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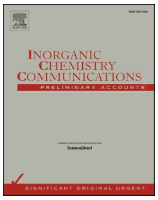

Short communication

Coloration of woven glass fabric using biosynthesized silver nanoparticles from *Fraxinus excelsior* tree flower

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ABSTRACT

A sustainable, fast, feasible, and controlled approach is reported for the synthesis of silver nanoparticle (AgNP). The extract from natural ash plant (*Fraxinus excelsior*) flower was used as bio-based reducing and stabilizing agent to synthesize AgNPs from $AgNO_3$ precursor. The quantitative analysis of AgNP was performed by X-ray fluorescence (XRF) and inductively coupled plasma optical emission spectroscopy (iCP-OES) for solid and liquid samples, respectively. The XRF analysis of untreated and treated glass fabrics were 0, 379 ± 16, and 630 ± 19 PPM, respectively; whereas iCP OES provided the results by 0, 240, and 830 mg/L AgNPs presence on liquid solutions. The Scanning electron microscopy analysis (SEM) exhibited uniform and clear distributions of AgNP on glass surface. The SEM-deployed energy-disruptive X-ray (EDX) and elemental mapping analysis also reflects the clear presence of AgNPs on the surface of glass. Both the treated and untreated glass fabrics were subjected to Fourier transform infrared spectroscopy (FTIR) analysis. The luminescent features of the AgNP treated glass fabrics were also further investigated to assess the color properties. The treated glass substrates also provided bright and brilliant color appearances after treating with AgNPs. Overall, this method of AgNP synthesis would be a benchmark to create new routes of ecofriendly materials for the coloration of woven glass fabrics.

1. Introduction

Green and eco-friendly processing methods in chemical technology field is gaining popularity for continuously increasing demands throughout the world to protect the surrounding environment [1–3]. The nanomaterials are getting attentions for diversified application such as coloration, functionalizing the materials in terms of thermal, mechanical, UV-protection, antibacterial property, wastewater treatment, and so on [4–8]. However, AgNP is getting more attentions for enhancing the bright and colorful appearances on textiles along with superior functional properties enhancement for their superior LSPR (localized surface plasmon resonance) optical properties. AgNP is considered as the significant nanomaterial in terms of optical, catalytic, mechanical, electronic, antibacterial, and biologically compatibility from commercial point of view. Different sectors like as biomedical, textiles, composites, containers of food storage, colouration houses are using AgNPs popularly [9,10]. Chen at el. [11] has mentioned that, around 320 tons of AgNPs were produced throughout the globe by 2011, which was then increased to around 500 tons by 2014, and 530 tons by 2018 [12,13]. Besides, the market volume of AgNPs were near about $1 billion throughout the world in 2015, which is going to be increased by $3 billion by 2024 (approximately) [13]. However, it is expected that the values of AgNP would be consumed by medical sector nearly $1 billion, whereas textile sector by $750 million, and beverage and food sector is going to surpass by $300 million within 2024 [13]. This quantity is going to be further increased with the expansion of scientific methods and technologies.

AgNPs has well known optical, thermal, antibacterial, and electrical properties. However, these properties are influenced significantly with the size of AgNPs (varying within 1–100 nm) [14]. There are various methods reported by the researchers to synthesize AgNPs such as biological, chemical, and physical approaches. Sol gel, chemical reduction, sputtering, *in situ* are some of the commonly used methods for metallic silver synthesis. Some of the approaches also required to use toxic chemicals or higher pressure, temperature, and radiation as the feasible synthesizing protocol [15,16]. So, a constant demand is rising towards emphasizing on green synthesis of nanosilver through avoiding any unhealthy effects from AgNP-based products. In this perspective, biological synthesis of silver has become more popular for eliminating any hazardous effects from the treated products. Although, the usage of

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Table 1
Color properties of control and treated glass fabrics.

Samples	(a) Recipe for nanotreatment	(b) Colorimetric values with associated color strength			(c) Color fastness		
	$AgNO_3$/APS (MW in % (v/v))	L*	a*	b*	CFW	CFR (dry)	CFR (wet)
G@C	0/0	84.81	0.22	4.35	–	–	–
G@Ag1	0.01/3.0-	35.58	5.57	27.88	4–5	3–4	3
G@Ag2	0.02/3.0	50.04	3.8	17.57	4	3	2–3

* APS-Ash plant solution; MW-Molar weight; CFL-Color fastness to light; CFW-Color fastness to wash; CFR-Color fastness to rubbing.

chitosan, algae, and fungi as potential stabilizing agent are also considered as the costly media, so the usage of extracts from plants are getting more attentions for minimized costs by the researchers [16,17]. We also have reported about the green synthesis of AgNPs in our previous studies by using chitosan and sodium alginate as the bio-based reducing and stabilizing agents [18–20].

