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Synergizing mechanical properties and vibrant aesthetics: Nanosilver-treated flax woven fabric reinforcement for polymeric composites

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ABSTRACT

Innovation and research in the field of natural fiber-reinforced composites have garnered significant attention in recent times due to the pursuit of sustainable materials for diverse applications in the automotive, building and construction, marine, defense, and aviation industries. Woven fabric lamination has emerged as a crucial reinforcement technique for fabricating structural composites using thermosetting polymers. Advanced characteristics of nanoparticles renders them instrumental in enhancing the thermomechanical and physical properties of composites. Researches are actively going on to explore alternative approaches to develop AgNPs that are safe, environmentally friendly, and conducive to their integration into composites. In this study, hornbeam leaves (*Carpinus betulus*), abundant in central European countries like Hungary, were employed as a stabilizing and reducing agent to in situ synthesize AgNPs onto flax woven fabrics at varying concentrations. The resulting nanosilver-coated and uncoated fabrics were then laminated with MUF (melamine urea formaldehyde) resin using five layers of flax, thereby producing nanocomposites. Extensive analyses were conducted on the compositted products, encompassing inductively coupled plasma optical emission spectroscopy (iCP-OES), morphological examinations of both fractured and intact samples, assessment of mechanical properties, evaluation of physical characteristics (including water absorption, moisture content, and thickness swelling), thermal stability using TGA (Thermogravimetric analysis)/DTG (Derivative thermogravimetry) study, FTIR (Fourier transform infrared spectroscopy), and colorimetric data. The nanocomposites treated with AgNPs exhibited vibrant colors and uniform dispersion of nanoparticles across the surface compared to the control samples. These results suggest that AgNPs were successfully incorporated into flax-woven fabrics, facilitating the production of sustainable composite production.

Introduction

Recently natural fibers are drawing tremendous attention in composite community to replace traditional synthetic fiber reinforced products in order to attain environmental sustainability feature. The rise in natural fibers consumption in order to produce biocomposite is showing a milestone for the sustainable products to future generations. Statistically, it was reported that almost 43,00 tonnes of natural fiber were used in 2003 by EU countries for composites manufacturing [1], which was then increased dramatically by 2010 (315,00 tonnes) [2] that risen in 2020 by 830,000 tonnes [3]. Like as many other natural fibers [4], flax is one of the most popular and widely considered fiber material in composite science [5,6]. Canada, France, Netherland, and Belgium are the highest producers of flax [7]. Like as other natural fibers, flax is also a lignocellulosic material composed of 62 to 72% cellulose, 18.6 to 20.6% hemicellulose, 2.2% pectin, 2.2% lignin, and 1.7% wax [8]. Mechanically, flax fiber also possess 1057 ± 462 MPa tensile strength, 66.9 ± 16.3 MPa Youngs modulus, 2.2 ± 0.8 failure at strain properties

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[9]. Therefore, flax fibers are getting more attentions to produce sustainable polymeric composites for developing different structural products in aeronautical, defense, packaging, automotive, and so on [5,6]. Flax fibers provide superior performance characteristics when reinforced with various thermoplastic and thermosetting polymers.

Nowadays, NPs are also playing significant roles to functionalize and improve thermomechanical performances of the materials [10–12]. NPs have smaller shape and sizes beside the surface ratio. Moreover, the attractive chemical and physical properties of NPs also attained considerable attentions by the researchers. There are various NPs like TiO_2, rGO (reduced graphene oxide), CNT (carbon nanotube), gold, cupper, and silver (Ag) found significant potential still now [12–16]. The journey of metallic colloids started nearly thousand years ago or more than this. However, nowadays with the expansion of scientific knowledge, new routes are getting explored. AgNP is getting more attention due to the significant performance characteristics (enhanced mechanical, thermal, anti-bacterial, UV protection, and so on). In this regards, different approaches like chemical, photochemical, laser ablation, electrochemical, and biological processes are gaining more popularity [17,18]. Although chemical-based synthesis protocols were getting introduced at the beginning of this century, however, plant-based synthesis methods are displaying more potentiality to meet the environmental standards imposed by different organizations and governments throughout the globe. Different parts of plants like stems, leaves, heartwoods, and flowers are reported by the scientists to develop and synthesize metallic green AgNP [19–22]. Additionally, metallic silver treated textiles could also be laminated with thermosetting and thermoplastic polymers to produce composite/biocomposite materials. This is why an attempt was taken to explore the possibility of in situ biosynthesis of green AgNPs from Hornbeam leaves over flax woven fabrics. The nanosilver coated fabrics were then laminated with MUF resin to produce multilayered composite panels.

