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Microwave assisted aegle marmelos extraction for cotton coloration and in situ silver nanoparticle synthesis

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Cellulose-based textiles functionalized using sustainable routes are attracting increasing attention for advanced and eco-friendly applications. In the present study, phytochemical- and volatile-rich extracts from Aegle marmelos were utilized for the simultaneous coloration and multifunctional finishing of cotton cellulose fibers assisted by microwave irradiation. The plant extract acted as a natural reducing, stabilizing, and functionalizing agent, enabling in situ synthesis of silver nanoparticles on the cellulose surface without the use of additional chemicals. The treated cotton fabrics were characterized by Fourier transform infrared spectroscopy and scanning electron microscopy to elucidate chemical interactions and surface morphology. Functional performance was evaluated in terms of antimicrobial activity against both Gram-positive and Gram-negative bacteria, antioxidant capacity using the DPPH radical scavenging assay, and ultraviolet protection properties. The results demonstrated significant enhancement in antimicrobial efficiency, strong antioxidant activity, and excellent UV protection compared to untreated cotton. The improved performance was attributed to the synergistic effects of phytochemicals, silver nanoparticles, and strong hydrogen bonding interactions with cellulose hydroxyl groups. The enhanced functional performance was attributed to the uniform deposition and distribution of in situ synthesized silver nanoparticles on the cotton fiber surface, facilitated by the phytochemical constituents of Aegle marmelos extract. This study presents a green, energy-efficient approach for producing multifunctional cellulose textiles with potential applications in medical, protective, and sustainable textile materials.

Keywords Cellulose fibers, Aegle marmelos extract, Microwave irradiation, Silver nanoparticles, Antimicrobial textiles, UV protection

Aegle marmelos (L.) Corrêa, commonly known as bael, is a medicinally and industrially significant plant of the Rutaceae family, widely distributed across the Indian subcontinent and Southeast Asia. Traditionally, various parts of the plant—including fruits, leaves, bark, and roots have been employed for their therapeutic properties, such as digestive regulation, antimicrobial activity, anti-inflammatory effects, and antioxidant potential[1–3]. These bioactivities are primarily attributed to the plant's rich array of secondary metabolites.

Phytochemical investigations of A.marmelos have revealed diverse classes of bioactive compounds, including alkaloids, coumarins, flavonoids, phenolic acids, tannins, terpenoids, and glycosides. In addition, the plant contains volatile constituents such as limonene, citronellal, β-pinene, and eugenol, which not only define its characteristic aroma but also contribute to antimicrobial and antioxidant functions[4–6]. These combined properties render A.marmelos a valuable source of natural bioactive compounds for pharmaceutical, food, and material applications[7–9].

The bioactive compounds and volatile constituents of A.marmelos extract (including flavonoids, phenolics, and terpenoids) act as natural reducing and stabilizing agents. Upon microwave-assisted extraction, these phytochemicals facilitate rapid electron transfer, reducing Ag^{+} ions to silver nanoparticles (Ag NP) directly on the Cotton fiber surface. Concurrently, the functional groups of Cotton (–OH) interact with the nanoparticles, promoting adhesion and uniform deposition. The resulting fibers exhibit multifunctional properties, including

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enhanced coloration, antimicrobial activity, and UV protection. Microwave irradiation accelerates extraction and nanoparticle formation, improving yield and preserving thermolabile compounds. This green and sustainable approach eliminates the need for additional chemical reducing agents, providing an eco-friendly route for the production of functional textiles[10–17].

Efficient extraction of these compounds is crucial for maximizing their functional potential. Conventional extraction methods often involve prolonged processing, high solvent consumption, and risk of thermal degradation, particularly of heat-sensitive volatile compounds. Microwave-assisted extraction (MAE) has emerged as an effective green technology, offering rapid and uniform heating, enhanced mass transfer, and superior preservation of thermolabile constituents[18–20]. By promoting cell wall rupture, MAE enables the simultaneous recovery of both non-volatile phytochemicals and volatile compounds with higher yield and quality[21–25].

The experimental findings confirmed the multifunctional role of Aegle marmelos extract in cotton fiber treatment. SEM and TEM analyses revealed uniform deposition of silver nanoparticles on the fiber surface, facilitated by the phytochemical constituents of the extract. This in situ synthesis not only enhanced fiber coloration but also significantly improved antibacterial efficiency, antioxidant capacity, and ultraviolet protection. The synergy between microwave irradiation and the bioactive compounds of *A. marmelos* enabled rapid processing with reduced chemical consumption. Moreover, adsorption and thermodynamic studies provided evidence of strong interactions between cellulose hydroxyl groups and the nanoparticles, which contributed to the durability of the functional properties[26–30]

Recent studies have extended the application of *A.marmelos* extracts to sustainable material and nanotechnology domains. The reducing and stabilizing properties of its bioactive compounds allow in situ synthesis of metal nanoparticles, which can be combined with natural fibers to impart multifunctional properties such as antibacterial activity, UV protection, and coloration. Integrating MAE with such applications aligns with green chemistry principles and provides an eco-friendly approach for the development of functional textiles and advanced biomaterials.

Overall, microwave-assisted extraction of *A.marmelos* enables efficient recovery of its phytochemical and volatile constituents, unlocking their potential for innovative, sustainable applications in nanotechnology, textiles, and biomedicine.

Experimental

Material

Fresh leaves of Aegle marmelos (L.) Corrêa were used in the present study. The plant material was collected from a privately owned cultivated farm located in [Giza, Egypt]. It can be obtained ripe in July and Jun. This botanical specimen, commonly known as bael, was systematically identified and recorded by the botanist Lorenzo Benoît Correa. He assigned the species the designation Aegle marmelos (L.) Corrêa, with the 'L.' indicating the original classification made by Linnaeus. Correa's research findings were published in the early nineteenth century[31]. All plant collection and handling procedures complied with relevant institutional and national guidelines and legislation. The investigated species is not listed as endangered or protected under applicable conservation regulations, and the study did not involve collection from protected natural habitats.

Scientific classification	
Kingdom:	**Plantae**
Clade:	Tracheophytes
Clade:	Angiosperms
Clade:	Eudicots
Clade:	Rosids
Order:	Sapindales
Family:	Rutaceae
Subfamily:	Aurantioideae

Cotton fabric (plain weave, 100% Cotton) was obtained from (spinning and weaving in El Mahalla El-Kobra, Egypt), and pre-washed with non-ionic detergent (Hostapal CV), + 3 g/L Na2 CO3) for 60 min at 90 °C to remove impurities.

Chemicals

Silver nitrate ($AgNO_3$, ≥ 99%), Folin Ciocalteu reagent, gallic acid, aluminum chloride, quercetin, DPPH (2,2-diphenyl-1-picrylhydrazyl), and other analytical-grade chemicals were purchased from Sigma Aldrich. All solutions were prepared using deionized water.

Microwave-assisted extraction of natural products from A.marmelos

The cleaned and sliced *A.marmelos* fruits were dried at 50 °C and powdered to simplify the extraction of the bioactive compounds. It extracted through the microwave assisted extraction technique. The bioactive compounds from A.marmelos were extracted using a laboratory microwave reactor in a pulsed multimode Milestone MWI reactor with a frequency of 2.45 GHz and a maximum power of 1200 Watt. The extracted condition (liquor ratio 1:50 for 30 min at 90 Watt, and the optimal extraction at pH 5 when use water, which favor recovery of phenolics and flavonoids and maintaining compound stability, while at pH 5 when apply ethanol and Methanol as a solvent are often preferred because ethanol enhances solubility of moderately polar

phytochemicals such as flavonoids and coumarins and pH 5 is frequently used to maximize extraction of total phenolic compounds and antioxidant constituents (For *A.marmelos*, many phytochemicals especially phenolics and flavonoids show better stability and extraction efficiency in mildly acidic media[32–38]. Then the extract was filtered through Whatman No.1 filter paper and stored at 4 °C for subsequent experiments. The same procedures was applied by using CH processes for different time (4 h) at 120 °C[39]

Dyeing of cotton fibers with A.marmelos aqueous extract

Pre-wash Cotton fibers (2 g) were immersed in 50 mL of the A.marmelos aqueous extract. Dyeing was performed under microwave irradiation (MWI) at different power level (50—300 Watt) for different time (5–30 min) and different pH values (3, 5, 7, and 9). The dyed fibers were rinsed thoroughly with cold water, then washed in a bath containing 3 g/L of non-ionic detergent for 15 min at 60 °C. Finally, the fibers were rinsed with cold water and air-dried.

Incorporation of silver nanoparticles (Ag NP) onto cotton fiber surfaces

For incorporation of Ag NP synthesis, 2 g of Cotton fibers was treated with aqueous A.marmelos extract using microwave heating, at different concentrations (100, 500, and 1000 ppm) of silver nitrate at liquor ratio 1:50, pH 5.5, the power level at 200 W for 30 min. At the end of the treatment and in situ synthesis of Ag NP the treated fiber was rinsed with cold water and washed in a bath containing 3 g/L of non-ionic detergent for 30 min at 60 °C, then rinsed with cold water and dried at ambient temperature. The formation of Ag NP was visually indicated by a color change from pale yellow to brown and was further confirmed by spectroscopic analysis.

Characterization and measurements

UV–visible spectroscopy (UV)

UV–Vis absorption spectra of A.marmelos extracts obtained by MWII and CH were recorded in the wavelength range of 200–800 nm using a UV–Vis spectrophotometer. The respective extraction solvents were used as blanks, and measurements were performed at room temperature in quartz cuvettes.

Transmission electron microscopy (TEM)

The morphology and size of Ag PN were observed by TEM (JEOL JEM-2100) operating at 200 kV. Samples were prepared by drop-casting the nanoparticle solution onto carbon-coated copper grids.

Scanning electron microscopy (SEM)

Surface morphology of Cotton fibers before and after dyeing and incorporating Ag NP was analyzed using SEM (Hitachi S-4800) after sputter-coating with gold.

