A FIRE-RETARDANT COATING FOR WOOD MADE FROM FOOD-INDUSTRY WASTE

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Abstract

Wood is regaining popularity due to increased environmental awareness but requires protection against humidity, UV radiation, stains, and fire. We have developed a sustainable and protective coating derived from biogenic residues. This non-flammable coating enhances thermal stability, delays combustion, and prevents flame spread. Additionally, it protects against moisture by forming a water-insoluble film after polymerization. The coating can be applied from an aqueous solution, adheres well to wood surfaces, and is made from renewable sources, ensuring ecological sustainability.

Keywords

Wood coating, fire-retardant, sustainability, food-industry waste, circularity.

1 INTRODUCTION

Wood has recently gained renewed attention as a construction material due to its sustainability and role in reducing carbon footprints. It serves as a viable alternative to conventional materials like steel and concrete, contributing to environmentally friendly building practices. However, its natural composition makes it highly susceptible to external factors such as humidity, ultraviolet radiation, staining, and most critically, fire. Given its organic structure, untreated wood ignites readily and supports combustion, posing significant safety risks in construction.

To mitigate these risks, fire-retardant treatments are commonly applied. Traditional solutions frequently rely on halogen-based compounds, which, while effective, have been associated with environmental persistence and potential health hazards. [1], [2] As a result, research has shifted towards biopolymer-based alternatives that provide flame resistance without compromising sustainability. Various approaches, including chitin [3], [4] and chitosan-derived formulations [5], have shown promising results. However, limitations such as water solubility, reliance on synthetic additives, and complex manufacturing processes, restrict their practicality.

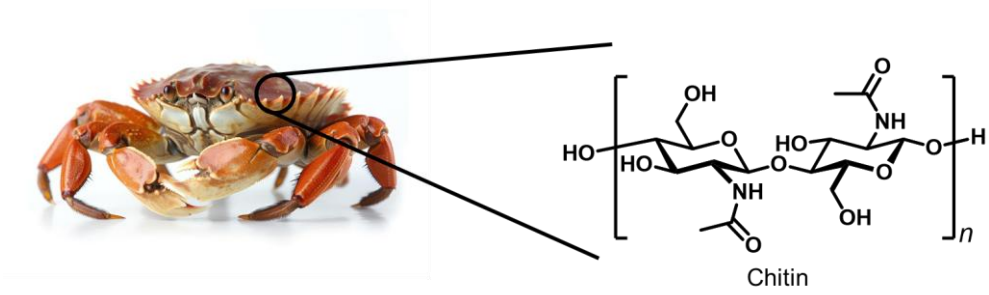

Fig. 1 Molecular structure of Chitin.

Chitosan, a biopolymer derived from chitin (Fig. 1), has emerged as a compelling candidate due to its abundance. The annual volume of biosynthesis amounts to approx. 1000 Gt, of which about 8 Mt p.a. is attributed to crustaceans' shells [6]. We have recently introduced a new type of wood coating based on chitosan itaconate, which uses waste materials from the food industry in combination with itaconic acid [7]. Due to its unsaturated carbon bonds, itaconate can be polymerised in a free radical reaction on wood surfaces to a colourless, water-insoluble protective layer (Fig. 2).

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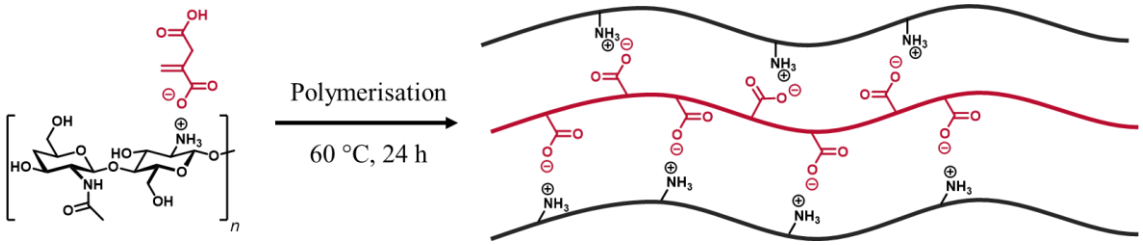

Fig. 2 Polymerisation of chitosan itaconate salt into an insoluble film.