Based on our current knowledge, no attempts have been made to utilize in situ biosynthesis for applying a green silver coating over flax woven fabrics using Hornbeam leaves. Furthermore, this current study reports a novel, easy, and cost-effective approach to develop AgNP-treated flax woven fabrics with MUF resin. This has implications for various industries, including automotive, building and construction, marine, defense, and aviation, where the demand for sustainable materials is on the rise. The research showcases a new pathway for sustainable nanocomposite development within the composite community, using flax woven fabrics and MUF resin.

Experimental

Materials and methods

Flax (*Linum usitatissimum*) woven fabric material was purchased from the Málitext located in Pecs, Hungary. The technical parameter of fabric material was 230 (g/m^2) density; twill structure, composed of 100% flax fiber; article number of product: LV 06506. The chemical reagent, $AgNO_3$ having 99.98% purity was bought from Sigma Aldrich Co., (St. Louis, America). The samples for composite manufacturing were prepared through cutting the fabrics 400 × 400 mm^2 before going to biocomposite fabrications, the flax materials were treated with AgNPs. The AgNPs were produced through stabilizing and reducing with Hornbeam (*Carpinus betulus*) leaves collected from the University Garden area (University of Sopron, Sopron, Hungary in summer, 2021). The MUF polymeric resin (Kronores MD MD 2141 J) was purchased from SC Kronospan Sebes SA, Romania.

In situ green synthesis of AgNPs over flax woven fibers

Generally, the in situ synthesis of AgNPs offers the advantage of achieving a uniform dispersion of metallic silver on cellulosic materials. This process is straightforward, cost-effective, and environmentally friendly, requiring minimal effort. Flax fiber is a well-known cellulosic fibers. The hornbeam leaves were washed with clean tap water to eliminate any related dirt and dust from the surfaces, then dried for 15 min at 100 °C. A laboratory grinder was used to crush all the dried leaves in powder form. The crushed powders (50 g hornbeam leaves in 500 mL water, 10% w/v) were boiled at boiling temperature 100 °C for 30 min. The solution (aqueous) was filtered using filter paper and refrigerated in a sealed beaker. The $AgNO_3$ reagent was measured (1.0, 2.0, and 3.0 mM, respectively) (w/v) and placed in different Erlenmeyer flasks. The added $AgNO_3$ solution was shaken continuously to ensure an uniform mixing. The isolated hornbeam solution was added to the flask dropwise and magnetically stirred for 10 min. The pieces of flax woven fabrics were also added to the flask and stirred continuously until the color of the liquid changed from colorless to colored state. The change in color states verify the successful synthesis of green AgNPs. However, the final AgNP coating was kept at 95 °C for 45 min. Initially, silver ions (Ag^+) are uniformly adhered to the surfaces of the flax fabrics. Subsequently, the silver ions undergo reduction to nanosilver with the aid of an aqueous solution of hornbeam leaves, which is applied to the surfaces of the flax fabrics. To eliminate any related nanoseeds on the surfaces, the fabrics were washed and rinsed with normal water. The fabrics were then dried in drier until the moisture content reached 3 to 4%. A schematic representation is shown in Fig. 1.

Lamination of developed composites

The control and AgNP coated fabrics were laminated with MUF polymeric resin through applying hand lay-up method followed by a low pressure (0.5 MPa) in pressing machine. Initially, a wax coated teflon paper sheet was placed over a steel plate where the flax woven fabrics were placed. Five different layers (F,F,F,F,F) of the fabrics were stacked one after another followed by a MUF resin coating. The stacked laminates were pressed by pressing machine and cured at environmental condition (where 65 ± 5% was relative humidity and 20 ± 5 °C was the temperature). The same procedures were followed for all the laminates as mentioned in Table 1. The control laminate was marked by FC1, whereas the AgNP coated samples by FAg2, FAg3, and FAg4 for 1 mM, 2 mM, and 3 mM AgNP treated fabrics. A schematic representation of lamination is shown in Fig. 1.

Characterizations of control and nanocomposite products

The quantitative analysis of greenly synthesized AgNPs on flax reinforcements were evaluated by iCP OES (iCP OES, iCAP^{TM} 600 series, Cambridge, UK) analysis in aqueous form. The morphological investigations of the developed composite products were characterized by SEM equipment (S 3400 N, Hitachi, Tokyo, Japan) at 15.0 kV. The coloration properties were evaluated by a spectrophotometer (Konica Minolta, CM-2600d, Japan) under a standard daylight source (D65) observer within 400 to 700 nm wavelengths. Mechanical properties of the composites/nanocomposites were investigated by Instron testing equipment (4208, USA) for both tensile and flexural properties. The crosshead movement speed was maintained at 10 mm/min for tensile testing whereas the standard adopted was EN310. The standard used for flexural properties was EN310. The moisture contents of the developed materials were checked by European standard (EN 322) after 2 h and 24 h drying at laboratory dryer. There were six test specimens taken under consideration to perform each tests. The bending test samples are shown in Fig. 1. The FTIR and thermal properties were investigated by a spectrophotometer (FT/IR-6300, Jasco, Japan) within 4000 to 500 cm^{-1} range wavenumber and TGA/DTG assessment equipment (Themys thermal analyser, Setaram Instrumentation, France) within 23 to 800 °C, respectively.

