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Sustainable fire-retardant coating for Nylon-cotton fabric from wastewater-recycled struvite

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Abstract

Nylon-cotton blend (NYCO) fabric is widely used in military uniforms and work-wear due to its durability and comfort, but it is highly flammable. Since the currently available fire retardant coatings release toxic fumes causing adverse health and environmental effects, there is a pressing need for a bio-safe fire retardant for NYCO. One potential solution is struvite—magnesium ammonium phosphate hexahydrate ($MgNH_4PO_4 \cdot 6H_2O$)—which can be recycled from wastewater. We investigated whether struvite, alone or combined with naturally occurring plant-based compounds, such as tannic acid (TA) and phytic acid (PA), could be developed as a sustainable and bio-safe fire retardant for NYCO. Using a simple dip-coating procedure, we treated the NYCO fabric with TA, PA, and/or struvite. We determined the surface functionalization of the treated NYCO fabrics utilizing gravimetry, Fourier-transform infrared spectroscopy, and scanning electron microscopy. We confirmed results from other studies that showed the fire-retardant properties of PA treatment on NYCO. We then demonstrated that NYCO fabric treated with struvite, alone or combined with TA, exhibited reduced flammability and a lower maximum temperature during burning, as assessed by a modified vertical flame test (as outlined by ASTM D-6413). Our study suggests that wastewater-recycled struvite, alone or combined with TA, may be an effective phosphorus-based bio-safe fire retardant for NYCO. Moreover, using struvite recycled from wastewater has an additional benefit of preventing residual phosphorous from reaching aquatic bodies; which in turn; may mitigate eutrophication.

Keywords

Struvite, Phytic acid, Tannic acid, Fire-retardant fabric, Nylon-cotton blend, NYCO, Recycle, Wastewater, Surface functionalization, Flammable

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Introduction

Nylon-cotton blend (NYCO) fabric is used extensively in military uniforms and work-wear because it combines the durability of nylon and the comfort of cotton (1, Figure 1A). However, NYCO is highly flammable due to the "scaffolding effect," where nylon melts at a lower temperature than the temperature at which cotton ignites, causing the melted nylon to adhere to the cotton and act as fuel for flames, which allows fire to spread across the fabric (2). Consequently, it becomes necessary to develop flame retardants specific to NYCO. Because the current fire retardants for NYCO release toxic fumes, such as halide gasses and formaldehyde, causing adverse health and environmental effects, there is a pressing need for a bio-safe fire retardant for NYCO (3).

Tannic acid (TA) is a naturally occurring plant-based polyphenol that has been investigated as a bio-safe fire retardant; it is thought that TA functionalizes fabrics such as cotton and nylon by forming hydrogen bonds via its multiple hydroxyl groups (4, Figure 1B). Phytic acid (PA) is another naturally occurring plant-based compound; it contains multiple phosphate groups, and the phosphorylation of cotton imparts fire retardancy (5, Figure 1B). Kulkarni et al. found that TA and PA could be combined with nylon and cotton fibers through hydrogen bonding and phosphorylation, respectively, to achieve flame retardancy in NYCO blends (6, Figure 1B).

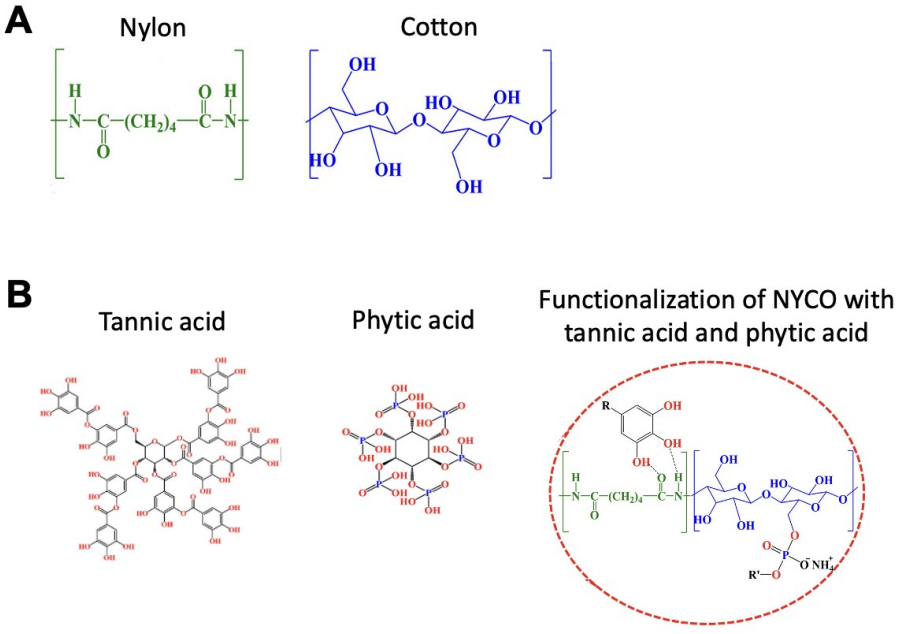

Figure 1. **A.** Chemical formula of nylon (left) and cotton (right), modified from Kulkarni, et al. (6). **B.** Chemical formula of tannic acid (left), phytic acid (middle), and functionalization of NYCO with tannic acid and phytic acid (right), modified from Kulkarni, et al. (6).