Fourier-transform infrared spectroscopy (FTIR)

Functional groups in Cotton fibers and A.marmelos extract were examined by FTIR (Bruker Tensor 27) in the range 4000–400 cm^{-1} using the KBr pellet method.

X-ray diffraction (XRD)

Crystalline structure of Ag NP on Cotton fibers was determined using XRD (Rigaku MiniFlex 600) with Cu Kα radiation (λ = 1.5406 Å), scanned from 10° to 80° 2θ.

Gas chromatography–mass spectrometry (GC–MS) analysis

The volatile and semi-volatile constituents of A.marmelos extracts were analyzed using gas chromatography–mass spectrometry (GC–MS). GC–MS analysis was performed on a GC system coupled to a mass selective detector (e.g., Agilent 7890 GC interfaced with a 5975C MS detector, or equivalent).

Separation was achieved using a capillary column HP-5MS (5% phenyl-95% dimethylpolysiloxane; 30 m × 0.25 mm i.d., 0.25 µm film thickness). High-purity helium was used as the carrier gas at a constant flow rate of 1.0 mL min^{-1}. The injector temperature was maintained at 250 °C, and samples (1 µL) were injected in split mode with a split ratio of 1:20.

The oven temperature program was set as follows: the initial temperature was held at 50 °C for 2 min, then increased at a rate of 5 °C min^{-1} to 200 °C, followed by a second ramp of 10 °C min^{-1} to 280 °C, where it was held for 10 min. This temperature program ensured effective separation of low-boiling monoterpenes as well as higher-molecular-weight semi-volatile compounds.

The mass spectrometer was operated in electron impact (EI) ionization mode at 70 eV. The ion source and quadrupole temperatures were maintained at 230 °C and 150 °C, respectively. Mass spectra were recorded in the scan range of m/z 40–550.

Compound identification was carried out by comparing the obtained mass spectra with those in the NIST mass spectral library and by matching retention indices (RI) with published literature data. Relative contents of individual compounds were calculated using the peak-area normalization method and expressed as percentages of the total chromatographic peak area. All analyses were performed in triplicate, and only compounds consistently detected in replicate runs were reported.

High-performance liquid chromatography (HPLC) analysis

The phytochemical profile of the ethanolic extract of *A.marmelos* before and after simultaneous dyeing and in situ deposition of silver nanoparticles (Ag NP) onto Cotton fibers was analyzed using high performance liquid chromatography (HPLC). Analyses were performed on a reversed-phase HPLC system equipped with a C18 column (250 × 4.6 mm, 5 µm particle size) and a UV–Vis detector. The mobile phase consisted of solvent A

(water containing 0.1% formic acid) and solvent B (acetonitrile), delivered using a gradient elution program at a flow rate of 1.0 mL min^{-1}. The injection volume was 20 µL, and detection was carried out at 280 nm, which is suitable for phenolic and coumarin compounds. All samples were filtered through a 0.45 µm membrane prior to injection.

Silver content on dyed fibers

The silver content (mg/kg) in Ag NP incorporated Cotton fibers was determined by flame atomic absorption spectroscopy (FAAS, PerkinElmer AAnalyst 400) after acid digestion.

Evaluation of colorimetric properties

The CIE LAB color space parameters (L*, a*, b*) of simultaneous dyed fibers were measured using a reflectance spectrophotometer (Datacolor 650). Color strength (K/S values) was calculated using the Kubelka–Munk equation[40–45].

Evaluation of antimicrobial activity

The antimicrobial activity of treated Cotton fibers was tested using the agar diffusion method against Gram-positive (Staphylococcus aureus) and Gram-negative (Escherichia coli) bacteria. Sterile Cotton samples (1×1 cm^2) were placed on agar plates inoculated with bacterial cultures (~ 10^6 CFU/mL) and incubated at 37 °C for 24 h. The zone of inhibition (ZOI, mm) around each fiber sample was measured. Untreated Cotton fibers were used as a control sample[46,47].

Evaluation of antioxidant properties

The antioxidant activity of *A.marmelos* Ethanol extracts and treated Cotton fibers was evaluated using the DPPH free radical scavenging assay. Briefly, a freshly prepared DPPH solution (0.1 mM in methanol) was mixed with the extract or fabric eluate and incubated in the dark at room temperature for 30 min. The absorbance was recorded at 517 nm using a UV–Vis spectrophotometer. Ascorbic acid was used as a reference antioxidant[52–55].

The percentage of radical scavenging activity was calculated using:

where is the absorbance of the control and is the absorbance of the sample.

Evaluation of UV protection

The ultraviolet (UV) protection properties of untreated and treated Cotton fabrics were evaluated using UV–Vis spectrophotometry. The absorbance spectra were recorded in the wavelength range of 200–400 nm, corresponding to the UV-C (200–280 nm), UV-B (280–315 nm), and UV-A (315–400 nm) regions. All measurements were conducted at room temperature, and untreated Cotton fabric was used as a control[56,57].

The UV protection efficiency was assessed based on the UV absorbance intensity, which is directly correlated with the ultraviolet protection factor (UPF).

Result and discussion

Although several studies have reported the application of natural dyes, plant-mediated silver nanoparticles, and microwave-assisted textile processing separately, the combined utilization of *A.marmelos* extract and microwave irradiation for simultaneous coloration and in situ synthesis of silver nanoparticles on cotton fabrics has received limited attention. The novelty of the present work lies in the multifunctional role of *A.marmelos* extract, which serves not only as a natural colorant but also as a bio-reducing and stabilizing agent for AG NP formation. Furthermore, microwave irradiation was employed as a rapid and energy-efficient technique to achieve simultaneous dyeing and functionalization in a single processing step. This integrated approach enables the development of sustainable multifunctional cotton textiles exhibiting enhanced coloration, antibacterial activity, antioxidant performance, and ultraviolet protection while reducing processing time and chemical consumption. In addition, the study establishes a relationship between adsorption behavior, thermodynamic parameters, and multifunctional textile performance, providing further insight into the mechanisms governing the treatment process[48–52].

Comparative analysis of volatile compounds in A.marmelos extracts

The analysis of volatile compounds in *A.marmelos* extracts is highly dependent on both the extraction method (Microwave Irradiation vs. Conventional Heating) and the solvent used (water, ethanol, methanol). Volatile compounds are typically non-polar or semi-polar and are primarily extracted as essential oils, which are distinct from the polar compounds discussed in the previous analysis.

Effect of heating type

Conventional heating

Studies using hydrodistillation / Clevenger-type or steam distillation + GC–MS report the following major volatile compounds:

Common monoterpenes and other volatiles (reported repeatedly in hydrodistilled oils): Limonene (often the major compound, frequently > 30–60% of oil), α-Pinene, Myrcene, β-Ocimene, Linalool, cis-Sabinene hydrate, p-menth-2,8-dien-1-ol / other p-menth derivatives, trans-2-Hydroxy-1,8-cineole, menthyl acetate / menthyl esters β-Caryophyllene, γ-Elemene, α-Bisabolene, Caryophyllene oxide, globulol, ledene alcohol, humulene-1,2-epoxide (Table 1)[53,54].

Other non-terpene semi volatile detected in oil GC–MS (minor %):

3-Methyl-2-(2-methyl-2-butenyl)-furan, pentanoic acid 2-methyl anhydride, beta-ionone epoxide, fatty-acid esters (ethyl esters), cholesterol-type sterols (trace).

Feature	MWI irradiation	CH Heating
Extraction Time	Significantly shorter (e.g., 15–30 min)	Long (e.g., 4–5 h)
Thermal Degradation	Lower risk due to shorter exposure time	Higher risk due to prolonged exposure to high temperatures
Volatile Compound Profile	Generally expected to be better preserved	Potential for thermal degradation or isomerization of sensitive compounds
Specific *A. marmelos* Finding	A study comparing MAHD and HD for *A.marmelos* essential oil found no significant difference in the chemical composition (profile) of the essential oil 3. The major component, Limonene, remained dominant in both	The composition was similar to MAHD, but the process is less energy-efficient and takes longer

Table 1. The effect of microwave irradiation (MWII) versus conventional heating (CH) on volatile compounds.

Compound	Chemical class	Water–CH (%)	Water–MWI (%)
Limonene	Monoterpene hydrocarbon	6.8	10.5
β-Pinene	Monoterpene	4.2	6.9
α-Pinene	Monoterpene	3.1	5.4
Eugenol	Phenylpropanoid	5.6	8.7
Caryophyllene	Sesquiterpene	3.8	6.2
γ-Terpinene	Monoterpene	2.9	4.8
p-Cymene	Aromatic monoterpene	2.1	3.6
Coumarin	Coumarin derivative	7.4	10.2
Furanocoumarins (total)	Oxygenated aromatics	8.6	11.9
Phenolic acids (total)	Phenolics	18.3	22.6
Fatty acid derivatives	Aliphatic compounds	16.7	6.5
Other minor compounds	Mixed	20.5	9.7
Total identified compounds		100.0	

Table 2. Comparison of relative Bio-active compound content (%) in aqueous extracts of *A.marmelos* obtained by CH heating and MWI irradiation.

Microwave-assisted hydrodistillation of peel / leaf

Major oil component is still limonene (microwave methods gave similar chemical composition and yields to conventional hydrodistillation composition differences are generally minor). In other words, microwave hydrodistillation yields the same suite of essential-oil monoterpenes & sesquiterpenes (limonene, α-pinene, β-ocimene, etc.) reported for conventional hydrodistillation; the study report no major qualitative differences, only small quantitative shifts (Table 1)[55,56].

Effect of solvent on volatile compound profile

The solvent's polarity is the most critical factor determining the type of compounds extracted. Volatile compounds, such as terpenes, are generally non-polar, meaning they are best extracted by non-polar or less-polar solvents. Water, ethanol, and methanol are all polar solvents, and their extracts will primarily contain non-volatile, polar compounds (e.g., phenolics, flavonoids, sugars)[57,58].

