

Green synthesis of nanoparticle and its application on cotton fabric using *Sterculia foetida* fruit shell extract

Abstract

Sterculia foetida fruit shell was used for the green synthesis of silver nanoparticles. Optimized reduction of silver ion (Ag^+) to metallic silver nanoparticles (Ag^0) was carried out at room temperature for 16hr. The reduced silver nanoparticles were characterized by UV-visible spectroscopy, particle size analyzer, FTIR and SEM analysis of treated cotton fabric not only showed superb coloration but also proven to be very good in antibacterial and ultraviolet protection properties even after ten washes.

Keywords: *Sterculia foetida* fruit shell, Silver nanoparticles, cotton, dyeing, antibacterial, UV protection

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Introduction

Biosynthesis of nanoparticles by microorganisms is a green and eco-friendly technology. The synthesis of nanoparticles could be extracellular in line with the positioning of nanoparticles.¹ For generating nanoparticles with plant extracts, the extract is combined with a solution of the metal salt at different temperatures and response times. The reducing agents associated comprise the other water-soluble plant metabolites (e.g., alkaloids, phenolic substances, and terpenoids) and also co-enzymes.² Extracts of a wide array of plant species have been successfully utilized in making nanoparticles. The green synthesis procedure is a simple, economical and environmentally friendly system of the synthesis of metal nanoparticles compare to physical and chemical processes.³ Synthesis approaches of nanoparticles are categorized into two different methods, top-down and bottom-up.⁴ In the top-down strategy, thin films or bulk substances are all scaled down to create nanodevices with other lithographic methods, e.g., grinding, milling, and thermal/laser ablation. The bottom-up approach utilizes small and easy building blocks (atoms, molecules, nanoparticles, etc.) that will self-assemble into larger, more intricate structures.⁵ In the bottom to top approach, nanoparticles can be synthesized using biological and chemical methods from the self-assembly of atoms to new nuclei, which grow into a particle of the nanoscale. Eco-friendly processes such as the synthesis of nanoparticles have gained momentum nowadays⁶ with nanotechnology likely to have a significant influence in different areas.^{7,8} Many techniques, such as thermal decomposition, microwave-assisted, etc., have been researched for its synthesis of silver nanoparticles. Biological procedures for synthesizing silver nanoparticles⁹⁻¹¹ are considered a valuable alternative to chemical or physical methods. In the present work, dried *Sterculia foetida* fruit shell extract (FSE) has been taken and use for the synthesis of this silver nanoparticle.¹²⁻¹⁴

Sterculia foetida open sections look a lot like woody, valentine-heart-shaped bowls. Alcoholic leaves extract also played a substantial part in anti-inflammatory and central nervous depressant action.^{15,16} There's been a lot of attention focused on producing medications and goods, which are organic, and its seed oil plays an essential role in this regard. Thus, the present study was done to know more about the phytochemical properties of *Sterculia foetida* fruit shells that are accountable for the medicinal and functional properties.¹⁷⁻²⁰

Material and methods

Material

The substance used for experimental work is prepared for dyeing cotton cloth (110 GSM). Precursors silver nitrate (AgNO_3) is among the analytical grade employed in this experimentation acquired from Merck, India.

Preparation of fruit shell extracts

The fresh and washed fruit shell of the required amount was cut into small bits and transferred to 500ml round bottom flask containing 100ml of distilled water. The refluxing procedure was utilized to get the colored solution/dye, which was used for dyeing. Then the temperature of this reaction system has been increased to 100°C and kept for 60minutes. Ultimately, the extract was filtered using nylon fabric and centrifuged for 15min at 4000rpm two times to find undissolved particles to be settled. The supernatant solution was used for dyeing in the laboratory rota dyer machine (Rossari Labtech).

Green synthesis of silver nanoparticles (H-AgNPs)

Silver nanoparticles were synthesized using fruit shell extracts of *Sterculia foetida* fruit shell extract (H-AgNPs). 1mM Silver nitrate solution of volume 80 ml was taken in a reaction vessel.

Then the required amount of fruit shell extract was added and mixed homogeneously on a magnetic stirrer at room temperature. Optimization of reaction time and concentration of fruit shell extract required to synthesize nanoparticles were carried out separately.

