

"Development of Nano-composite Textiles for Bio-medical Application"

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2.0 BRIEF RESEARCH METHODOLOGY

2.1 OBJECTIVES OF RESEARCH:

The present study has been designed to develop and optimize green synthesis technique for AgNPs using CP leave and gauge the performance of the prepared prototype antibacterial nonwoven @AgNPs/CP nanocomposites for various bio-medical applications. The goals set up for the research are:

- a) Lab-scale production of the silver nanoparticles and their validation via chemical and green synthesis techniques.
 - i) Elementary investigation of the AgNPs produced by a chemical synthesis technique.
 - ii) Elementary investigation of the AgNPs produced by a CP extract based green synthesis technique.
- b) Application of bio-synthesized AgNPs on nonwoven fabric and Confirmation of Nano-composite's antibacterial activity.
 - i) Loading bio-synthesized AgNPs on a PV (Polyester-Viscose) non-woven to produce nano-composite textile materials.
 - ii) Confirming antibacterial activity of PV-AgNPs/CP nanocomposite.
 - iii) Evaluating characteristics of the nanocomposite by ESEM, EDAX, Nanoparticles size distribution, FTIR, Physical parameters e.g. GSM and Thickness; Low-stress properties e.g. Bending modulus and Crease recovery angle; Comfort-related properties e.g. Air permeability and Overall moisture management capability (OMMC); and UV Transmission properties.
- c) Investigating optimum feed levels for the constituents in the AgNPs/CP colloidal synthesis based on antibacterial activities potential in nonwoven @PV-AgNPs/CP Nano-composites.
 - i) Major scale add-on trials
 - ii) Minor scale add-on trials
- d) Development of nanocomposites using optimum AgNPs/CP colloidal and the best 10% CP extract with popular nonwoven base materials and evaluating their basic physical, low stress, comfort associated as well as functional properties as per ASTM standards.

2.2 RESEARCH IN BRIEF:

The entire experimental work was divided into four phases for the step-by-step evaluation of the newly designed product (Figure 2.1).