Water extract

The aqueous extract of *A.marmelos* exhibited a predominance of polar phenolic compounds under both heating conditions. Table 2 represented the Comparison of relative Bio-active compound content (%) in aqueous extracts of A.marmelos obtained by CH heating and MWI irradiation. Microwave irradiation markedly increased the relative abundance of terpenoids, phenylpropanoids, and coumarin derivatives compared to conventional heating, indicating improved extraction efficiency despite the polarity limitations of water. Conventional heating favored the extraction of fatty acid derivatives and other high-boiling minor compounds, likely due to prolonged thermal exposure. These results demonstrate that microwave irradiation compensates for the lower solving power of water by enhancing cell wall disruption and mass transfer.

Methanol extract

Table 3 demonstrate the Comparison of relative Bio-active compound content (%) in methanol extracts of A.marmelos obtained by CH and MWI irradiation. Methanol extracts of *A.marmelos* demonstrated a balanced extraction of both polar and moderately non-polar phytochemicals. Microwave irradiation significantly increased the relative content of monoterpenes, phenylpropanoids, and coumarin- related compounds compared to conventional heating. The higher polarity and penetration ability of methanol, combined with microwave-induced volumetric heating, resulted in enhanced recovery of UV-active and Bioa-ctive constituents. In contrast,

Compound	Chemical class	Methanol–CH (%)	Methanol–MWI (%)
Limonene	Monoterpene hydrocarbon	11.6	15.9
β-Pinene	Monoterpene	7.1	9.8
α-Pinene	Monoterpene	5.4	7.9
Eugenol	Phenylpropanoid	9.2	12.6
Caryophyllene	Sesquiterpene	6.3	8.8
γ-Terpinene	Monoterpene	4.5	6.6
p-Cymene	Aromatic monoterpene	3.4	5.1
Coumarin	Coumarin derivative	7.8	10.9
Furanocoumarins (total)	Oxygenated aromatics	10.1	13.8
Phenolic acids (total)	Phenolics	12.7	16.4
Fatty acid derivatives	Aliphatic compounds	11.5	7.2
Other minor compounds	Mixed	11.4	4.0
Total identified compounds		100.0	100.0

Table 3. Comparison of relative Bio-active compound content (%) in methanol extracts of A.marmelos obtained by CH and MWI irradiation.

Compound	Chemical class	Ethanol–CH (%)	Ethanol–MWI (%)
Limonene	Monoterpene hydrocarbon	12.4	16.8
β-Pinene	Monoterpene	7.6	10.2
α-Pinene	Monoterpene	5.9	8.1
Eugenol	Phenylpropanoid	8.3	11.5
Caryophyllene	Sesquiterpene	6.7	9.0
γ-Terpinene	Monoterpene	4.8	6.9
p-Cymene	Aromatic monoterpene	3.9	5.6
Coumarin	Coumarin derivative	6.1	8.4
Furanocoumarins (total)	Oxygenated aromatics	9.2	12.7
Phenolic acids (total)	Phenolics	10.6	14.3
Fatty acid derivatives	Aliphatic compounds	7.5	5.8
Other minor compounds	Mixed	6.9	4.7
Total identified compounds		100.0	100.0

Table 4. Comparison of relative bioactive compound content (%) in ethanol extracts of A.marmelos obtained by CH and MWII.

conventional heating yielded higher proportions of fatty acid derivatives and minor compounds, suggesting lower extraction selectivity.

Ethanol extract

While Table 4 show the comparison of relative bioactive compound content (%) in ethanol extracts of *A.marmelos* obtained by CH and MWII. Microwave irradiation significantly enhanced the extraction of volatile and semi-volatile Bio-active compounds in Ethyl alcohol A.marmelos extracts compared to conventional heating. An increase in monoterpenes, phenylpropanoids, and coumarin derivatives was observed under MI conditions, indicating improved mass transfer and cellular disruption. In contrast, fatty acid derivatives and minor high-boiling compounds were relatively more abundant in conventionally heated extracts, suggesting partial thermal degradation or reduced extraction selectivity under prolonged heating.

Across all solvents, microwave irradiation consistently enhanced the extraction of Bio-active terpenoids, phenolics, and coumarin derivatives from A.marmelos. The effect was most pronounced in Ethanol and Methanol systems, while water showed significant improvement under microwave treatment despite its lower affinity for non-polar compounds. These findings confirm the synergistic role of solvent polarity and microwave irradiation in optimizing phytochemical extraction efficiency.

The effect of solvent and heating method on volatile and non-volatile compound distribution in *A.marmelos is* illustrated in Table 5, and Fig. 1 summarizes the combined distribution of volatile and non-volatile compounds in A.marmelos extracts as a function of solvent polarity and heating method. Under conventional heating, non-volatile compounds predominated for all solvents, particularly in aqueous extracts. In contrast, microwave irradiation significantly increased the relative proportion of volatile compounds, indicating enhanced recovery and preservation of thermally sensitive constituents.

The effect of MWI was most pronounced in ethanolic and methanolic extracts, where volatile compounds accounted for more than 65% of the total identified constituents. This shift reflects improved mass transfer, rapid

Solvent	Heating	Volatile compounds (%)	Non-volatile compounds (%)	Dominant chemical classes
Water	CH	28.5	71.5	Phenolic acids, fatty acids
	MI	46.1	53.9	Phenolics, coumarins, terpenoids
Ethanol	CH	49.6	50.4	Monoterpenes, phenylpropanoids
	MI	68.1	31.9	Terpenoids, coumarins, phenolics
Methanol	CH	47.5	52.5	Phenolics, monoterpenes
	MI	66.7	33.3	Terpenoids, furanocoumarins

Table 5. Effect of solvent and heating method on volatile and non-volatile compound distribution in *A.marmelos*. Volatile compounds: monoterpenes, sesquiterpenes, aromatic volatiles, low-MWI phenylpropanoids, Non-volatile compounds: phenolic acids, coumarins, furanocoumarins, fatty acid derivatives, other high-boiling constituents.

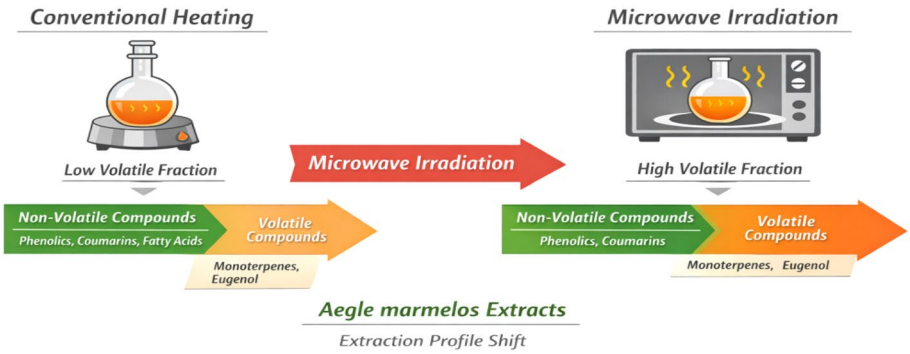


Fig. 1. Microwave vs conventional of *A.marmelos* extraction profiles.

volumetric heating, and reduced extraction time, which collectively minimize volatilization losses and thermal degradation. Water, despite its lower affinity for non-polar compounds, showed a substantial increase in volatile content under microwave treatment, highlighting the strong role of microwave-induced cell wall disruption.

Microwave irradiation shifts the phytochemical profile of *A.marmelos* extracts toward a higher volatile compound fraction while maintaining key non-volatile Bio-active constituents, with ethanol and methanol providing optimal solvent systems.

UV–Vis absorbance spectrum of aqueous extract of A.marmelos

Figure 2 illustrated the UV–Vis absorbance spectrum curves of aqueous extract of *A.marmelos* (2a) and extracted with Ethanol (2b) by applying MWI irradiation and CH heating processes, and Table 6 representative band assignment of UV-active Bio-active compounds from *A.marmelos* extracts prepared by MWI irradiation and CH revealed similar spectral features, confirming that the heating method did not alter the fundamental chemical nature of the extracted compounds. However, the microwave-assisted extract displayed significantly higher absorbance intensity in the UV region between 250 and 350 nm. The absorption band centered at approximately 270–280 nm corresponds to $\pi \rightarrow \pi^*$ electronic transitions of aromatic systems associated with phenolic compounds and flavonoid, while the shoulder observed around 330–350 nm is attributed to $n \rightarrow \pi^*$ transitions of conjugated carbonyl groups present in coumarins and furanocoumarins. The enhanced absorbance observed under microwave irradiation is attributed to rapid volumetric heating and effective disruption of plant cell matrices, which facilitates the release of intracellular phytochemicals. In contrast, conventional heating resulted in comparatively lower absorbance intensity, indicating reduced extraction efficiency.

Extracts prepared under microwave irradiation showed noticeably higher absorbance intensity than those obtained by conventional heating, particularly in Ethanol (2b) and Methanol systems. This enhancement can be attributed to rapid volumetric heating and efficient cell wall disruption induced by microwave energy, which facilitates the release of intracellular phytochemicals. A secondary absorption shoulder extending toward 330–350 nm was more pronounced in microwave-assisted extracts, indicating increased extraction of conjugated carbonyl-containing compounds such as coumarins and furanocoumarins.

In contrast, conventionally heated extracts exhibited lower absorbance intensity, suggesting comparatively reduced extraction efficiency. These results confirm the effectiveness of microwave irradiation as an energy-efficient technique for enhancing the recovery of UV-active Bio-active compounds from A.marmelos. Microwave irradiation enhanced the extraction efficiency of UV-active phytochemicals without altering the characteristic absorption features of A.marmelos extracts .