Dyeing procedure

Dyeing was carried out in a beaker at room temperature, and a laboratory rota dyer machine was used with programmable time and temperature control. The required amount of dye was taken according

to the dyeing shade on the weight of fabric (o.w.f.). Dyeing trials were carried out both at neutral pH and alkaline pH. In contrast, the dyeing conditions were room temperature for 60 min and exhaust dyeing at 90°C for 60min in the rota dyeing machine (Figure 1).

Particle size analysis

The average particle size of nanoparticles was measured by the laser diffraction method using Shimadzu SALD-7500 analyzer at room temperature.



Figure 1 Extraction procedure of raw material to natural dye.

UV-visible spectroscopy analysis

The UV-vis spectra of nanoparticles were recorded at room temperature using Shimadzu UV-1800 Spectrophotometer. The absorption spectra of fruit shell extract synthesized nanoparticles were recorded in the range of 290nm to 800nm, with distilled water as the reference.

Colour characteristics analysis

Cotton fabric treated with nanoparticles showed a coloration effect and samples were evaluated for the depth of color and colour difference by reflectance method using 10° observers with D65 illuminant. The reflectance of nanoparticle-treated samples was measured on Rayscan Spectra scan 5100+ equipped with reflectance accessories

UV protective properties of the fabric

UV protection characteristics of fabrics were determined by measuring ultra-violet protection factor (UPF) rating, according to the Australian and New Zealand (AS/NZS 4399) standard by using UV-vis spectrophotometer (UV-2600, Shimadzu, Japan).

Antibacterial properties

The antibacterial efficacy of a compound will vary as per its presence in the solution or on the textile substrate. The quantitative assessment of antibacterial activity exhibited on the finished cotton fabric was carried out by AATCC Test 100-2004.

SEM-EDS analysis

The untreated and treated sample were analyzed utilizing Field Emission Gun-Scanning Electron Microscope (FEG-SEM, TESCAN). Specimen size of 5×5mm² was shot and the conductive agent employed was sputter-coated for its 600s length. The beam voltage of 10 kV, 2000X magnification and 6mm working space for examining the sample had been utilized.

Results and discussions

Green synthesis of silver nanoparticles using fruits shell extract

In a typical synthesis process, fruit shell extract causes the reduction of silver ions (Ag⁺) in an aqueous solution. It leads to the

formation of metallic silver (Ag⁰), which is further nucleated to form silver nanoparticles. The nanoparticle size increases or decreases on nucleation.²¹ Thus, to synthesize particles having a minimum size, synthesis parameters such as extract concentration and reaction time were optimized with silver nitrate concentration (1mM) constant. The color change is noted by virtual observation in *Sterculia foetida* fruit shell extract incubated with an aqueous sol of 1mM AgNO₃. The color changed from the extract color yellowish-brown due to the reduction of silver ions, thus exhibiting the formation of silver nanoparticles, as shown in Figure 2.

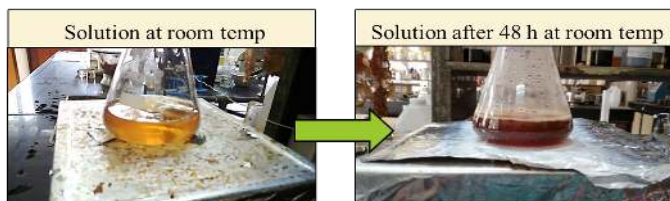


Figure 2 Colour change of the fruit shell extract after 48h.

Particle size analysis of reduced silver nitrate concerning the time

The results indicate that with increasing the concentration of the extract, particle size increased. This is attributed to increasing the rate of reaction, which caused higher nucleation of metallic silver leading to an increase in particle size. Hence, 10ml of extract concentration was taken as optimum for the synthesis of silver nanoparticles using *Sterculia foetida* fruit shell extract. Using a concentration of 10ml, the time of reaction was varied. The particle size decreased significantly with an increase in time. Larger particles are formed during longer reaction times due to the agglomeration of particles. Hence, to get smaller particles the time of reaction was optimized at 16 hours. The particle size distribution graph of the sample at a different time is shown in Figure 3 and Table 1.

The reaction for the synthesis of fruit shell extract-AgNPs was also carried out at 50ml of extract concentration with the time of response 16h at pH 7 and pH 10. The particle size distribution graph of the optimized sample at 50 ml concentration of FSE is shown in Figure 4 and Table 2. The distribution graph showed that the particles were uniform in size with an average particle size of 51 nm in pH 7 and 57nm in pH 10 for 16h.

