in the figure, normalized in the same way to obtain independent strength values regardless the initial strength or wood substrate.

A first comparison can be made for the base resins, without fire retardants. The PRF and MUF adhesives provide a higher performance baseline for comparison, where a near-linear loss of strength is obtained from the room-temperature to 250 °C. The standard urea resin, conversely, have a lack of thermal stability resistance, resulting in an approximately zero strength at 200 °C. For the case of the unmodified MDI resin the behaviour is not linear. It shows a significative reduction between room temperature and 100 °C, equal to 52%, and an additional 16% reduction between 100 and 200 °C.

The most relevant fire-retardant formulations are additionally presented in the Figure 14. MDI formulations with 4 and 2% APP showed to be the most effective formulations at 100 and 200 °C, respectively. The results show that the addition of both APP formulations significantly improves the relative strength of the MDI system, eliminating the significative decrease until 100 °C. With both MDI plus fire retardant APP formulations is possible to obtain normalized strengths higher than the ones from resins PRF and MUF, for temperatures until 200 °C.

The experimental findings show a direct relationship between the change in failure modes with increasing temperature and the variation of shear strength. In most

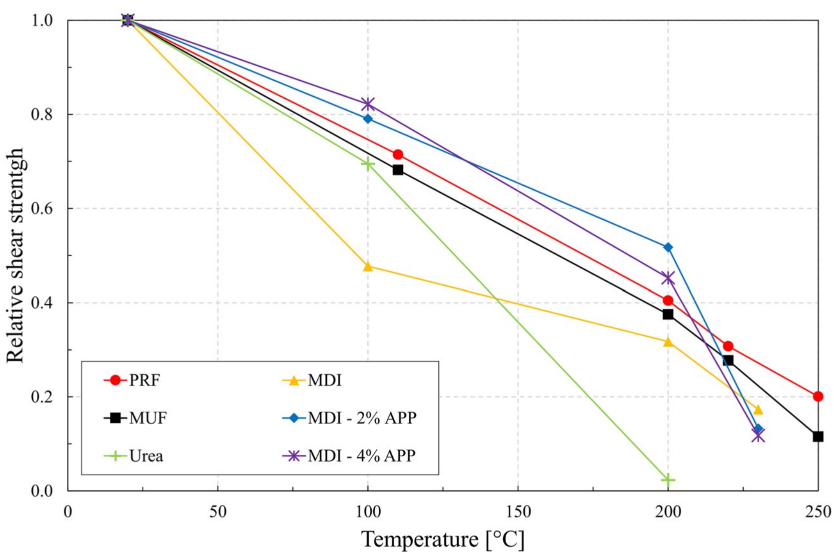

Figure 14. Relative shear strength of MDI and urea adhesives versus PRF and MUF data from [53].

cases, at room temperature and 100 °C, the adhesive bond strength was higher than the substrate, which led to the panel tearing. However, there was a noticeable change towards cohesive failure within the adhesive layer at 200 °C and particularly at 230 °C, indicating that the adhesive thermal degradation became a limiting factor for joint integrity. This behaviour was especially noticeable for MDI-based systems, which, even though they were more stable at high temperatures than urea resins, loose a significant amount of their cohesive strength as soon as the polymer matrix started to break down significantly, as highlighted in a recent review by Lee et al. [12]. The shear strength data trends are directly supported by this change in failure mode, which also demonstrates that adhesive cohesion, not the wood substrate, determines adhesion performance above a particular temperature threshold.

Fire retardants' effects on the thermomechanical behaviour of bonded joints showed a significant temperature dependence that was controlled by competing mechanisms. According to the mechanisms outlined by Lee et al. [12] and experimentally verified by Chen et al. [31], the addition of solid particles that function as physical fillers and produce local stress concentration sites is responsible for the overall decrease in bond strength at room temperature that occurs when APP, borax, or EG are added, breaking the polymer matrix continuity, reducing strength and ductility. However, protective effects dominate at intermediate temperatures (100–200 °C): EG expands to form a physical insulating barrier, APP produces a stable intumescent char, and borax can form a molten glassy surface layer, mechanisms also described in [12]. Because of these effects, the adhesive's thermal degradation is delayed, allowing MDI-based formulations with APP to exceed the reference resin's shear strength without fire retardants. A change take place at 230 °C, in which the fire retardants effect became negligible, mainly due to the catalytic degradation, described in [12] and illustrated by Wu et al. [54], in which degradation of byproducts from fire retardants accelerated polymer breakdown.