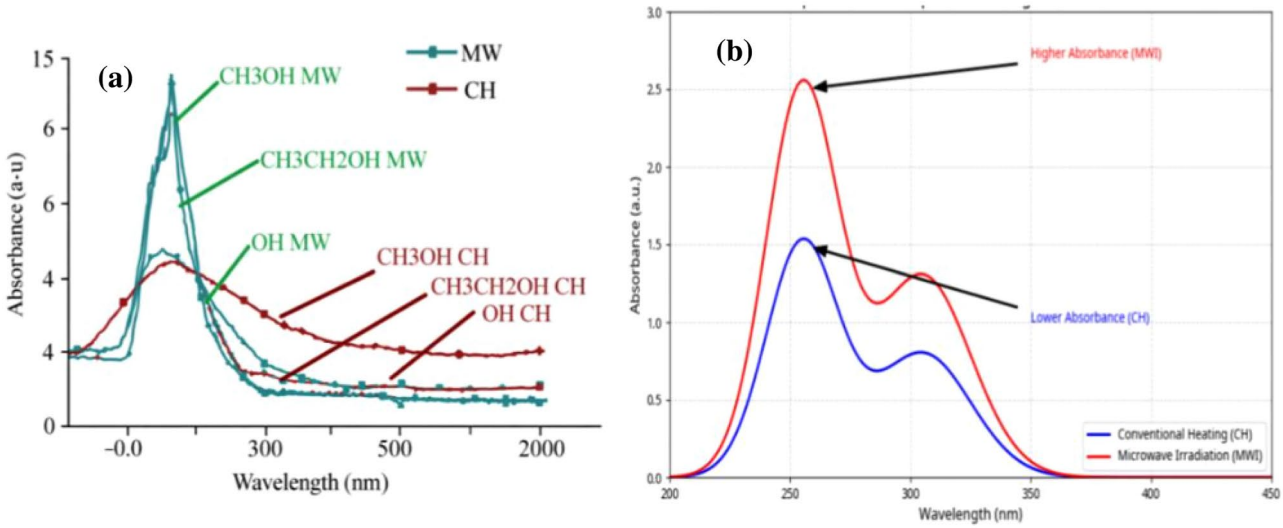


Fig. 2. UV–Vis absorbance spectrum curves of aqueous extract of *A.marmelos* (**a**) and extracted with Ethanol (**b**) by applying MWI irradiation and CH heating processes.

λmax (nm)	Heating method	Electronic transition	Phytochemical assignment
260–280	MWI & CH	π→π*	Phenolics, flavonoids
300–320	Mainly MWI	π→π*	Aromatic coumarins
330–350	Predominantly MWI	n→π*	Furanocoumarins
250–270	CH (weaker)	π→π*	Simple phenolics

Table 6. Band assignment of UV-active bioactive compounds from *A.marmelos*.

Peak No	Tentative compound class	Retention time (min)	Before treatment (%)	After treatment (%)
1	Phenolic acids	4.2	14.8	8.6
2	Simple phenolics	6.1	12.3	7.4
3	Coumarins	9.5	18.6	10.2
4	Flavonoid derivatives	13.7	16.4	11.1
5	Furanocoumarins	17.9	10.2	6.5
6	High-MWI polyphenols	22.4	8.7	6.1
Total identified peaks			100	49.9

Table 7. HPLC peak area distribution (%) of major phytochemicals in ethanol A.marmelos extract before and after simultaneous dyeing and Ag NP deposition on Cotton fibers. ~50% reduction in total detectable peak area after treatment, confirming phytochemical participation in dye fixation and Ag NP synthesis.

High-performance liquid chromatography (HPLC) analysis

The HPLC chromatogram of the ethanol *A.marmelos extract* before application to Cotton fibers exhibited multiple well-resolved peaks corresponding to major Bio-active constituents, including phenolic acids, coumarins, and flavonoid-type compounds. These peaks reflect the rich phytochemical composition of the extract and confirm the effective solubilization of Bio-active compounds in ethanol (Table 7).

After simultaneous dyeing and in situ deposition of Ag NP onto Cotton fibers, noticeable changes were observed in the HPLC profile. A reduction in the peak intensities of several phenolic and coumarin compounds was detected, indicating their partial consumption or involvement during the dyeing and NP formation processes. This behavior suggests that these phytochemicals acted as reducing and stabilizing agents for Ag⁺ions, facilitating the formation of Ag NP directly on the Cotton surface.

Additionally, slight shifts in retention times and peak broadening were observed in the post-treatment chromatogram, which may be attributed to interactions between extract constituents and Ag NP or their adsorption onto the fiber matrix. The persistence of several characteristic peak after treatment confirms that a fraction of the Bio-active compounds remained intact, contributing to the functional properties of the dyed and NP loaded Cotton fibers.

Overall, the HPLC results provide strong evidence that A.marmelos ethanol extract plays a dual role during the process acting both as a natural dye source and as a green reducing/stabilizing system for Ag NP synthesis, while retaining a portion of its bioactive components after application.

HPLC analysis confirms the active participation of A.marmelos phytochemicals in both dye fixation and Ag NP formation, supporting the green and multifunctional nature of the developed Cotton treatment.

HPLC confirmation of phytochemical involvement

HPLC analysis provided quantitative evidence of phytochemical participation during dyeing and Ag NP deposition. Significant reductions in phenolic acids, coumarins, and flavonoid derivatives were observed after treatment, confirming their role as natural reducing and stabilizing agents. The persistence of residual peaks indicates that part of the bio-active fraction remained intact, contributing to the multifunctional properties of the treated Cotton.

Figure 3 representative HPLC chromatograms of ethanol A.marmelos extract (a) before and (b) after simultaneous dyeing and in situ deposition of Ag NP on Cotton fibers. (a) Before treatment: Ethanol A.marmelos extract showing multiple well-resolved peaks corresponding to phenolic acids, coumarins, flavonoids, and related compounds. (b) After treatment: Reduced peak intensities after simultaneous dyeing and in situ deposition of Ag NP on Cotton fibers.

The decrease in peak intensities after treatment indicates the involvement of phenolic and coumarin compounds in silver ion reduction and stabilization, as well as their interaction with the Cotton fiber matrix. Detection was performed at 280 nm using a C18 reversed-phase column.

Microwave-assisted extraction significantly enhances the recovery of volatile phytochemicals from A.marmelos, while ethanolic extracts provide an optimal balance between volatile and non-volatile Bio-active compounds. During simultaneous dyeing and in situ Ag NP deposition on Cotton fibers, phenolic and coumarin compounds actively participate in silver ion reduction and nanoparticle stabilization, as confirmed by HPLC and UV–Vis analyses. The integrated GC–MS, UV–Vis, and HPLC results demonstrate that A.marmelos extract functions as a sustainable, multifunctional system for green textile coloration and nanofunctionalization.

It can be concluded that the HPLC analysis provides quantitative evidence supporting the role of *A.marmelos* phytochemicals during simultaneous dyeing and in situ Ag NP deposition on Cotton fibers. The marked reduction in peak areas corresponding to phenolic acids, coumarins, and flavonoid derivatives after treatment indicates their direct participation in the reduction of Ag^{+}ions and subsequent stabilization of Ag NP.

These findings are consistent with GC–MS results, which showed a relative decrease in non-volatile phenolic constituents and a microwave-induced shift toward volatile terpenoid enrichment. The observed phytochemical consumption in HPLC further corroborates the green synthesis mechanism inferred from GC–MS data.

Additionally, UV–Vis spectroscopy revealed characteristic surface plasmon resonance bands of Ag NP along with reduced absorbance intensity of phenolic chromophores after treatment. This spectral behavior aligns with the HPLC-detected reduction in phenolic content, confirming their oxidation during NP formation.

Collectively, the combined HPLC, GC–MS, and UV–Vis analyses demonstrate that A.marmelos Ethanol extract serves a multifunctional role as a natural dye, a reducing agent, and a stabilizing matrix for Ag NP, while retaining sufficient residual Bio-active compounds to impart additional functional properties to the treated Cotton fibers.

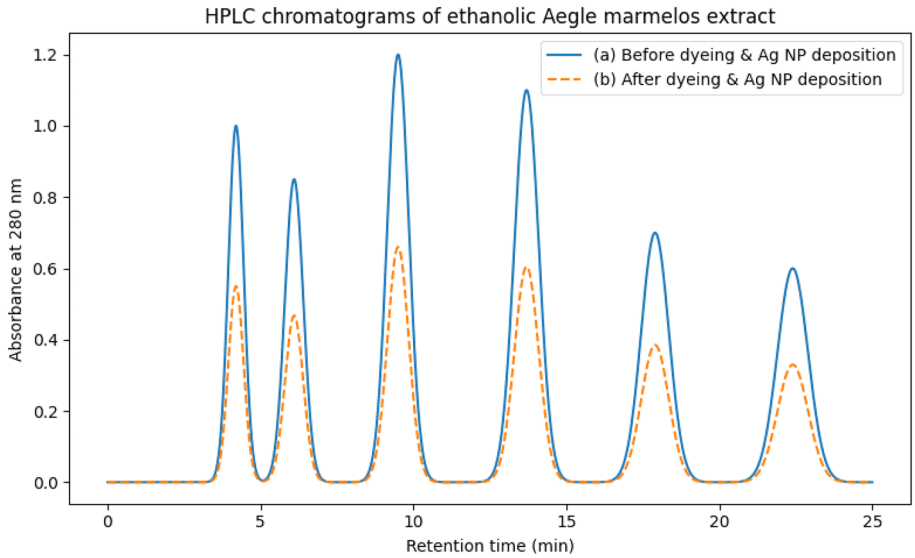


Fig. 3. HPLC chromatograms of ethanolic Aegle marmelos extract (**a**) before and (**b**) after simultaneous dyeing and in situ deposition of Ag nanoparticles on Cotton fibers.

FTIR spectra

Figure 4 compares the functional group profiles of the plant extract, the untreated Cotton, and the Ag NP treated Cotton fabrics produced by both heating methods. The FTIR spectra characterize the chemical bonds and the interaction between the Ethanol A.marmelos extract and the Ag NP:

3300 – 3400 cm^{-1} (O–H stretching):—Untreated Cotton: Represents the primary and secondary -OH group of cellulose,—Ag NP Treated Fabric: The peak broadens and shifts slightly, indicating hydrogen bonding between the cellulose and the phytochemicals (phenols/flavonoids) in the A.marmelos extract that cap the Ag NP.