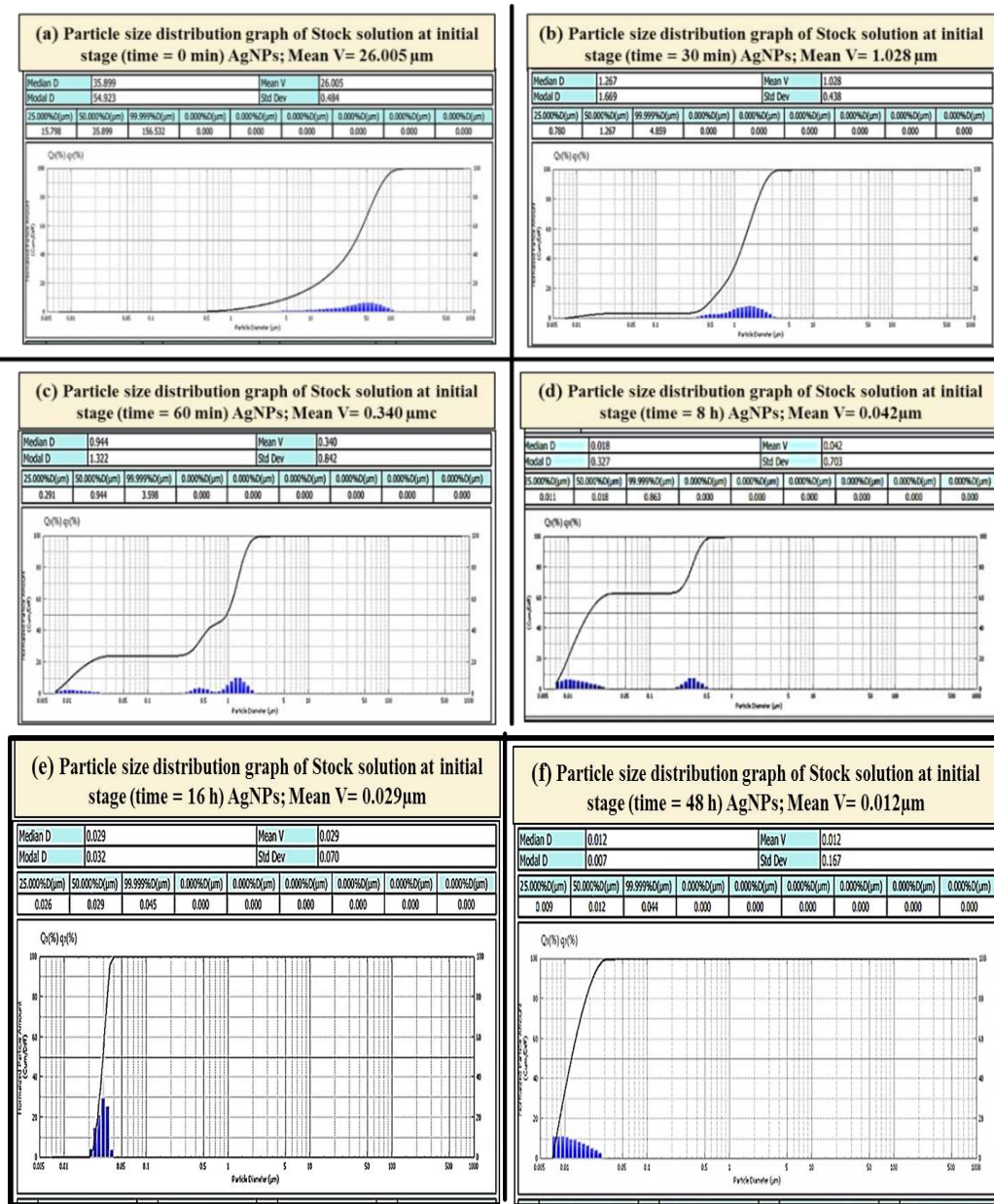


Figure 3 Particle size analysis of silver in fruit shell extract at (a) 0min (b) 30min (c) 60min (d) 8h (e) 16h and (f) 48h.