In addition to the impetus for developing less hazardous chemicals as fire retardants, there has been a greater interest in sustainably sourced fire retardants (7). One promising sustainable fire retardant candidate is struvite, magnesium ammonium phosphate hexahydrate ($MgNH4PO4 \cdot 6H2O$) that can be recovered from wastewater (8, Figure 2A). With its high phosphorus and nitrogen content, struvite has been developed as a slow-releasing fertilizer (9). Additionally, struvite, in its powdered form or as a component of hydrogels, has also been explored as a potential fire-extinguishing agent (10, 11). Chandrasekaran et al. showed that the economic valuation of wastewater-derived struvite as a fire retardant is higher than its use as a fertilizer (12, Figure 2B). Besides addressing the need for sustainable fire-retardant chemicals, the production of struvite from wastewater prevents phosphorus- and nitrogen-containing wastewater from reaching aquatic bodies. Such accumulation of N and P can result in a decrease in oxygen in bodies of water—a process known as eutrophication—and cause significant environmental harm (13).

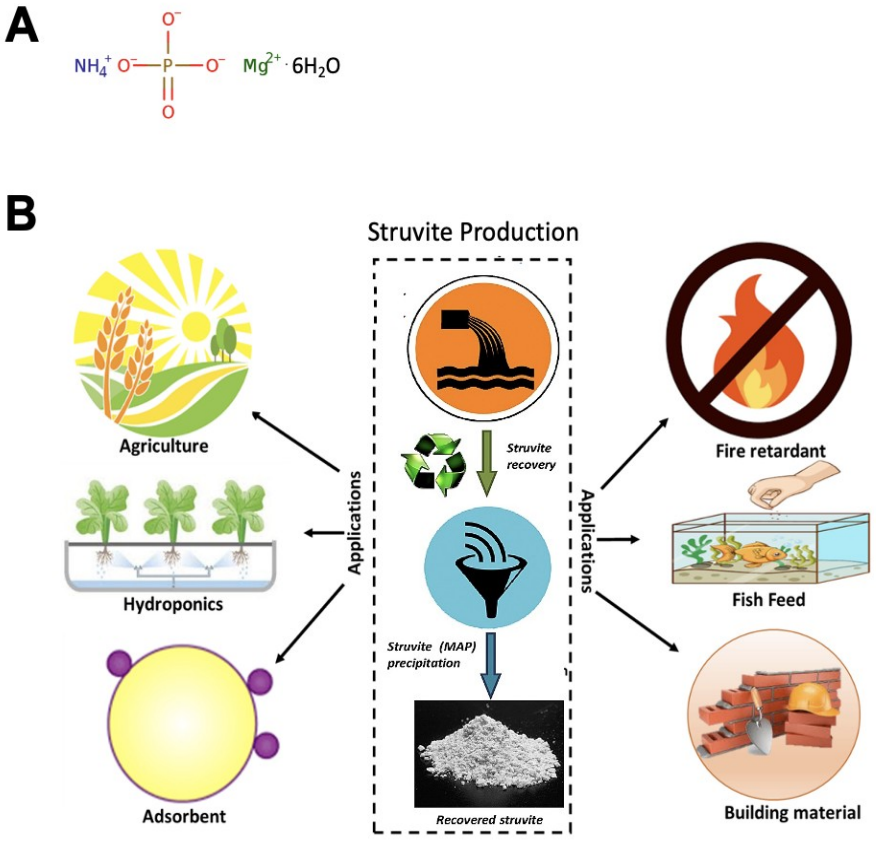

Figure 2. A. Chemical formula of struvite. **B.** Production of struvite from wastewater and its use, modified from Yetilmezsoy, et al., 2020.

Importantly, struvite has not yet been evaluated as a fire-retardant coating for fabrics such as cotton or nylon. We hypothesized that wastewater-recycled struvite may affect flame suppression in the NYCO fabric by phosphorylating the hydroxyl groups in the cellulose that constitutes cotton fibers, similar to the action of PA, another phosphorus-rich compound. In this study, we investigated whether struvite, by itself or in combination with TA, could be developed as a sustainable and bio-safe fire retardant for NYCO fabric.