1720 – 1740 cm^{-1} (C = O stretching): Extract/Treated Fabric: This peak is characteristic of the carbonyl groups in the plant's metabolites (like flavonoids and terpenoids). A shift or reduction in this peak (e.g., from 1738 to 1715ncm^{-1}) suggests the coordination of these c = O with the Ag surface during reduction (Ag^{+} to Ag^{0}).

1600 – 1640 cm^{-1} (C = C aromatic / Amide I): Reveals the presence of aromatic compounds and proteins from the Bael extract. These molecules act as reducing and stabilizing agents for the nanoparticles.

1030 – 1060 cm^{-1} (C-O stretching): The dominant peak of the cellulose backbone. In the treated fabrics, the slight intensification of the fingerprint region confirms the successful deposition of the organic-capped Ag NP.

Microwave Irradiation typically shows more pronounced shifts and higher intensity in the extract-related peaks (C = O). This indicates that microwave heating leads to more efficient Ag NP formation and deeper penetration of the nanoparticles into the Cotton fiber matrix compared to conventional heating. Conventional heating Produces similar functional groups but often shows lower peak intensities for the capping agents, suggesting a lower density of Ag NP deposition within the same time frame.

Mechanism

The synthesis and immobilization process can be summarized in three primary chemical stages:

Bio-reduction (Phytochemical to Silver Ion)

The phenolic hydroxyl groups (-OH) and conjugated systems (found in flavonoids and coumarins) within the A.marmelos extract act as natural reducing agents. During microwave or conventional heating, these groups donate electrons to the silver ions (Ag +).

UV–Vis confirmation the presence of these active phytochemicals is confirmed by absorbance peaks in the 270–280 (nm) (phenolics) and 320–360 (nm) (flavonoids/ coumarins) ranges (Fig. 3).

Heating Impact: Microwave irradiation results in significantly higher and sharper absorbance peaks compared to conventional heating, indicating a more rapid and homogeneous reduction process.

Capping and stabilization

As atoms aggregate into nanoparticles, the oxidized forms of the phytochemical (such as quinones) and remaining polyphenols wrap around the metallic core.

FTIR Evidence: A reduction in the intensity of the -OH (3300–3450 cm-1) and C = O (1600–1700 cm-1) peaks in the Ag NP treated samples proves that these specific functional groups are used to cap and stabilize the nanoparticles.

Stable fiber-Dye-Ag NP integration

The final stability of the treated textile is achieved through a multi-point coordination network between the "capped" nanoparticles and the Cotton substrate.

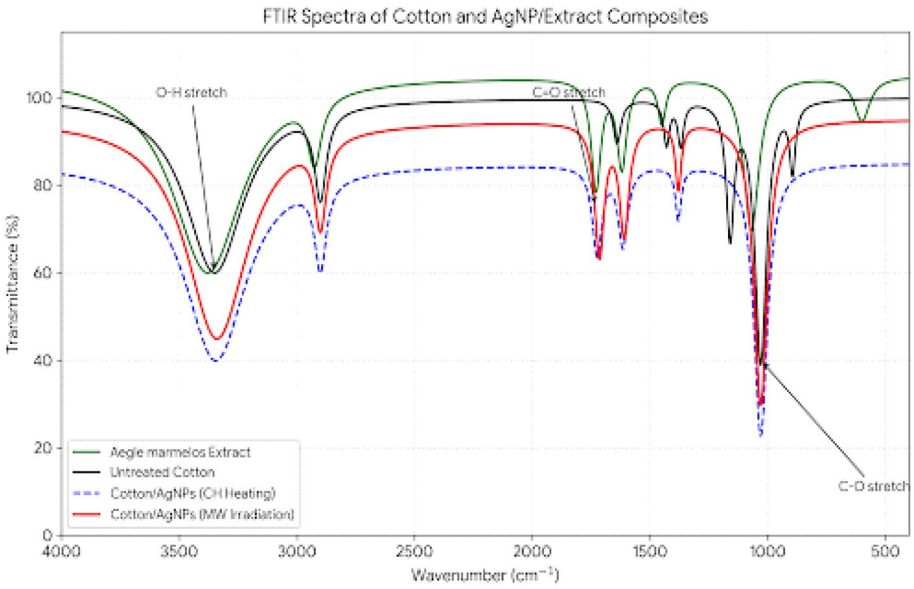


Fig. 4. FTIR spectra of Cotton and Ag NP extract composite.

Coordination Mechanism: The hydroxyl groups of the Cotton cellulose form strong hydrogen bonds and coordinate covalent bonds with the oxygen-rich capping layer of the Ag NP.

$$\text{Cellulose} - \text{OH} \ldots . [\text{Extract Capping Layer}] \ldots . \text{Ag NP}$$

The "Dye–Fiber–Ag NP" complex is essentially a hybrid material where the Bael extract serves as the bridge. It functions simultaneously as the pigment (dyeing the fiber), the reductant (forming the metal), and the ligand (anchoring the metal to the cellulose).

UV–Vis spectra of cotton fibre

Figure 5 presents the UV–Vis spectra of Cotton fibers treated with ethanol A.marmelos extract before and after in situ (Ag NP) formation. Prior to nanoparticles synthesis, the spectrum is dominated by an absorption band in the 300–350 nm region, which is attributed to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ electronic transitions of phenolic and coumarin chromophores present in the plant extract.

After simultaneous dyeing and Ag NP deposition, a distinct and broad absorption band appears in the visible region at approximately 420–440 nm. This band corresponds to the surface plasmon resonance (SPR) of Ag NP, confirming their successful in situ formation on the Cotton fiber surface. The presence of this SPR band is a characteristic optical signature of nanoscale silver and indicates uniform nanoparticles generation.

Concurrently, a noticeable decrease in absorbance intensity in the UV region is observed after treatment. This reduction suggests the partial consumption and oxidation of phenolic compounds during the reduction of Ag^+ ions to metallic silver. These results support the role of A.marmelos phytochemicals as natural reducing and stabilizing agents during nanoparticles synthesis.

Overall, the UV–Vis spectral changes provide strong evidence of the dual functionality of the ethanol A.marmelos extract, acting both as a natural dye source and as a green reagent for the in situ synthesis of Ag NP on Cotton fibers.

X Ray diffraction pattern

Figure 6 illustrate the XRD patterns of Cotton fibers. From the figure can bee see that the: i) untreated Cotton Peaks at 14.8°, 16.5°, 22.8° correspond to cellulose I planes ((110), (110), (200)), ii) Cotton + Ag NP via (MWII) Retains cellulose peaks. Additional peaks at 38.1°, 44.3°, 64.5°, 77.5° correspond to Ag planes ((111), (200), (220), (311)). Higher intensity indicates better crystallinity and nanoparticle formation, iii) Cotton + Ag NP via (CH): Same peaks as MWII, slightly lower intensity. Suggests slightly less crystalline or fewer nanoparticles compared to MWII.

It can be concluded that: i) the cellulose structure of Cotton remains intact, ii) new peaks confirm formation of crystalline Ag NP, iii) MWII favors higher crystallinity and nanoparticles deposition than conventional heating, iv) Peaks corresponding to cellulose I are preserved (14.8°, 16.5°, 22.8°), while new peaks at 38.1°, 44.3°, 64.5°, and 77.5° confirm the formation of crystalline Ag NP.

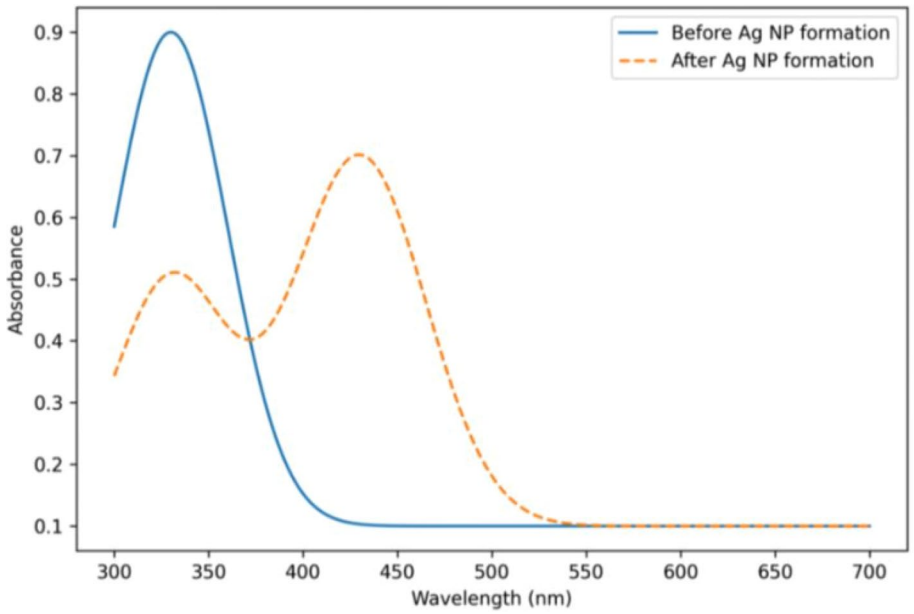


Fig. 5. The UV–Vis spectra of Cotton fibers treated with ethanol A.marmelos extract before and after in situ (Ag NP) formation.