Table 1 Effect of different parameters on the particle size of silver nanoparticles synthesized using *Sterculia foetida* fruit shell extract (FSE)

Code	Extract conc. FSE (ml)	Time (min)	Mean particle size	Change in (pH)
			(μ meter)	
(a)	10	0	26.005	5.9-6.5
(b)	10	30	1.028	6.5
(c)	10	60	0.34	6.7
(d)	10	480 (8h)	0.042 (42nm)	6.8
(e)	10	960 (16h)	0.029 (29nm)	7
(f)	10	2880 (48h)	0.012 (12nm)	7

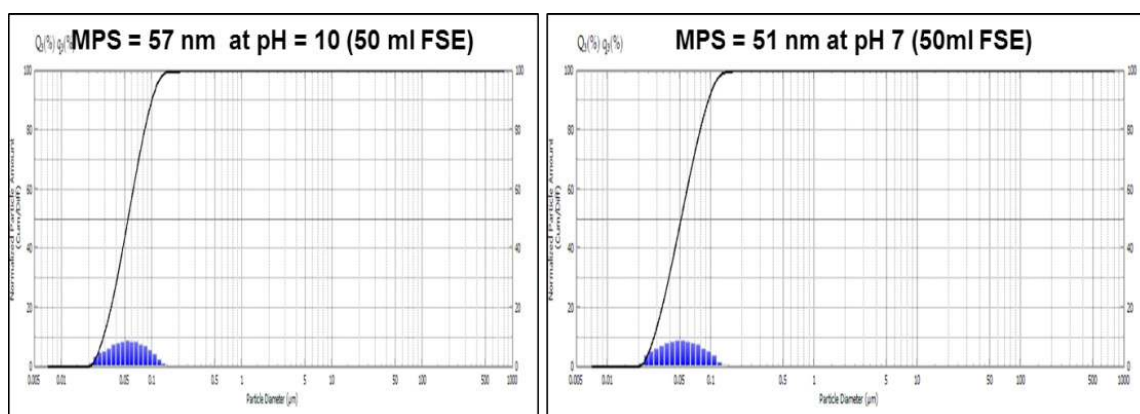


Figure 4 Particle size analysis of 50 ml of fruit shell extract at different pH for 16h.

Table 2 Mean particle size for optimized process parameters at different pH

Extract Conc. (ml)	Time (h)	Mean particle size (nm)	pH
10	16	29	7
10	16	63	10
50	16	51	7
50	16	57	10

Mean particle size results have been reported in Table 2 for 16 h and further work for dyeing and finishing effect on cotton fabric has been carried out at two different concentrations 10ml and 50 ml FSE in two different pH 7 and pH 10 at room temperature and 90°C for 1h.

UV visible absorption studies of silver nanoparticles

Measurement on UV–visible spectra is a technique to confirm the formation of metal nanoparticles.²² An absorption band was found for the silver nanoparticles in the range of 390–450nm, generated by surface plasmon vibrations of conducting electrons of silver. The UV-visible spectra of a colloidal solution of optimized 10% fruit shell extract concentration for 16h and 50% fruit shell extract concentration for 16h AgNPs are shown in Figure 5 and it showed a strong absorption band of 430nm, which is somewhat close to the reported work (Figure 6).

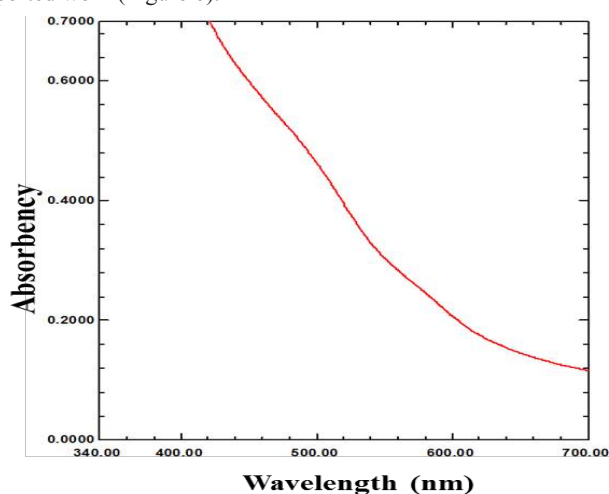


Figure 5 UV–visible spectra of *Sterculia foetida* fruit shell extract solution.

