



Wet chemical preparation of herbal nanocomposites from medicinal plant leaves for enhanced coating on textile fabrics with multifunctional properties

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Abstract

The present study is aimed at the synthesis of different herbal nanoparticles from *Acalypha indica*, *Azadirachta indica*, *Piper betle*, *Tridax procumbens* and *Aloe vera* plant leaves through wet processing method i.e., solution based synthesis for the development of a biocompatible nanomaterials with excellent medicinal properties. The efficiency of herbal nanoparticles are determined by subjecting a comparative assessment among the various herbal nanoparticles in virtue of their physico-chemical as well as their functional properties when coated on cotton fabrics. The prepared five different herbal nanoparticles show amorphous nature having an average particle size distribution ranges from 21 to 27 nm. *Tridax procumbens* nanoparticles exhibit higher antioxidant activity (98.17%) while tested against DPPH assessment. Nevertheless, the herbal nanocomposites prepared from *Aloe vera* with chitosan polymer shows higher protection (UPF = 62.3) than the fabric coated with other herbal nanoparticles. The superhydrophobic properties (154.5°) and higher antibacterial properties against *Escherichia coli* (33.13 mm) and *Staphylococcus aureus* (35.62 mm) for the *Azadirachta indica* nanocomposite coated cotton fabrics shows comparably more effectiveness than that of the other counterpart. The present study helps to identify the appropriate processing methods as well as herbal nanoparticles for enhanced the self-cleaning, UV-protection, antibacterial and antioxidant activity in biomedical textiles.

Keywords Herbal nanoparticles · Cotton fabrics · Antimicrobial activity · UV-protection · Antioxidant activity · Hydrophobicity

1 Introduction

Nanotechnology, the study of the technological aspects of nanoparticles is playing a major role in terms of textile due to the ease of surface modification of these materials [1] and its higher surface to volume ratio [2]. The growing public awareness of contagious pathogens facilitates

the need of new surface modification of cotton fabrics, because of its large surface area and moisture retainment ability resulting in an excellent medium for microbial growth [3, 4]. The development of new cotton fabrics based nanocomposites to wide spectrum applications in different fields such as antimicrobial [5], wound healing [6], water repellence [7, 8], hygienic application [9]

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and UV-protection clothing [10, 11]. Synthetic chemical compounds like Triclosan (2, 4, 4-hydrophenyl trichloro (II) ether), quaternary ammonium compounds, and metallic compounds are used in textile industries to impart antibacterial activity on cotton fabric [12]. Chemical antibacterial finishing prevents microbial growth, but it also has some negative characteristic features such as toxic, non-biodegradable, non-eco-friendly, and cost effective, etc. Hence, it is necessary to change non-toxic, non-allergic, eco-friendly resources for textiles with antibacterial finishing. The abovementioned aspect is possible by use of biological materials particularly with herbal plants. Additionally, the utilization of plant extracts and bioactive compounds encompasses a range of antibiotic properties, which is conventionally utilized in therapeutic treatments. Active compounds present in the phytoconstituents of plants also helps in the inhibition of growth of microbes [13].

Acalypha indica (*A. indica*) of the genus *Acalypha* belongs to euphorbiaceae family is a small annual shrub which generally occurs as a troublesome weed in garden or roadside and throughout plains in India and has being used in South India over decades as a traditional medicinal plant [14, 15]. The *A. indica* plant consists phytochemical compounds like 1H-Pyrrole-2, 5-dione, 1-ethenyl-, Cysteine, 3, 8-Nanodiene-2-one, (E)-, Proline, 3,4-didehydro-, 4-Amino-3-methoxypyrazolo [3,4-d] pyrimidine, Propanenitrile, 3-(5-diethylamino-1-methoxy-3-pentynyl)- and Kaempferol compounds are recorded through GC-MS [16]. Hence, the *A. indica* plant components is chosen for active textile coating applications as a functional nanocomposite reported by Saravanan et al. and Karthik et al. [17, 18].

Azadirachta indica (*Az. indica*), is a neem tree belongs to the family Maliaceae. It is a well known plant showing versatile and wide spectrum biological activities. In Indian medicinal codex, Azadirachtina steroid-like tetranortriterpenoid (limonoid) in neem plant is one of the main reasons for insecticidal activity [19, 20]. Along with it, other phytochemicals in neem plant is also reported to show antibacterial properties like isoprenoids, carbohydrates, sulphurous compounds; polyphenolics such as flavonoids and glycosides, tannins, and aliphatic compounds [21]. In addition, some tetranortriterpenes, including nimbin, nimbinin, nimbidinin, nimbolide and nimbidic acid have been isolated from neem leaves [22]. The chemical compound of nimbolide also shows antibacterial activity against *Staphylococcus aureus* (*S. aureus*) and *Staphylococcus coagulase* (*S. coagulase*) [23]. In addition, *A. indica* leaf used for nano herbal coated cotton fabrics developed by Muhammad et al. and Karthik et al. [13, 24].

Piper betle (*P. betle*) is one of the most commonly used masticatory leaves in Asian countries like India due to its

strong pungent aromatic flavour [25]. GC-MS study of the *P. betle* shows more than 32 different components whereas it is seen that eugenol and its ester derivative acetyleugenol are the major biomolecules. But some researchers stated that phenolics like chavibetol and chavibetol acetates, safrol and 4-allyl-2-methoxy-phenolacetate, 3-allyl-6-methoxyphenol are the prime components that can be seen in the plant [26].

Tridax procumbens (*T. procumbens*) is commonly known as coat buttons of genus *Tridax* and family *Tridax* is one of the most common traditional household medicinal remedies in South India. Attempts are made to show its antidiabetic property antioxidant property as well as its potential usage to evaluate hypoglycemia and antihypoglycemia conditions [27, 28].

The other plant assessed in this study is *Aloe vera* (*A. vera*) a species from genus *aloe*. The most important chemical compounds which are biological activities are namely n-Hexadecanoic acid, Oleic acid, 1,2-Benzenedicarboxylic acid, diisooctyl ester, Squalene, 1-Heptanol, 2-propyl, 1,2-Benzenedicarboxylic, butyl octyl ester, and Tetradecanoic acid. *A. indica* and *A. vera* leaf extracts are responsible for the biological activities like antimicrobial, anti oxidant, anti-inflammations, pesticides, etc. [29, 30].

Polysaccharides obtained from the inner leaf parenchymatous tissue is being attributed to many medicinal effects [31]. Researchers have also reported and isolated phytochemicals such as Aloe-emodin, aloetic-acid, anthranol, aloin A and B, isobarbaloin, emodin, polymanans which results in the plants antibacterial activities [32, 33]. *Aloe vera* nanoparticles chitosan composite coated fabric for developed by Karthik et al. [34]. Due to the aforementioned reason, *A. vera* is one of the versatile plants for this study.

Cotton fabrics can be modified and improved by use of suitable surface finishing with nanomaterials [18, 35]. Chitosan have diverse properties like non-toxic, biodegradable and biocompatible and substitute for other chemicals [36, 37]. Hence, it is aimed to develop herbal nanocomposite coated fabrics for enhanced functional and medical properties. Application of herbal plants and its extracts in medical field and textile is one of the fascinating approaches to confer multi functionalised cotton fabrics development. Polymer based nanocomposites like chitosan are widely used to produce biocompatible and adhesive coating on fabrics.

In the present investigation, *A. indica*, *Az. indica*, *P. betle*, *T. procumbens* and *A. vera* plant leaves nanoparticles are obtained by wet processing method (solution processing). The prepared five different plant leaves nano powders are subjected to optimization of their properties through XRD, FT-IR spectroscopy, UV-Vis spectroscopy, Particle size analyser (DLS), SEM-EDX and TEM studies.