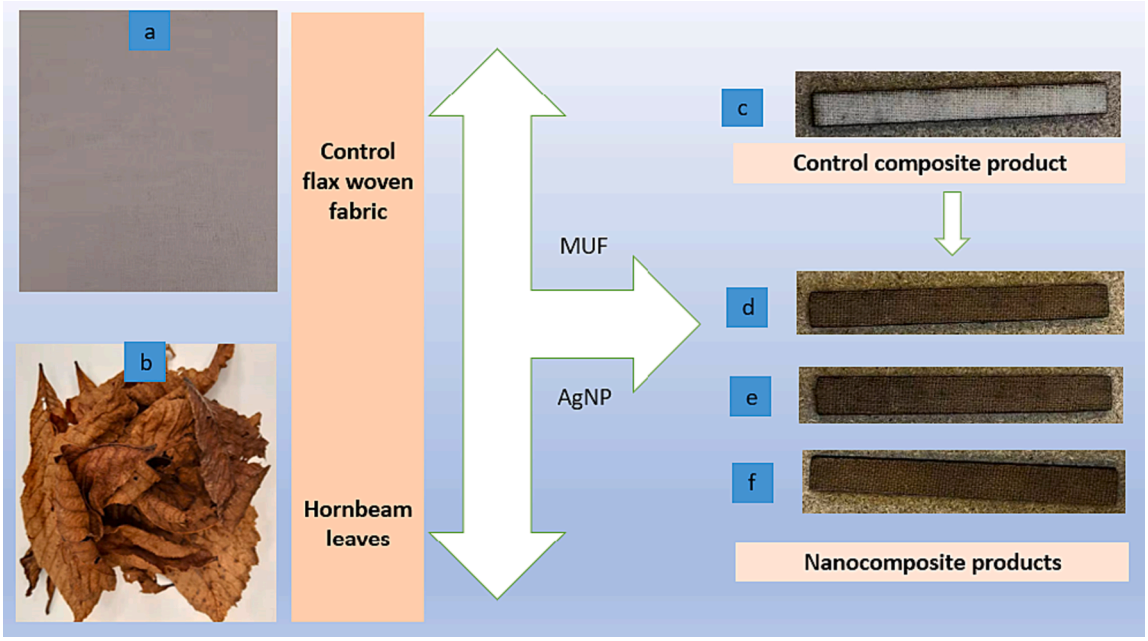

Fig. 1. A schematic representation for AgNP treated flax woven material reinforced MUF composites: (a) Hornbeam leaves; (b) Flax woven shaped material; (c) Control composites bending sample; (d) 1 mM AgNP treated composites bending sample; (c) 2 mM AgNP treated composites bending sample; (c) 3 mM AgNP treated composites bending sample.

Table 1
Different parameters of composites materials (Mean values with the standard deviations in parentheses).

Title of Laminates	Reinforcement characteristics	Fabric stacking sequence	Thickness of laminates (mm)	Density (kg/m^3)
FC1	100% control flax	Fl,Fl,Fl,Fl,Fl	4.7 (0.04)	664.2 (10.7)
FAg2	1 mM $AgNO_3$ treated flax	Fl,Fl,Fl,Fl,Fl	4.2 (0.02)	722.3 (8.00)
FAg3	2 mM $AgNO_3$ treated flax	Fl,Fl,Fl,Fl,Fl	4.9 (0.12)	1018.7 (11.00)
FAg4	3 mM $AgNO_3$ treated flax	Fl,Fl,Fl,Fl,Fl	3.8 (0.01)	736.5 (12.2)

*Fl means Flax woven materials.

Results and discussions

Quantitative investigation of control AgNP treated flax reinforcements

The quantitative value of in situ biosynthesized AgNPs on flax reinforcements in the aqueous solutions were quantified. The tested values of AgNPs were found to have 0, 38, 47, and 53 mg/L concentrations demonstrating an increased value with the increase in silver salt incorporation in the treatment system Fig. 2 . The obtained values also goes in line with some of our previous studies [11,20]. The results explicitly confirm the successful biosynthesis of metallic silver over the flax reinforcements used for laminated composites production.