Materials

Ripstop weave NYCO (50-50 weight %) was obtained from Allender and Company, Inc. (Ferndale, MI, USA). Struvite recovered from domestic wastewater in the form of Crystal Green® (99.6% purity) was sourced from Ostara Nutrient Recovery Technologies (St. Louis, MO, USA) and was finely crushed before use. Tannic acid (TA) (powdered, ACS Reagent Grade) and acetic acid (glacial, Reagent Plus Grade) were obtained from Sigma-Aldrich (St. Louis, MO, USA). Phytic acid (PA) (50 weight % in H_2O, ACS Grade) was obtained from Tokyo Chemical Industry (Tokyo, Japan); according to the Certificate of Analysis, the phosphorus content was between 13.5 to 14.6 %. Deionized (DI) water at a conductivity of 0.2 μS/cm was produced with MilliQ Lab Water System from Millipore Sigma (Burlington, MA).

Methods

Pretreatment of NYCO

Samples were weighed using a Mettler-Toledo XS105 Scale with a sensitivity, d = ± 0.01 mg (Columbus, OH). The NYCO fabric was cut into 3”x10” strips. These fabric swatches were washed by heating in DI water at 60 °C for 1 hour. The fabric swatches were then placed in a hot-air oven (Sheldon Laboratory Vacuum Oven, Model #1465, Cornelius, OR) at 80 °C for 8 hours to dry, after which the initial weight (IW) was measured.

Surface functionalization of NYCO with TA

A PASPORT pH Sensor from PASCO Scientific (Roseville, CA), calibrated with 4.01, 7.00, and 10.01 pH buffer solutions from ThermoScientific (Waltham, MA) was used for pH measurement. A 1.25% w/v solution of TA in DI water was prepared (1.25 g TA per 100 mL DI water) in a quantity sufficient to treat 2.5 g fabric with 100 mL TA solution. The pH of the TA solution was adjusted to 3.0 by adding acetic acid (1 M). Fabric swatches were boiled (in a 2.5 g fabric to 100 mL TA solution ratio) in the TA solution at 90 °C for 1 hour, followed by drying the fabric swatch samples in a hot-air oven at 80 °C for 8 hours. After drying, the final weight (FW) was immediately measured.

Surface functionalization of NYCO with PA

PA is supplied as a solution of 50% w/v H_2O. A solution of PA was prepared at 37.5% v/v (37.5 mL PA per 100 mL DI water) in a quantity sufficient to treat 90 g fabric with 100 mL PA solution. Urea was dissolved into the PA solution at 40% w/v (40 g urea per 100 mL PA solution). Fabric swatches were dipped in this solution at room temperature for 10 minutes, then dried at room temperature for 30 minutes. These PA/urea exposed swatches were then cured by air-drying in an oven at 155 °C for 45 minutes. These cured swatch samples were washed in DI water at 60 °C for 20 minutes with constant stirring, removing all visible excess of

solid material. The samples were then dried in a hot-air oven at 80 °C for 8 hours, then immediately weighed to record the final weight (FW).

Surface functionalization of NYCO with struvite

A 37.5% w/v solution of struvite in water was prepared (37.5 g struvite per 100 mL DI water) and urea was dissolved in this solution at 40% w/v (40 g urea per 100 mL DA water). Then we added 5.5% w/v (~2 mL) acetic acid (1 M) and the mixture was heated in a microwave oven for 30 seconds to enable the struvite to dissolve completely. Fabric swatches were dipped in this solution at 50% w/v (50 g fabric per 100 mL of struvite solution) at room temperature for 10 minutes, then dried at room temperature for 30 minutes. The air-dried swatches were then cured in an oven at 155 °C for 45 minutes. The cured samples were washed in DI water at 60 °C for 20 minutes with constant stirring, removing all visible excess of solid material. The samples were then air dried in a hot-air oven at 80 °C for 8 hours, then immediately weighed to record the final weight (FW).

Scanning Electron Microscopy (SEM)

Fabric samples were gold sputtered in a Desk V Sample Preparation System from Denton Vacuum (Moorstown, NJ). Morphological characterization of treated and dried NYCO samples was performed using a FEI Quanta 200 3D SEM from FEI Company (Hillsboro, OR).

Fourier Transform Infrared (FTIR) spectroscopy

Surface characterization of treated and dried NYCO samples was performed using a Perkin Elmer Spectrum 400 FTIR spectrometer equipped with a germanium crystal in the attenuated total reflectance (ATR) mode. Washed and dried fabric samples did not undergo any additional pre-treatment for the FTIR and were pressed onto the germanium crystal using a built-in pressure arm. Samples were scanned over the range of 2000 to 700 cm^{-1}. Each spectrum was obtained with 16 scans at a resolution of 4 cm^{-1}.