The herbal nanoparticles and their herbal nanocomposite coated fabric is assessed for antibacterial properties against *Escherichia coli* (*E. coli*) and *Staphylococcus aureus* (*S. aureus*) and antioxidant activity was obtained by using DPPH assay. Functional properties such as UV protection and water repellence were carried out for herbal nanocomposite coated fabrics. Therefore, our study will help to understand and model the nanocomposites coated fabrics with exotic functional and biomedical properties for biomedical textile industries.

2 Materials and methods

2.1 Samples collection

Clean and fresh plant leaves of *A. indica*, *Az. indica*, *P. betle*, *T. procumbens* and *A. vera* were collected from different places of Tiruchengode area, Tamil Nadu, India. The collected plants leaves were separately washed thoroughly with tap water and then double distilled water to remove dust particles on the surface of the plant leaves. Following the washing process, the obtained plant leaves were shade dried for 15 days in dust free environment at room temperature.

2.2 Synthesis of herbal nanoparticles

To get a coarse powder, the shade dried *A. indica*, *Az. indica*, *P. betle*, and *T. procumbens* leaves were grounded using domestic grinder. To further decrease the size of the herbal particles, Wet-Chemical method was used where the coarse ground powder was added to a solution containing 50% H_2SO_4 and a mixture of $KMnO_4$ (0.1 M) and Sodium nitrate (0.1 M) under vigorous stirring. The formed solution was dried at 80 °C and then, the *A. indica*, *Az. indica*, *P. betle*, and *T. procumbens* nanoparticles were obtained (hereafter termed as AINPs, AZINPs, PBNPs, and TPNPs respectively), were used for further characterization.

2.2.1 vera herbal nanoparticles

The dried gel of the *A. vera* plant was grinded to obtain a coarse grained particles using domestic grinder. The obtained coarse ground *A. vera* particles were further treated with a solution containing 50% H_2SO_4 and a mixture of $KMnO_4$ (0.1 M) and sodium nitrate (0.1 M) under vigorous stirring to further reduce the particle size. The prepared *A. vera* nanoparticles are hereafter termed as AVNPs and it was used for further characterization.

2.3 Characterisation

Powder X-Ray diffraction technique was used to determine the phase purity and crystalline nature of all the herbal nanoparticles. X-ray diffractometer having a long fine focus of Cu anode at 40 kV and 30 mA operating in Bragg–Brentano geometry was used to acquire the XRD spectrum of the plant herbal nanoparticles. The XRD spectra acquired were in a 2θ range between 10° to 80° having a step size of 0.02° (2θ). The FTIR spectra of all the prepared herbal nanoparticles was obtained using Fourier Transform Infrared Spectrophotometer (Spectrum 100; PerkinElmer, USA) having a range of 4000–400 cm^{-1} using KBr as a reference matrix. KBr and the herbal nanoparticles were mixed in a concentration ratio of 200:1 to obtain the KBr matrix. This matrix was used to analyse the functional groups of the herbal nanoparticles.

UV–visible spectrophotometer (Cary 8454, Agilent, Singapore) operating in the UV near to IR spectral region (180–800 nm) was used to record the absorption spectra of the herbal nanoparticles. The samples used for the analysis was prepared by diluting 0.1 mL of the sample in a cuvette to 2 mL of deionising water. A sub-micrometer particle size analyser (Nanophox, Sympatec, Germany) was used to determine the particle size distribution of the prepared nanoparticles using Dynamic light scattering method in a size range of 1–1000 nm at a scattering angle of 90°. The qualitative analysis of the herbal nanoparticles was made using an X-ray Fluorescence spectrophotometer (EDX-720; Shimadzu, Japan). To determine the morphological, microstructure and the elemental composition of the prepared herbal nanoparticles, a Scanning electron microscope combined with Energy dispersive X-ray Spectroscopy (JSM 6360; JEOL, Japan) was used. Crystallite size and surface morphology of all the prepared herbal nanoparticles was observed under the transmission electron microscopy (TEM, CM200; Philips, Eindhoven, The Netherlands) operated at a potential of 120 kV. The average particle size and diffraction pattern of the herbal particles was also refined from TEM analysis.

2.4 Preparation of herbal nanocomposites

Chitosan solution was prepared by adding 1% of chitosan (Himedia, India (GRM9358)) dissolved in 1% acetic acid under continuous stirring. Then, it was kept overnight so as to remove the bubbles formed during stirring. To the obtained chitosan solution (100 mL), 5 g of the herbal nanoparticles (AINPs, AZINPs, PBNPs, TPNPs and AVNPs) were dispersed separately under constant stirring until a clear homogeneous solution was obtained. The prepared herbal nanocomposites prepared from the different herbal nanoparticles (AINPs, AZINPs, PBNPs, TPNPs and AVNPs)

are respectively named as AI-NC, AZI-NC, PB-NC, TP-NC and AV-NC.

2.5 Herbal nanocomposites coating on cotton fabrics

The prepared herbal nanocomposites were coated on a fine and medium weight bleached 100% cotton woven fabric (plain weave 138.84 g m^{-2} , 116 ends per inch, and picks per inch 84). $60 \times 60 \text{ cm}$ pieces of cotton fabrics (CF) were cut and used for the coating applications. Herbal nanocomposites solutions were coated on the cotton fabrics using Pad-dry cure method as per the method described in Karthik et al. [38]. The cotton fabrics separately coated with the different herbal nanocomposites are named as AI-NC-CF, AZI-NC-CF, PB-NC-CF, TP-NC-CF and AV-NC-CF respectively. The coated cotton fabrics were dried for 20 min at $80 \text{ }^\circ\text{C}$ and used for the comparative analysis of its functional properties.

2.6 UV protection on cotton fabrics

UV transmission spectra (Lambda 35; Perkin Elmer, USA) were used to test the UV blocking property of the herbal nanocomposites coated fabrics with the wavelength region from 280 to 400 nm as reported from our previous studies [39].

2.7 Antimicrobial studies

2.7.1 Collection of microorganisms and culture preparation

Bacterial culture of Gram-positive *S. aureus* (ATCC 6538P) and Gram-negative *E. coli* (ATCC 9677) were obtained from the National Collection of Microorganisms (NCM), National chemical laboratory, Pune, India. Subsequent to the sub-culturing at room temperature at $37 \text{ }^\circ\text{C}$ for 24 h, the bacterial cultures were maintained at $4 \text{ }^\circ\text{C}$ in a nutrient agar medium (M001, HiMedia, Mumbai, India) for further experiments. Mueller–Hinton agar (M391, HiMedia, India) medium was prepared and plated. Bacterial inoculums was prepared by introducing a loopful of test organisms into nutrient broth and incubated at $37 \text{ }^\circ\text{C}$ for 4–5 h till a moderate turbidity was developed. A loopful of the bacterial inoculums was swabbed on the prepared Mueller–Hinton agar plate to be assessed for the antibacterial properties of fabrics.

2.7.2 Agar well diffusion assay

The qualitative antibacterial assessment against *S. aureus* and *E. coli* was performed using disc diffusion method [40].

The formation of the zone of inhibition for the prepared nanoparticles at different mass concentrations 25, 50, and 100 mg mL^{-1} was recorded. The un-coated and nanocomposites (AI-NC-CF, AZI-NC-CF, PB-NC-CF, TP-NC-CF and AV-NC-CF) coated cotton fabrics were cut into pieces in the standard size (10 mm). The obtained specimen is gently pressed transversely across the inoculums of streaks. The plates were incubated at $37 \text{ }^\circ\text{C}$ for 12–24 h.