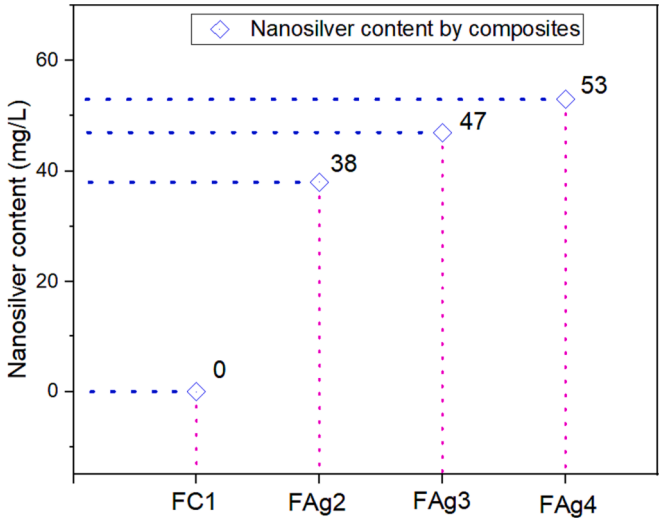

Fig. 2. iCP OES study of control and AgNP treated flax reinforcements.

Morphological studies of control composites and nanocomposites

The SEM photographs of the developed composites were investigated and observed further before and after the fracture (tensile testing). The test specimens of tensile properties are shown in Fig. 3, whereas the first sample (Fig. 3**a**) is belong to the control one and the other samples (Fig. 3 c,e,g) are related to the physical images of AgNP treated composites reinforced with MUF polymer. As SEM images are showing the surface photography of samples, hence the flat, uniform, and smooth surfaces of composites are seen in Fig. 3 d, f, h. However, still there is light presence of flax fibers are observed in SEM samples before the fractures. Additionally, the presence of flax fibers are more explicitly noticed in the fractured surfaces (Fig. 4). It is further noticed that AgNP treated composites are seemingly displaying more fractured surfaces compared to the untreated composites demonstrating a strong bonding is anchored by the NPs between the reinforcements (flax woven fabric) and MUF resin. The similar effects also seen in our previous report for nanosilver treated hemp/glass woven fabric reinforced epoxy composites [11].

