Novel zinc carbonate synthesis using olive wood ash extract: a sustainable approach for enhancing cotton flame retardancy

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ABSTRACT – REZUMAT

Novel zinc carbonate synthesis using olive wood ash extract: a sustainable approach for enhancing cotton flame retardancy

This study presents a novel, sustainable, and cost-effective method for synthesising zinc carbonate ($ZnCO_3$) via the precipitation of zinc acetate using an alkaline extract derived from olive wood charcoal ash, which stabilises at approximately pH 13.0. The formation of the desired ($ZnCO_3$) flame retardant was confirmed using XRD and Fourier-transform infrared (FTIR) spectroscopy.
The synthesised ($ZnCO_3$) was applied to cotton fabric via a pad-dry-cure process at two concentrations (0.5 wt. % and 1.4 wt. %), resulting in final add-on percentages of 6.0% ± 0.3% and 8.0% ± 0.4%. Flammability tests demonstrated significant fire resistance improvement: the burning rate of the fabric was reduced by up to 63%, decreasing from 0.62 cm/s (untreated) to 0.23 cm/s for the 8.0% treated sample, increasing total burning time from 48 s to 126 s. Mechanical characterisation revealed that the 8.0% treated fabric exhibited an enhanced tensile strength, increasing by an average of over 22% without significant fabric shrinkage. These findings highlight olive charcoal ash as a promising, sustainable, zero-cost alkali source and ($ZnCO_3$) as an effective, eco-friendly flame-retardant coating for reinforced cotton textiles.

Keywords: *zinc carbonate, sustainable synthesis, ash water, flame-retardant cotton*

O nouă metodă de sinteză a carbonatului de zinc cu ajutorul extractului de cenușă din lemn de măslin: o abordare sustenabilă pentru îmbunătățirea proprietăților ignifuge ale bumbacului

Acest studiu prezintă o metodă inovatoare, sustenabilă și rentabilă de sintetizare a carbonatului de zinc ($ZnCO_3$) prin precipitarea acetatului de zinc cu ajutorul unui extract alcalin obținut din cenușa de lemn de măslin, care se stabilizează la o valoare a pH-ului de aproximativ 13,0. Formarea substanței ignifuge dorite ($ZnCO_3$) a fost confirmată prin spectroscopie XRD și spectroscopie în infraroșu cu transformată Fourier (FTIR).
Carbonatul de zinc ($ZnCO_3$) sintetizat a fost aplicat pe țesătura de bumbac printr-un proces de fulardare-uscare-condensare la două concentrații (0,5% în greutate și 1,4% în greutate), rezultând procente finale de adăugare de 6,0% ± 0,3% și 8,0% ± 0,4%. Testele de inflamabilitate au demonstrat o îmbunătățire semnificativă a rezistenței la foc: viteza de ardere a țesăturii a fost redusă cu până la 63%, scăzând de la 0,62 cm/s (netratată) la 0,23 cm/s pentru proba tratată cu 8,0%, ceea ce a dus la creșterea timpului total de ardere de la 48 s la 126 s. Caracterizarea mecanică a relevat că țesătura tratată cu 8,0% a prezentat o rezistență la tracțiune îmbunătățită, crescând în medie cu peste 22%, fără o contracție semnificativă a țesăturii. Aceste rezultate evidențiază cenușa de lemn de măslin ca o sursă de alcalii promițătoare, durabilă și fără costuri, precum și ($ZnCO_3$) ca un strat ignifug eficient și ecologic pentru țesăturile din bumbac consolidate.

Cuvinte-cheie: *carbonat de zinc, sinteză durabilă, apă de cenușă, bumbac ignifug*

INTRODUCTION

Textiles with multifunctional properties, especially flame resistance, play a crucial role in safety-oriented applications such as protective workwear, medical fabrics, and home furnishings, where reducing flammability can help prevent injuries and property damage [1–8]. The textile industry has increasingly shifted toward safer and more sustainable alternatives to traditional flame retardants, with carbonate-based compounds, such as zinc carbonate ($ZnCO_3$), emerging as promising eco-friendly options. This compound functions by releasing non-flammable gases, such as carbon dioxide (CO_2), upon heating, which dilutes flammable gases and suppresses combustion [9]. Additionally, they promote char formation, creating a protective barrier that limits heat and oxygen transfer to the material. Known for their low toxicity, environmental compatibility, and cost-effectiveness, carbonate-based flame retardants offer a significant advantage over conventional halogenated systems, making them a viable solution for sustainable textile applications.