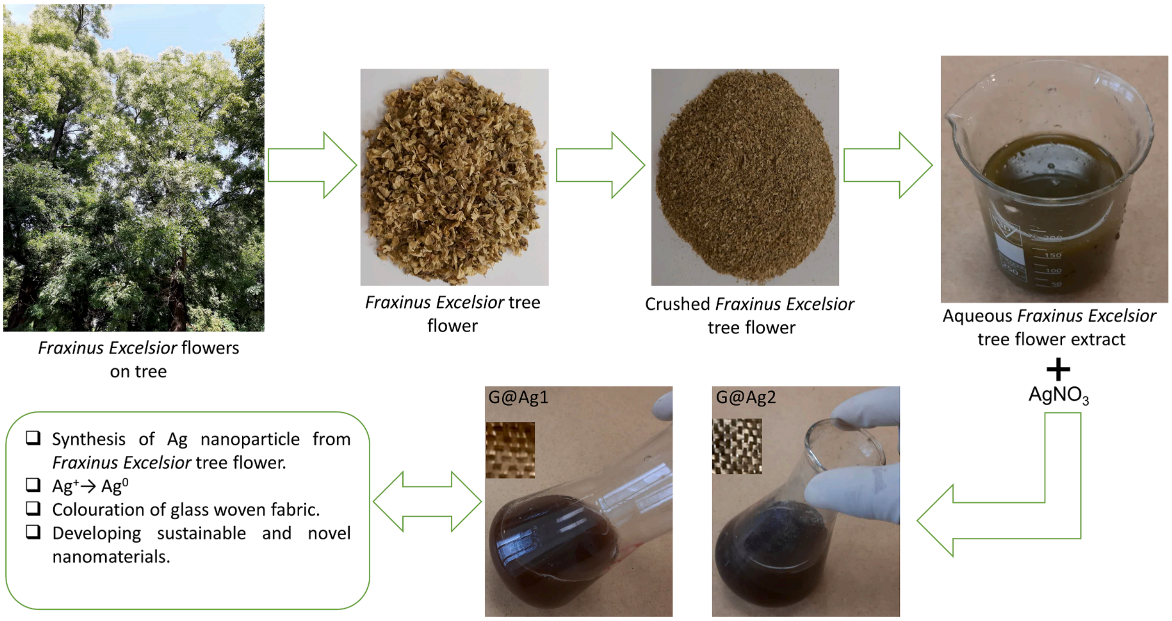
The demands on super fiber is increasing throughout the world with the continuous advancements of science and technology. Glass woven fabrics are widely used for composite productions with multifaceted applications having diversified functionalities, especially for mechanical and thermal property enhancements in structural design [21–23]. Besides, fiberglass is also popular for its superior strength, water resistant finishes, and lighter weight features [24]. Some other reports also have mentioned about the potential usage of glass as an ultraviolet ray protective materials against sunlight by using greenhouse panels made of glass [25]. Besides, considering the huge potentiality, it is tried to investigate and develop a new route of nanosilver synthesis to color the glass woven fabrics. So, we have greenly synthesized the nanosilver on glass fabrics to produce colorful textile fabrics. On the other hand, the flowers from ash plant (*Fraxinus excelsior*) is not yet studied for AgNP synthesis. Ash plant is a widely available plant in central European countries like Hungary. There are plenty of flowers found during the summer season from this tree. So, it is tried in this current research to synthesize the AgNPs from *Fraxinus excelsior* plant flower extracts. The synthesized silver metals provided brilliant color appearances on the surfaces of glass. The methods discussed here is simple, facile, economical, and afterall sustainable.

2. Materials and methods

The woven E-glass fabric with 255 density (g/m^2) was collected from Tolnatext company (Tolna, Hungary). The glass fabrics were prepared in rectangular size and measured for 5 g to each types before the treatments. Silver nitrate ($AgNO_3$, 99.98%) and Vinyltrimethoxysilane ($C_5H_{12}O_3Si$, 98%) were procured from Sigma-Aldrich Co., (St. Luis, USA). The glass fabrics were treated with $C_5H_{12}O_3Si$ for 15 min at room temperature before going to the coloration process. The flowers of ash plant were collected near the campus area (University of Sopron, Sopron, Hungary) in last summer (August 2020).