2.7.3 Percentage reduction test

The un-coated (CF) and herbal-nanocomposites coated fabrics (AI-NC-CF, AZI-NC-CF, PB-NC-CF, TP-NC-CF and AV-NC-CF) were cut into different pieces as recommended by American Association of Textile Chemists and Colonists (AATCC 100) standard size (10 mm radius) with round shape. The coated test samples were saturated in a sterile bacteriostatic broth and the test organisms was inoculated [41]. The soaked coated samples were incubated at $37 \text{ }^\circ\text{C}$ for 18–24 h, diluted appropriately and 0.1 mL of sample from each dilution was plated on the sterile AATCC bacteriostasis agar plates by spread plate method and again incubated at $37 \text{ }^\circ\text{C}$ for 24 h. After incubation, final concentration of cell in control and the test samples were calculated using viable cell count method. As the final number of existing cells (B) much lower than the initial cell concentrations (A) is calculated through the viable cell count method [34]. The percentage of bacterial reduction was calculated using the following formula:

$$\% \text{ Bacterial Reduction} = \left[\frac{(A - B)}{A} \right] \times 100 \quad (1)$$

2.8 Antioxidant activity

2.8.1 Herbal nanoparticles

DPPH assessment was performed to analyse the dose dependent antioxidant behavior of the prepared herbal nanoparticles (AINPs, AZINPs, PBNPs, TPNPs and AVNPs) as reported earlier [42]. 50 μL of the different dilution (5, 10, and 100 mg) of the prepared herbal nanoparticles was added to 0.001 M methanol solution of DPPH (di (phenyl)-trinitrophenylimino azanium). After an incubation period of 1 h, the sample absorbance was calculated at 517 nm using an UV–Vis spectrometer (Cary 8454, Agilent Technologies, Singapore).

$$\text{DPPH Scavenging (\%)} = \frac{CA - TA}{CA} \times 100 \quad (2)$$

where CA and TA are control absorbance and test absorbance respectively. For the assessment 1.95 mL of the prepared radical solution was added to 50 μL antioxidant

solution loaded with various doses of different herbal nanoparticles such as AINPs, AZINPs, PBNPs, TPNPs and AVNPs. At first the excitation intensity of the DPPH is measured using UV–Vis spectrometer (Agilent technologies, Cary 8454; Agilent, Singapore) having wavelength between UV to Visible range of light then the antioxidant solution was added and the intensity is calculated. Then, the comparative assessment of antioxidant activity of the various plant herbal nanoparticles was done by determining the decrease in absorbance intensity at different concentrations by using the equation already explained.

2.8.2 Herbal nanoparticles chitosan nanocomposite coated cotton fabrics

The un-coated (CF) and herbal-nanocomposites coated fabrics (AI-NC-CF, AZI-NC-CF, PB-NC-CF, TP-NC-CF and AV-NC-CF) were cut into 2 cm × 2 cm and kept in 0.001 M methanol solution of DPPH under dark condition. After an incubation period of 1 h, the sample absorbance was calculated at 517 nm using an UV–Vis spectrometer (Cary 8454, Agilent Technologies, Singapore). Without fabric served as blank. All samples analyses carried out using triplicates. DPPH scavenging activity calculated using following formula [43]:

$$\text{DPPH Scavenging (\%)} = \frac{C - S}{C} \times 100 \quad (3)$$

where as C—control (CF) and S—sample (herbal-nanocomposites coated fabrics).

3 Results and discussion

3.1 Characterisation of herbal nanoparticles

The prepared five different herbal nanoparticles (AINPs, AZINPs, PBNPs, TPNPs, and AVNPs) are extensively characterised for their physico-chemical properties. Then, they are ascertained their antimicrobial and antioxidant property herbal nanoparticles for various biomedical applications. The XRD pattern of the prepared herbal nanoparticles namely, AINPs, AZINPs, PBNPs, TPNPs and AVNPs are shown in Fig. 1. The absence of any sharp crystalline diffraction peaks except the observed broad band at the 2θ value ranging from 20 to 30° confirms that the prepared are amorphous in nature.

The FTIR spectra of the AINPs, AZINPs, PBNPs, TPNPs and AVNPs samples show similar peaks with little dissimilarity and absence of change in the wave numbers of the IR peaks infersto the presence of similar functional groups as it can be evidently seen from the Fig. 2. The broad peak observed at 3401 cm^{-1} is assigned to the presence of

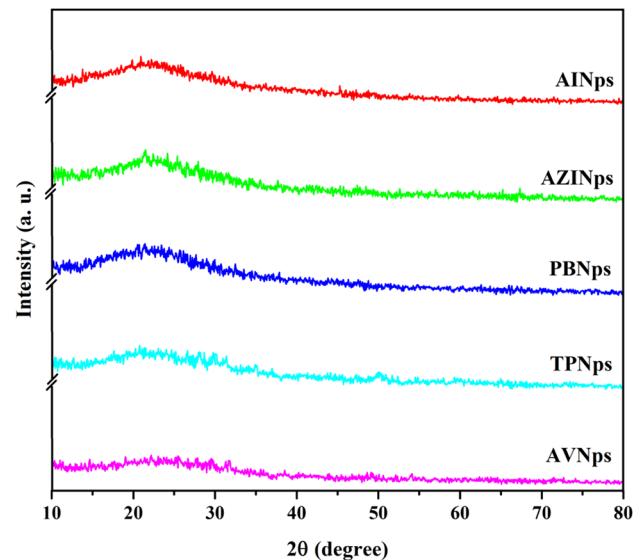


Fig. 1 XRD spectra of different herbal nanoparticles

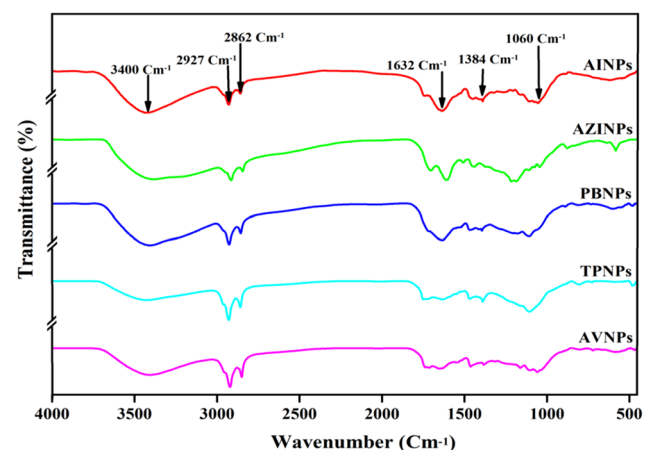


Fig. 2 FTIR spectra of five different leaf nanoparticle

superficially absorbed water or stretching mode of OH/ NH group. The peaks seen at 2927 cm^{-1} and 2862 cm^{-1} resembles the asymmetric stretching vibration of aliphatic and aromatic C–H groups, possibly terpenoids which is present in *A. indica* as reported by Kokila et al. [42] which confirms the antimicrobial property [44]. Comparatively, narrow bands at 1716 cm^{-1} corresponds to the presence of carbonyl functional groups such as ketones, aldehyde, and carboxylic acid and the peak observed between 1630 and 1660 cm^{-1} is assigned to the carbonyl stretching vibration of the amide group in plant proteins. The peaks observed at 1384 cm^{-1} attributes to the presence of flavonoids, indicating good antimicrobial activity. Along with the aforementioned peaks the herbal nanoparticles shows additional peaks such as, 1060 cm^{-1} corresponds to

C–N stretching and vibration respectively [45]. The study shows that the herbal nanoparticles synthesised through wet chemical route shows similar functional groups as that of the herbal nanoparticles synthesised through ball milling in our previous study [18]. Hence, it can be inferred that the change in the synthesis process does not result in alteration of the functional group of the herbal nanoparticles, resulting in exhibition of various properties such as antibacterial, antioxidant, etc. by the herbal nanoparticles.