The use of Zinc Carbonate $ZnCO_3$ specifically its basic form, hydrozincite, as a textile flame retardant has been explored extensively through conventional chemical precipitation methods [9, 10]. These established routes typically involve dissolving a soluble zinc precursor (like zinc acetate or zinc sulfate) in water and introducing a commercially sourced alkaline agent, such as Sodium Hydroxide (NaOH) or Potassium Carbonate (K_2CO_3), under controlled pH and temperature conditions to induce the precipitation of $ZnCO_3$ [11, 12]. This chemical approach yields nanoparticles with good control over morphology and size, enabling uniform application to fabrics [13]. Studies have demonstrated that $ZnCO_3$ synthesised this way provides effective flame inhibition via its endothermic decomposition, leading to reduced heat release and enhanced char formation [14, 15].

However, the economic feasibility and environmental profile of these methods are often hampered by the reliance on high-purity, expensive commercial chemical reagents, which directly contributes to the overall material cost and chemical waste burden of the finishing process.

Consequently, contemporary research has decisively shifted toward developing non-toxic, eco-friendly alternatives. This strategic shift emphasises the valorisation of waste materials and the use of sustainable inorganic compounds. Research into environmentally friendly bio-based flame retardants has attracted considerable interest to address these environmental safety issues, such as using pumpkin extract (Cucurbita maxima) [16], Banana (Musa Cavendish) Pseudostem Sap extract [17], spinach leaf juice [18], and chicken eggshell powder [19]. Harnessing agricultural waste as a chemical precursor represents a vital strategy in this context, directly supporting the transition to a sustainable circular economy, which aims to convert liabilities (waste) into high-value assets.

Olive wood charcoal ash (OWCA) is a sustainable by-product of charcoal combustion which contains alkaline compounds such as calcite and calcium oxide (CaO), along with trace minerals like potassium, magnesium, and phosphorus. These components make OWCA valuable for soil enhancement, pest control, and composting in agriculture [20]. Moreover, ash water has been employed in green synthesis methods, such as the production of zinc oxide (ZnO) catalysts [21].

The choice to utilise olive wood charcoal ash extract instead of conventional alkaline reagents (like NaOH or K_2CO_3) is primarily justified by principles of sustainability and long-term economic advantage, despite the initial requirement for filtering and leaching [22]. This method employs a zero-cost agricultural waste product as the primary chemical precursor, which strategically eliminates the need to purchase the most expensive component of the synthesis: high-purity commercial alkaline chemicals. This waste valorisation not only drastically lowers the material cost footprint, making the $ZnCO_3$ synthesis more competitive and scalable, but also minimises environmental pollution by converting a high-volume waste liability into a valuable resource, aligning the entire process with the fundamental goals of the circular economy and Green Chemistry.

The critical research gap addressed by this study is the development of a fully circular and cost-effective synthesis route. This work is the first to utilise Olive Wood Charcoal Ash Extract, a zero-cost agricultural waste, as a sustainable and eco-friendly alkali source with the aim of applying it as a flame retardant coating for cotton cloth. This novel approach demonstrates a superior, economical, and environmentally sustainable pathway to provide a sustainable, cost-effective solution for enhancing the safety and functionality of textiles, while promoting the use of biomass-derived materials in advanced applications.

METHODS AND MATERIALS

Preparation of OWCA water extract and characterisation

Olive Wood Charcoal is commonly used in rural Tunisian families for cooking. The ash of this Charcoal was collected for this study and was mixed with distilled water at varying concentrations (10%, 20%, and 40% w/v) to prepare the ash water extract. Each mixture was stirred continuously for 100 minutes at room temperature, 40 and 70°C to facilitate the dissolution of soluble minerals. The solutions were filtered by centrifugation at 3500 rpm for 5 minutes, followed by filtration through (Whatman No.1) filter paper to remove impurities. The pH of each extract was measured using a calibrated pH meter (S-610H), and the extracts were stored in sealed containers at room temperature for further use. This approach was intentionally chosen to demonstrate a sustainable, waste-to-value synthesis route. While requiring initial steps for extraction and filtration, the use of this zero-cost agricultural waste eliminates the need for expensive, commercially purchased alkaline reagents (such as NaOH), thereby enhancing the environmental and economic viability of the overall finishing process.

Synthesis of zinc carbonate

Zinc carbonate was synthesised via a precipitation method using zinc acetate dehydrate and water extracts of olive wood ash. In a typical procedure, 0.1 g of zinc acetate dihydrate was dissolved in 200 mL of olive wood ash water extract (prepared at a concentration of 10% w/v). Immediately after the addition of the zinc acetate solution, the reaction started, and the solution became white and cloudy. The reaction continued for 2 h, and then the suspension produced was kept still overnight to precipitate. After the reaction, the resultant precipitate was allowed to settle for 48 hours at room temperature. The precipitate was then separated from the reaction solution by centrifugation at 4000 rpm for 10 minutes and repeatedly washed with deionised water to

remove any soluble impurities. Finally, the purified product was dried in an oven at 80°C for 24 hours to yield zinc carbonate as a fine powder (figure 1).