Mechanical characteristics of control and nanocomposites

The mechanical properties of the developed products were also

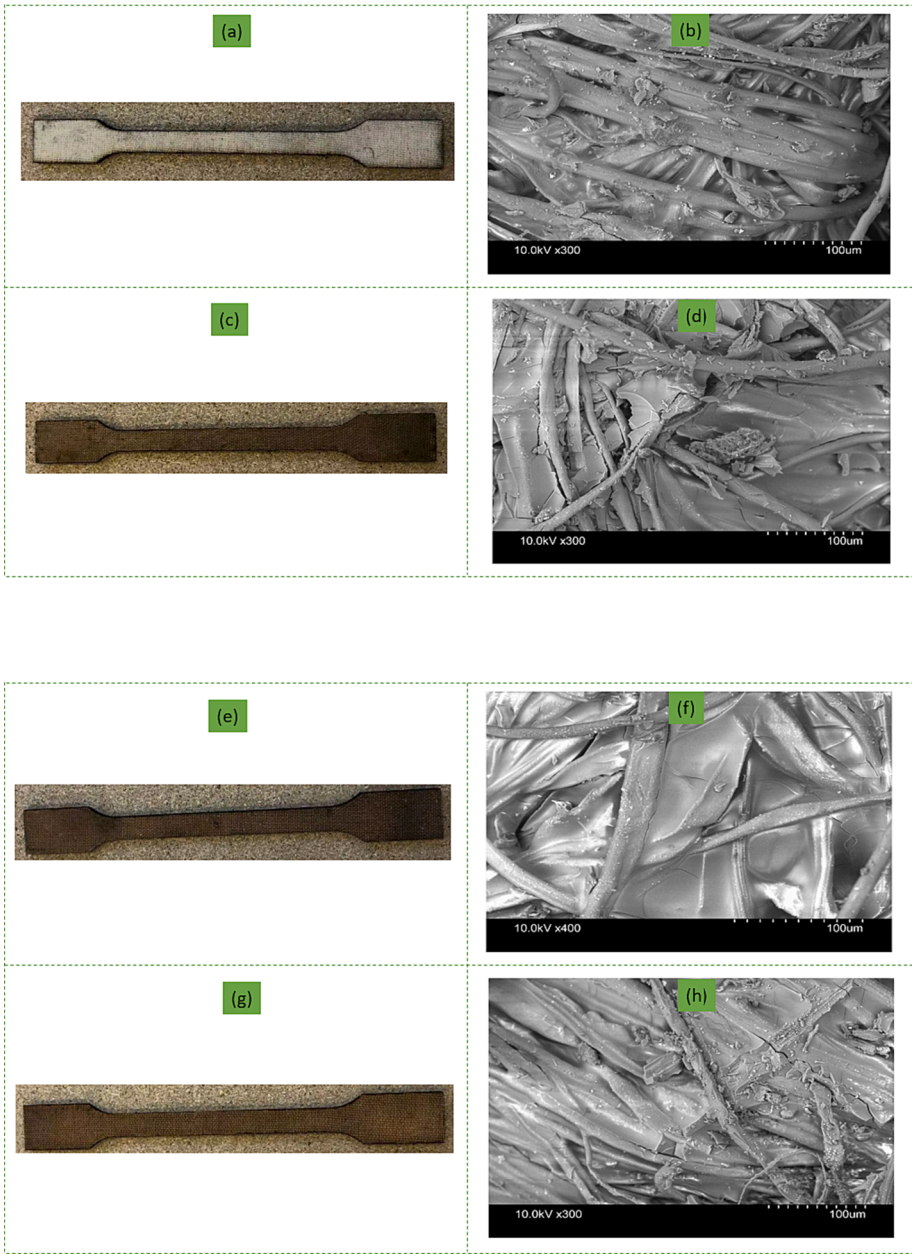

Fig. 3. Physical and SEM photographs of control and AgNP treated flax woven materials reinforced composites: tensile test specimen for FC1 (a), tensile test specimen for FAg2 (c), tensile test specimen for FAg3 (e), and tensile test specimen for FAg4 (g). SEM pictures of FC1 (b), FAg2 (d), FAg3 (f), and FAg4 (h) at 100 μm, respectively.

studied further to understand their performance characteristics in terms of tensile and bending properties Table 1. It is noticed that there is an increasing pattern in tensile strength and modulus found with the increase in nanosilver loading, however it is continued until a certain limit 2 mM. The tensile properties again started to decline after 3 mM loading of $AgNO_3$. Similar effects are also seen in case of bending properties (both for strength and modulus). The tensile strength obtained for untreated composite was 5.8 (0.3) MPa, whereas the 1 and 5 mM silver precursor treated samples displayed 7.5 (0.4) and 8.1 (0.2) MPa strengths. However, 3 mM silver precursor treated samples showed 6.7 (0.7) MPa tensile strengths. It seems that the incorporation of increased nanosilver loading is consequence agglomerations, hence the mechanical properties started to decline again at higher loading of $AgNO_3$. Conversely, in case of flexural strengths 1 and 5 mM silver precursor treated samples provided 12.8 (0.8) and 30.1 (0.1) MPa value; whereas the control and 3 mM precursor treated samples provided 3.7 (0.3) and 15.8 (0.4) MPa value. The Youngs modulus in both the cases also found to have similar trends, whereas control composite shown 1.3 (0.1) GPa value, and FAg2, FAg3, and FAg4 samples provided 1.6 (0.1), 1.9 (0.2), and 1.7 (0.1) GPa values. Conversely, flexural modulus was showing the values by 1.4 (0.1), 1.7 (0.1), 3.3 (0.2), and 2.5 (0.1) GPa, respectively for FC1, FAg2, FAg3, and FAg4 composite samples Table 2. The effects of nanolsilver loading on laminated composites were also further investigated in terms of Coefficient of determinations (R^2) and found all the numerical values are equal or higher than 0.79 signifying a strong relationship between the treatments of flax woven fabrics with AgNPs and associated mechanical properties. The improved pattern in mechanical properties with the increased loading of AgNP was also reported in our previous studies [11].