Figure 3 shows the UV–visible spectra of the prepared AINPs, AZINPs, PBNPs, TPNPs and AVNPs dispersed in DD water show high absorbance at 355, 269, 253, 264 and 289 nm respectively. From the graph, it can be observed that the AVNPs plant shows higher UV absorbance than that of other (AINPs, AZINPs, PBNPs, TPNPs) herbal nanoparticles confirming as an efficient UV-protective material. Alike other inorganic nanoparticles such as silver, gold, and ZnO, these herbal nanoparticles also show high UV-protective property, and antimicrobial property but with lesser toxicity than that of conventional inorganic nanoparticles [46].

The details of particle size distribution of herbal nanoparticles prepared via ball milling techniques are given in Fig. 4. The DLS assessment of herbal nanoparticles shows almost same average particle size distribution. The observed particle size distribution of the prepared herbal nanoparticles AINPs, AZINPs, PBNPs, TPNPs and AVNPs are 21, 22, 26, 28 and 27 nm respectively.

SEM and TEM images of the AINPs, AZINPs, PBNPs, TPNPs and AVNPs are shown in Figs. 5 and 6 respectively. Topographical characterization of the prepared nanoparticles observed in TEM shows that the herbal nanoparticles (AINPs, AZINPs, PBNPs, TPNPs and AVNPs) are uniform in

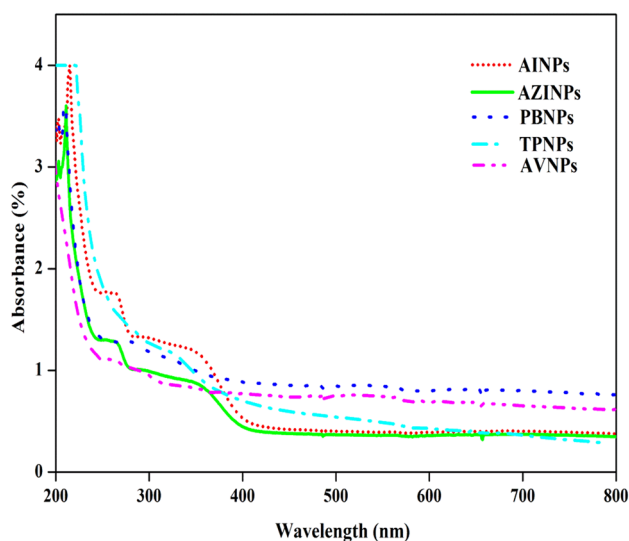


Fig. 3 UV–visible spectra of five different leaf nanoparticles

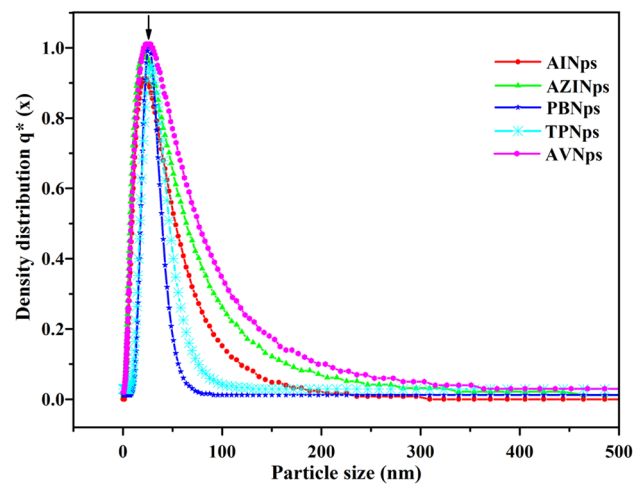


Fig. 4 Particle size distributions of different leaf nanoparticles

structure. SEM image of the nanoparticles shows discrete distribution of herbal nanoparticles at higher magnification. The elemental composition of the herbal nanoparticles performed using an Energy dispersive X-Ray spectroscopy (Fig. 5b) shows that the nanoparticles consists of C, Ca, K, and O ions which shows the presence of inorganic constituents in the plant nanoparticles.

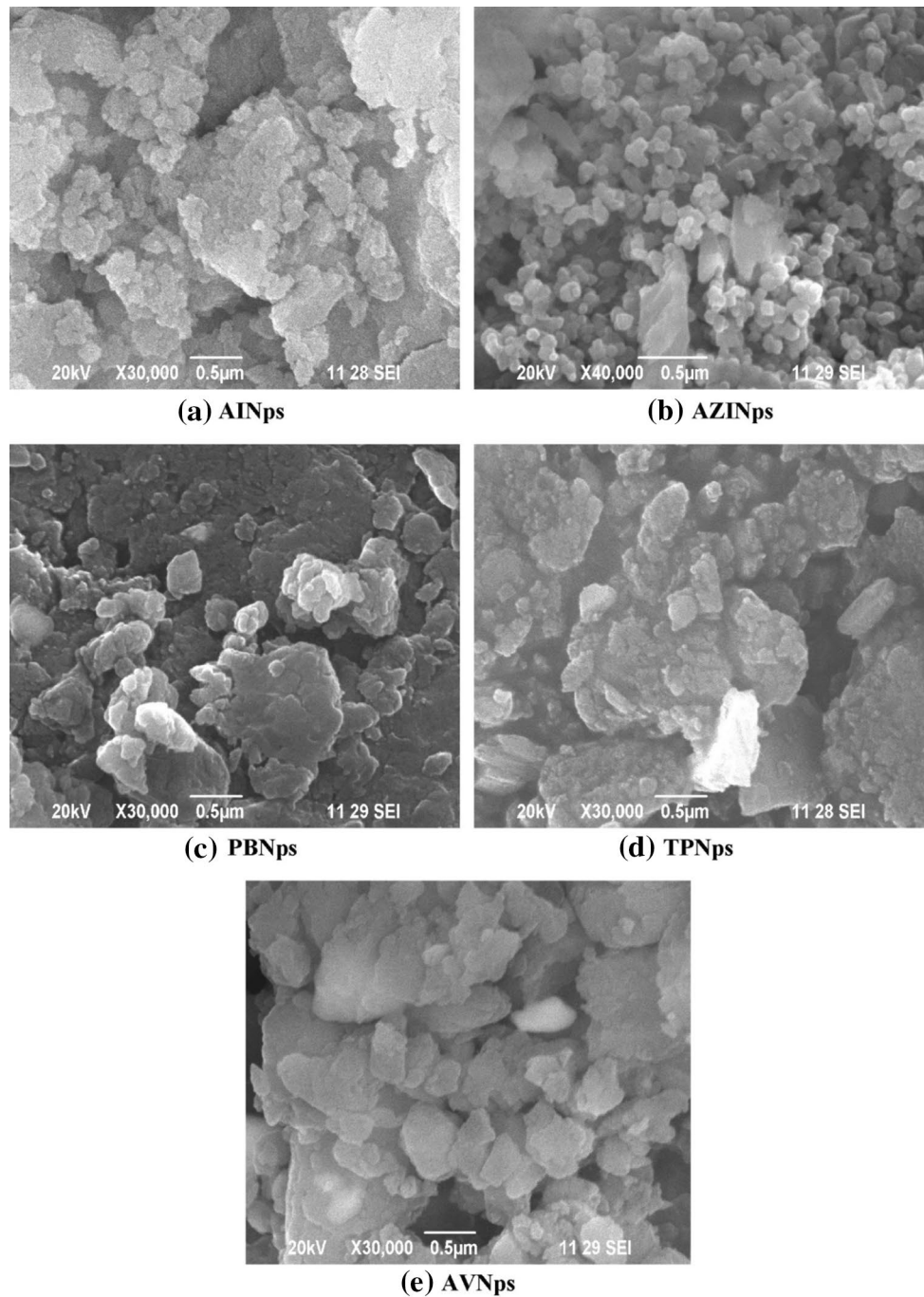
Along with the aforementioned fact, it can be seen from the SEM and TEM images that the herbal nanoparticles from the *A. indica* only tends to show distinct spherical nanostructure where as the other nanoparticles sample shows no distinct structures.

