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Green synthesis of copper oxide NPs using different medicinal plant Extracts for the applications of textile industries

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Abstract : In this present work, the simple, eco-friendly and cost effective method is adopted for preparation of copper Oxide NPs (CuO) using various traditional medicinal plants like Andrographis paniculata (Nilavembu), Aegle Marmelos (Vilva), Moringa Oleifera (Moringa) plant leaves extract and copper sulphate. The X- ray diffraction studies reveal the end-centered structure of CuO NPs and the grain size ranges from 4 nm to 60 nm. The FTIR analysis showed that the functional groups present in the plant extract which may involve in the green CuO NPs synthesis. From UV-visible spectroscopy we find the absorbtion peak to be around 360nm and the calculated band gap is found to be at 3.58 eV.SEM image shows that the NPs are orthorombic in shape. EDX analysis confirmed to some chemical elements is present in our sample .Antibacterial activity of CuO NPs were carried out for both gram positive (MRSA, Staphylococcus epidermidis) and gram negative (E-Coli, pseudomonus aeruginosa and Serratia marcescens) bacteria by using Agar well method. It concludes that gram negative bacteria shows significant activities compared to gram positive bacteria. Hence, it may be utilized as finishing agent in textile industries.

Keywords : Textile applications, Antibacterial activity, copper NPs, Plants extract.

1. Introduction:

In this competitive world, a textile industry gives many challenges to the researches. The rapid growth in technological textiles and their uses has produced many opportunities for the application of finishing process. Antimicrobial textiles was improved their functions and find a lot of applications [1, 2] such as health and hygiene products. Particularly the textiles worn close to the skin and need to many medicinal applications such as infection control [3] and maintanence of fiber quality.

The use of natural medicinal product, such as natural dyes, for antimicribial finishing of textile industries has been widely reported. Other natural herbal products such as tea tree, tulsi, neem leaves extracts can also be used for this purpose [4]. There is a vast source of medicinal plants with active antimicrobial ingredients. The study of their use in textiles is very limited and not reported well. Most of the areas concentrate on technical details of applying natural agents such as neem extract and herbal products on their testing process.

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In the last few years, the researcher has been motivated to work with the increase in new antimicrobial fiber technologies with healthy life style. A range of textile product based on microbial agents such as metallic compound containing copper [5], silver [6, 7], gold [8], magnesium [9] and Zinc [10]. It also makes fabrics resist growth of bacteria. The use of CuO NPs offers durable antibacterial agents that are used in textile finishing. CuO acts as an antimicrobial agent, when it was synthesized using medicinal plants extracts, to apply the processes of finishing, coatings in textiles. The main classes of antimicrobial agents are disinfectants, which kill a wide range of microbes on living surfaces to prevent the spread of illness, antiseptics and antibiotics [11]. Antibacterial agent slow down or stall bacterial growth. It has resulted in solutions that can go beyond simply inhibiting microbial growth. Finishing refers to the processes that convert the knitted cloth into usable one. Antimicrobial finish causes fabrics to inhibit the growth of microbes. The humid and warm environment found in textile fibers increases the growth of microbes. It causes some cross-infection in skin [12] and loss of fiber quality by pathogens. Therefore, textile industries aim to protect the skin of fabrics. So antimicrobial finishing is applied to textile industries.

Metal [13] and metal oxide [14] NPs have found wide range of uses including medicinal applications. The different applications of metal NPs varied with morphology and size. Among the metals NPs, copper NPs showed more activity against pathegenic bacterias like E.Coli [15]. There is no exclusively work on CuO NPs with natural plants extract like nilavembu, vilva, moringa leaves based on antimicrobial finishing agent for the applications in the field of textiles. Therefore, in this present work, the green synthesis of CuO NPs is reported using medicinal plant extracts for the antibacterial finishing agents in textiles.

2. Materials and Experimental methods:

2.1 Plant Materials:

The herbal plant leaves were collected from surrounding area at Virudhunagar district, Tamilnadu. Many medicinal plants and herbs are active against antimicrobial activities due to presence of some chemically active substances that produce a definite physiological action on the human body. These plants have numerous medicinal applications and high antibacterial activity. It cures skin disease, dysentery, allergies, asthma, fever, cholera, diabetes, influenza and swelling. Textile industry aims to protect the skin of fabrics. They are used to antibacterial finishing process in textiles. So, it is decided to use these herbal plants for this work.