All chemicals, including zinc acetate dihydrate (99.99%) and non-toxic polyacrylate-based blinder solution with 20% solid content (acrylamide/Acrylic Acid copolymer), were sourced from ALDRICH and used as received.

The alkaline agent for the precipitation was derived from the olive charcoal ash extract (providing OH^- and CO_3^{2-} ions). The synthesis involves the precipitation of the basic form of zinc carbonate, Hydrozincite, rather than pure $ZnCO_3$, which is typical under the high pH conditions established by the extract. The balanced chemical reaction for the primary synthesis is as follows:

$$5Zn^{2+} + 2CO_3^{2-} + 6OH^- \xrightarrow{\text{Precipitation}} Zn_5(CO3)_2(OH)_6$$

Textile cotton fabric modification

A 100% cotton fabric with a plain weave structure and density of (118 g/m^2) cut along the chain direction has been used for the entire study.

The treatment bath for fabric impregnation consisted of 10 mL of water and 20 mL of acrylic binder. The cotton fabric was padded with the prepared solution using double impregnation method to apply the prepared solution, followed by double squeezing with a lab padder at a fixed rate. The fabric was then dried in an oven at 80°C for 3 min (figure 2). To quantify the effective mass absorption after coating, the percentage of addition was calculated using equation 1.

$$Add\text{-}on\ (\%) = \frac{W_{after\ treatment} - W_{before\ treatment}}{W_{before\ treatment}} \times 100 \quad (1)$$

The amount of liquid solution retained by the fabric was controlled and quantified using the Wet Pickup (WPU) measurement. *WPU* was determined by weighing three replicate samples ($n = 3$) before (dry) and immediately after (wet) passing them through the padder, using the following equation:

$$WPU\ (\%) = \frac{W_{wet} - W_{dry}}{W_{dry}} \times 100 \quad (2)$$

The padder speed and pressure were carefully calibrated to maintain consistency across all treatment groups (both $ZnCO_3$ concentrations and both additive levels). Across all samples, the coating process achieved a mean WPU of 57% ± 1%, demonstrating the high reproducibility of the liquid transfer step.

Characterization techniques

X-ray diffraction (XRD) spectra were obtained using an X'Pert Pro Panalytical X-ray diffractometer and a D8 ADVANCE system from Bruker, USA. The diffractograms were processed with X'Pert HighScore Plus software and were performed with a resolution of 0.02° and 30 scans. The functional groups of the samples were identified using the ATR-FTIR spectroscopy technique within the range of 400–4000 cm^{-1}.