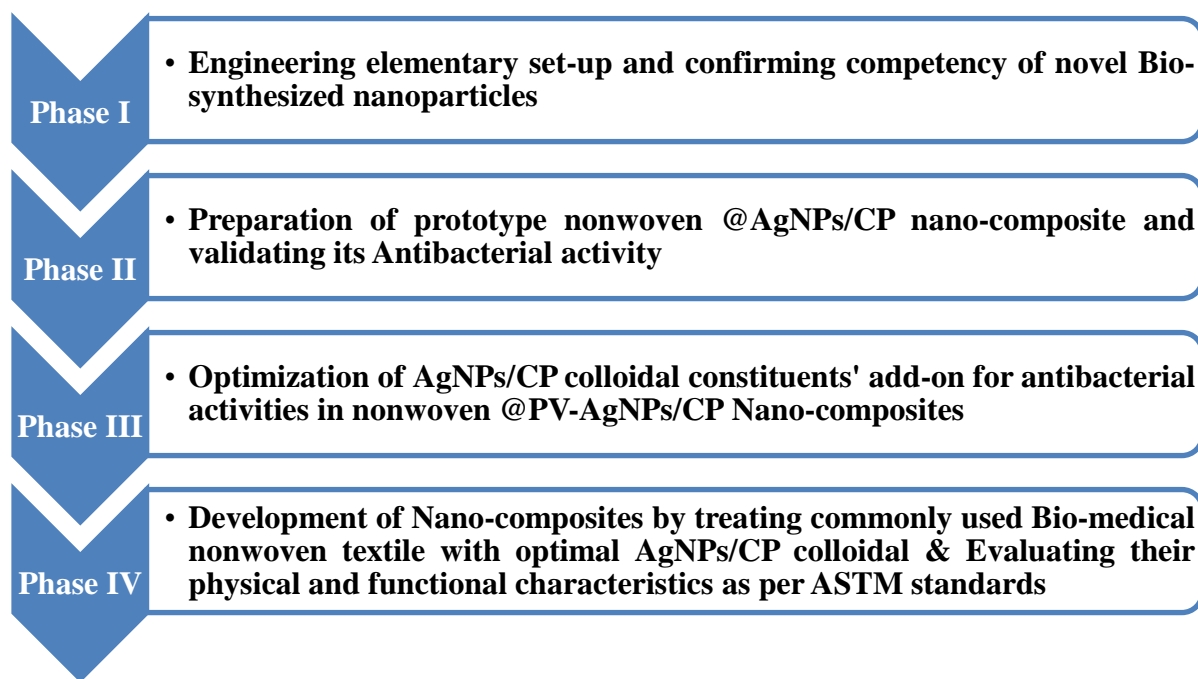


Figure 2.1: Plan of experimental work

The first phase of study was designed for elementary investigation of the silver nanoparticles. They were synthesized via commercial chemical technique and pioneering green channel; using Calotropis Procera (CP) leave extract using forward, reverse and reduction approaches. The reverse method (Bottom-up approach) has produced excellent nanoparticle during green synthesis in line with the chemical technique. The preliminary but subjective colour change observation and SEM analysis both have confirmed successful formation of desirable nanoparticles on green-way synthesis.

The second phase was designed to develop the prototype eco-friendly nano-composites for medical health care applications. The simple and effective cold dipping technique was used for AgNPs/CP under in-situ deposition on PV-Nonwoven; the most popular bio-medical textile material. The EDAX and FTIR test results have confirmed presence of AgNPs in PV-nonwoven structure on treatment. Whereas, the ESEM images have endorsed a virtuous deposition of AgNPs on the PV-nonwoven by even covering of constituent fibres. Antibacterial activity potential was realized from the significant

antibacterial activity noted against both the bacterial cultures *Staphylococcus aureus* (23.75mm) and *Escherichia coli* (24.5mm) for the newly engineered PV-AgNPs/CP material. The favourable shift in moisture management, low-stress, and comfort-related characteristics were detected on loading of AgNPs/CP.

After realizing efficacy of novel green channel, the third phase of experimentation was conducted to optimize qualitatively and quantitatively silver nitrate (AgNO_3) salt add-on and CP leave extract concentration during synthesis process. Major scale and minor scale add-on pilot trials were conducted for the purpose being and synthesized colloidal were analysed for nanoparticle formation and stability, particle size analysis and antibacterial activities. They were also checked for the susceptible toxicity of the participating elements by MTT assay cytotoxicity test. The sorted optimized combination was further evaluated for its antibacterial efficacy into prototype PV based bio-medical nonwoven nano-composite. The colloidal solution E10S0010 produced with 10% CP leave extract and 10mM AgNO_3 was found optimum with formation of a very good nanoparticle size distribution of 25.8nm. This was also endorsed by its UV-visible spectra showing a narrower and more (sharp) strong peak with maximum absorption in the wavelength range 400–450nm. The nanoparticles have retained their stability well up to six month and possessed substantial 84% cytotoxicity. The optimum PV-AgNPs/CP nano-composite has exhibited the highest average ZOI value, with 18.0mm against SA and 21.7mm against EC bacterial culture. Contribution of optimal green medium in the success of AgNPs/CP performance was also analysed. The GC-MS analysis of the optimum 10% concentrated CP leave extract has revealed the presence of N-Decanoic acid ($\text{C}_{10}\text{H}_{20}\text{O}_2$) responsible for antibacterial activity as well as other components contributing effectively for reduction and capping of the nanoparticles. The extract has shown 92% cell viability during the MTT assay as well as significant antibacterial activity against SA (16mm) and EC (19.3mm) bacterial cultures.

The novel and ecologically strengthened AgNPs/CP nonwoven composites for vivid bio-medical applications were worked out in the fourth phase of the study. Two most popular bio-medical textile materials; PP-Nonwoven and PV-Nonwoven of their commonly used GSM; PP30, PP45, PP50 and PV40, were used as base materials for the purpose being. The innovative composites were meticulously evaluated for their basic physical, comfort-related, morphological, and elemental as well as the targeted antibacterial potential. Efficacy of CP leave extract in the absence of AgNPs in the nonwoven composites was also worked out in

parallel. It was noted that GSM as well as thickness has shown significant increase at the nano-level AgNPs/CP loading mainly due to participating heavier silver particles. This was substantiated by the non-significant changes noticed on loading only CP leave extract in each case. The similar behaviour was noticed for comfort-associated and low-stress properties. However, the resultant values in either of the cases were well within the acceptance range for the targeted end uses. The favourable significant rise in UPF value has been noticed on AgNPs/CP loading invariably for all the samples under consideration. ESEM and FTIR have endorsed presence and desirable uniform distribution of AgNPs in the constituent fibers for all the base textile materials. All together has resulted into good antibacterial efficacy for the selected samples. Apart from these, only CP treated nonwoven textile materials also have shown remarkable antibacterial activity due to its inherent chemical constituents as mentioned in phase-III for GC-MS test results.