Furthermore, the nanoparticles synthesised from the *A. indica* plant only shows uniform size distribution. The high surface to volume ratio of the herbal nanoparticles synthesised from *A. indica* plant due to its spherical structure can result in its enhanced physico-chemical property than that of the other herbal nanoparticles. The average particle size of the herbal nanoparticles analysed using the TEM is about 50 nm for AINPs, AZINPs, PBNPs, TPNPs and AVNPs which is close to the value obtained from the DLS study.

3.2 Antibacterial property

The assessment of the antibacterial activity of the prepared nanoparticles are carried out using well diffusion method by determining the inhibition zone formed against gram positive and gram negative bacteria at different particle concentrations, (25, 50 and 100 mg mL⁻¹) and they are tabulated in Table 1. The agar well containing the highest concentration (100 mg mL⁻¹) of the herbal nanoparticles of i.e. AINPs, PBNPs, TPNPs and AVNPs shows the maximum zone of inhibition against the *S. aureus* and *E. coli*. It can be evidently seen from the Table 1 that the inhibition zone of the prepared herbal

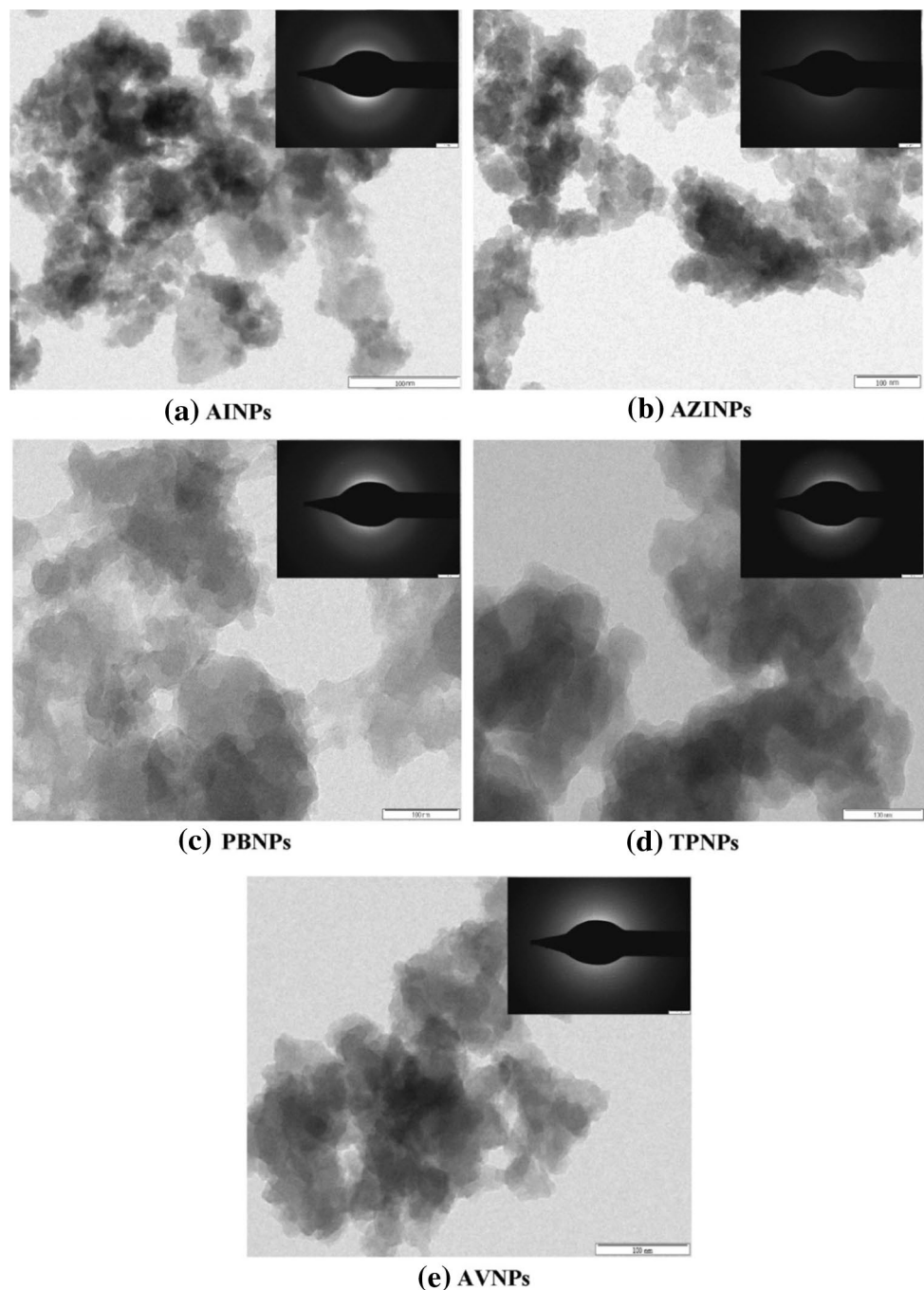
Fig. 5 SEM images of five different herbal nanoparticles



AZINPs is slightly higher than that of the other herbal nanoparticles (AINPs, PBNPs, TPNPs and AVNPs) against *E. coli* (24.55 ± 0.52 mm) and *S. aureus* (28.53 ± 0.72 mm). This might be due to the fact that the AZINPs consist of phytochemical compounds responsible for synergistic action for exhibiting higher antimicrobial property than other four herbal nanoparticles. Furthermore, the spherical morphology seen in the AZINPs herbal nanoparticles might also be responsible for the enhanced antibacterial activity.

The existence of spherical morphology in case of the herbal nanoparticles acquired from the *A. indica* helps in the easy penetration of the bacterial cell wall. This in turn, causes a change in the chemical constituents in the bacteria resulting in the restriction of DNA replication. Hence, it leads to the formation of higher inhibition zone than that of the other nanoparticles. And also the phytochemical present in *A. indica* might have a more effective antibacterial activity than that of the other nanoparticles synthesised.

Fig. 6 TEM and SAED patterns images of five different herbal nanoparticles



3.3 Antioxidant property of herbal nanoparticles

3.3.1 Antioxidant properties of herbal nanoparticles

In DPPH assessment, the prepared herbal nanoparticles are used to reduce the free radical 2, 2-diphenyl-1-picrylhydrazyl, which shows a strong absorption peak at 517 nm [47]. As seen from the Fig. 7 it was seen that the antioxidant activity is shown by almost all the prepared herbal nanoparticles but the scavenging activity of the TPNPs

nanoparticles is high. Also from the above results a direct comparative assessment between the plant nanoparticles in virtue of its synthesis parameters shows a variation in the antioxidant activity. From the previously reported antioxidant activity of the herbal nanoparticles i.e., AINPs, PBNPs, TZINPs and AVNPs shows that the synthesis process employed during the synthesis of the aforementioned nanoparticles through wet processing shows higher antioxidant activity than that of the before mentioned investigations.