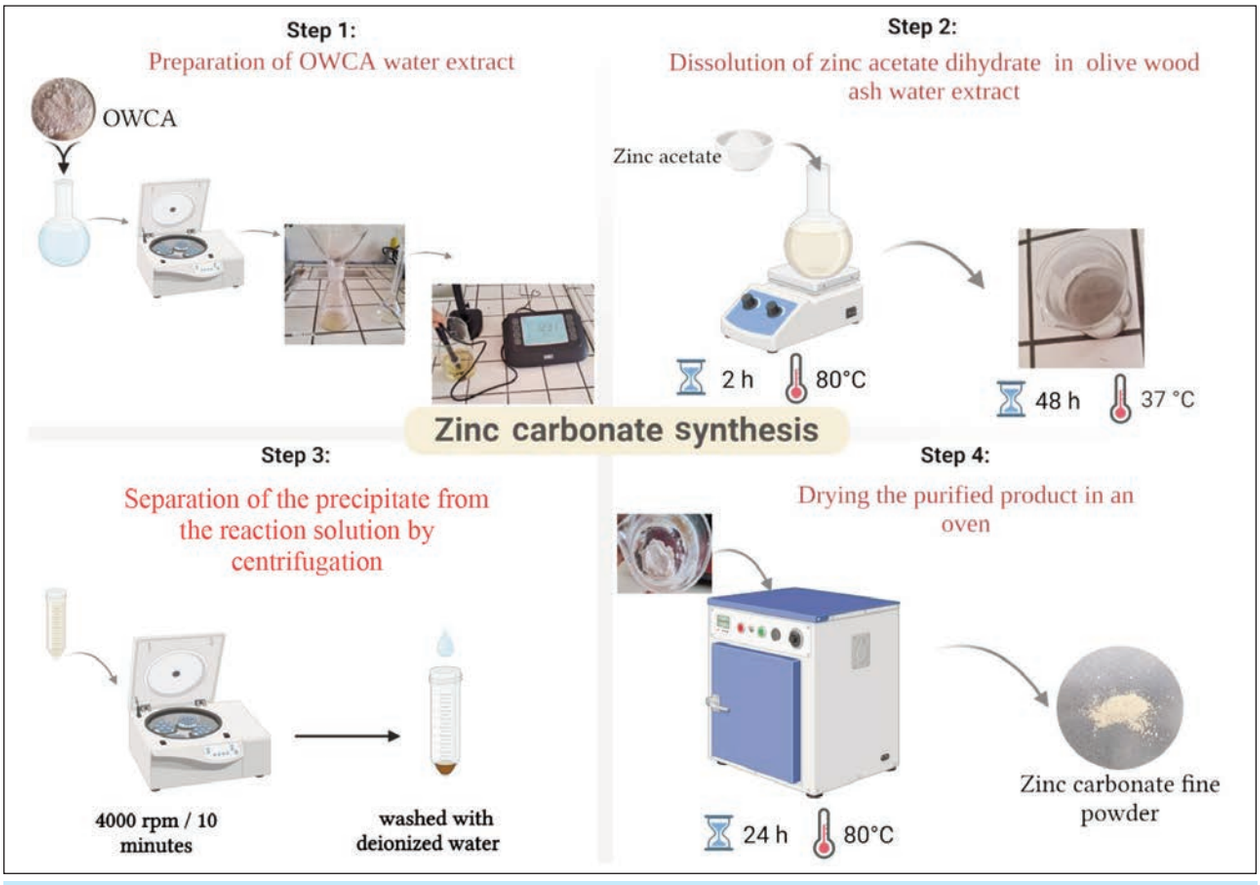


Fig. 1. Illustration of the green process of the synthesis of zinc carbonate

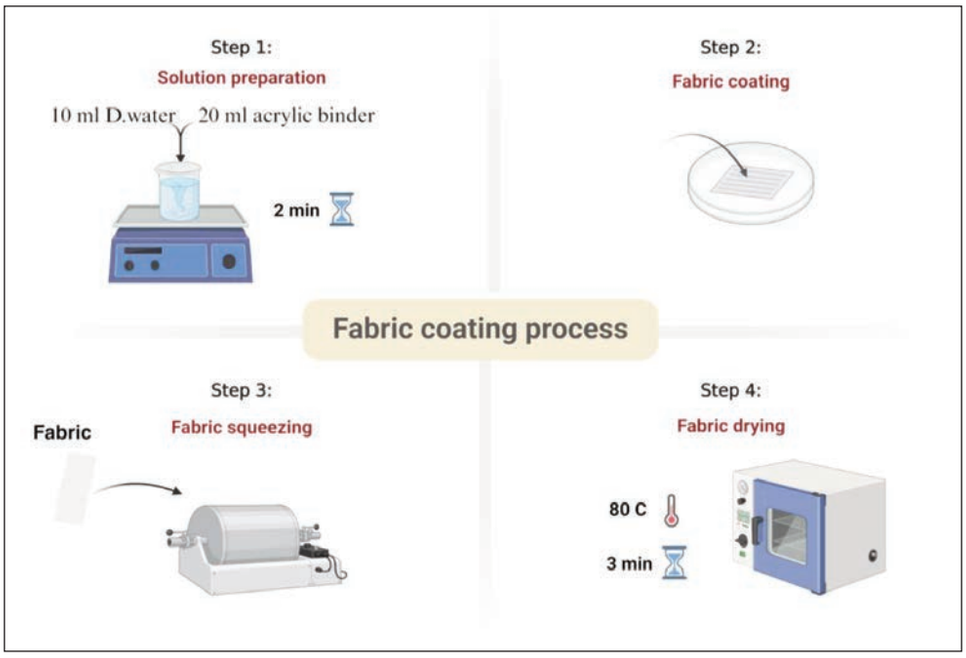


Fig. 2. Fabric coating process

Scanning Electron Microscopy (SEM) images of the selected samples were captured. The flame retardancy of untreated and treated fabrics was evaluated in accordance with ASTM D6413 (Standard Test Method for Flame Resistance of Textiles, Vertical Test). Fabric samples (300 mm × 75 mm) were vertically mounted and exposed to a flame with a calibrated length of 38 mm for 12 seconds to measure ignition resistance and burning behaviour. Five replicate samples ($n = 5$) were tested for each treatment condition. In parallel, the tensile strength and elongation of both control and treated fabrics were assessed following ASTM D5034 (Standard Test Method for Breaking Strength and Elongation of Textile Fabrics, Grab Test), using a universal testing machine to apply uniaxial force until rupture. Five replicate strips ($n = 5$) were tested in both the warp and weft directions for each sample.