Before the extraction, the flowers were washed with distilled water to remove all the associated dust, debris, and mud from the surface. The flowers were then air dried at environmental temperature and crushed with a grinder for 10 min in fine particle forms. The aqueous extraction of flowers were prepared by mixing 30 g fine particles of the flowers with 200 mL water, which was then followed by 45 min boiling. The boiled extracts were cooled down and filtered with a filter paper (Whatman). The extracted solution was stored in a sealed beaker at 4 °C temperature for the purpose of future usage. The biosynthesis of AgNPs were carried out into Erlenmeyer flask by adding 0.01 M and 0.02 M $AgNO_3$ as shown in Table 1. Besides, 3% (v/v) ash flower aqueous solutions were also added to each flask and stirred continuously. The untreated glass was marked by G@C, whereas the treated samples were indicated by G@Ag1 and G@Ag2, respectively for 0.01 M and 0.02 M $AgNO_3$. The woven glass fabric samples were also immersed into the solution simultaneously to proceed with *in situ* synthesis method. The detailed synthesis protocol of the NPs are illustrated in Scheme 1. The overall synthesis protocols were continued for 30 min at 95 °C temperature. After the treatment process, the samples were then washed away to remove the unfixed nanoseeds from glass surfaces. Finally, the glass samples were dried in an oven drier for 10 min at 60 °C.

The coated NPs on woven glass fabrics were characterized by using XRF instruments to measure silver content on the solid surface of it. The SEM machine was used to investigate the surface morphology of the nanotreated fabric samples, whereas SEM mediated elemental mapping and EDX spectrum also employed for assessing the presence of chemical elements into the control and treated materials. The FTIR analysis were also further carried out to check the chemical bonding/elements of the control and nanotreated samples. Atomic absorption spectroscopy (iCP



Scheme 1. Mechanism of greenly synthesized AgNPs deposition on glass surface.