Modified vertical flame test

The flammability of the treated fabrics was evaluated by a vertical flame test as outlined by the ASTM D6413-15 test method using a custom-built vertical flame test setup inside the fume hood (Figure 3A). Briefly, the fabric was placed onto the testing setup while ensuring that the bottom of the fabric was ¾" from the top of a butane torch. The butane torch was then ignited to a flame length of 5". The lit torch was then placed directly below the fabric. The timer and the video recording was started as soon as the flame made contact with the fabric. After 12 seconds, the butane torch was removed from the set up and the burning behavior of the fabric was observed. During the vertical flame test, the temperature of the fabric was continuously measured with a ThermoPro TP 30 infrared thermometer with an appropriately calibrated emissivity setting (Toronto, Canada). The emissivity of cotton and nylon is typically ~ 0.88 (14).

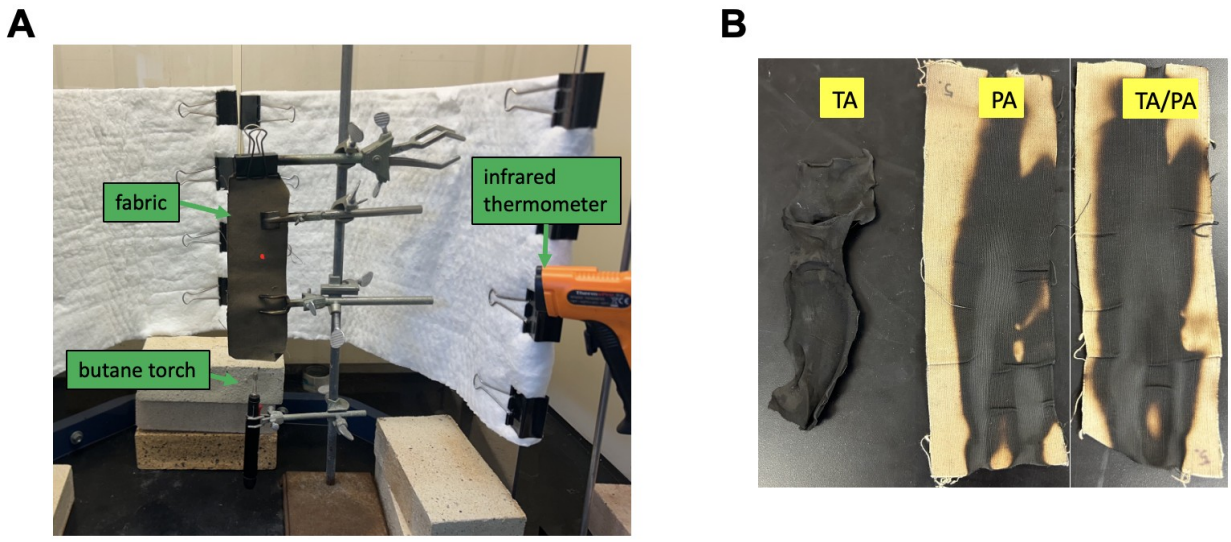

Figure 3. A. Vertical flame test set up. A butane torch is the flame source and is placed just below the fabric. An infrared thermometer is pointing to the fabric to monitor the temperature continuously (a red dot). **B.** Images of NYCO fabric samples subject to the vertical flame test. TA (left) PA (middle) TA/PA (right).

Data analysis

The weight gain after functionalization and the mole ratios were calculated using equations 1 and 2 respectively. In these equations, IW = initial weight and FW = final weight. Molar mass of phytic acid = 660 g.mol^{-1}, molar mass of tannic acid = 1701 g.mol^{-1}, molar mass of struvite = 245 g.mol^{-1}, molar mass of NYCO = 194 g.mol^{-1} (~ as the average of molar mass of cellulose monomer = 162 g.mol^{-1} and molar mass of nylon66 monomer = 226 g.mol^{-1}).

$$\textit{Weight gain after functionalization}(\%) = \frac{FW - IW}{IW} \times 100 \qquad \text{(eq.1)}$$

$$\textit{Mole ratio NYCO monomer to functionalizing agent} = \frac{\textit{Molar mass functionaliing agent}}{\textit{Molar mass NYCO monomer}} \times \frac{IW}{FW - IW} \qquad \text{(eq.2)}$$

Results

Confirmation of the fire retardancy of NYCO by TA and PA treatment

We first replicated the findings of the study by Kulkarni et al. (6), which showed that a combination of TA and PA treatment affected flame retardancy to NYCO. In our study, the NYCO fabric treated with TA and/or PA gained weight after the treatment, indicative of; but not confirmatory of; successful functionalization (Table 1). We calculated the mole ratios of the NYCO to the functionalizing agents based on the weight gain. The calculation yielded ~ 372 NYCO monomers per molecule of TA and 42 NYCO monomers per molecule of PA (Table 1).