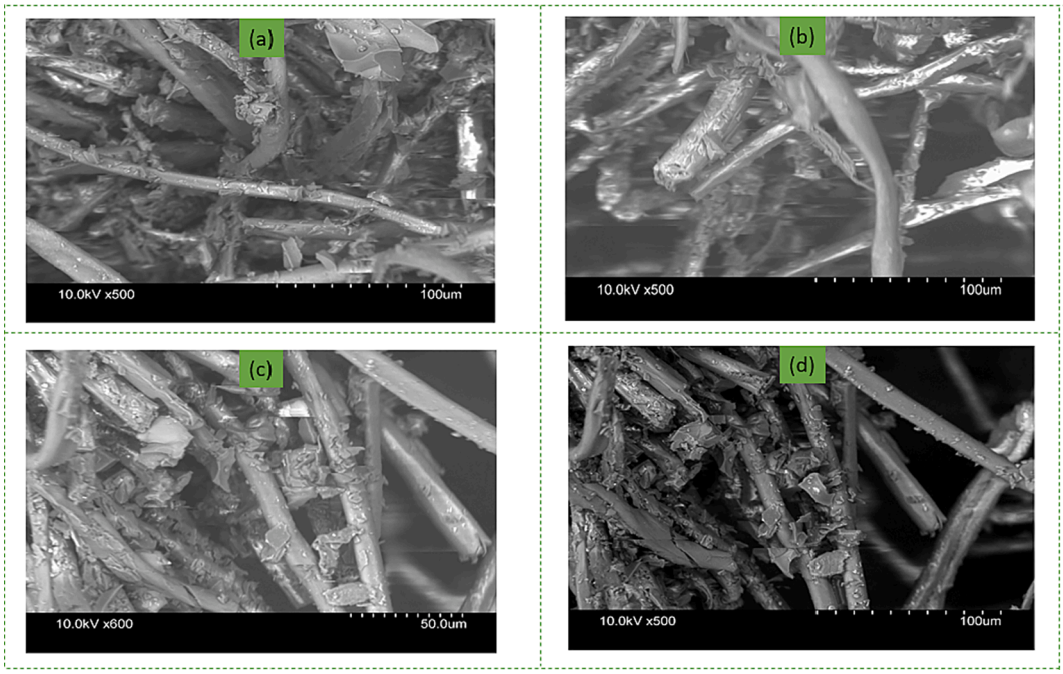

Fig. 4. SEM photographs of control and AgNP treated flax woven materials reinforced composites (fractured surfaces): FC1 (a), FAg2 (b), FAg3 (c), and FAg4 (d) at 100 μm, respectively.

Table 2
Mechanical properties of AgNP-coated flax woven fabric reinforced composites.

Laminated composites	Tensile strength (MPa)	Youngs modulus, E (GPa)	MOR (MPa)	MOE (GPa)
FC1	5.8 (0.3)	1.3 (0.1)	3.7 (0.3)	1.4 (0.1)
FAg2	7.5 (0.4)	1.6 (0.1)	12.8 (0.8)	1.7 (0.1)
FAg3	8.1 (0.2)	1.9 (0.2)	30.1 (0.1)	3.3 (0.2)
FAg4	6.7 (0.7)	1.7 (0.1)	15.8 (0.4)	2.5 (0.1)
Coefficient of determinations (R^2)	0.97	0.79	0.93	0.93

Colorimetric properties investigation of control and nanocomposites

The incorporation of AgNP through in situ protocol also imparts magnificent color appearance [11] to the materials besides providing the adequate strengths. The coloration characteristics of the developed laminated materials also investigated further to assess their colorimetric values within the visible wavelengths. The localized surface plasmon resonance characteristics of the AgNPs are responsible for brilliant coloration effects to the products. The laminated composites even after fabrication displays brilliant coloration effects as shown in Fig. 3 c, e, g compared to the control sample Fig. 3 a. The control sample shown 78.18 L* value, the lightest one compared to the AgNP treated composites (57.72, 50.54, and 32.82, respectively for composite 2, 3, and 4). The results further demonstrate that the higher loading of silver precursor provided more darker color appearances and vice versa. Similarly, color strength (K/S) values also displaying the similar strength followed by L* values (0.19, 0.97, 1.62, and 5.49, respectively for composite 1, 2, 3, and 4). Furthermore, a* and b* values also show positive integer revealing yellowish/reddish/brown color appearance on coordinates. Finally, the perceived color on the composites is lighter yellowish reddish to darker brown yellowish in visual appearances also confirmed further by the coloration characteristics as seen in Table 3.

Table 3
Colorimetric studies of control and nanosilver treated flax woven fabric reinforced composites.

Composite specimens	L*	a*	b*	K/S
FC1	78.18	1.49	9.91	0.19
FAg2	57.72	5.95	26.11	0.97
FAg3	50.54	9.03	23.65	1.62
FAg4	32.82	12.26	24.77	5.49

L* Lightness/darkness; a*– Reddish/greenish; b*–Yellowish/bluish; K/S–Color strength.

The similar coloration consequences were also found in previous reports for AgNP treated materials [11,23–26].

Moisture content investigation of control and nanocomposites

The moisture content analysis of natural fiber reinforced composite is another important characteristic as natural cellulosic fibers absorb some moisture from the atmosphere due to the presence of some hydrophillic components like –OH, –COOH, and so on in their polymeric composition [27,28]. It is found from the Fig. 5 that control composite absorb 3.22 (0.18)% moisture, whereas 1 mM $AgNO_3$ treated sample absorb 2.7 (0.20)% moisture wwhich is slightly lower than control one. However, there is an increase in moisture content is noticed after 1 mM $AgNO_3$ loading, like as 3.06 (0.02) for 2 mM $AgNO_3$ loading and 3.17 (0.03)% for 3 mM $AgNO_3$. The similar trend was also observed in case of 24 h duration of drying in the dryer. Generally, NP treated samples exhibit less moisture compared to control samples [27,29], which is also reflected to our current study for the samples untill 3 h drying. However, the sample 3 and 4 absorbed more moisture after 24 h compared to control sample which is providing different phenomenon maybe for the presence of cellulosic flax fibers in the composite system.