Our research demonstrates that polymerised chitosan itaconate films exhibit inherent fire resistance, remaining intact without igniting or melting when exposed to flames. Furthermore, when applied to wood, the coating significantly enhances fire protection by slowing ignition and preventing sustained burning. These results suggest that chitosan itaconate coatings offer a sustainable, high-performance alternative for fire protection in wood-based applications.

2 METHODOLOGY

The chitosan flakes (200–600 mPa·s, 0.5 % in 0.5% acetic acid at 20 °C) were acquired from TCI Deutschland GmbH and were freeze-dried to a constant weight. To determine the degree of acetylation (DA), elemental analysis was performed using a Vario EL Cube (Elementar Analysensysteme GmbH, Germany), with the DA calculated based on the carbon-to-nitrogen mass ratio following the method of dos Santos et al. [8]. The degree of acetylation was found to be 11.99%.

The chitosan was milled using a Fritsch Pulverisette 14 Premium Line mill, employing a sieve insert (0.2 mm hole size). For solution dispersion, the IKA T 25 digital ULTRA-TURRAX® was utilised.

Aqueous hydrochloric acid (1 mol·L^{-1}) and sodium hydroxide were obtained from Sigma Aldrich. Itaconic acid was sourced from Alfa Aesar. Potassium peroxodisulphate (PPS) was acquired from Thermo Scientific. The wood samples (beech and spruce), which were planned and cut, were purchased from Rocholl GmbH.

A 2.5 wt.% chitosan itaconate solution was prepared according to our previous work [7]. Therefore 32.5 g (250.0 mmol) of itaconic acid was dissolved in 500 mL of deionized water. The exact concentration was determined by potentiometric titration with a 0.1 M sodium hydroxide solution. A monosodium itaconate solution was created by adding 125 mmol (5.0 g) of sodium hydroxide and stirring until completely dissolved. Separately, 42.163 g of chitosan was dissolved in 220 mL of 1 M hydrochloric acid and 924 mL of demineralized water. These two solutions were then combined and mixed at 25,000 rpm for 1–2 minutes using the IKA T25 digital ULTRA-TURRAX, resulting in a viscous, colourless solution. Fresh batches were prepared and immediately used for each experiment since chitosan itaconate solutions have a limited shelf life.

For chitosan itaconate film preparation, 5 mol% relative to the chitosan's free amine groups of potassium peroxydisulfate (PPS, 12.0 mmol) were mixed with the 2.5 wt.% chitosan itaconate solution. 500 µL of this solution was placed onto a polyimide-coated petri dish and dried at 60 °C for 24 hours [7].

After incorporating 12.0 mmol of PPS (5 mol% relative to free amine groups) into the chitosan itaconate solution, wood samples (beech or spruce) were coated with a 120 mL·m^{-2} of the resulting solution [7]. It was uniformly applied using a bristle brush. After application, the samples were placed in an oven and dried at 60 °C for a period of 24 hours. They were then conditioned at 23 °C and a 50% relative humidity for 48 hours prior to further analysis.

The fire resistance tests were performed according to the UL 94 HB standard ("ANSI/UL 94," 2018) by Underwriters Laboratories Inc., utilizing a UL94 horizontal and vertical flammability tester TF328 from TESTEX. Wooden test specimens with dimensions of 125 × 13 × 3 mm were uniformly coated with 120 mL·m^{-2} of a chitosan itaconate solution containing 5 mol % PPS relative to the free amine groups in chitosan. The coated samples were then dried vertically at 60 °C in a drying oven to promote polymerization. Afterward, they were conditioned for 48 hours at a temperature of 23 ± 1 °C and a relative humidity of 50 ± 3 % before testing. This process was repeated

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to achieve the desired number of coating layers. The specimens were finally labelled in accordance with the UL 94 HB standard. During the test, the samples were placed horizontally and exposed to a 2 cm blue methane gas flame at the tip for up to 30 seconds, or until the flame reached the 25 mm mark. If the flame reached this point, the time taken was recorded. If the sample continued to burn after the flame source was removed, the duration until the flame either reached the endpoint or self-extinguished was noted. To ensure stable testing conditions, the flammability tester was ventilated for five minutes before each test.