RESULTS AND DISCUSSION

XRD

The X-ray diffraction patterns of zinc carbonate, untreated cotton, and cotton treated with zinc carbonate are shown in figure 3. The resulting diffraction pattern corresponds reasonably well to that of Zinc carbonate in its basic form (zinc carbonate hydroxide or hydrozincite [14–15]. The XRD analysis revealed characteristic reflection peaks at (200), (201), (310), (020), (311), (401), (510), (112), (330), (512), (332), and (313), which are consistent with the monoclinic crystal structure of hydrozincite (reference pattern: JC-PDF: 00-019-1458) [14]. Untreated cotton shows two dominant peaks around 2θ = 15.3, 17° and 23.4° and 32.4°, which correspond to crystalline planes of cellulose in cotton fabric [15]. Upon treatment with zinc carbonate, the treated cotton retains the characteristic cellulose peaks, suggesting that the fundamental crystalline structure of the cotton fibres remains intact after treatment. However, the treated cotton pattern also exhibits additional peaks at 2θ = 21.4° and 22.6°, which correspond to the reflections of zinc carbonate, confirming its successful incorporation onto the textile surface. The presence of these $ZnCO_3$ peaks, albeit with lower intensity compared to the pure sample, indicates that zinc carbonate is present in a dispersed form rather than large, well-ordered crystals.

FTIR

The FTIR spectra of untreated cotton, Zinc carbonate and Zinc carbonate-treated cotton (figure 4) provide valuable insights into their chemical compositions

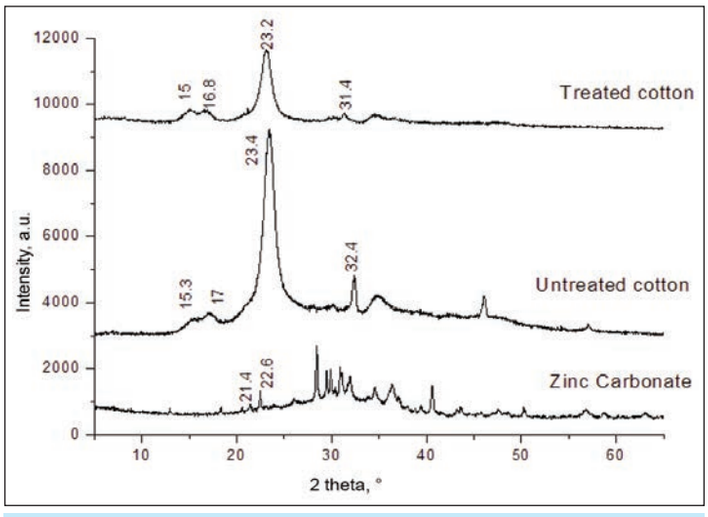


Fig. 3. XRD of zinc carbonate, untreated and treated cotton textile

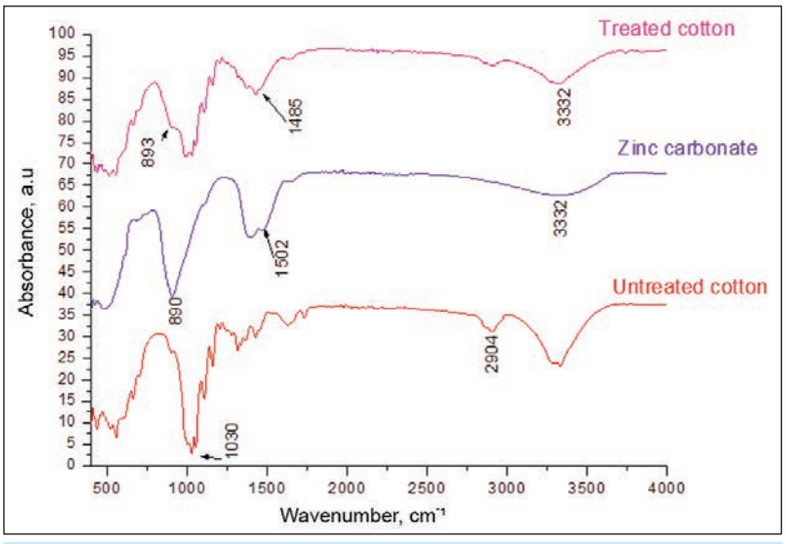


Fig. 4. FTIR Analysis of untreated cotton (red), zinc carbonate (blue) and zinc carbonate-treated cotton (purple)

and interactions. The spectrum of zinc carbonate exhibits characteristic peaks associated with carbonate functional groups, including a strong asymmetric stretching band at 1502 cm^{-1} and an out-of-plane bending mode at 889 cm^{-1}, confirming the presence of CO_3^{2-} ions. Additionally, a broad absorption band around 3332 cm^{-1} corresponds to O-H stretching, indicating surface hydroxyl groups or water molecules in the structure. In contrast, untreated cotton shows a distinct cellulose fingerprint, with a broad O-H stretching peak around 3300–3400 cm^{-1}, highlighting its hydrophilic nature, and a C-H stretching band near 2900 cm^{-1} from aliphatic groups in the cellulose backbone. The characteristic C-O-C and C-O stretching vibrations between 1050 cm^{-1} and 1160 cm^{-1} represent the glycosidic bonds within cellulose, while a peak at 1635 cm^{-1} is attributed to bound water molecules [13].