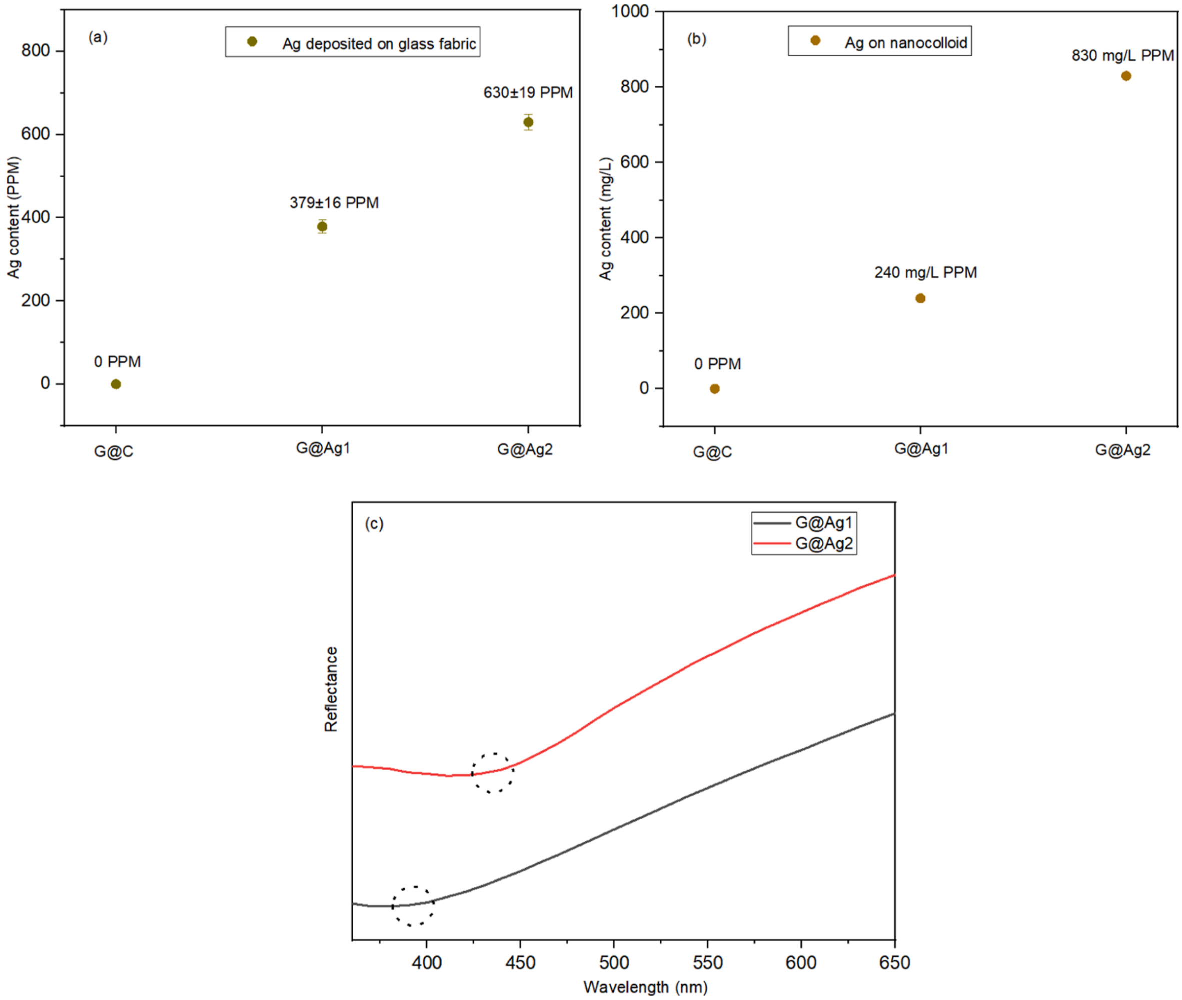


Fig. 1. (a) XRF analysis of control and treated glass fabrics, (b) iCP OES analysis of nanocolloid, and (c) reflectance spectra of treated glass fabrics.

OES, iCAP™ 6300 series) helped for finding out the silver content on nanocolloid. Furthermore, A spectrophotometer was used for assessing the color values for G@C, G@Ag1, and G@Ag2 samples. The colorimetric properties were measured by Konica Minolta 6000d spectrophotometer within 400–700 nm wavelength under daylight (D65). All of the measurements and tests were conducted as per our previous studies [10,20].

3. Results and discussions

The plant extracts like flowers could reduce the Ag^+ to Ag^o through functioning as a reducing agent and generates a thin layered coating of particles as a stabilizing agent simultaneously [26]. The formation of AgNPs were initially confirmed by the color change of solutions in the beaker and associated glass fabrics from clear watery to pale yellow and nearly brown color. The surface plasmon resonance property of AgNP [19] is responsible for this color changing phenomenon. It is also observed (Scheme 1) that, the color is turning to yellow/light brown to brown color at 0.01 M and 0.02 M nanocolloid which was consequently deposited onto the glass fabrics. In both cases, the leaf extracts were remained constant by 3%. Here, the variation in colors were regulated by controlling the concentrations of silver precursor ($AgNO_3$), which were subjected to synthesized AgNPs. After the treatment process, unfixed nanoseeds were washed away from the glass surfaces. The concentration of AgNPs on nanocolloids were investigated further by using the iCP OES measurement and found 240 and 830 mg/L for using 0.01 M and 0.02 M nanosilver precursor, respectively. It is also observed that, the concentrations of silver content of nanocolloid is increased with the increase of $AgNO_3$. The similar phenomenon was also observed by XRF analysis on solid glass surface. As expected, control glass did not exhibit any NPs presence on the surface; however, treated fabrics provided 379 ± 16 PPM and 630 ± 19 PPM silvers content for G@Ag1 and G@Ag2 samples, respectively.

However, the appearances of silver on glass surfaces were further investigated by SEM morphology as illustrated in Fig. 2(*a_2 and a_3; b_2 and b_3; c_2 and c_3*). It is apparent from the SEM images that, the AgNPs are uniform and evenly distributed throughout the surfaces of glass (G@Ag1 and G@Ag2), whereas the control glass is exhibiting plane and smooth surface (G@Ag). These observations clearly confirm the successful deposition of AgNPs on glass surface. The spherical shaped particles are appeared throughout the treated surfaces through homogeneous distributions of nanosilver along with the presence of fewer clusters as well. It is also found that, the size of nanosilver increases with the increase of silver precursor. The similar effect was also reported by Samari et al. [27] for mango leaf extracted NP synthesis. The overall SEM investigations indicate a very strong nanosilver binding and grafting aptitude into the glass fabric.