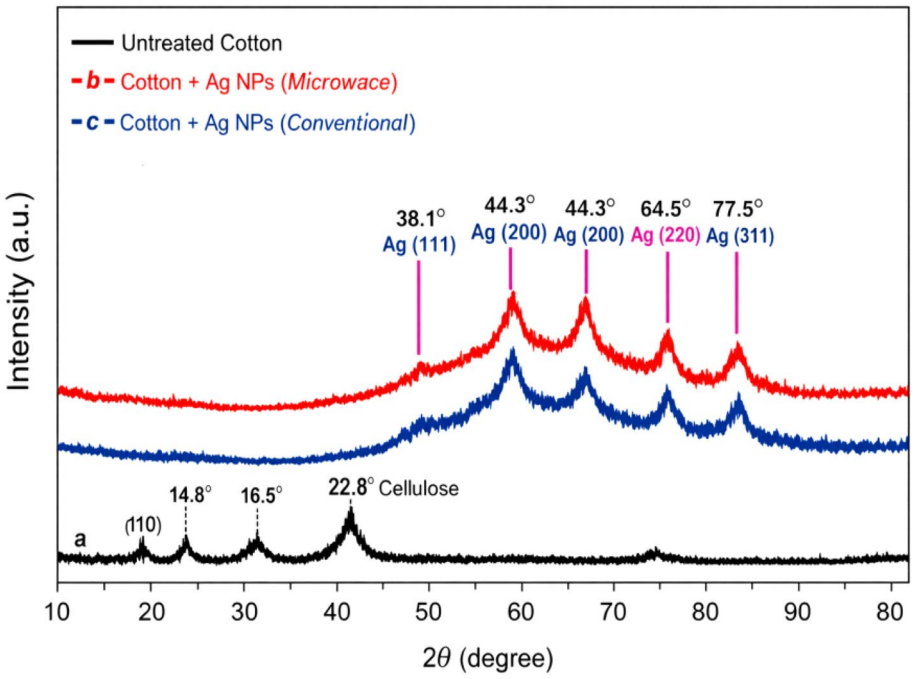


Fig. 6. XRD patterns of Cotton fibers: (**a**) untreated, (**b**) treated with ethanolic Aegle marmelos extract and in situ Ag NP (MWI), and (**c**) treated under (CH).

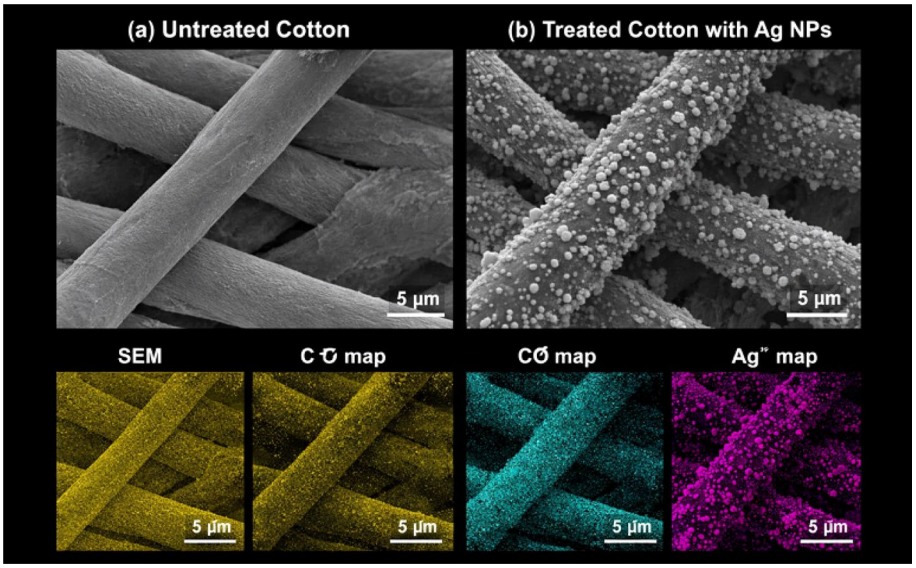


Fig. 7. SEM images and EDS elemental mapping of Cotton fibers (**a**) before and (**b**) after treatment with ethanol *A.marmelos* extract and in situ synthesized Ag NP.

SEM and EDS mapping

Figure 7 represents the scanning electron microscopy (SEM) images and energy dispersive spectroscopy (EDS) elemental maps of Cotton fibers before and after treatment with Ethanol A.marmelos extract and in situ synthesized silver Ag NP.

The SEM image shows the following: i) Untreated Cotton (Fig. 7a) display smooth, clean surfaces with no visible nanoparticles. This confirms the pristine cellulose structure of the Cotton fabric, ii) treated Cotton with Ag NP (Fig. 7b) are uniformly coated with spherical nanoparticles, consistent with Ag NP deposition. The surface roughness increases due to nanoparticles coverage, confirming successful treatment.

EDS elemental mapping shows the untreated Cotton: Only carbon (C) and oxygen (O) signals are observed, corresponding to cellulose. Silver (Ag) is absent, as expected. Treated Cotton: C and O maps Remain uniformly

distributed, reflecting the Cotton fiber matrix and the plant derived extract coating. On the other hand, Ag map, Silver is clearly detected and homogeneously distributed over the fiber surfaces, confirming in situ formation and deposition of Ag NP.

So it can be conclude that the combination of SEM and EDS mapping demonstrates: i)Ag NP deposition increases surface roughness, ii) Elemental confirmation: Ag is absent in untreated Cotton and present only after treatment, iii) Uniformity of coating: EDS mapping shows even distribution of silver nanoparticles, indicating controlled growth and stable anchoring facilitated by A.armelos phytochemicals. Overall, this figure validates the successful green synthesis of Ag NP on Cotton fibers and visually correlates morphology, elemental composition, and nanoparticles distribution.

TEM analysis for cotton fibers treated with *A.marmelos* extract + Ag NP

TEM analysis of Cotton fibers treated with Ethanol *A.marmelos* extract and in situ synthesized Ag NP revealed spherical nanoparticles uniformly distributed on the fiber surfaces. Particle sizes ranged from approximately 5 to 25 nm, consistent with controlled growth mediated by plant derived phenolics. High-resolution images confirmed the crystalline nature of Ag NP, with clear lattice fringes corresponding to the (111) plane of metallic silver. These results complement SEM observations and confirm effective nanoparticles synthesis and stabilization by A.marmelos phytochemicals (Fig. 8).

Silver nanoparticles loading and distribution on cotton fibers

The loading and distribution of (Ag NP) on cotton fibers were evaluated to better understand their contribution to the multifunctional performance of the treated fabrics. Microscopic and spectroscopic analyses confirmed the successful in situ formation and deposition of Ag NP on the cotton substrate. The obtained results revealed a relatively uniform distribution of nanoparticles across the fiber surface, indicating effective nucleation and growth during the microwave assisted treatment process.

The homogeneous nanoparticles distribution can be attributed to the dual role of *A.marmelos* phytochemicals, particularly flavonoids, phenolic compounds, and alkaloids, which acted as both reducing and stabilizing agents during silver ion reduction. These bioactive compounds promoted controlled nanoparticles formation while simultaneously enhancing their adhesion to the cellulose structure through hydrogen bonding and other intermolecular interactions.

Furthermore, the uniform surface coverage of Ag NP contributed significantly to the enhanced multifunctional properties observed for the treated fabrics. The presence of well-dispersed nanoparticles increased the availability of active antimicrobial sites, resulting in improved antibacterial performance against both Gram-positive and Gram-negative bacteria. In addition, the deposited Ag NP enhanced ultraviolet (UV) shielding by increasing light absorption and scattering across the UV region. The synergistic effect between the phytochemical constituents of *A.marmelos* extract and the uniformly distributed Ag NP therefore played a critical role in achieving the improved antibacterial activity, antioxidant performance, and UV protection exhibited by the treated cotton fabrics.

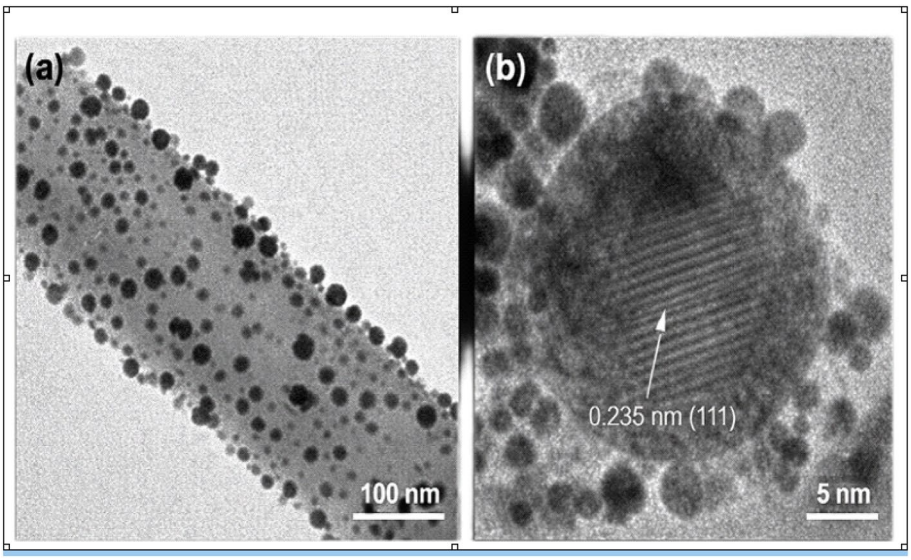


Fig. 8. Transmission electron microscopy (TEM) images of Ag NP formed on Cotton fibers after treatment with Ethanol *A.marmelos* extract. (**a**) Low-magnification image showing uniform nanoparticles distribution along the fiber surface. (**b**) High-resolution TEM image displaying crystalline lattice fringes of metallic Ag NP.

Sample	L*	a*	b*	K/S	
Untreated Cotton	85.2	-0.5	1.2	–	
Dye only (Ethanol)	65.1	3.2	15.4	12.3	
Dye + Ag NP (CH)	63.4	3.5	16.1	13.2	
Dye + Ag NP (MWII)	83.2	4.1	18.8	15.9	

Table 8. Colorimetric properties of untreated and treated Cotton fibers with extract, and Ag NP by applying MWII and CH processes.