Upon modification with zinc carbonate, the treated cotton spectrum reveals a combination of both materials, confirming successful deposition. The carbonate peaks at ~870 cm^{-1} and 1430 cm^{-1}, which are prominent in pure zinc carbonate, appear clearly in the treated cotton, indicating its presence on the textile surface. Meanwhile, the cellulose peaks, including C-H (~2904 cm^{-1}) and C-O (1030 cm^{-1}) stretching, remain visible, suggesting that the fundamental structure of cotton is preserved. However, slight shifts in peak positions and intensity variations suggest possible interactions between zinc carbonate and the cotton groups**,** likely through hydrogen bonding or surface attachment mechanisms**.**

SEM

As shown in figure 5, the treated cotton fibres appear as elongated, cylindrical structures, with the zinc carbonate deposits visible as irregularly shaped clusters adhering to their surfaces. It is observed that some agglomeration of the zinc carbonate particles has occurred, leading to a somewhat uneven distribution across the fibre surfaces. This phenomenon can likely be attributed to the high concentration of the precursor solution, which increased the frequency of particle-particle collisions during the deposition process. While this agglomeration represents a degree of unevenness, the coating is still sufficiently distributed to impart a significant flame-retardant effect. This successful surface modification acts as a protective barrier, as evidenced by the substantial improvement in flame resistance observed in our combustion tests. Therefore, despite the visible particle aggregation, the coating effectively inhibits flame spread and provides the intended protection to the textile.

Mechanical strength of the treated fabric

The mechanical properties of the fabric were evaluated by measuring the tensile strength in both the warp and weft directions, with the full data presented in table 1. The untreated cotton fabric showed base tensile strengths of 189 N (warp) and 168 (weft). Upon treatment with 1.4 additive levels ($ZnCO_3$), the tensile strength increased significantly in both axes, reaching 235 N in the warp direction and 205 N in the weft direction. This represents an average increase of 24.3%in the warp and 22.0% in the weft direction. This finding aligns with the results reported by Sharma et al., who observed a 12 % increase in tensile strength upon treatment of cotton with zinc carbonate nanoparticles [13]. The observed increase is attributed to the reinforcing effect of the nanoparticles and the presence of the polymeric binder, which enhances fibre-to-fibre bonding and load distribution within the textile structure, thereby confirming that the zinc carbonate treatment did not negatively impact the fabric's intrinsic strength. It is also important to note that no significant fabric shrinkage was observed through macroscopic inspection following the pad-dry-cure process. Therefore, the observed increase in tensile strength is attributed to the reinforcing action of the $ZnCO_3$ nanoparticles and the polymeric binder promoting enhanced load distribution and fibre-to-fibre adhesion, rather than an artefact of dimensional stabilisation.

Table 1

MEAN TENSILE STRENGTH (N) AND PERCENTAGE INCREASE IN WARP AND WEFT DIRECTIONS

Sample	Direction	Tensile Strength (N)	Tensile increase (%)
Untreated	Warp	189 ± 5	N/A
Untreated	Weft	168 ± 3	N/A
Treated	Warp	235 ± 4	24.3
Treated	Weft	205 ± 5	22