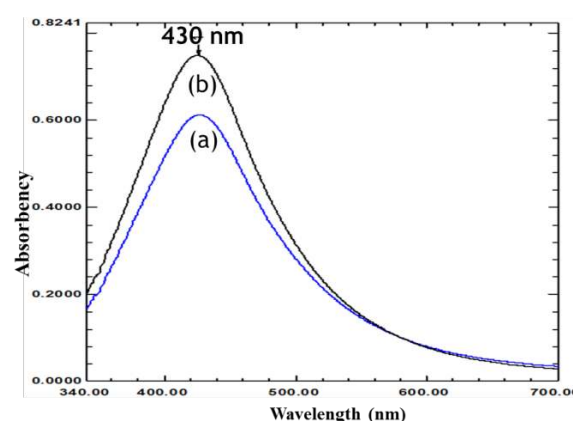


Figure 6 UV–visible spectra of (a) 10 % extract concentration for 16h and (b) 50 % extract concentration for 16h.

Relating these data with particle size, the absorption bands showed a redshift from 404 to 430nm due to an increase in particle size.²³ Absorption bands showed a single, sharp and intense peak suggesting the formation of mono-dispersed and spherical shaped nanoparticles. The color changed from the extracted color to yellowish-brown due to the reduction of silver ions, thus exhibiting the formation of silver nanoparticles. Fruit shell extract without silver nitrate solution did not show any color change. The color change of extract was seen after 16 at room temperature. Thus, the absorption spectrum at different wavelengths ranging from 340–700nm revealed a peak of λ_{max} at 430nm confirmed reduced silver nanoparticles.

Dyeing of cotton fabric

Dyeing of FSE synthesized silver nanoparticles of cotton fabric at room temperature for 1h: Coloration imparted for treated cotton fabric using optimized synthesized silver nanoparticles for 10% and

50% fruit shell extract was analyzed for the depth of shade concerning K/S and CIE Lab Parameters L^* , a^* and b^* when applied at room temperature for 1h and results are mentioned in Table 3. It is found from Table 3 that coloration is imparted at room temperature for 1h.

However, colour fading obtained were more after 5 washes. The highest colour retained (K/S=0.8156, pH 10) in the case of fabric treated with silver nanoparticles synthesized using 50 % *Sterculia foetida* as shown in Table 4.

Table 3 Dyeing of cotton fabric at room temperature for 1h before washing

Sample	pH	L^*	a^*	b^*	K/S
Cotton treated with 10% FSE-AgNPs	7	58.383	9.58	18.29	0.963
Cotton treated with 10% FSE-AgNPs	10	75.156	4.74	16.933	2.015
Cotton treated with 50% FSE-AgNPs	7	58.359	6.64	17.701	1.116
Cotton treated with 50% FSE-AgNPs	10	56.217	6.67	14.781	2.813

Table 4 Dyeing of cotton fabric at room temperature for 1h after 5 washing

Sample	pH	L^*	a^*	b^*	K/S
Cotton treated with 10% FSE-AgNPs	7	70.216	3.63	10.379	0.6335
Cotton treated with 10% FSE-AgNPs	10	69.959	3.24	10.235	0.5992
Cotton treated with 50% FSE-AgNPs	7	74.176	7.85	18.533	0.7791
Cotton treated with 50% FSE-AgNPs	10	73.669	7.83	18.162	0.8156

Assessment of fastness properties of the dyed cotton fabric (room temperature)

The fastness ratings of cotton fabric dyed with silver nanoparticles at different concentrations are presented in Table 5. Before washes,

it was found that washing fastness of the dyed cotton fabrics using *Sterculia foetida* fruit shell was moderate to good for cotton fabric. Light fastness was of the grade good to moderate for cotton fabric. However, after 10 washes overall fastness properties were found to be reduced.

Table 5 Fastness properties of dyed cotton fabric at room temperature for 1h

Dye conc., % owf	pH	Initial (before washes)				After 10 washes			
		Wash fastness	Light fastness	Rubbing fastness		Wash fastness	Light fastness	Rubbing fastness	
				Dry	Wet			Dry	Wet
Cotton treated with 10% FSE-AgNPs	7	3	5-6	5	5	2	5	5	4-5
Cotton treated with 10% FSE-AgNPs	10	3	5-6	5	5	2	5	5	4-5
Cotton treated with 50% FSE-AgNPs	7	3-4	4-5	5	5	2-3	4	5	4
Cotton treated with 50% FSE-AgNPs	10	3-4	4-5	5	4-5	2-3	4	5	4

Dyeing of FSE synthesized silver nanoparticles of Cotton fabric at 90°C for 1h

Coloration imparted for treated cotton fabric using optimized synthesized silver nanoparticles for 10% and 50% fruit shell extract was analyzed for the depth of shade concerning K/S and CIE Lab Parameters L^* , a^* and b^* for treatment at 90°C for 1h and results are mentioned in Table 6. It was found from Table 6 that coloration is imparted at 90°C for 1h and color decreases slightly after 5 washes. The highest color was obtained (K/S=2.1734, pH 10) in the case of fabric treated with silver nanoparticles synthesized using 50% *Sterculia foetida* extract. The result showed that there were slight changes in K/S values even after 5 washes whereas, at a room temperature treatment, the color loss was more, as shown in Table 7; this might be due to the unfixed dye molecule on the fabric.