The EDX spectrums (Fig. 3) also ensures about the AgNPs presence on the glass surface. The typical peaks at 3.0 keV, indicate the existence of AgNPs on G@Ag1 and G@Ag2 samples due to the surface plasmon resonance [28,29]. Kambale et al. biosynthesized the AgNPs through using plant extracts and found the sizes were within 15–115 nm through TEM analysis providing the EDX peak at 3.0 keV [30]. As in our current study, the EDX peaks for Ag is also found at 3.0 keV; hence, it could be

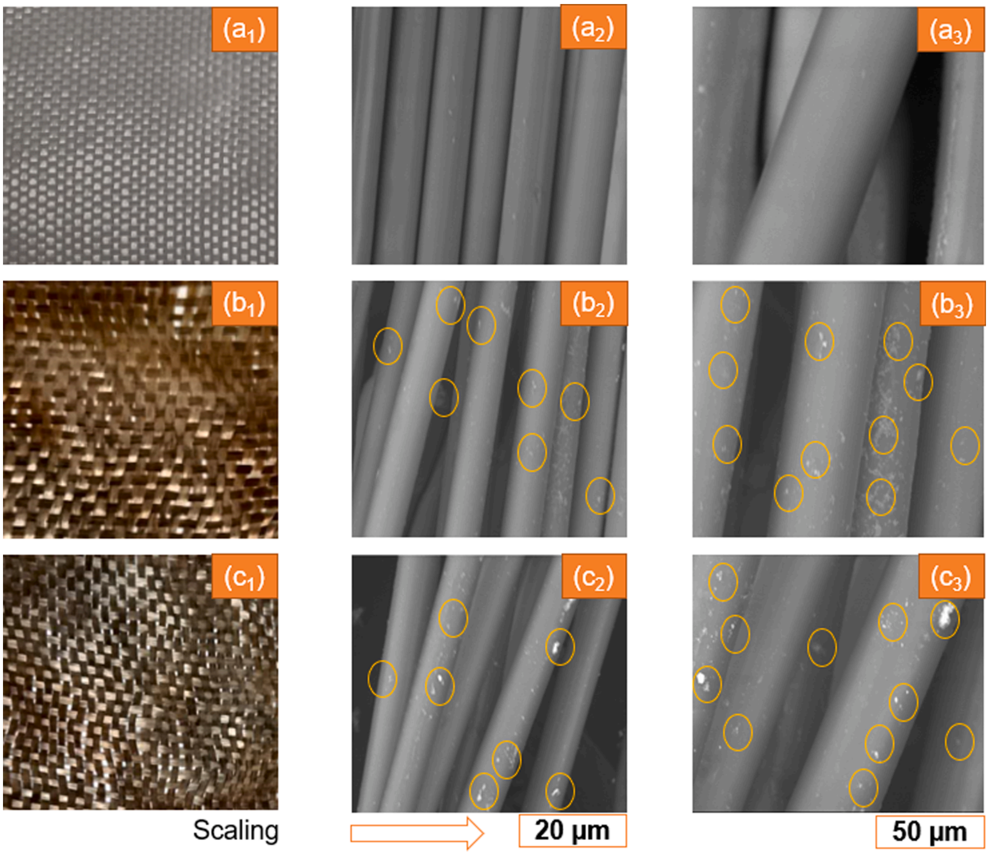


Fig. 2. Photographs of G@C (a$_1$), G@Ag1 (b$_1$), and G@Ag2 (c$_1$). SEM images of G@C (a$_2$ and a$_3$), G@Ag1 (b$_2$ and b$_3$), and G@Ag2 (c$_2$ and c$_3$) at 20 and 50 µm, respectively.

mentioned that the possibility of biosynthesized NP dimensions are also within this range. The nanosilver presence was also further confirmed by the signal of elemental mapping (Fig. 4) taken by using SEM -assisted equipment. The mapping also signaled for carbon and oxygen, which maybe attributed for the capping of stabilization by the extracted ash flower on glass. Besides, the signals for silicon (Si), aluminum (Al), fluorine (F), and calcium (Ca) both in treated and untreated fabrics are associated with the different oxides of glass [31].