Table 1 UPF value and contact angle measurement for the herbal nanoparticles coated and un-coated fabric samples

Herbal nanoparticles	Test organisms	Concentrations of herbal nanoparticles [zone of inhibition (mm)]		
		25 mg mL ⁻¹	50 mg mL ⁻¹	100 mg mL ⁻¹
AINps	<i>E. coli</i>	14.71 ± 0.41	20.42 ± 0.34	23.22 ± 0.51
	<i>S. aureus</i>	21.10 ± 0.61	23.14 ± 0.68	26.12 ± 0.49
AZINps	<i>E. coli</i>	15.14 ± 0.39	22.27 ± 0.76	24.55 ± 0.52
	<i>S. aureus</i>	22.64 ± 0.70	25.65 ± 0.31	28.53 ± 0.72
PBNps	<i>E. coli</i>	13.12 ± 0.12	21.21 ± 0.21	23.35 ± 0.42
	<i>S. aureus</i>	21.34 ± 0.14	23.57 ± 0.14	26.37 ± 0.51
TPNps	<i>E. coli</i>	14.82 ± 0.26	22.28 ± 0.51	24.01 ± 0.13
	<i>S. aureus</i>	22.13 ± 0.33	24.31 ± 0.23	27.62 ± 0.24
AVNps	<i>E. coli</i>	13.66 ± 0.52	21.42 ± 0.47	23.06 ± 0.60
	<i>S. aureus</i>	21.33 ± 0.46	24.11 ± 0.37	27.77 ± 0.72

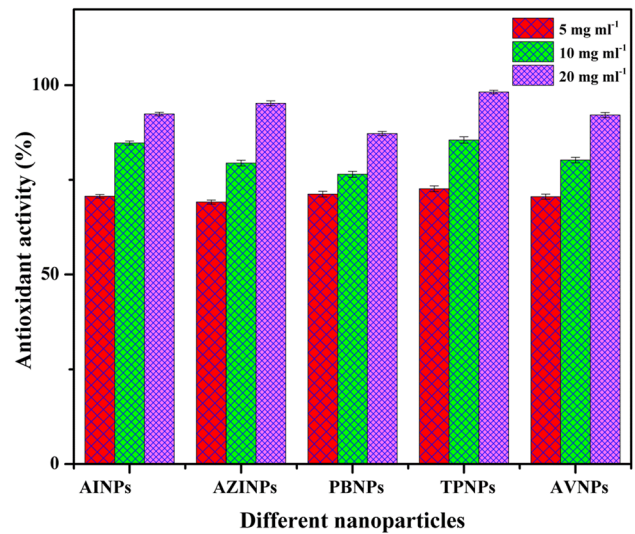


Fig. 7 Antioxidant activity of five different herbal nanoparticles

T. Procumbens shows high antioxidant activity (98%) due to it contains hydrogen donors and singlet oxygen quenchers. In addition, phytochemicals phenol, flavanoids, tannins, catechins [48, 49], followed by *Az. indica* (95%), *A. indica* (92%) and *A. vera* (92%) because it also contains phytochemicals polyphenols, alkaloids, steroids, flavanoids, and saponins, tannins and amino acids [50–53]. *P. betle* (87%) contains phenols and polyphenols responsible for antioxidant activity [25]. This increase in the antioxidant property of the herbal nanoparticles might be due to the change in the structure or morphology of the mentioned nanoparticles through wet processing. It is seen that from the previously done studies on antioxidant

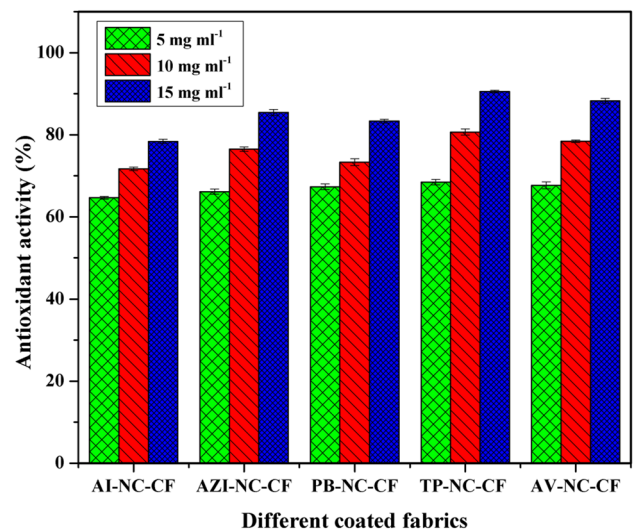


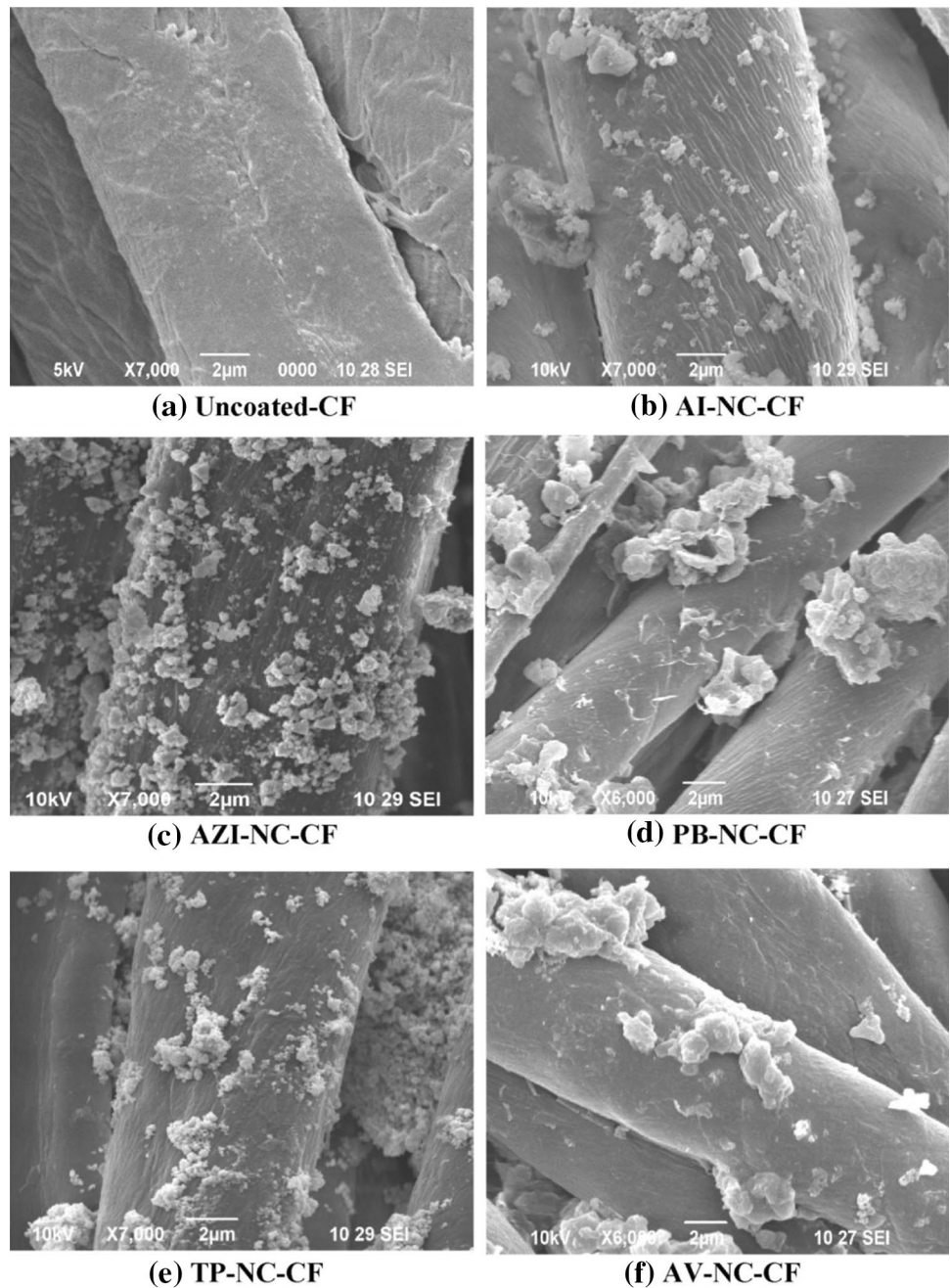
Fig. 8 Antioxidant activity of herbal nanocomposites coated fabrics