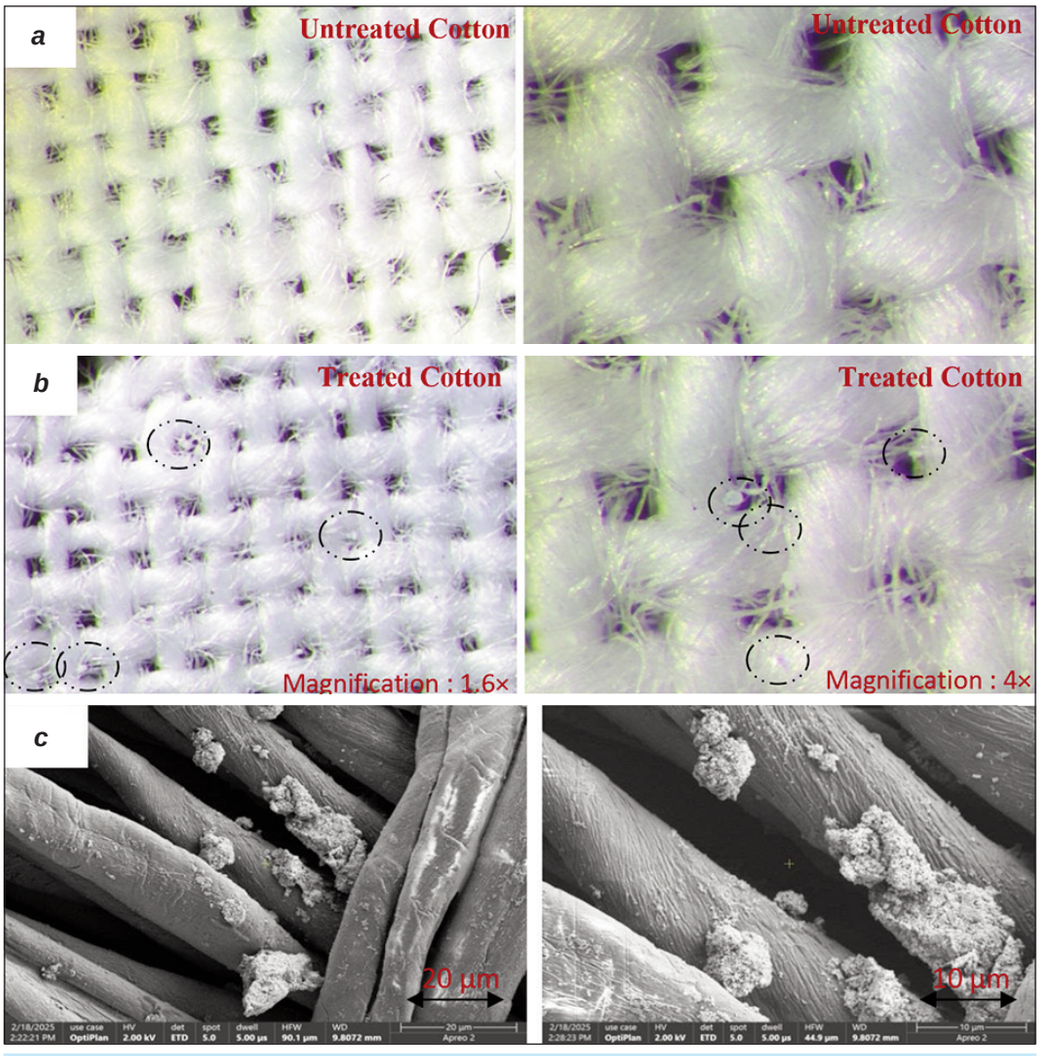


Fig. 5. Microscopic analysis comparing untreated and treated cotton fabric: *a* – optical micrograph of untreated fabric; *b* – optical micrograph of treated fabric; *c* – SEM images of treated fabric

Flammability analysis of the treated fabric

Cotton fabric was treated with synthesised zinc carbonate. Subsequently, the treated fabric and control fabric were evaluated for fire retardant properties.

It was observed that the untreated cotton fabric burned quickly, with flashes and flames, within a minute. After it finished burning, a light, fragile, whitish ash remained. However, while the control fabric exhibited an afterglow after burning, the zinc carbonate-treated cotton fabric showed no such afterglow. The key results of this study are summarised in the following table. Burning rates were calculated by dividing the sample length (30 cm) by the burning time.

The fire-retardant mechanism of zinc carbonate ($ZnCO_3$) is based on various synergistic effects. When exposed to heat, $ZnCO_3$ decomposes endothermically according to the following reaction:

$$ZnCO_3 \longrightarrow ZnO + CO_2$$

As shown in studies involving thermal decomposition, transformation of basic zinc carbonates to ZnO begins at relatively low temperatures (noticeable decomposition at 150–200 °C) and leads to ZnO as a solid product [15]. Then, the formed ZnO in the condensed phase enhances substrate protection by

Table 2

ADD-ON PERCENTAGE AND VERTICAL FLAMMABILITY PERFORMANCE OF UNTREATED AND $ZnCO_3$ TREATED COTTON FABRIC

Sample	Additive (%)	Add-On (%)	Burning time (s)	Burning rate (cm/s)
A	untreated	-	48 ± 1.0	0.62 ± 0.05
B	0.5	6 ± 0.3	81 ± 3.5	0.37 ± 0.02
C	1.4	8 ± 0.4	126 ± 4.2	0.23 ± 0.01

favouring char formation, which is denser and more cohesive, acting as a thermal shell providing a heat barrier and mass transfer inhibitor. Experimentally, $ZnCO_3$ treatments on cotton have shown a significant improvement in LOI and high char mass retention (LOI value of 30 and a specific char length of 40 mm with more than 40% char mass retention at 600°C) [13].