FTIR investigation was carried out further to find out the chemical interactions between the glass fabric and AgNPs before and after the coloration as illustrated in supplementary files (Fig. S1). The peaks around 924 cm^{-1} is associated with Si—O—Si stretching vibration, which is the characteristic of glass fibers [32,33]. The bands at 428 cm^{-1} is also related with the Si—O stretching [33] in G@C samples. However, this peak is not clearly appeared for the 0.02 M nanosilver treated glass fabrics (G@Ag2) although there is a slight appearance for 0.01 M AgNP treated fabrics. The absorption band at 1243 cm^{-1} reflects about the C—O—C stretching vibrations for the treatment with $C_5H_{12}O_3Si$ [33]. The peak at 2361 cm^{-1} is responsible for C—O—O, which is comparatively less broad in case of G@Ag1 and G@Ag2 than the G@C sample. It maybe for the treatment of nanoparticles. However, the above discussions correspond to the strong and tight binding of nanosilver on glass fabrics.

The control glass fabric have become colored (Fig. 2) after the treatment with NPs, which maybe for the strong encompassing of AgNPs. So, the reflectance spectra of the fabrics were investigated for understing the associated color propperties. The reflectance peak of G@Ag1 (0.01 M silver precursor) nanotreated glass fabric was found nearly at 400 nm; whereas 430 nm was found for G@Ag2 (0.02 M silver precursor) samples. It is also seen that, reflectance spectras varied with the different NPs present into the glass samples. As expected, G@Ag1 exhibitted more lighter tone compared to G@Ag2 sample. The color strength (K/S) value of G@Ag1 (1.78) was also comparatively lower than G@Ag2 (4.72). This phenomenon is observed maybe for the differences in AgNPs between the two samples (Fig. 1). Besides, L* values of control sample is relatively higher along with lower values of a* and b* values compared to the AgNP treated fabrics, which is nearly the white color. However, the L* value is reduced significantly after the nanosilver treatment from 84.81 to 35.58 and 50.04, respectively for G@Ag1 and G@Ag2 samples. Through investigating above data it is found that the AgNP treated glass becomes more darker with the increase of silver content. If the CIE a* value is positive, it indicates reddish tone; whereas negative value stands for the greeinsh tone. On the other hand, positive b* stands for yellow color and negative b* for blue color. As all the perceived b* values (Table 1) are positive here, so the appeared color for G@Ag1 is nearly yellow, whereas with the increased silver content G@Ag2 is turned to nearly brown color. The similar phenomenon of nanosilver treated fabric was also described by other researches [7].

The color stability on textile material is also another prominent criteria need to fulfill for ensuring the quality of color in terms of durability. The color durability were investigated further in terms of color fastness to wash (CFW) and color fastness to rubbing (CFRR, both in dry and wet conditions). The perceived colorations on glass has shown the ratings for CFW within 4–5 expressing “very good” to “excellent” color color stability in case of G@Ag1 samples, whereas “very good” color durability was found for G@Ag2 samples having the fastness rating 4. The similar trends also found for rubbing fastness results. However,