Table 1. Weight gain of TA and/or PA treated NYCO fabrics (mean ± SD, n = 6).

Functionalizing agent	Weight gain (%)	Mole ratio of NYCO monomer to functionalizing agent
TA	2.82 ± 1.21	372.4 ± 186.3
PA	8.08 ± 0.57	42.3 ± 2.8
TA/PA	10.01 ± 2.26	N/A

We then subjected PA or TA/PA-treated NYCO to the vertical flame test. The Control NYCO fabric burnt away completely with a negligible amount of fine ash (photo not shown). The TA treated NYCO fabric burnt away completely but left behind a thick char (Figure 3B). In contrast, PA or TA/PA-treated NYCO fabric only partially burnt away (Figure 3B). This result shows that TA and PA treatment conferred fire retardancy to NYCO fabric, with PA appearing to be a more effective fire retardant, based on a visual unburnt area qualitative examination of the burnt fabric.

Functionalization of NYCO by TA and struvite treatment

Next, we investigated the fire-retardant properties of struvite-treated or TA/struvite-treated NYCO fabric. The NYCO fabric treated with TA and/or struvite gained weight after the treatment; again being indicative, but not confirmatory of successful functionalization (Table 2). We calculated the mole ratios of the NYCO to the functionalizing agents based on the weight gain. The calculation yielded ~ 317 NYCO monomers per molecule of TA and 1.6 NYCO monomers per molecule of struvite (Table 2).

Table 2. Weight gain of TA and/or struvite treated NYCO fabrics (mean ± SD, n = 3).

Functionalizing agent	Weight gain (%)	Mole ratio of NYCO monomer to functionalizing agent
TA	2.78 ± 0.16	316.8 ± 17.2
struvite	77.75 ± 6.31	1.62 ± 0.14
TA/struvite	60.42 ± 2.22	N/A

Next, we characterized the surface morphology of the control NYCO and the struvite-treated NYCO using SEM (Figure 4A). No apparent differences between control and struvite-treated NYCO were observed in the SEM images. This finding is in contrast to SEM images presented by Smith et al. which showed that PA/chitosan- and TA/chitosan-treated NYCO had minimal difference compared to untreated NYCO, but PA/ chitosan/ TA/ chitosan-treated NYCO exhibited more material on the surface (Figure 4B) (15).

In the FTIR spectra, the apparent functionalization by TA was manifested as an absorbance peak at ~ 1719 cm^{-1} corresponding to the carbonyl (C=O) stretching of TA. The functionalization by struvite was manifested as a disappearance of 'upward peak' at 1230 cm^{-1}, suggesting an appearance of a downward peak

by struvite functionalization, corresponding to the P=O stretching of the newly phosphorylated cellulose that now constituted the cotton fibers (Figure 4C).