Lastly, the effects can be seen in the more general terms of metallic additives, with many publications noting that metal oxides (including ZnO) encourage catalytic carbonisation and formation of a dense char layer that significantly decreases the polymer area exposed to air, thereby improving condensed-phase protection and smoke removal [23].

This study demonstrates that the incorporation of zinc carbonate is an effective flame retardant for cotton fabric. At a 1.4% additive level, our treated fabric showed a significant increase in burning time of 162.5% and a decrease in burning rate of 63% compared to the untreated cotton. To contextualise these results, we compared our findings with similar studies in the literature. Sharma et al. synthesised zinc carbonate nanoneedles using a conventional direct precipitation method with zinc sulfate and sodium carbonate [13]. Their study reported a burning rate of 0.05 cm/s, but this was achieved with a significantly higher additive level of 4% under alkaline conditions. Although their burning rate was lower, our findings are highly competitive, achieving a comparable flame-retardant effect with a much lower additive level (1.4%), which is beneficial for maintaining fabric properties and reducing cost. Conversely, our results showed superior performance compared to the work of Tseghai et al., who utilised chicken eggshell powder as an additive. Their fabric, with a 0.3% additive level, had a burning rate of 1.4 cm/s, which is significantly higher than the 0.23 cm/s achieved in our study [19]. These comparisons highlight the effectiveness of our green-synthesised zinc carbonate in imparting a strong flame-retardant property to cotton fabrics, while also showcasing the advantages of our eco-friendly approach. Notably, our study demonstrates that our green synthesis of zinc carbonate, applied via the dip-coating method, achieves optimal flame retardancy with a relatively low additive level of 1.4%. This constitutes a significant contribution to the field. Furthermore, an important practical advantage is the low add-on percentage of our treatment, which is 0.6% for a 0.5% additive level and 8% for a 1.4% additive level, indicating uniform deposition of the $ZnCO_3$/acrylic binder preparation for all replicates. This parameter allows for comparability of flammability results by confirming an identical coating weight was applied to all samples. This is considerably lower than the typical 20–30% required for many commercial flame-retardant treatments [13]. The use of a lower add-on percentage not only reduces costs but also helps to better preserve the natural properties and hand-feel of the cotton fabric.

From a sustainability and economic perspective, the synthesised $ZnCO_3$ finishing offers distinct advantages over conventional systems. The method adheres strongly to the principles of the circular economy by utilising Olive Wood Charcoal Ash extract, a zero-cost agricultural waste, to successfully replace costly, high-purity commercial alkaline precursors (e.g., NaOH) needed for precipitation. This waste valorisation significantly reduces the material cost footprint and the need for fresh raw chemical resources. Furthermore, by providing an effective, non-toxic $ZnCO_3$ coating, the technology offers a viable alternative to widely used toxic or persistent flame retardants (such as halogenated or formaldehyde-based systems), thereby mitigating environmental pollution and health risks.

CONCLUSION

In this study, we successfully developed and validated a novel, sustainable route for synthesising an effective textile flame retardant by using an alkaline extract derived from olive wood charcoal ash. This methodology effectively performs waste valorisation by utilising a zero-cost agricultural byproduct to replace expensive, purchased commercial alkaline reagents, thereby establishing a proof-of-concept for a truly circular economy approach to chemical synthesis. The alkaline extract exhibited pH values near 13 under varying conditions, enabling efficient precipitation of zinc carbonate. XRD analysis confirmed the formation of the basic form of Zinc carbonate, as evidenced by characteristic diffraction peaks corresponding to its crystalline structure. However, the presence of non-indexed peaks suggests compositional complexity, likely arising from residual components in the ash-derived alkaline agent used during synthesis. The resulting $ZnCO_3$ coating offers significant improvements in textile properties: the 8.0% ± 0.4% treated fabric exhibited substantial FR performance, reducing the burning rate by up to 63% and increasing tensile strength by over 22% across both the warp and weft directions, without inducing significant fabric shrinkage. These findings position the $ZnCO_3$ coating as a highly viable, eco-friendly, and economically sound alternative to conventional, resource-intensive FR textile finishes.

However, a primary limitation identified was the durability of the coating to standard laundering. Future research will therefore focus strategically on process optimisation through techniques like sonication to improve particle uniformity, and the investigation of alternative, environmentally benign cross-linking agents to significantly enhance the adhesion and wash durability of the $ZnCO_3$ coating, thereby validating the long-term commercial potential of this sustainable textile finish.

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