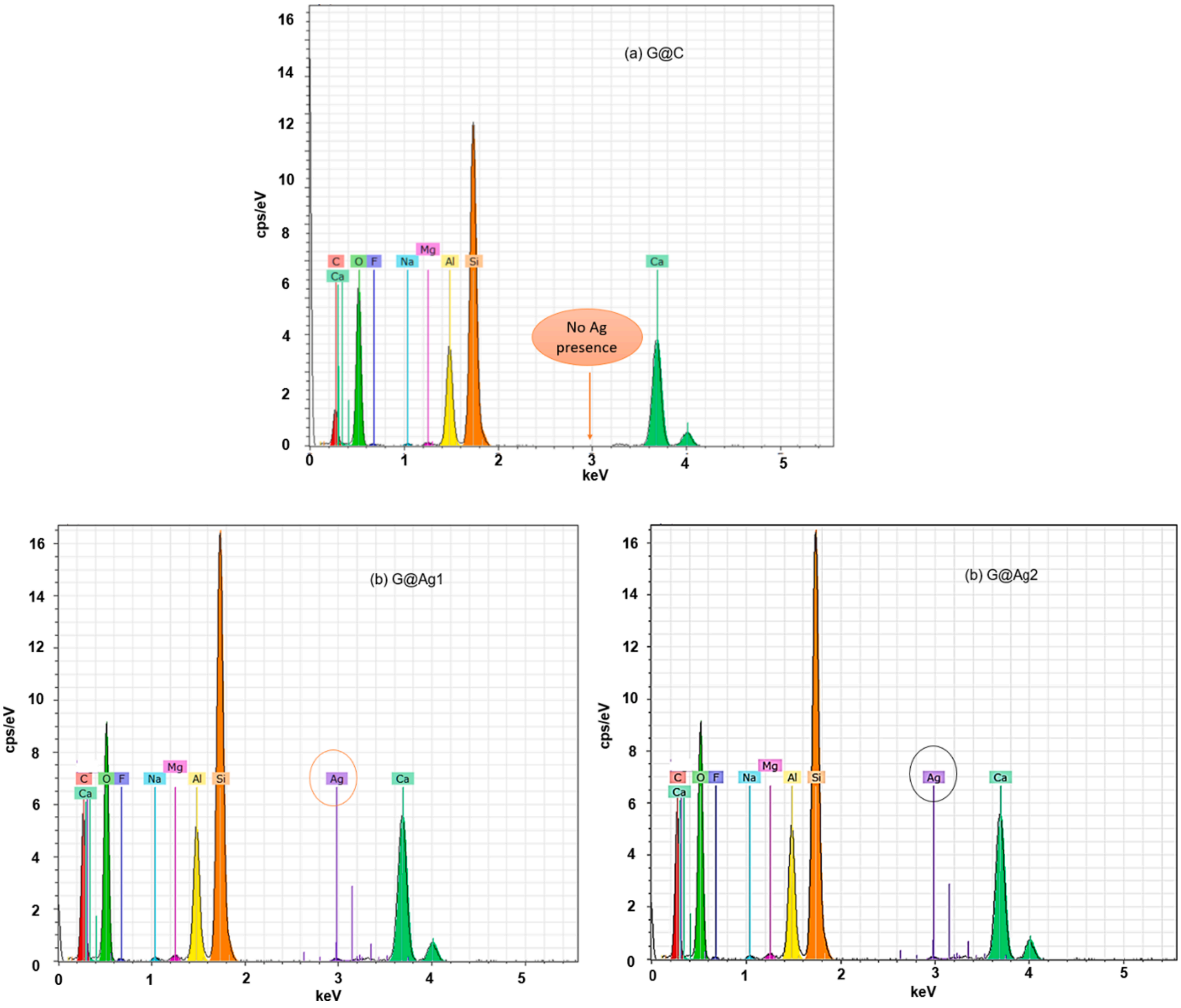


Fig. 3. EDX linear spectra of G@C (a), G@Ag1 (b), and G@Ag2 (c).

color fastness to rubbing (wet) is shown the lowest values 2 to 3 (expressing fair to good) for G@Ag2 sample, whereas 3 (expressing good) for G@Ag1 samples. The better performances in terms of color stability is found for G@Ag1 compared to G@Ag2. It maybe that, the AgNPs are strongly entrapped into the glass fibers monofilament network after the coloration and drying for the molecular force [7,20,29]. However, the overall circumstances discussed above confirms about the successful deposition of AgNPs on glass surface with enhanced stability and durability.

The convetional methods for synthesizing AgNPs with chemical reduction approach were under threats of human toxicity or environmental pollutions. The treated glass fabrics were washed for 15 times with detergent and the AgNPs were measured again by using XRF instrument. The obtained XRF results after the washing were 356 PPM for G@Ag1 and 582 PPM for G@Ag2 samples. Therefore, it is found that around 6% AgNPs were released in case of G@Ag1 sample and approximately 7.5% for G@Ag2 samples. However, the release of nanosilver is very low if considered for every single wash. There is no cytotoxic effects on the cells of human if the AgNPs are released to the liquid medium less than 11 PPM [34]. Consequently, it could be summarized that the as synthesized AgNPs exist no toxicity on human health and the environment as well. This current study associated with the chemical reduction less biosynthesized AgNPs approach on glass woven fabrics is safer, environment-friendly, and commercially feasible.

4. Conclusions

The *Fraxinus excelsior* flower mediated AgNP was successfully synthesized using *in situ* synthesizing protocol on glass woven fabric in an eco-friendly way. The flowers of *Fraxinus excelsior* tree worked as a bio-based stabilizing and reducing agent to ensure the reduction of $AgNO_3$ (metalllic precursor), which is free from any toxic ingredint. The synthesized NPs quantitaive existences were monitored and confirmed by XRF and iCP-OES investigation. Besides, EDX spectrum also ensures about the presence of nanosilver on glass surface. SEM-deployed elemental mapping also confirmed the nanosilver existence on the glass surface. The interaction between the AgNP and glass fabric along with different functional groups were confirmed by FTIR analysis. The color properties of the nanosilver treated glass also confirms the brilliant color appearances through successful deposition of AgNPs. The nanotreatment on glass woven fabrics provided stable and durable colors having no toxicity on human health and environments. This eco-friendly and green synthesis of AgNPs would help the coloration industries to be turned into more sustainable products through minimizing environmentally hazardous chemicals.