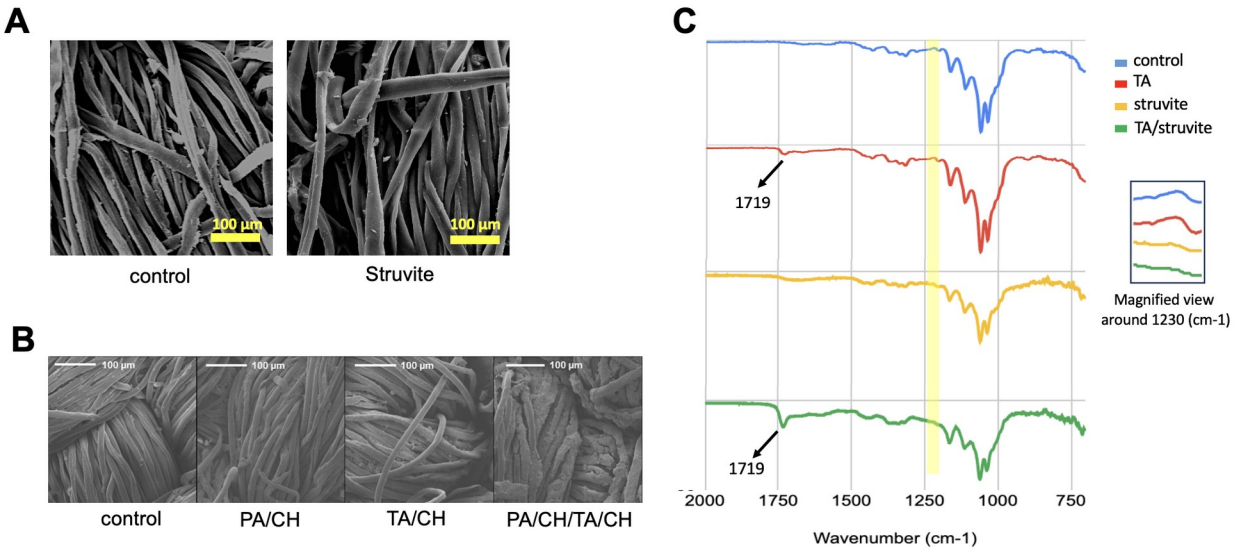

Figure 4. Characterization of struvite-treated NYCO. **A.** SEM images of control NYCO (left) and struvite-treated NYCO (right). **B.** SEM images of control NYCO, PA/chitosan (CH) treated NYCO, TA/CH treated NYCO, and PA/CH/TA/CH treated NYCO (from left to right). Chitosan is another bio-sourced fire retardant. The figure was from Smith et al., 2022. **C**. FTIR spectra of control NYCO and struvite-treated NYCO. The arrow points to a new downward peak at 1719 cm^{-1} indicating TA functionalization. The inset shows a magnified view of the light-yellow shaded area around 1230 cm^{-1} on the FTIR spectra. The disappearance of an upward peak (implying a new downward peak) at 1230 cm^{-1} indicates the phosphorylation of cellulose by struvite.

Investigation of the fire retardancy of NYCO by TA and struvite treatment

We then subjected the struvite-treated and TA/struvite-treated NYCO fabrics to the vertical flame test. We video recorded the burning behavior. The control NYCO caught fire and burned away completely, but the TA/struvite-treated NYCO only charred partially in 60 seconds after the flame source was removed (Figure 5A). We also measured the temperature of NYCO samples over time (Figure 5B). Both the control and TA-treated NYCO samples reached their maximum temperature ~30 seconds after the fabric came into contact with the flame. By 80 seconds, the control NYCO sample, and to a lesser extent TA-treated NYCO sample turned to ash hence the temperature could not be measured reliably beyond that time point. In contrast, the struvite- and TA/struvite-treated NYCO samples took until ~ 70-80 seconds to reach their peak temperatures, demonstrating that the struvite-treatment delayed flame propagation. Additionally, the maximum temperatures during the burning of the struvite- and TA/struvite-treated NYCO fabric were significantly lower compared to the control NYCO fabric (Figure 5C). This finding suggests a reduction in the maximum intensity of flame by struvite

treatment, which is an additional effect to delaying of the flame propagation.