The analysis of combustion residues was carried out using an Agilent 5100 ICP-OES instrument from Agilent Technologies. Chitosan and chitosan itaconate films were incinerated in a muffle furnace at a temperature of 500 °C. In order to prepare the sample, 0.2 g of the resulting ash was combined with about 40 mL of double-distilled water, forming a suspension. The mixture was left to stand for 10 minutes to facilitate phase separation. The supernatant was then extracted with a 25 mL syringe and transferred to a 50 mL centrifuge tube using a 0.45 µm PES filter. For each sample, 1 mL of a 65 % nitric acid solution was added to 50 mL of double-distilled water. For the blank sample, only double-distilled water was used, which was then acidified with the 65% nitric acid solution.

3 RESULTS

The fire-resistance of chitosan itaconate coatings was assessed on six wooden specimens following the UL 94 HB standard. Uncoated samples of the same dimensions served as controls. According to the UL 94 HB standard, the burning rate must not exceed 40 mm·min^{-1}, or the fire must self-extinguish before reaching the 100 mm mark.

Tab. 1 Results of the UL 94 HB fire test of untreated and coated test specimens.

	Flaming time, in s	Burning rate, in mm·min^{-1}	Burned, in %
Uncoated	13	78	100
Chitosan itaconate coated	60	0	0

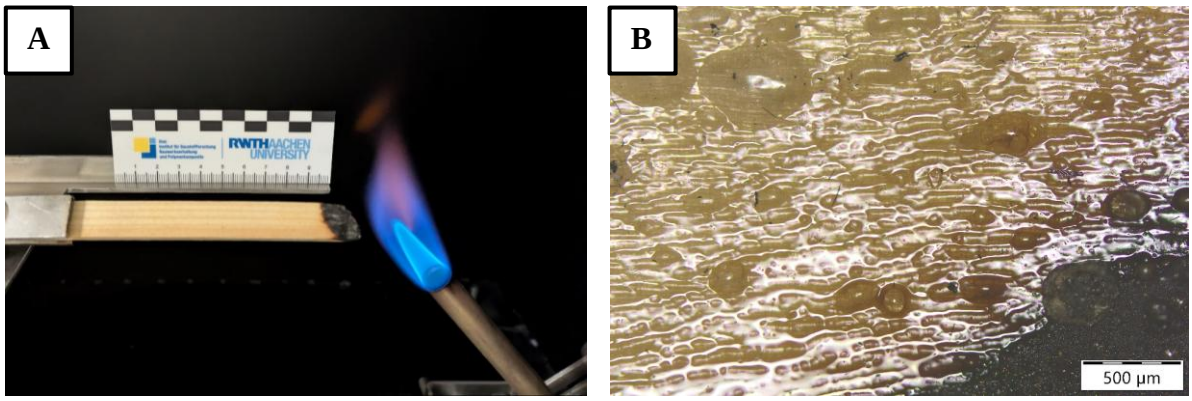

Fig. 3 UL-94 fire testing of coated wood specimen (A) and microscopy image of partially burned chitosan itaconate coating (B).

The polymerised chitosan itaconate film was burned and the ashes were subject to inductively coupled plasma optical emission spectrometry (ICP-OES) in order to analyse the inorganic components in the stating material and the film (Tab. 2).

Tab. 2 Results of the ICP-OES measurement of incinerated pure chitosan and chitosan itaconate film.