In contrast, urea-based resins responded differently to fire retardants addition due to the sensitivity of their polycondensation reaction to chemical interference. According to Khan et al. [55], the presence of inorganic fillers can change the resin system's pH and interfere with its curing kinetics. This prevents a fully cross-linked polymer network from forming, which results in intrinsically weaker adhesive bonds. Zhang et al. [56] also noted this phenomenon for MUF-based systems. While FRs reduced the shear strength of urea adhesives even at 100 °C, they appeared to preserve rigidity relative to the unmodified resin, likely by acting as a rigid dispersed phase within the softened matrix. This explains why the modulus loss was somewhat mitigated even though the absolute shear strength decreased. According to these results, MDI formulations with APP provide the best strength retention for extended service at 100–200 °C, while pure MDI is ideal for fire-resistance applications needing maximum structural integrity at higher temperatures.

5. Conclusion

The performance of two adhesives, urea-based resin and MDI-based resin, with three different fire-retardants (APP, borax and EG) was evaluated at both room and elevated temperatures. The shear tests utilized lap joints with pinewood-MDF combinations, and the experimental procedures were adapted from EN 205 to determine the shear strength of the bonded joints.

The results showed that the effect of the fire retardants on bond performance varies with temperature. The MDI resin demonstrated markedly higher thermal stability and shear strength compared to the urea resin, which lost nearly all its integrity at 200 °C. At room temperature, the addition of retardants tended to slightly decrease strength, acting as defects in the polymer matrix. However, at intermediate temperatures (100 and 200 °C), the retardants improved the shear strength of the MDI resin. At 230 °C, this effect changed, and the retardants start to reduce strength, which suggests the onset of catalytic degradation of the polymer. This adhesive degradation at higher temperatures was confirmed by the shift in failure mode from panel tear (at lower temperatures) to cohesive failure in the bond line.

Comparison with literature data for structural adhesives such as PRF and MUF showed that the tested MDI formulations are competitive. At room temperature, the MDI adhesive without retardants demonstrated greater strength, and at 100 °C, the retardant-containing formulations performed better than the reference adhesives. At 200 °C, the strength of MDI was similar to that of PRF, and the addition of 2% APP resulted in superior performance.

The analysis of the shear modulus indicated a decrease in adhesive stiffness with increasing temperature. For the urea resins, the retardants, despite decreasing the ultimate strength, helped maintain higher resistance at 100 °C compared to the reference resin.

For fire-resistance applications, where structural integrity at the highest critical temperature is the main objective, the reference MDI resin (without fire retardants) proved to be the most robust, maintaining the highest shear strength of 1.98 MPa at 230 °C. On the other hand, other formulations are better for applications that need the strongest bond possible at a consistent service temperature. The MDI formulation with 4% APP provides the best performance for service near 100 °C (7.10 MPa), while the MDI with 2% APP is the most effective for service near 200 °C (4.23 MPa). Therefore, the thermomechanical performance of MDI adhesives can be adapted to particular uses, where the unmodified resin performs better at the highest tested temperatures while the addition of specific retardants is very effective at the intermediately tested temperatures.

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Authors' contributions

CRediT: **Luana Silva**: Conceptualization, Investigation, Methodology, Writing – original draft, Writing – review & editing; **Matheus Alves**: Investigation, Writing – original draft; **Djems Andrade**: Investigation, Writing – original draft; **Débora Macanjo Ferreira**: Supervision, Writing – original draft; **Paulo Piloto**: Supervision, Writing – original draft; **Luis Mesquita**: Conceptualization, Methodology, Supervision, Writing – original draft, Writing – review & editing.

Disclosure statement

No potential conflict of interest was reported by the author(s).

ORCID

Luana Silva http://orcid.org/0000-0001-5321-5257
Débora Macanjo Ferreira http://orcid.org/0000-0002-5195-8265
Paulo Piloto http://orcid.org/0000-0003-2834-0501
Luis Mesquita http://orcid.org/0000-0002-2385-4282

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