The innovative CP based green synthesis of AgNPs and its incorporated both PP based as well as PV based nonwovens have shown targeted sustainable antibacterial efficacy along with other physical, mechanical, low-stress and comfort-associated characteristics. Thus, it has opened new horizon for ecological bio-medical textile materials preparation technique.

3.0 CONCLUSIONS AND RECOMMENDATIONS

3.1 CONCLUSIONS

Development of hygiene nano-composites, using the green-way synthesized silver nanoparticles (AgNPs) with CP leave extract was aimed in the present study. The outcomes for the series of experiments are summarized here.

1. The chemical synthesis (CS) of the AgNO_3 has produced a noteworthy amount of nanoparticle by bottom-up (reverse) method (CS-2) compared to forward and reduction approaches. The colloidal colour was changed from colourless to yellowish and confirmed occurrence of nanoparticles. Going in accordance, the reverse green synthesis (GS) method has generated admirable nanoparticles (GS-2). The subjective colour change validation was supported by the SEM micrographs. A uniform dispersion of AgNPs with rarely occurring aggregation was noticed even though ten times more CP extract was consumed than AgNO_3 during reverse mode of synthesis. This pro-rata of participants also persuaded economy to the newly synthesized colloidal.
2. A prototype PV-nonwoven nano-composite @PV-AgNPs/CP was successfully prepared using a cold dipping technique. The ESEM micrograph obtained for AgNPs/CP loaded fabric using the 'ImageJ 1.53a' program has revealed the average particles size of 160nm without an accumulation. It has also witnessed a good level of AgNPs deposition on the PV-nonwoven fabric.
3. An elemental analysis (EDAX) of PV-AgNPs/CP has illustrated a prominent peak of metallic silver nanoparticles for AgNPs/CP treated sample 01 and endorsed penetration of AgNPs in the nonwoven structure.
4. The FTIR spectrum characterization peaks for the PV-AgNPs/CP nano-composite has exhibited a similar chemical composition in case of untreated (sample 00) and treated (sample (01) due to major share of common base material. However, additional chemical compositions, such as aromatic amine (C-N) stretching vibration and phenolic (O-H) bending vibration endorsing presence of AgNPs/CP, were also visualized.

5. The PV-AgNPs/CP nano-composite (Sample 01) has executed considerable antibacterial activity against both bacterial cultures *Staphylococcus aureus* (23.75mm) and *Escherichia coli* (24.5mm), compared to values realized for usual chemical based AgNPs loaded composites. This has proven qualitative strength of new green channel of AgNPs synthesis.
6. The physical parameters revealed no remarkable change in fabric thickness after treatment, but GSM has shown a small rise even at the nanoscale addition, which is contradictory to the normal trend.
7. The selected PV-nonwoven with the added prototype green functional elements has shown arbitrary but not much higher fluctuations in machine-direction (MD) and cross-direction (CD) bending modulus. Thus, rendered the material suitable for bio-medical applications.
8. Air permeability of the base fabric was reduced in quite a low amount; by 1.99% only on AgNPs/CP deposition. It was more expectedly to be the outcome of inbuilt structural variations of the material itself rather than treatment. This will not affect adversely its use as a bio-medical textile material.
9. The PV-AgNPs/CP nano-composite has executed preferred 'excellent' moisture management capabilities for bio-medical textile and is classified as a 'moisture management fabric' than the reference untreated fabric.
10. Application of the AgNPs/CP nanoparticles has not positively contributed in the UPF rating of the PV-nano-composite. However, percent UVA and UVB transmittance was reduced to 20.75% and 9.18% and made the material more protective against UVA and UVB type radiations.
11. Colour of all the colloidal solutions synthesized with major scale silver nitrate (AgNO_3) salt add-on was turned from brown to black, indicating formation of the undesirable macro molecules due to agglomeration of unstable nanoparticles after 24

to 48 hours of synthesis. Thus, pointed towards inappropriateness of higher level AgNO₃ and CP extract add-ons in newer synthesis.