	Concentration, in µg·L^{-1}									
	B	**S**	**Mg**	**Ca**	**Sr**	**Ba**	**Cr**	**Fe**	**Cd**	**Hg**
Chitosan	8	2742	4582	2180	20	2	36	14	0	0
Chitosan itaconate	41	2856	6	21	19	2	35	4	0	0

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4 DISCUSSION

Untreated wood samples did not fulfil the UL 94 HB standard. Both beech and spruce burned entirely, though ignition was slightly faster for spruce, whereas beech burned at a significantly higher rate. The application of chitosan itaconate notably decreased the number of samples that were completely burned. Beech and spruce samples, each coated with chitosan itaconate, were exposed to a direct flame for 60 seconds (Fig. 3A). In both cases, combustion was limited to the area in direct contact with the methane flame and the fire extinguished immediately after the flame source was removed (Tab. 1). The edges and smaller end surfaces appeared to be particularly vulnerable. Chitosan itaconate enabled a complete self-extinguishing of all samples. This protective effect was also evident in increased ignition times and reduced burning rates.

Microscopic analysis of coated spruce samples after the fire test revealed a transparent, slightly reflective layer in undamaged areas (Fig. 3B). Heavily burned sections showed that the coating foamed upon exposure to heat, forming charred bubbles. When these bubbles burst, the underlying wood surface became exposed. However, in the observed cases, these newly revealed areas remained unaffected by the fire, suggesting that the coating had partially penetrated the wood. This fact indicates that the coating formed a slightly intumescent layer, which shielded the wood from atmospheric oxygen.

Compared to other biopolymer-based fire retardants [3], [4], [5], [9], [10], the chitosan itaconate coating offers distinct advantages. Previous studies have demonstrated that chitosan-based fire retardants can improve flame resistance but often relied partially on petrochemicals, making them not entirely bio-based [9]. Additionally, their production processes frequently involve complex solvent-based methods, limiting their sustainability [10]. Many existing chitosan-based fire retardants are also partially or completely water-soluble, restricting their durability. In contrast, the chitosan itaconate system used in this study forms a water-insoluble, well-anchored protective layer, providing long-lasting fire resistance.

As marine organisms, crustaceans are susceptible to the accumulation of heavy metals that could end up in chitosan samples [11]. Therefore, the chitosan and the chitosan films were burned completely and investigated in the ICP-OES. The ashes show a significant reduction in the magnesium and calcium content of the films. Such deviations can, however, be explained by matrix effects. It has previously been reported that easily ionisable elements such as sodium and potassium can impair the determination of magnesium and calcium in ICP-OES [12]. Since the present film contains high levels of sodium and potassium, the matrix effect is the most likely cause for the striking reduction in calcium and magnesium.

The increase in the boron content is unusual and may have been introduced by itaconic acid or the initiator. The elevated levels of sulphur could indicate that traces of protein are still present in the sample (Tab. 2). Since the chitosan used was originally extracted from crab shells, the organism had apparently absorbed the chromium from the seawater. Other heavy metals such as Ag, As, Co, Cu, Mn, Mo, Ni, Pb, U, V, or Zn (not shown in Tab. 2) were not found in the samples.

5 CONCLUSION

The conducted flammability tests demonstrated the effectiveness of chitosan itaconate coatings in enhancing the fire resistance of wood.

- Significant reduction in burning rate.
- Coated wood samples achieved complete self-extinguishing properties.
- Fire propagation was only observed at edges and smaller surfaces, likely due to coating distribution inconsistencies from vertical drying.
- The coating formed charred bubbles upon exposure to heat, creating a barrier that limited oxygen access to the wood surface.
- Completely bio-based approach.
- No toxic heavy metals remain as residues after combustion.

Potential applications include fire-resistant coatings for wooden components such as beams, panels, and flooring, enhancing safety in residential and commercial buildings. It could also be used in the furniture industry to produce fire-resistant wooden products that are both aesthetically pleasing and meet environmentally friendly standards.

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Additionally, chitosan itaconate can be integrated into environmentally conscious construction projects by providing fire-resistant coatings for green building applications. These findings highlight chitosan itaconate as a promising bio-based flame retardant for wood applications, offering enhanced fire resistance combining waste reduction and circularity.

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