Vilva leaf

(Aegle Marmelos)



Nilavembu leaf (Andrographis paniculata)

Moringa leaf (Moringa Oleifera)

2.2 Preparation of the plant leaf extract:

The required amount of plant leaves were taken and thoroughly washed 3 - 4 times in normal and distilled waters respectively to remove impurities. The cleaned leaves were dried under sunshade ambient to remove moisture completely. Then, the dried leaves were gushed by adding drop of distilled water. After crushing, the leaf extract was filtered using wattman filter paper. Then, this extract was kept in cool for using further work.

2.3 Preparation of copper NPs:

Copper sulphate and distilled water were used without further purification. In this experiment, a 0.1 mM of copper sulphate with 100 ml distilled water was kept under constant stirring using magnetic stirrer to completely dissolve the copper sulphate for four hours. After complete dissolution of copper sulphate, 10 ml plant leaf extract solution was added under high speed constant stirring, drop by drop touching the walls of the vessel. The reaction was allowed to proceed for 2 hours after complete addition of leaf extract. The beaker was sealed at this condition for 4 hours. After the completion of the reaction, the solution was allowed to settle for overnight and further the supernatant solution was separated carefully. Then, the sediments are dried at room temperature. During this process, the colour change was absorbed and this represents the formation of CuO NPs. The collected NPs were subjected to various characterizations in order to understand its quality as well as properties.

3. Characterization studies:

XRD (X-ray diffraction) patterns of the CuO NPs were investigated to study the crystalline nature and crystallite size of the NPs using X-ray diffractometer (X PERT PRO) with the step size of 0.05° from the diffraction range 10 to 80°. Cu K_a radiation of wavelength $\lambda = 1.5406$ Å was used as a diffraction source. The morphology studies of samples were studied by Scanning Electron Microscope (SEM) with an operating voltage 20 kV with the magnification range of 7500 - 30,000X. FTIR spectra of the prepared samples were recorded in the range 400-4000 cm⁻¹ using a Shimadzu FTIR spectrophotometer. UV-Vis spectra were recorded using UV-1700 series. The antibacterial studies of green synthesized CuO NPs were established against both gram negative and gram positive pathogenic bacterias using Agar well method.

3.1 XRD Analysis:

The crystal structure and average particle size of CuO NPs using plant leaf extract has been analysed by using XRD spectrum. The XRD pattern of prepared green CuO NPs is shown in Fig 1.The peaks of XRD spectrum are characteristics of the end-centered structure. Using Scherrer's formula the average grain size of the synthesized CuO NPs is calculated and its value is 4nm for sample 1, 3nm for sample 2 and 60 nm for sample 3 respectively. The diffraction data were in good co-ordination with the Joint Committee on Powder Diffraction Standards card (no:78-1588).This spectrum shows distinct diffraction peaks around 15.22(010,) 16.25(101), 18.84(002), 22.30(111), 33.75(200), 44.61(220) for sample1, 15.51(010), 16.31(101), 19.13 (002), 22.60 (111), 33.04(200), 43.75(220) for sample2 and 15.51(010), 16.25(101), 18.84(002), 22.3(111), 32.59(200), 44.734(220) for sample3 respectively.



Fig 1: XRD spectrum of green CuO NPs

3.4 SEM Analysis:

Surface morphology of Cu NPs was represented in Figs. 4 (a-c). It exhibits that almost all the NPs looks like orthorhombic in shape.



Fig 4b



3.5 EDX-Analysis:

Fig. 5 shows the EDX spectrum of CuO NPs and the elemental composition of green synthesized CuO NPs using various medicinal extracts were represented in Table. From the spectrum, it is revealed that the prepared CuO NPs have some additional related peak (S) which confirms the presence of untreated plant extracts in the samples.