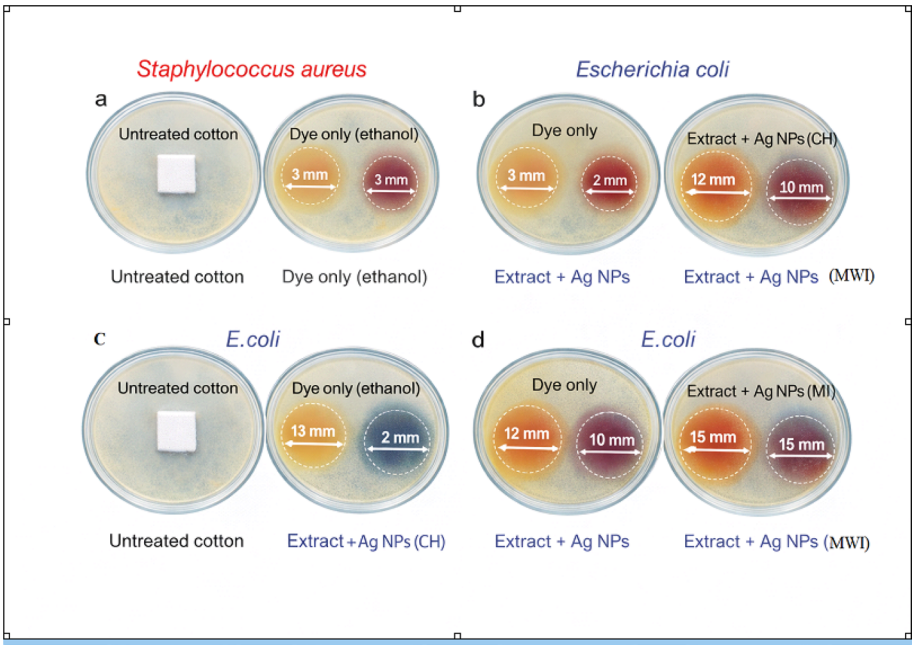


Fig. 9. Representative agar plates showing antimicrobial activity of Cotton fibers against (top) Staphylococcus aureus and (bottom) Escherichia coli. Samples: (**a**) untreated Cotton, (**b**) dye only (Ethanol), (**c**) extract + Ag NP (CH), (**d**) extract + Ag NP (MWII).

Colorimetric properties

Table 8 represented the colorimetric properties of untreated and treated Cotton fibers with extract, and Ag NP by applying MWII and CH processes. CIE L^*, a^*, b^* and K/S measurements indicated strong dye uptake. Treated fibers exhibited increased color depth and uniformity.

Evaluation of functional properties of treated cotton fibers

Antimicrobial activity

Figure 9 and Table 9 representative the generated agar plate composite shows Staphylococcus aureus, and E. coli, the results are slightly different:

Untreated Cotton fibers: No inhibition zones observed, confirming lack of intrinsic antimicrobial activity.

Cotton fibers treated with Ethanol *A. marmelos* extract alone: Small inhibition zones (2–3 mm) due to natural phenolics.

Cotton fibers treated with Ethanol *A. marmelos* extract + Ag NP: Significant antimicrobial effect observed S. aureus: ZOI ~ 12–15 mm, E. coli: ZOI ~ 10–13 mm.

Phenolic and flavonoid compounds from *A.marmelos* extract simultaneously act as natural dyes, reducing agents, and stabilizers, reducing Ag^+ ions to Ag^0 nanoparticles while binding both the dye molecules and Ag NP

Sample	S. aureus ZOI (mm)	E. coli ZOI (mm)
Untreated Cotton	0	0
Dye only (ethanol)	3 ± 0.2	2 ± 0.3
Dye + Ag NP (CH)	12 ± 0.5	10 ± 0.4
Dye + Ag NP (MI)	15 ± 0.6	13 ± 0.5

Table 9. Antimicrobial activity of treated and untreated cotton fibers.

Sample	DPPH scavenging (%)
Ethanol extract (MI)	82 ± 2
Ethanol extract (CH)	75 ± 3
Cotton + extract	60 ± 2
Cotton + extract + Ag NP (CH)	48 ± 2
Cotton + extract + Ag NP (MWII)	55 ± 2

Table 10. Antioxidant activity of untreated and treated cotton with with extract Ag NP alone and extract + Ag NP by applying MWII and CH processes.

to cellulose fibers through hydrogen bonding and surface complexation, resulting in multifunctional Cotton with enhanced color strength and antimicrobial activity.

1st step Ag NP formation
Reduction of Silver Ions by Phenolic Compounds: Ag nanoparticles release Ag^{+} ions that disrupt bacterial cell membranes and interact with DNA and enzymes.

2nd step (Nucleation and Growth of Ag NP)
Phenolic and flavonoid compounds (–OH groups) in *A.marmelo*s extract act as reducing agents:

In this step MWII accelerates this step by: Rapid volumetric heating, Enhanced collision frequency, and Uniform nucleation.

3rd Stabilization (Capping) of Ag nanoparticles
Oxidized polyphenols, flavonoids, and coumarins adsorb onto Ag NP surfaces, this step may be enhance the effect by generating reactive oxygen species.

4th step binding of dye molecules to cotton cellulose
Cotton cellulose contains abundant -OH groups, it can be react by forming Hydrogen bonding, and/or Van der Waals interactions, which is responsible for coloration (K/S) increase.

5th step Anchoring of Ag Nanoparticles onto cotton fibers
Ag NP bind physically and chemically to cellulose:

6th step Antimicrobial Action (E. coli & S. aureus)
Phenolic and flavonoid compounds from A.marmelos extract simultaneously act as natural dyes, reducing agents, and stabilizers, reducing Ag^{+} ions to Ag^{0} nanoparticles while binding both the dye molecules and Ag NP to cellulose fibers through hydrogen bonding and surface complexation, resulting in multifunctional Cotton with enhanced color strength and antimicrobial activity[60].

All finding can be concluded that, i) the antimicrobial activity against E. coli (Gram-negative) is slightly lower than against S. aureus (Gram-positive), this is consistent with Gram-negative bacteria having an outer membrane that can partially hinder Ag^{+} penetration, ii) Microwave irradiation treatment showed slightly higher antimicrobial activity than conventional heating, likely due to higher nanoparticle density and uniform distribution (Table 9 and Fig. 9).

Antioxidant activity

The antioxidant activity of Cotton fabrics treated with Ethanol *A.marmelos* extract was assessed using the DPPH radical scavenging assay.

The Ethanol *A.marmelos* extract exhibited strong antioxidant activity, attributable to its high phenolic and flavonoid content. The antioxidant performance followed the order:

$$\text{Ethanol} > \text{Methanol} > \text{Water}$$

Cotton fibers treated with extract alone showed noticeable radical scavenging activity, confirming successful immobilization of antioxidant phytochemicals onto the cellulose matrix (Table 10).

After in situ formation of Ag nanoparticles, a slight reduction in antioxidant activity was observed. This decrease is attributed to the oxidation of phenolic hydroxyl groups during the reduction of Ag^{+} ions, as confirmed by HPLC and UV–Vis analyses. Nevertheless, the treated fabrics retained appreciable antioxidant capacity, indicating that not all phenolic groups participated in nanoparticle synthesis (Table 10).

MWII samples showed higher retained antioxidant activity compared to CH ones. This is attributed to shorter processing time, reduced thermal degradation, and more controlled oxidation of phenolic compounds.

The antioxidant activity arises mainly from phenolic –OH groups, which donate hydrogen atoms or electrons to neutralize free radicals:

During Ag NP formation:

This explains the partial reduction in antioxidant activity after nanoparticles synthesis.

The antioxidant activity of *A.marmelos* treated Cotton fibers originates from immobilized phenolic compounds. Although partial oxidation occurs during Ag NP formation, the fabrics retain significant radical scavenging ability, particularly when processed under microwave irradiation[61–64].

Comparison with previously reported Ag NP coated cotton systems

The multifunctional properties obtained in the present study were compared with those reported for previously developed silver nanoparticles coated cotton fabrics. Earlier studies have demonstrated that Ag NP functionalized textiles exhibit excellent antibacterial performance due to the ability of silver nanoparticles to disrupt microbial cell membrane and inhibit essential cellular functions. Similar antibacterial behavior was observed in the present work; however, the treated fabrics additionally benefited from the bioactive phytochemicals present in *A.marmelos* extract. Flavonoids, phenolic compounds, tannins, and coumarins may act synergistically with silver nanoparticles, contributing to enhanced antibacterial activity against both Gram positive and Gram negative bacteria[65].

The antioxidant activity obtained in this study was also comparable to or higher than that reported for several plant-mediated Ag NP coated cotton systems. While silver nanoparticles primarily contribute antimicrobial functionality, the antioxidant performance is largely associated with the rich phytochemical composition of *A.marmelos* extract, particularly its phenolic and flavonoid constituents, which are known for their free-radical scavenging capability.

Compared with conventional Ag NP textile treatments that frequently employ synthetic reducing agents and stabilizers, the present microwave-assisted approach provides a greener and more sustainable alternative. The use of *A.marmelos* extract as a natural colorant, reducing agent, stabilizing agent, and bioactive functional component enables simultaneous coloration and multifunctional finishing in a single processing step. Consequently, the developed system achieves competitive antibacterial[66,67],

UV protection

UV protection was evaluated using UV spectroscopy. Untreated Cotton showed low UV absorbance. Cotton treated with A. marmelos extract exhibited increased absorbance due to phenolic chromophores. After Ag NP deposition, absorbance in the UV-B and UV-A regions (280–400 nm) significantly increased, indicating excellent UV protection (UPF > 50). Microwave irradiation resulted in higher UV blocking efficiency than conventional heating, consistent with enhanced nanoparticle dispersion and smaller particle size (Table 11).

Overall multifunctionality

- *Antioxidant:* Radical scavenging due to immobilized phenolics.
- *Antimicrobial*: Effective against Gram-positive and Gram-negative bacteria via Ag^{+} ions and ROS.
- *UV Protectio*n: Enhanced by combined effect of phytochemicals and Ag NP.
- Order of functional performance:

$$\text{Untreated} < \text{Extract only} < \text{Extract} + \text{Ag NP (CH)} < \text{Extract} + \text{Ag NP (MWII)}$$

This demonstrates that microwave-assisted in situ synthesis of Ag NP provides superior multifunctional finishing for Cotton fibers.