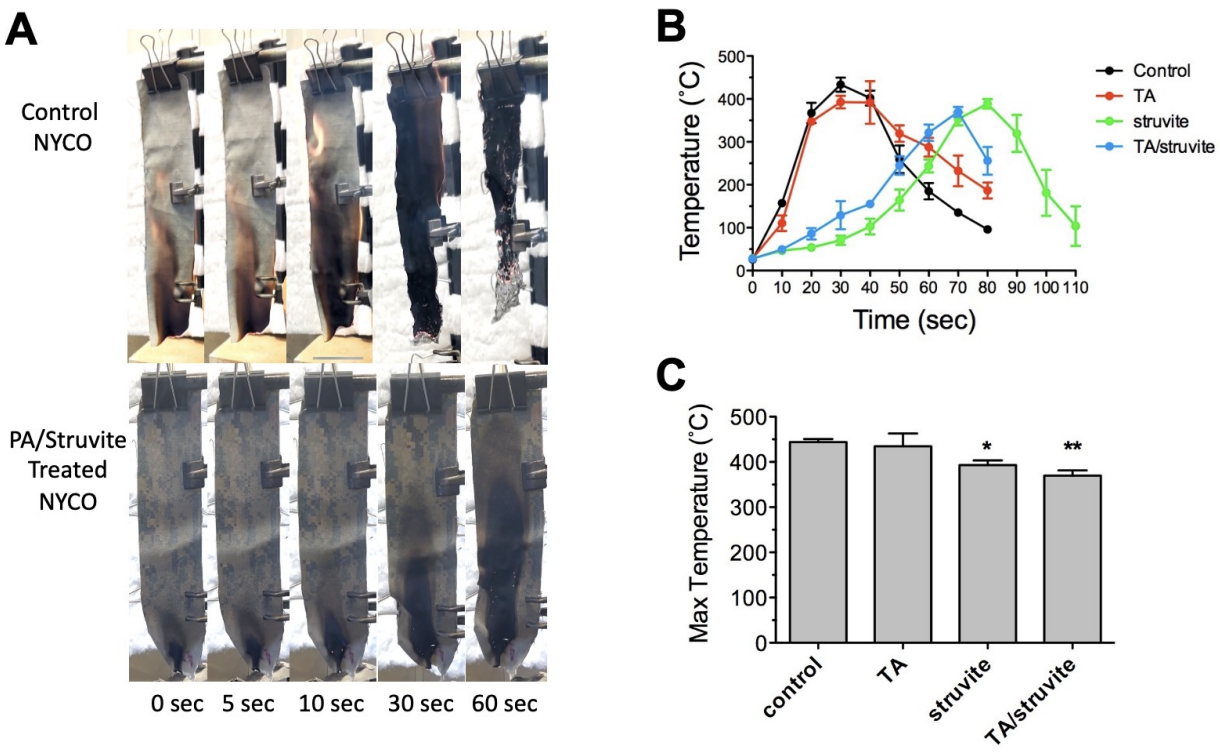

Figure 5. The vertical flame test of struvite-treated NYCO. **A.** Representative images of NYCO fabric samples over 60 seconds after the butane torch was removed. Top row: control NYCO. Bottom row: TA/struvite-treated NYCO. **B.** Time series of the temperatures of fabrics during the vertical flame test. Each data point represents the mean ± standard error (n = 3 for each treatment). **C.** Maximum temperatures recorded during the vertical flame test. Each bar represents the mean ± standard error (n = 3 for each treatment). ANOVA followed by Bonferroni post-hoc pair-wise comparisons. * $P < 0.05$, ** $P < 0.01$.

Discussion

In this study, we demonstrated that treating NYCO fabric with struvite using a dip-and-cure method resulted in weight gain and the appearance of a new peak at 1230 cm^{-1} in the FTIR spectra, indicative; but not confirmatory of successful functionalization of the NYCO fabric with struvite. The NYCO fabric functionalized with struvite, whether used alone or in combination with TA, exhibited a reduced maximum temperature during combustion and exhibited fire-retardant behavior.

A potential chemical mechanism for the fire retardancy of NYCO through struvite modification may involve the phosphorylation of hydroxyl groups in cotton, akin to the effects seen with PA, another phosphorus-rich compound (Figure 6). In numerous studies since the 1950s, including the study of Kulkarni et al., urea was added in the solution containing a

phosphate source such as phytic acid, to phosphorylate cellulose (6, 16, 17). Urea causes the cellulose fibers to swell, allowing phosphorylating agents a better access to the hydroxyl groups on the cellulose (17). In addition, urea can act as an activator to facilitate the reaction between cellulose and the phosphate source (17). In the presence of urea in solution, the phosphate group from phosphorylating agents such as PA and struvite is thought to form a covalent bond with the hydroxyl group on cellulose (P=O). A phosphate group does not bind the carbonyl group on urea, although it may form a hydrogen bond with a positively polarized NH proton (16). The phosphorylation of cellulose by phosphorylating agents such as PA and struvite requires high temperature, which may be affected by curing at 155 °C for 45 minutes (16).

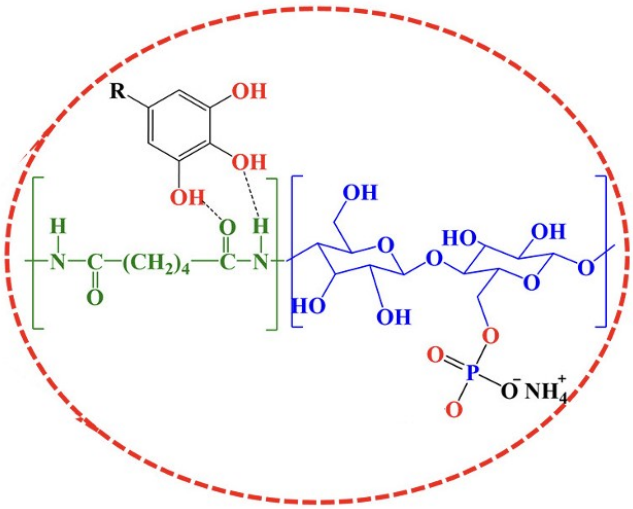

Figure 6. Functionalization of NYCO with tannic acid and struvite. The figure was modified from Kulkarni, et al., 2021.

Additionally, the hydrogen bonding of nylon with TA could contribute to the overall fire-retardant effect of struvite and TA (Figure 6). It is possible that the phosphate groups of PA or struvite can phosphorylate the hydroxyl groups of TA. However, the method of functionalization of NYCO; first with TA and then with PA or struvite sequentially, instead of mixing TA and PA (or struvite) at the same time, likely ensures that PA or struvite will functionalize the hydroxyl groups of cellulose in the absence of the hydroxyl groups of TA. Moreover, Smith et al postulated that additional interaction between PA and TA may stabilize the PA and TA coating of NYCO, contributing to the fire-retardant effect (15).