Rubbing fastness was found to be in the range of 4-5 to 5 that indicates that it is in the range of very good to excellent for the dyed cotton fabric.

Assessment of fastness properties of the dyed cotton fabric at 90°C /1h

The Fastness ratings of cotton fabric using silver nanoparticle dyed in different dye concentrations are shown in Table 8. These results indicate the washing fastness of dyed cotton fabrics was good to very good in range. Light fastness was also found to be in the field of good to very good. Rubbing fastness is in the range of 4-5 to 5, i.e., very good to excellent for dyed cotton using silver nanoparticles. This indicates that dye fixed through dyeing may be caused by the creation of metal chelates in the presence of tannin which was present to a substantial extent in *Sterculia foetida* fruit shell extract. The tannins having

phenolic construction, promote the creation of metal chelate together and silver nanoparticles. Hence, dyed with silver nanoparticles these tannins present in *Sterculia foetida* fruit shell extract become insoluble in water and improve washing fastness properties.

Table 6 Dyeing of cotton fabric in rota dyer at 90°C for 1h before wash

Sample	pH	L*	a*	b*	K/S
Cotton treated with 10% FSE-AgNPs	7	58.652	9.097	17.706	0.902
Cotton treated with 10% FSE-AgNPs	10	74.569	4.6125	16.698	2.215
Cotton treated with 50% FSE-AgNPs	7	70.632	4.9582	11.515	1.125
Cotton treated with 50% FSE-AgNPs	10	58.261	8.214	17.877	2.3464

Table 7 Dyeing of cotton fabric in rota dyer at 90°C for 1h after 5 washes

Sample	pH	L*	a*	b*	K/S
Cotton treated with 10% FSE-AgNPs	7	58.864	8.463	17.804	0.7904
Cotton treated with 10% FSE-AgNPs	10	75.495	4.424	16.241	2.1243
Cotton treated with 50% FSE-AgNPs	7	70.672	4.4138	11.218	1.0236
Cotton treated with 50% FSE-AgNPs	10	57.216	7.0154	15.822	2.1734

Table 8 Fastness properties of dyed cotton fabric in rota dyer at 90°C /1h

Dye conc., % owf	pH	Washing fastness	Light fastness	Rubbing fastness	
				Dry	Wet
Cotton treated with 10 % FSE-AgNPs	7	4-5	5	5	5
Cotton treated with 10 % FSE-AgNPs	10	4-5	5-6	5	5
Cotton treated with 50 % FSE-AgNPs	7	4	5-6	5	4-5
Cotton treated with 50 % FSE-AgNPs	10	4	5-6	5	4-5

Ultraviolet protective properties

The Ultraviolet protection factor (UPF) values of the silver nanoparticles treated cotton fabric by the rota dyeing (exhaust) method dyeing and their durability of UV protection efficiency are shown in Table 9. Cotton treated with exhaust process at 90°C for 1h

offered excellent protection from UV radiation as compared to the untreated sample. The UPF values and protection data of the silver nanoparticles treated cotton fabric by rota dyeing showed adequate UV protection in a higher concentration of extract solution, as shown in Table 9.

Table 9 Ultraviolet protection properties for dyed cotton fabric

Parameters		Ultraviolet Protection Factor (UPF) 290–400nm			Protection Category
Sample	pH	Initial	After 5 washes	After 10 washes	UPF rating After 10 washes
Untreated cotton	-	4.49	4.49	4.49	No protection
Cotton treated with 10% FSE-AgNPs	7	18.51	17.21	15.31	Good
Cotton treated with 10% FSE-AgNPs	10	21.59	19.91	17.22	Good
Cotton treated with 50% FSE-AgNPs	7	29.42	26.56	25.34	Very Good
Cotton treated with 50% FSE-AgNPs	10	38.95	34.21	31.71	Very Good

UPF of untreated cotton fabric at 4.49 has no protective abilities and it allows transmission of solar radiation. There is an improvement of the UPF rating of cotton fabric after dyeing with synthesized nanosilver using fruit shell extract both in the case of pH 7 and pH 10 for 10 % and 50 % FSE concentration. Analysis of results in Table 9 reveals that cotton treated with 50% FSE-AgNPs at pH 10 showed maximum UPF value in an excellent rating even after ten washes.