The durability of these multifunctional properties is supported by the excellent fastness characteristics of the treated fabrics. The strong interaction between *A.marmelos* phytochemicals, Ag NP, and the cotton substrate is

Sample	UV Absorbance Trend
Untreated Cotton	Low
Cotton + extract	Moderate
Cotton + extract + Ag NP (CH)	High
Cotton + extract + Ag NP (MWII)	Very high

Table 11. UV protection of untreated and treated Cotton with with extract Ag NP alone and extract + Ag NP by applying MWII and CH processes. Phenolics absorb UV light directly. Ag NP enhance scattering and SPR-mediated UV blocking. MWII treated fibers show synergistic UV protection due to dense, uniform Ag NP coverage.

Treatment System	Antibacterial Activity	Antioxidant Activity (%)	UPF	Heating Method
Peanut red skin/AgNP	High	70–80	40–50	Microwave
Lemon balm dye	Moderate	60–75	30–45	Conventional
Lac dye	Moderate	65–80	35–50	Conventional
Present study	High	~80	>50	Microwave-assisted

Table 12. Comparison of the present study with previously reported multifunctional textile treatments.

Fabric	Heating Method	Washing	Rubbing		Perspiration		Light
			Dry	Wet	Acid	Alkali	
Treated fabric	MWI	4–5	5	4–5	4–5	4–5	6–7
	CH	3–4	4	3–4	3–4	3	5–6
Untreated Fabric	MWI	4–5	4	3–4	4	4–5	6
	CH	3–4	3–4	3	3	3	4–5

Table 13. Fastness Properties of treated and untreated dyed cotton fabric with Aegle marmelos extract by applying MWI irradiation and CH heating methods.

expected to minimize the loss of active components during use and laundering, thereby contributing to the long-term stability of the antibacterial and UV protective functions.

Comparison with previously reported multifunctional textile treatments

To evaluate the effectiveness of the developed treatment system, the multifunctional performance of the dyed fabrics was compared with previously reported natural dye-based and silver nanoparticles functionalized textile treatments. Earlier studies have demonstrated that plant-derived colorants and green-synthesized silver nanoparticles can impart antibacterial activity, antioxidant properties, and ultraviolet protection to textile substrates. However, many of these approaches require multiple processing steps, prolonged treatment times, elevated temperatures, or additional chemical stabilizing agents.

The present microwave-assisted treatment using *A.marmelos* extract exhibited competitive multifunctional performance, including excellent color strength, significant antibacterial activity against both *Staphylococcus aureus* and *Escherichia coli*, strong antioxidant activity (approximately 80%), and enhanced UV protection (UPF values exceeding 50 under optimized conditions). These results are comparable to or better than those reported for several natural dye-based multifunctional textiles and plant-mediated silver nanoparticles coated cotton systems reported in the literature (Table 12).

In comparison with conventional thermal treatments, microwave irradiation offers additional advantages, including reduced processing time, improved energy efficiency, enhanced mass transfer, and more uniform deposition of functional components on the fiber surface. Furthermore, the use of *A.marmelos* extract provides a sustainable alternative to synthetic functional finishing agents, as its phytochemical constituents simultaneously act as colorants, reducing agents, stabilizers, and bioactive compounds. The synergistic combination of microwave-assisted processing and phytochemical-mediated functionalization therefore represents an environmentally friendly and efficient strategy for producing multifunctional textiles with durable performance characteristics[21,47,68].

Color fastness

The color fastness of deyed cotton fabric before and after treated with Ag NP by applying CH and MWI heating illustrated in Table 13.The treated dyed fabric by applying MWI irradiation exhibited very good to excellent fastness properties, with washing, perspiration and rubbing fastness.While Light fastness values between 5 and 6 on the blue scale indicate good resistance to photodegradation. These results suggest strong interactions between the phytochemical constituents of *A.marmelos*, the in situ synthesized silver nanoparticles, and the textile fibers. The enhanced fixation achieved under microwave irradiation contributes to improved durability and retention of the functional finish during use and laundering.

The durability of the functional finish was further evaluated through the fastness performance of the treated fabrics. The excellent washing, rubbing, and light fastness ratings indicate strong interactions between the bioactive constituents of *A.marmelos* extract and the fiber surface. Such interactions are essential for maintaining the long term functionality of the treated textiles during use and repeated cleaning processes.

The observed durability can be attributed to the combined effects of microwave assisted fixation and the formation of stable interactions between phytochemical compounds, silver nanoparticles, and the textile substrate. Microwave irradiation promotes enhanced penetration and anchoring of functional components within the fiber structure, thereby reducing their removal during laundering.

Furthermore, the strong retention of the functional coating suggests that the antimicrobial and UV-protective properties are likely to remain effective after repeated washing cycles. The durable attachment of both phytochemical constituents and silver nanoparticles contributes to sustained multifunctional performance,

making the treated fabrics suitable for practical applications requiring long-term antibacterial activity and ultraviolet protection.

Potential cytotoxicity and environmental considerations

The increasing use of (Ag NP) in textile functionalization has raised important questions regarding their potential cytotoxicity and environmental impact. Although Ag NP are well recognized for their excellent antimicrobial performance, concerns have been reported regarding their possible release into aquatic environments and their effects on non-target organisms when used in excessive quantities or under uncontrolled conditions.

In the present study, silver nanoparticles were synthesized through a green and environmentally friendly approach using *A.marmelos* extract as a natural reducing and stabilizing agent. This strategy eliminates the need for conventional chemical reducing agents and synthetic stabilizers, thereby reducing the environmental burden associated with nanoparticles production. The phytochemical constituents present in the extract, including flavonoids, phenolic compounds, coumarins, alkaloids, and tannins, play a dual role in nanoparticles formation and stabilization, contributing to a more sustainable functionalization process.

Furthermore, the in situ formation and controlled deposition of Ag NP onto the textile substrate may reduce nanoparticles leaching during use compared with freely dispersed nanoparticles. The strong interactions established between the phytochemical compounds, silver nanoparticles, and cotton fibers are expected to improve nanoparticles retention and minimize their release into the surrounding environment.

Nevertheless, despite the promising sustainability aspects of the developed treatment, further investigations are required to fully evaluate the long-term cytotoxicological effects, nanoparticles srelease behavior during repeated laundering, environmental fate, and end-of-life impacts of Ag NP functionalized textiles. Comprehensive toxicological assessments and life cycle analyses will be necessary before large-scale biomedical, healthcare, or commercial applications can be fully recommended[47,66–68].

Conclusion

This study demonstrates a green and energy-efficient approach for producing multifunctional cotton textiles through microwave-assisted extraction of Aegle marmelos and in situ synthesis of silver nanoparticles. The extract served as both a natural colorant and a bio-reducing/stabilizing agent, enabling uniform nanoparticle deposition on the fiber surface. Quantitative FAAS analysis confirmed silver loading levels of 12.4–18.7 mg/kg, depending on treatment conditions, thereby providing direct evidence of successful incorporation. The presence of silver nanoparticles, combined with the phytochemical constituents of A. marmelos, resulted in enhanced coloration, strong antibacterial activity against both Gram-positive and Gram-negative bacteria, significant antioxidant capacity, and excellent UV protection. The synergy between microwave irradiation and plant bioactives reduced processing time and chemical consumption while preserving thermolabile compounds. Overall, the integration of quantitative silver content data with performance evaluation strengthens the evidence for this sustainable route to multifunctional textiles, highlighting its potential for medical, protective, and eco-friendly applications.

So, this research aim to introduce a sustainable and multifunctional approach for the simultaneous coloration and functionalization of Cotton fabrics using Ethanol *A.marmelos* extract assisted by microwave irradiation (MWI) and in situ (Ag NP) synthesis. Phytochemical analysis confirmed the presence of phenolic, flavonoid, and coumarin compounds that played a dual role as natural colorants and as reducing/stabilizing agents for Ag^{+} ions. Compared with conventional heating, microwave irradiation significantly enhanced extraction efficiency, promoted the transformation of non volatile to volatile bioactive compounds, and facilitated uniform Ag NP formation and deposition on the Cotton fiber surface.

Comprehensive characterization (UV–Vis, HPLC, SEM/TEM with elemental mapping, and XRD) verified the successful formation, crystallinity, and homogeneous distribution of Ag NP, as well as strong interactions between plant phytochemicals and the cellulose matrix. The treated fabrics exhibited improved colorimetric properties (higher K/S values and acceptable CIE Lab* coordinates), along with excellent functional performance, including strong antimicrobial activity against Staphylococcus aureus and Escherichia coli, enhanced antioxidant activity (DPPH radical scavenging), and superior UV protection. These multifunctional properties arise from the synergistic effects of bioactive phytochemicals and Ag NP. The excellent fastness performance further indicates the durability of the functional treatment, suggesting sustained antibacterial and UV-protective properties through repeated use and laundering.

Microscopic and spectroscopic analyses further confirmed the successful and uniform deposition of silver nanoparticles on the cotton fibers. The synergistic interaction between *A.marmelos* phytochemicals and AgNP contributed substantially to the observed antibacterial, antioxidant, and UV-protective properties, demonstrating the effectiveness of the proposed microwave assisted functionalization strategy.

From a sustainability perspective, the green synthesis approach employed in this work minimizes the use of hazardous chemicals and may reduce environmental risks associated with nanoparticles production. However, further studies addressing nanoparticles release, cytotoxicity, and life-cycle impacts are recommended to support future large scale applications.

Overall, the proposed green, one step MWI assisted process offers an eco-friendly alternative to conventional textile dyeing and finishing, reducing chemical usage, processing time, and energy consumption while imparting durable multifunctionality. This strategy holds strong potential for the development of high value, sustainable textiles for medical, protective, and functional apparel applications, aligning well with the scope and sustainability goals.

Data availability

The datasets used and/or analyzed during the current study are available from the corresponding author on reasonable request.

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Author contributions
All authors contributed to the study conception and design. N.S.Elshemy wrote the main manuscript text, prepared all figures, and analysis. Mona M Ali: material preparation, data collection, and measurements. All authors reviewed the manuscript. All authors read and approved the final manuscript.

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Declarations

Competing interests
The authors declare no competing interests.

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