FTIR study of control and nanocomposites

The FTIR spectra of control and AgNP treated samples are shown in Fig. 6. The prominent peak on the left side indicates the presence of

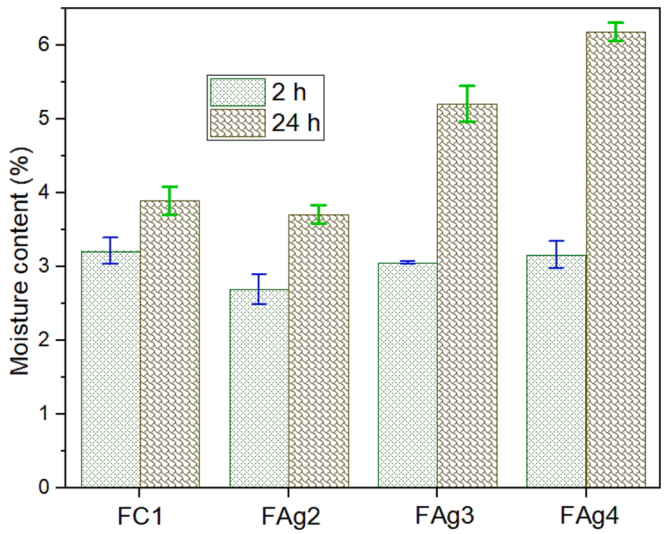

Fig. 5. Moisture content analysis of control and AgNP treated flax woven fabric reinforced composites.

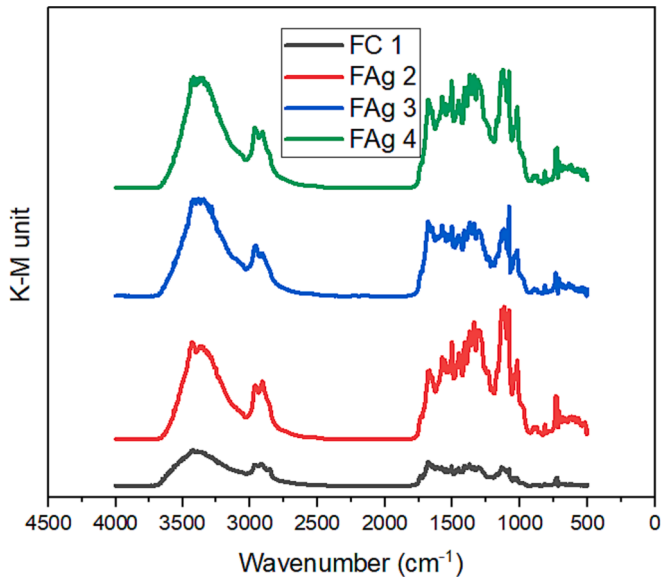

Fig. 6. FTIR analysis of control and AgNP treated flax woven fabric reinforced composites.

hydroxyl groups in the flax fiber fiber reinforced. These hydroxyl groups are primarily located in various components such as cellulose, lignin, and hemicellulose within the fiber's structure. The absorption bands in the spectrum appear broad due to the incorporation of –OH groups, which are naturally present in lignocellulosic polymers, at different wavenumbers. Specifically, the broad IR band observed at 3400 cm^{-1} is responsible for the absorption of –OH groups in this case [30]. The peaks observed around 2908 cm^{-1} correspond to the stretching vibrations of asymmetric CH_2 bonds. Similarly, the peaks around 2854 cm^{-1} are associated with the symmetric stretching of CH_2 bonds. These peaks are attributed to the conjugated C-O stretching in quinones, coupled with the C = O stretching of various functional groups such as flavones. The bands around 1670 cm^{-1} for the control flax is associated with H_2O and CO stretching in the conjugated system. Additionally, the spectrum reveals the presence of symmetric C-O-C stretching at 1135 cm^{-1}, along with aromatic C-H deformation, glucose, and ring vibrations. Furthermore, the C-O stretching originating from cellulose I is also observed in the control flax fiber sample. The similar phenomena for K-M unit-based absorption of lignocellulosic wood based material was reported by a Csanady et a. [30]. However, when the control flax samples were treated with AgNPs, the resulting spectra displayed broader peaks compared to the untreated control samples, as depicted in Fig. 6. However, the chemical structure is not not changed after the production of nanocomposites compared to that of the control composites.