Deep dyeing as a result of the existence of tannin also can help to increase the UPF rating. Additionally, the “good to excellent” UPF score after dyeing with *Sterculia foetida* fruit shell extract might be due to the coated layer on the dyed cotton fabric, which can be seen from the SEM image (Figure 7). EDS analysis also reveals the presence of an elemental peak of magnesium, silicon, phosphorus, calcium, sulphur, chlorine and phosphorus on dyed cotton fabric using fruit shell extract alone also supports the ultraviolet protection of cotton fabric.

Antibacterial test of *Sterculia foetida* fruit shell extract cotton fabric

The quantitative analysis of the percentage reduction in gram-negative bacteria was done for cotton using nanosilver dyed with fruit shell extraction, and results are given in Table 10. The results indicate that the dyed fabric using silver nanoparticles inherently showed excellent antibacterial property against *E. coli*. After five and ten washes, this level of colonies reduction percentage gradually decreased. Compared to *S. aureus* good results were obtained for *E. coli* bacteria. As seen in either the concentration after five washes and yet the overall reductions in colony growth were greater than 85% both for *S. aureus* and *E. coli*.

The observed antibacterial property of the treated cotton fabric is due to the presence of silver, which is well known and reported

in the literature. Moreover, it showed excellent results might be due to the tannins present in fruit shell extract, which was discovered to be 4.71 %. These polyphenolic compounds that bind to cellulosic material have been shown to have antibacterial action. The presence of flavonoids, terpenoid, and saponin, was also found to be present in fruit shell extract act as an antibacterial agent against *S. aureus* and *E. coli*. The EDS analysis also showed that *Sterculia foetida* fruit shell extract comprises metal ions like chromium, nickel, magnesium, calcium, potassium along with chlorine and silicone molecules which may be responsible for getting suitable antibacterial property even after ten washes for dyed cotton fabric.

SEM-EDS analysis

SEM micrograph of untreated and treated cotton fiber is depicted in Figure 7. As exhibited in Figure 7(a), the cotton fiber had a smooth and clear surface free of surface imperfections. After the treatment of the synthesized silver nanoparticles depicted in Figure 7(b) to Figure 7(e) showed the top layer of the material has the residue of the coating. It was found from the 50 % FSE-AgNPs at pH 10 from Figure 7(e) that treated cotton fabric surfaces have more deposition as a coated layer compared to treated with 10% FSE. A coated layer on the treated cotton might help for good ultraviolet protection and antibacterial properties.

Elemental peaks of *Sterculia foetida* fruit shell extract using its atomic and weight percentages are reported in Table 11. *Sterculia foetida* fruit shell extract showed several elemental peaks of carbon, oxygen, magnesium, chromium, chromium, calcium, potassium, sulphur, chlorine and phosphorus. The components within the dried fruit shell extract might be in the form of either metallic oxide or metallic chloride. It has been found that the fruit shell extract along with nano silver particles dyed cotton fabric might also be responsible for the excellent antibacterial and UV protection properties.