We observed that the weight gain from the TA treatment was less than 3%, which is less than half of the 6% reported by Kulkarni et al (6). It is possible that the degree of functionalization of NYCO with TA in our experiments was less

effective, indicating a need for adjustments to the treatment conditions. Additionally, the weight gain from the struvite treatment exceeded 70%, which is significantly greater than the typical 8-15% weight gain observed with other successful fire retardants. We suspect that this substantial weight gain may be partly due to the physical deposition of struvite onto the fabric, in addition to the phosphorylation of the cotton fibers by struvite. Despite the weight gain, we did not observe apparent differences between control and struvite-treated NYCO in the SEM images, whereas Smith et al (15) found signs of attached material in the SEM images of PA/TA treated NYCO. It is possible that struvite treatment of the NYCO fabric may have created a uniform film over the fibers that was be too smooth or too transparent to electrons to create a distinguishable texture or contour difference at the resolution of the SEM. For future studies, we plan to optimize the conditions for struvite phosphorylation of cotton by varying different parameters such as pH, urea concentration, curing temperature, and curing duration.

In addition, we calculated the mole ratios of NYCO to functionalizing agents based on the weight gain from functionalization. The results showed more than 300 NYCO monomers per molecule of TA, more than 40 NYCO monomers per molecule of PA, and 1.6 NYCO monomers per molecule of struvite. These mole ratios are largely expected since a PA molecule contains multiple phosphate groups and a TA molecule has multiple hydroxyl groups that can interact with multiple NYCO monomers (although stearic hindrances and geometries still almost completely negate this effect), whereas a molecule of struvite is likely interact with one NYCO monomer at the most. Moreover, the mole ratio suggests that a functionalization of NYCO by struvite may be more efficient than TA or PA, leading to improved fire-retardancy.

The current study did not compare the TA/struvite-treated NYCO fibers with other currently available fire-retardants, such as Roscoflamex, for Synthetic Fibers. Therefore, we do not know whether the TA/struvite coating is a more effective fire-retardant. The publicly available safety data sheet for Roscoflamex for Synthetic Fibers only lists urea as the active ingredient (1.0 - 5.0 %)(18). The curing process of the struvite-treated NYCO fabric with urea in solution can generate urea fumes, which are irritant. However, in our study, we hypothesize – but cannot definitively prove - that after washing and drying of the functionalized NYCO fabric, urea was not a component of the fire retardant. Thus the composition of our fire-retardant may be dis-similar from Roscoflamex for Synthetic Fibers, which lists urea as an ingredient.

Limitations

An important factor in the fire-retardant coating of fabric is the durability of the fire-retardancy from repeated laundering (19), which was not evaluated in our study. Since, we hypothesize that our functionalization created a stable covalent ester bond between the phosphate and cellulose, surfactants in laundry detergents are unlikely to cause dephosphorylation, but at an alkaline pH with almost all currently available detergents that contain enzymes, the hydroxyl anion could be enzymatically facilitated to break a P-O bond to remove phosphate. Kulkarni et al. (6) found that laundering caused a decrease in the fire-retardancy of PA-treated NYCO, even though the phosphate content of the fabric was

retained, indicating that the phosphate groups from PA may remain attached to NYCO (6). It was postulated that the chelation of the salts from water causes the inhibition of the fire-retardant action of PA (6). It would be interesting to investigate whether struvite-treated NYCO demonstrates the similar durability decreases in future studies.

Some fire-retardants work by creating a less air permeable protective barrier, which slows access to oxygen and heat, but this decreased air permeability can reduce comfort (20). Kulkarni et al. noted a decrease in air permeability of NYCO with TA- or PA-treatment (6), but we did not perform the experiments to assess air permeability in our study. Consequently, we do not know what proportion of the fire-retardancy provided by our treatment was due to a decrease in air permeability versus due to chemically altered inhibition.

Our analysis of the burning behavior of treated fabric was also limited to visual observation and temperature measurement during the vertical burning test, since we did not have access to a Differential Scanning Calorimeter (DSC) for a detailed quantitative assessment of thermal processes.

Lastly, our analysis was limited to speculation, rather than confirmation, of functionalization. This was because we did not have access to additional equipment, such as inductively coupled plasma-optical emission spectrometry (ICP-OES) or X-ray photoelectron spectroscopy XPS) for definitive elemental analysis and nuclear magnetic resonance spectroscopy (NMR) for structural confirmation after the treatment with TA and struvite/PA, although we utilized SEM for surface characterization and FTIR for functional group identification.

Conclusions

Our study demonstrated that wastewater-recycled struvite, or magnesium ammonium phosphate hexahydrate, by itself or in combination with TA, is a phosphorus-based bio-safe fire retardant for NYCO, similar to another phosphorus-based fire retardant, PA. Moreover, using struvite recycled from wastewater has an additional benefit of preventing residual phosphorous from reaching aquatic bodies; which in turn; may mitigate eutrophication.

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