Thermal properties testing

The thermal characteristics of both control and AgNP-treated flax woven fabric reinforced composites were thoroughly investigated as seen in Fig. 7. The AgNP-treated samples exhibited slightly improved thermal stability compared to the control samples. The thermogravimetric curves revealed distinct degradation steps for each composite and nanocomposite product. Initially, some decomposition occurred, likely due to the evaporation of moisture from the flax surfaces [31]. Notably, significant decomposition in the control composite started at 265 °C and followed a consistent pattern until 365 °C, eventually resulting in approximately 75% weight loss up to 800 °C. In contrast, the AgNP-loaded composites displayed a similar pattern at the beginning, but a shift in the trend was observed from 200 °C onward. The nanocomposites exhibited enhanced thermal stability compared to the control samples. The weight loss around 400C is responsible for the degradation of cellulose from the reinforcement fibers of the composite products. Specifically, the 1 mM and 2 mM silver precursor-loaded samples showed nearly 74% weight loss, while the 3 mM silver precursor-loaded samples displayed 72% weight loss at 800 °C. This decomposition is primarily attributed to the dehydration of the internal area in the reinforcement fibers. However, in the case of nanosilver-loaded samples, this tendency was reduced due to the increased loading of nanosilver. The higher loading of nanosilver led to more collisions between the fibers and the metallic nanoparticles, contributing to the overall improvement in thermal stability [32]. Overall, all the samples loaded with nanosilver exhibited higher thermal stability compared to the control samples, showcasing the potential benefits of incorporating nanosilver into flax woven fabric reinforced composites.

Conclusions

This research presents a viable, cost-effective, and convenient protocol for the development and production of innovative nanocomposite materials using flax woven fabric reinforced with melamine-urea–formaldehyde (MUF) composites and greenly synthesized silver nanoparticles (AgNPs) anchored by hornbeam leaves. The concentration of the produced nanosilver materials ranged from 0 to 53 mg/L, corresponding to control, 1, 2, and 3 mM $AgNO_3$ concentrations. The developed materials were subjected to comprehensive morphological, mechanical, and colorimetric analyses, revealing significant improvements in performance. The composite plates exhibited notable enhancements in mechanical properties up to a silver precursor loading of 2 mM, after which a decline was observed with the incorporation of 3 mM $AgNO_3$. Sample 2, with a 2 mM $AgNO_3$ precursor, demonstrated the highest tensile strength of 8.1 (0.2) MPa, while sample 3, with a 3 mM silver precursor, exhibited the highest flexural strength of 30.1 (0.1) MPa. Conversely, the control specimens displayed the lowest performance. Fractured surfaces of the nanosilver-coated composites further confirmed the strong reinforcement effect between the flax fiber and MUF polymer in the lamination system. The developed products showcased varying color appearances ranging from light yellow to darker brown due to the localized surface plasmon resonance optical properties of AgNPs. Moreover, significant thermal stability was also noticed from the nanocomposites. Overall, the findings underscore the significant potential of AgNP-treated flax woven fabrics as sustainable reinforcement materials within the composite community. These materials hold promise for a wide range of applications, offering an eco-friendly and efficient approach to enhance composite properties.

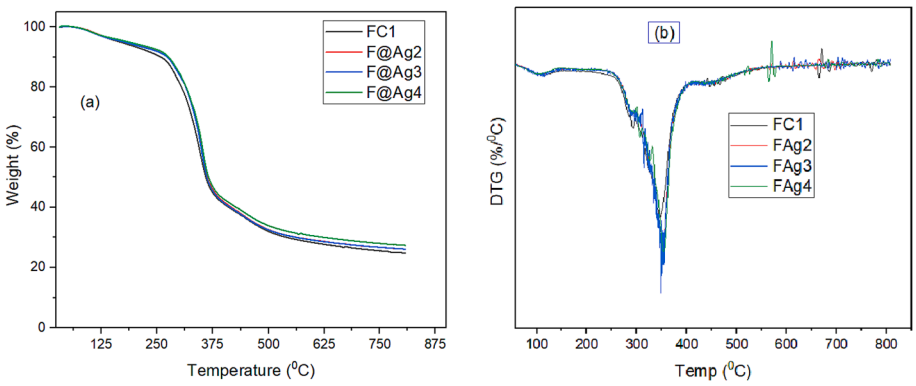

Fig. 7. TGA/DTG analysis of control and AgNP treated flax woven fabric reinforced composites.

CRediT authorship contribution statement

K. M. Faridul Hasan: Data curation, Writing – original draft. **KM. Noman Al Hasan:** Data curation, Writing – original draft. **Péter György Horváth:** Project administration, Validation. **Bernadett Bolodár Varga:** Data curation, Characterization. **Zsófia Kóczán:** Data curation, Characterization. **Bejo Laszlo:** Project administration, Validation. **Miklós Bak:** Data curation, Characterization. **Tibor Alpár:** Project administration, Validation.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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