Declaration of Competing Interest

The authors declare that they have no known competing financial

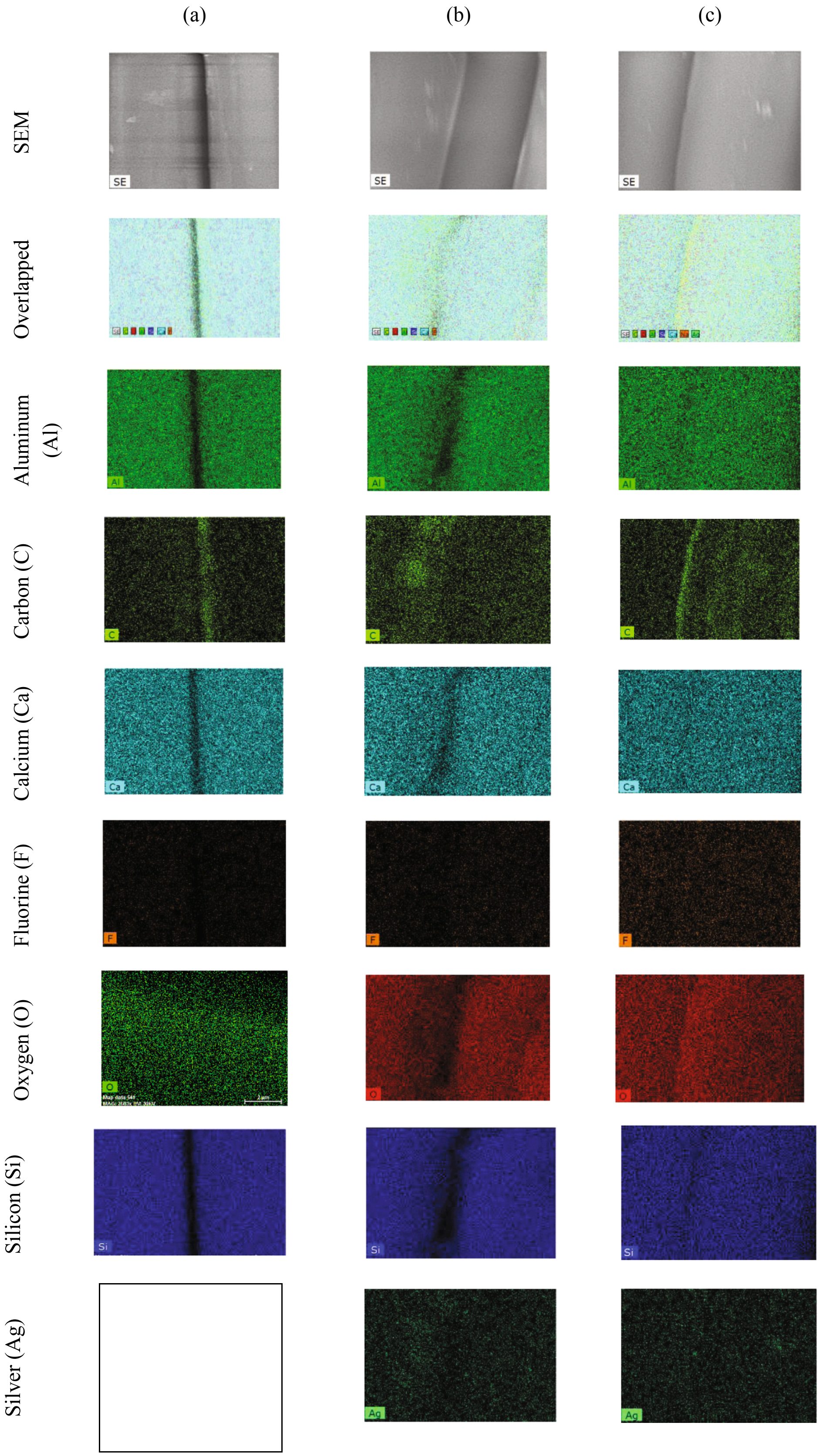


Fig. 4. Elemental mapping photographs of G@C (a), G@Ag1 (b), and G@Ag2 (c).

interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary material

Supplementary data to this article can be found online at https://doi.org/10.1016/j.inoche.2021.108477.

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