Fig 5: Energy dispersive X-ray spectrum of CuO NPs

3.2 FTIR Analysis:

Fig. 2 shows that the FTIR spectra of CuO NPs synthesized using various medicinal plant extracts. From the spectra, it is revealed that the broad absorption bands around at 3819 and 1651 cm⁻¹ may be attributed to the presence of O-H stretching vibrations. A small transmittance peak at ~2361 cm⁻¹ is due to C-N stretching vibration. The vibration band at 1519.91cm-1 is due to N-H stretching vibrations. The vibration band at 1107.14cm-1 is due to C-O stretching vibration. The vibration band at~ 883cm-1 is due to C- N vibrations. The band at 617.22 cm⁻¹ is due to Cu-O stretching vibrations. The FTIR analysis showed that the functional groups present in the plant extracts which may involve in the copper oxide NPs synthesis.



Fig 2: IR spectrum of green CuO NPs

3.3 UV-visible spectrometer:

Fig 3 a. shows UV-Visible absorption spectra of green synthesized CuO NPs using various plant extracts. The reduction of $CuSO_4$ to CuO NPs during exposure of plant extracts was observed as a result of the colour change. The colour change is due to the surface Plasmon resonance (SPR) phenomenon. The metal oxide NPs have free electron which give the SPR absorption band due to the combined vibrations of electrons of metal NPs in resonance with light wave. The sharp bands of Cu NPs were observed around 360 nm in case of Andrographis Paniculata, 359 nm in case of Aegel marmelos and 362 nm in case of moringa leaf. It confirms the formation of CuO NPs. The peak value was found to be gradually decreased with increase in wavelength. From fig 3b, the calculated band gap is found to be at 3.58 eV.



Fig 3a: UV-Vis. absorption spectra of green CuO NPs



Fig 3b: Band gap energy graph of green CuO NPs

3.6 Antibacterial activity:

Antibacterial activity was done by using Agar well method .From this method we determine the diameter of inhibition growth zone and to test the extent to which bacteria are affected by those antibiotics. This method gives many advantages such as simplicity, low cost,eco-friendly. This study has the great interest in patients who suffer from bacterial infections. Antimicrobial agent such as plants extract diffuses in the agar medium and inhibits the growth of the microbial strain and measure zone of inhibition area. The effectiveness of antibiotics can be measured using their zone of inhibition. From this we conclude the best antibiotics to use against new patheogen. Fig 6 shows that zone of inhibition (ZOI) for different pathogen of copper oxide NPs. This result was effective when the size of copper oxide NPs was observed to be increased with decreased in the zone of inhibition. However the zone of inhibition was observed to be more in gram negative bacteria when compared to gram positive bacteria. From table 1 the maximum ZOI values is observed as 20mm in E-coli bacteria for 100 μ l concentration of copper oxide NPs using nilavembu plants leaves extract. This significant type of green copper oxide NPs used to antimicrobial finishing process in textile industries.





Fig 6: Zone of Inhibition (ZOI)

Table 1: Zone of inhibition value

	Zone of inhibition(mm)					
Microbes	Vilva leaf extract nps		Moringa leaf extract nps		Nilavembu leaf extract nps	
	50 µl	100 µl	50 µl	100 µl	50 µl	100 µl
MRSA(gram positive)	10mm	11 mm	6mm	7mm	8mm	10 mm
E-Coli(gram negative)	12mm	15 mm	6mm	8mm	15mm	20mm
pseudomonus aeruginosa(gram negative)	11mm	13 mm	8mm	9mm	10mm	12mm
Serratia marcescens(gram negative)	14mm	14 mm	7mm	8mm	12mm	14mm
Staphylococcus epidermidis(gram positive)	10mm	12mm	8mm	10mm	7mm	9mm

4. Conclusion:

Present study conclude that the plants Andrographis paniculata (Nilavembu), Aegle Marmelos (Vilva), Moringa Oleifera (Moringa) can be used as an excellent source for preparing CuO nps.From antibacterial activity is more in gram negative bacteria when compared to gram positive bacteria. Among these three nps ,CuO nps with Nilavembu and Vilva leaf extract has small in size (3nm,4nm) and also gives maximam zone of inhibition it says good antibacterial activity aganist some pathogenic bacterias.This significant type of green copper NPs used to antimicrobial finishing process in textile industries.The NPs offer a larger surface area compared to bulk particles [16]. Being in the nanometer range, the particles are transparent. So they do not blur colour or alter brightness of textiles substracts. So this type of green NPs is used to colour maintanence process in textiles.

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