12. All of the major scale AgNO₃ add-on colloidal samples except sample A1 (E05S00) have shown good bacterial inhibition against both SA and EC bacterial cultures up to 72 hours. The highest average ZOI value has gone in the account of colloidal solution 'E10S01 (B2)' made with 10% CP extract (green medium) concentration and 100mM AgNO₃. This was attained due to presence of higher quantum of AgNO₃ and CP extract inherently antibacterial in nature.
13. Sample A1 (E05S00) prepared without AgNO₃ was failed to achieve bacterial inhibition against SA bacteria but demonstrated average ZOI of 12mm against EC bacteria in the initial 24 hours. However, antibacterial activities were diminished to zero after the next 24 hours holding time.
14. The MTT assay cytotoxicity test displayed the favourable much higher viability of the cell line at the chosen 5 to 20 percent concentrations of the CP leave extract than the CP pure latex. The viability of the cell line was decreased with the increase in the concentration of CP leave extract. So, cell survival point of view the lowest 5 percent concentration of CP leave extract was considered as best amongst all. But its ineffectiveness against bacteria has made next higher-level CP concentration; E10 with highest antibacterial potential as an optimum choice.
15. All of the colloidal solutions formed in the first set of the minor scale trails have shown a fuzzy milky brownish colour change to validate the formation of AgNPs at the moment of synthesis during visual judgment test. However, stability of the colloidal solutions E10S0020 and E10S0040 was only persisted for a week and sustained up to a month only. Thus, substantiated poor efficacy in meeting the targeted performance.
16. The UV-VIS Spectroscopy conducted for the first minor set samples has shown broad but weak peak between 400 and 600nm (or <600nm) with increased molarity of the AgNO₃. This witnessed presence of undesirable poor-quality AgNPs on agglomeration of basically small or medium-sized nanoparticles.

17. The DLS spectra for all of the first minor scale colloidal solutions under observation have shown a broad polydisperse type of distribution with the preferable AgNPs size less-than 100nm (range from 28.8nm to 58.7nm). This has indicated a large diversity of particle sizes existence.
18. Colloidal solutions E10S0020 (E1) and E10S0040 (E2) have demonstrated good antibacterial activity against SA and EC bacterial cultures amongst the first minor set samples. This behaviour has supported better antibacterial activities with the use of low add-on of AgNO₃ for constant 10% concentration of CP leave extract.
19. Formation of AgNPs in second minor scale colloidal was demonstrated by a milky-fuzzy brownish colour at the time of synthesis. Colloidal solutions E10S0010 and E10S0015 have demonstrated good AgNPs stability even after six months with a little but still acceptable colour change. Except these two, rest of the sample's stability lasted maximum for a week.
20. The UV spectra peak observed for the sample E10S0010 was stronger, sharper, and narrower, with a maximum absorption in the 400–450nm wavelength range, advocated good quality smaller nanoparticles formation without agglomeration in the group. A wider and stronger peak between 190 and 400nm usual for CP leave extract was noted in the sample E10S0000. The DLS spectra for E10S0010 (F4) colloidal solution have shown the most ideal silver nanoparticle size distribution of 25.8nm. This has endorsed optima for AgNO₃ as 10mM in the group.
21. The highest average ZOI value; 18.0mm against SA and 21.7mm against an EC bacterial culture, was recorded with the colloidal solution E10S0010 (F4). The sample E10S0000 (F1) synthesized using CP leave extract (10%) only has shown average ZOI value of 16.0mm against SA and 19.3mm against EC bacterial cultures and proven its individual antibacterial strength.
22. The colloidal solution E10S0010 has an 84% cell survival rate, which is regarded to be non-toxic (>80% is significant). The cell viability of the colloidal solutions prepared with very low AgNO₃ add-on E10S0001 and E10S0005 was quite high (90%

and 88%, respectively). The optimum CP leave extract (E10) has demonstrated 92% cell viability witnessed its non-toxic characteristic. Thus, both the participating constituents in prototype green synthesis were found not harmful to the user if employed in a bio-medical product.