activity of *Az. indica* it is seen that with change in the synthesis parameters causes a change in the effectivity of the herbal nanoparticles. It is seen that the herbal nanoparticles synthesised through our solution processing method shows higher antioxidant activity.

Even in the case of the *P. betle* the solution processing parameter shows higher antioxidant activity from the previously mentioned studies on the plants antioxidant activity and also the antioxidant activity of the *A. vera* herbal nanoparticles slightly increases in case of the wet processing method comparing tom the other previously

done study. It can be seen that the herbal nanoparticles acquired from the *T. procumbens* shows very high affective antioxidant comparing to other previously mentioned processing methods but also highly effective comparing to the other herbal nanoparticles. From aforementioned Fig. 7 it can be safely concluded that not only the increase in the concentration of the plant herbal nanoparticles but also change in the plant source and the change in the synthesis process can result an increase in the scavenging activity of all the herbal nanoparticles against DPPH.

Fig. 9 SEM images of uncoated and herbal nanocomposites coated fabrics



3.3.2 Antioxidant properties of herbal nanoparticles coated cotton fabrics

Antioxidant activity of herbal nanocomposites fabrics (AI-NC-CF, AZI-NC-CF, PB-NC-CF, TP-NC-CF and AV-NC-CF) coated cotton fabrics represented in Fig. 8. Free radical scavenging activity (90.42%, 88.26%, 85.32%, 83.46% and 78.35%) of the herbal nanocomposites coated cotton fabrics (TP-NC-CF > AV-NC-CF > AZI-NC-CF > PB-NC-CF > AI-NC-CF) respectively. The phytochemicals such as phenol, polyphenols, alkaloids, steroids flavanoids, tannins, saponins, catechins are reasonable for antioxidant activity of herbal nanocomposites fabrics [25, 48–53]. The antioxidant activity of chitosan due to its polymer chain contains formation of strong intermolecular and intra-molecular hydrogen bonds [25].

3.4 Functional property of cotton fabrics

Figure 9 shows the SEM images of the herbal nanocomposites fabrics (AI-NC-CF, AZI-NC-CF, PB-NC-CF, TP-NC-CF and AV-NC-CF) coated cotton fabrics. Herbal nanocomposites seen are to be clearly engrossed on the fabrics surface area. The surface morphological features of uncoated fabrics are shown in Fig. 9a. The chitosan coated fabrics with the herbal nanocomposites prepared by dry ball milling are clearly demonstrated in our previous studies [13].

Based on the ASTM D6603 standard data, the calculated UV-protection Factor (UPF) value for the coated and uncoated fabrics are shown in Table 2. All herbal nanocomposites (Chi-AINPs, Chi-AZINPs, Chi-PBNPs, Chi-TPNPs and Chi-AVNPs) coated cotton fabrics able to block UV-A and UV-B radiations that show high UPF value of about 50, indicating high restriction UV-rays.

The Chi-AVNPs coated cotton fabrics shows higher effectiveness near restricting of UV-ray when compare to other four herbal nanocomposites (Chi-AINPs, Chi-AZINPs, Chi-PBNPs, and Chi-TPNPs) coated cotton fabrics (AI-NC-CF, AZI-NC-CF, PB-NC-CF, and TP-NC-CF). The particular herbal nanoparticles (AVNPs) might have higher optical

Table 2 Antimicrobial activity of the five different nanoparticles from well diffusion assay

Samples	UPF	Contact angle (°)
UC-CF	13.9	0
CF-Chi	42.2	151.4 ± 3
AI-NC-CF	60.1	153.6 ± 4
AIZ-NC-CF	59.6	154.5 ± 1
PB-NC-CF	55.8	153.3 ± 3
TP-NC-CF	57.2	153.4 ± 2
AV-NC-CF	62.3	154.2 ± 1

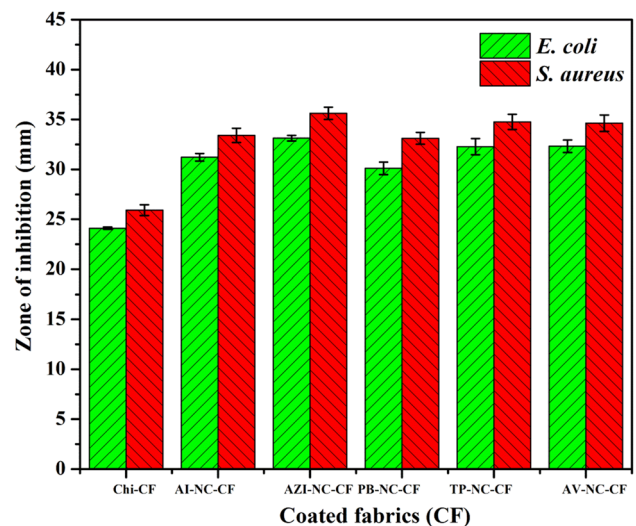


Fig. 10 Antimicrobial activity of herbal nanocomposites coated fabrics against *E. coli* and *S. aureus*

polarisation resulting in the higher restriction of the UV-radiation. AINPs nanocomposites coated fabrics have good UV-blocking properties as revealed in our previous studies [13]. This enhanced UV-protection activity of the Chi-AVNPs is due to the fact that the phytoconstituents present in the AVNPs nanoparticles consists of photochemical active compounds which show higher affectivity in blocking UV-B radiation than that of the other nanocomposites.