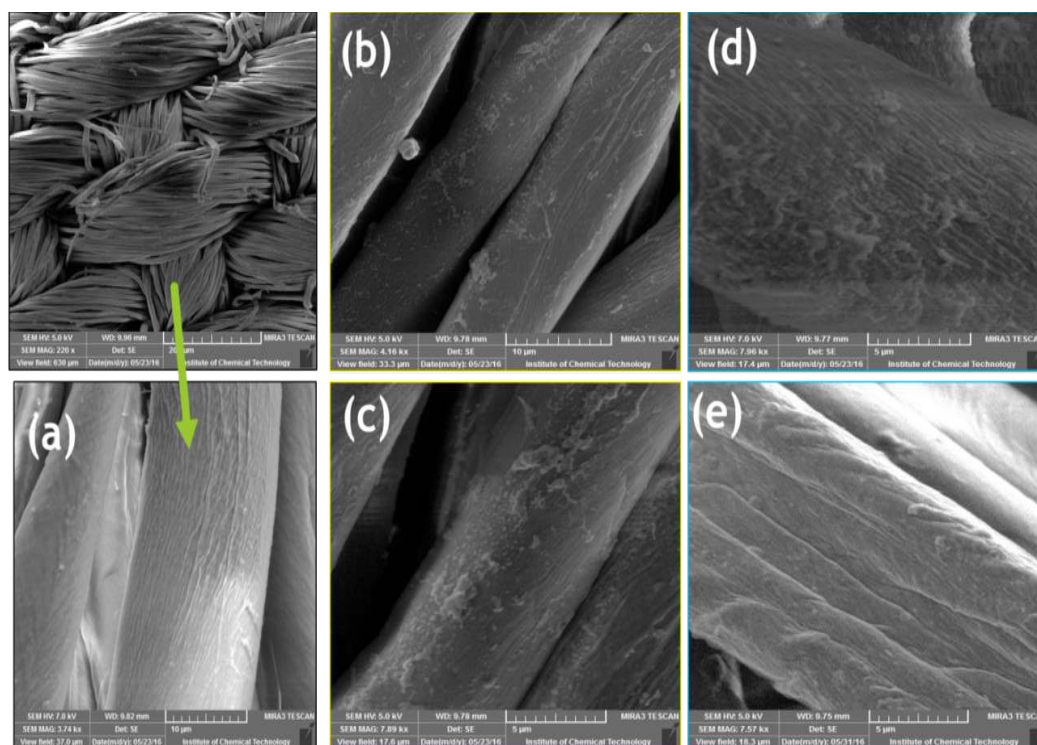


Figure 7 SEM analysis of (a) untreated and treated cotton fabric for (b) 10% FSE-AgNPs at pH 7 (c) 10% FSE-AgNPs at pH 10 (d) 50% FSE-AgNPs at pH 7 (e) 50% FSE-AgNPs at pH 10.

Table 10 The antibacterial property of the dyed cotton fabric

Sample	pH	Reduction (%) <i>E. Coli</i>			Reduction (%) <i>S. Aureus</i>		
		Initial	After 5 washes	After 10 washes	Initial	After 5 washes	After 10 washes
Rota dyer at 90°C/1h							
Cotton treated with 10% FSE-AgNPs	7	90.06	86.89	84.14	92.06	89.34	83.45
Cotton treated with 10% FSE-AgNPs	10	92.96	88.44	86.65	94.96	91.32	87.56
Cotton treated with 50% FSE-AgNPs	7	97.67	94.63	88.73	96.67	92.32	89.43
Cotton treated with 50% FSE-AgNPs	10	98.34	96.21	91.42	99.14	94.56	90.87

Table 11 EDS analysis of dried fruit shell extract for quantification of elements

Quantification of elements	<i>Sterculia foetida</i> fruit shell extract	
Element	Weight (%)	Atomic (%)
Carbon, C	21.96	33.18
Nitrogen, N	--	--
Oxygen, O	44.01	49.91
Sulphur, S	0.59	0.33
Sodium, Na	0.38	0.3
Silicon, Si	0.41	0.26
Phosphorus, P	0.81	0.47
Potassium, K	26.96	12.51
Calcium, Ca	1.02	0.46
Chlorine, Cl	1.32	0.67
Magnesium, Mg	2.54	1.9

Conclusion

The present work demonstrated that *Sterculia foetida* fruit shell extracts were capable of producing Ag⁰ from an aqueous solution of Ag⁺. Reduction of silver ion (Ag⁺) to metallic silver nanoparticles (Ag⁰) was done at room temperature and the minimum sizes of synthesized silver were 15nm. UV-visible spectroscopy and FTIR characterized the reduced silver nanoparticles. Cotton was dyed with the *Sterculia foetida* fruit shell extract using synthesized silver nanoparticles. The treated fabric was characterized for K/S, color fastness to washing, light, and rubbing and the results were found to be very good at 90°C for 1h. Treated cotton fabric using synthesized nanoparticles showed excellent antibacterial and ultraviolet protection properties both at room temperature and at 90°C for 1h. However, after 10 washing fabrics treated in the rota dyeing method at 90°C for 1h found to achieve very good antibacterial and UV protective properties. Hence, *Sterculia foetida* fruit shell extract is promising enough to be used as a green reducing agent for the synthesis of nanoparticles with good natural coloration and multi functionalised textile fabrics.

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Conflicts of interest

The authors have no conflicts of interest regarding the publication of this paper.

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