23. The GC-MS analysis endorsed presence of N-Decanoic Acid ($C_{10}H_{20}O_2$) responsible for antibacterial activity in the 10% concentration CP leave extract. Presence of amino acids, citric acid, flavonoids, phenolic compounds, terpenoids, heterocyclic compounds, enzymes, peptides, polysaccharides, saponins, tannins, etc. were also detected in the extract found responsible for reduction and capping of nanoparticles. Altogether proven potential of the innovative medium in AgNPs synthesis.
24. A virtue-based technique, similar to the one used in phase-II, was used successfully to deposit AgNPs on the hydrophobic PP and hydrophilic PV non-woven fabrics most popularly used in different bio-medical applications. So, this method was regarded as an appropriate for the prototype bio-medical nano-composites development.
25. The Physical parameters, low-stress qualities, comfort-associated attributes, ESEM micrographs, and FTIR spectrum characterization peaks all have shown more or less comparable behaviour with reference parent samples. The additional component (AgNPs) density caused significant but end use point of view tolerable changes in GSM and thickness. The observation was supported by the lesser changes seen with the lighter CP extract loaded composite values.
26. The statistical analysis has shown that there were no statistically significant differences occurred between the AgNPs/CP and CP extract treatments for low-stress characteristics measured in the Machine-wise Direction and Cross-wise Direction.
27. The air permeability of treating nonwoven textile materials with CP leave extract and AgNPs/CP was lowered due to coverage of higher specific surface by nanoparticles and found statistically significant also. But not to the extent which can hamper their performance as a bio-medical textile material.

28. Most of the composite samples including the control have shown 'very poor' rating for OMMC. The base materials were also classified as 'water proof fabrics', with poor moisture management, and thereby graded as 'very poor'. Only the 'moisture management fabric' @PV40-AgNPs/CP nano-composite has shown better capacity to handle water in the group mainly due to presence of hydrophilic viscose fibers in the structure.
29. The efficiency and durability of the antibacterial activity mapped against SA and EC bacteria were zero invariably for all the selected base fabrics as per expectation. However, they were visualized in good quantum for all the CP treated composites which has endorsed potential of the selected novel green channel in developing hygiene bio-medical materials. The extent of antibacterial activities got further enhanced in the presence of silver; AgNPs/CP treated samples as anticipated. The ZOI values noted in each category of sample are much higher than the chemically synthesized AgNPs treated bio-medical materials (~10 – 15mm) in commercial use.
30. The applications of CP leave extract and AgNPs/CP have shown preferable statistically significant improvements in the UV transmittance profile of the studied nonwoven textile materials.
31. The ESEM micrographs have substantiated the AgNPs deposition method adopted in the present study as a satisfactory for a virtuous deposition of AgNPs on the difficult to treat kind PP as well as more user-friendly PV non-woven fabric.
32. The FTIR spectrum peaks associated with phenolic and aromatic amine groups seen clearly have advocated success about the incorporation of AgNPs/CP into PV based composites as well as difficult to induced type all the PP based nano-composites.

3.2 RECOMMENDATIONS/SCOPE FOR THE FUTURE WORK

- I. The present work was focused to develop disposable class of bio-medical textile materials only. So, the extension works with the non-disposable woven and knitted structures can be targeted. The performance sustainability of the AgNPs/CP into these

structures on immediate loading and after prescribed number of washing cycles need to be worked out.

- II. The base material selection was biased by the type of fabric; nonwoven used during the study. Thereby, extent of hygiene attains and retains with other popular materials like cotton, polyester, woollen etc. used in different formats in hospital environment need to be explored with this novel green channel.
- III. The CP leave extract has proven its antibacterial efficacy in the absence of silver also. So, it should be further analyzed to avoid undue utilization of costlier and toxic silver or other metallic mediums.
- IV. The healing capabilities of CP extract in wound care and diabetes is well-known and also mentioned by various investigators in literature. The limitations of medical permissions restricted to explore this region during the present study but the collaborative work with medical field can give new heights to this valuable medicinal but wild plant.
- V. It is possible to produce the antibacterial bio-medical products with lower material and production costs due to the abundant and free availability of CP leave. In light of the mass production of CP treated and AgNPs/CP treated fabrics used in various bio-medical applications, studies for cost efficiency can be conducted.

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