3.5 Antimicrobial assessment of coated fabrics

To perform a comparative assessment between uncoated and herbal nanocomposites (AI-NC-CF, AZI-NC-CF,

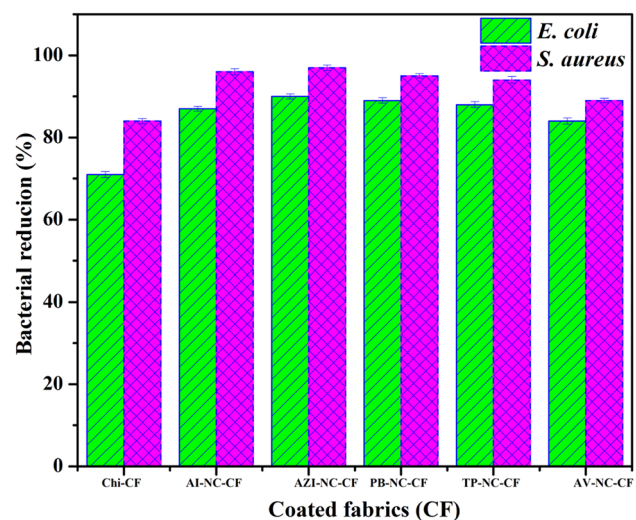


Fig. 11 Bacterial reductions of herbal nanocomposites coated fabrics against *E. coli* and *S. aureus*

PB-NC-CF, TP-NC-CF and AV-NC-CF) coated cotton fabrics in virtue of their antimicrobial property, we execute disc diffusion assay against *S. aureus* and *E. coli*. It is seen that there is no antibacterial property in case of the un-coated fabric whereas in the coated fabric exhibits effective zone of inhibition. This observation conveys that the antimicrobial property of the herbal nanoparticles is retained during its change to nanocomposites (Fig. 10). As seen from the graph, the zone of inhibition for the AZI-NC-CF is slightly higher zone of inhibition for both *E. coli* and *S. aureus* (33.13 ± 0.28 mm and 35.62 ± 0.62 mm) than that of other nanocomposites (AI-NC-CF, PB-NC-CF, TP-NC-CF and AV-NC-CF).

This enhanced antibacterial activity of the *A. indica* nanocomposite is due to the fact that the *A. indica* nanocomposite is more strongly adhered to the cotton fabrics than that of the other nanocomposites. Along with it the spherical structure of the *A. indica* nanoparticles results in a higher antibacterial activity along with it the phytochemical present in *A. indica* nanocomposite shows higher antibacterial activity than that of the other nanocomposites prepared.

3.6 Percentage reduction test for coated fabrics

Figure 11 shows the quantitative estimation of the antibacterial action for the UC-CF and herbal nanocomposites

(AI-NC-CF, AZI-NC-CF, PB-NC-CF, TP-NC-CF and AV-NC-CF) after percentage reduction test assessment.

The bacterial percentage reduction of *E. coli* and *S. aureus* treated with AZI-NC-CF fabrics is shows better results (90 and 97%), while AI-NC-CF (87 and 96%), PB-NC-CF (89 and 95%), TP-NC-CF (88 and 94%) and AV-NC-CF (84 and 89), CF-Chi (71 and 84%) respectively, The bacterial percentage reduction is found to be more, which ensures better functional properties of herbal nanoparticles coated fabrics.

3.7 Water repellent property

To achieve a comparative assessment of the nanocomposites coated cotton fabrics (AI-NC-CF, AZI-NC-CF, PB-NC-CF, TP-NC-CF and AV-NC-CF) for water repellent property, the surface contact angle using sessile drop method is adapted. Most often un-coated fabrics show high degree of hydrophilic behaviour due to the fact that the un-coated fabric has more number of hydroxyl groups on its surface. It can be furthermore confirmed by placing a water droplet over the cotton fabrics, the water droplet gets easily absorbed by the un-coated fabrics as shown in Fig. 12. In contrast, the cotton fabrics coated with herbal nanocomposites show enhanced superhydrophobicity for AI-NC-CF (153.6°), AZI-NC-CF (154.5°), PB-NC-CF (153.3°), TP-NC-CF (153.4°) and AV-NC-CF (154.2°) while chitosan

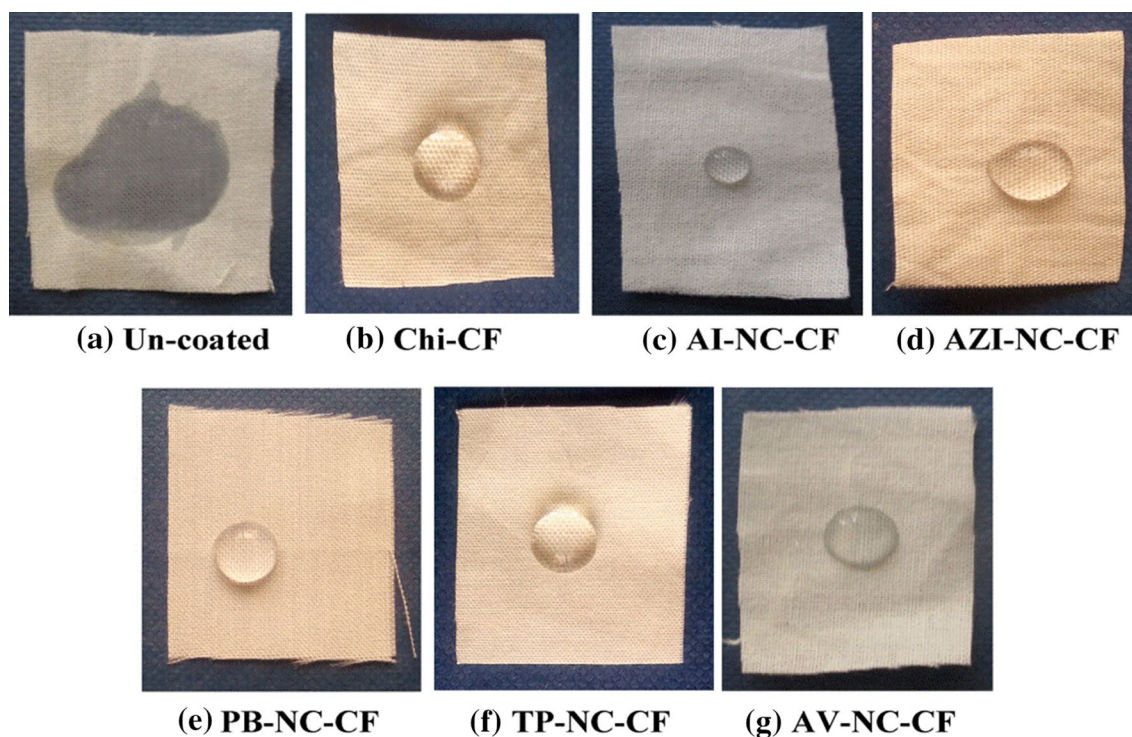


Fig. 12 Water repellent property of un-coated and coated fabrics surface

coated fabrics have 151.4°. The enhanced hydrophobicity of the cotton fabric could be due to the lower surface energy of the herbal nanoparticles, superhydrophobic nature of chitosan and also due to the formation of strong adherent layer over the surface of the cotton fabrics resulting in a net reduction of pore size as seen in SEM image [18]. Therefore, the superhydrophobic nature of nanocomposites favours the use of AZINPs for self-cleaning applications.

4 Conclusion

Herbal nanoparticles synthesized from various plant sources such as *Acalypha indica*, *Azadirachta indica*, *Piper betle*, *Tridax procumbens* and *Aloe vera* through wet processing method were screened and assessed to explore their physico-chemical, biological as well as functional property to understand the possible applications for these herbal nanoparticles in biomedicine. By reviewing all the biological and functional properties of five herbal nanoparticles, the efficacy is identified i.e., a broad spectrum (water repellency (154.5°) in the particles prepared from *Az. indica* and antimicrobial activity against *E. coli* (24.55 mm) and *S. aureus* (28.53 mm)), *T. procumbens* (high anti oxidant activity (98.17%)), and *A. vera* (high UV-protection (UPF = 62.3)). In addition, solution processing method of particular synthesis is found to be better than dry milling techniques in terms of controlling the particle formation and dispersibility. This study will help in the tuning of the herbal nanoparticles in virtue of its plant sources and processing method for the use in textile and biomedical applications.

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Compliance with ethical standards

Conflict of interest Authors declare no conflict of interest.

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