Materials and its Characterization

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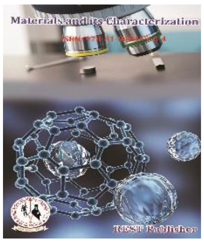

Biosynthesis of Copper Oxide Nanoparticles Using Plant Extract as Stabilizing Agent and Their Characterization

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Abstract: *In this paper, Copper oxide nanoparticles (CuO NPs) were synthesized using Tea, Peppermint and Psidium guajava leaves extract as stabilizing agent. Structure of the green synthesized CuO nanoparticles were characterized by Fourier transform infrared spectroscopy (FTIR), X-ray diffraction pattern (XRD), Scanning electron microscope (SEM) coupled with energy dispersive X-ray diffraction study. Further, the above analysis methods confirmed the formation of CuO nanoparticles. The synthesized nanoparticles exhibit zone of inhibition against pathogenic bacteria such as Escherichia Coli and Bacillus subtilius*

Keywords: *Tea leaves, Psidium guajava, Peppermint, CuO nanoparticles*

1. INTRODUCTION

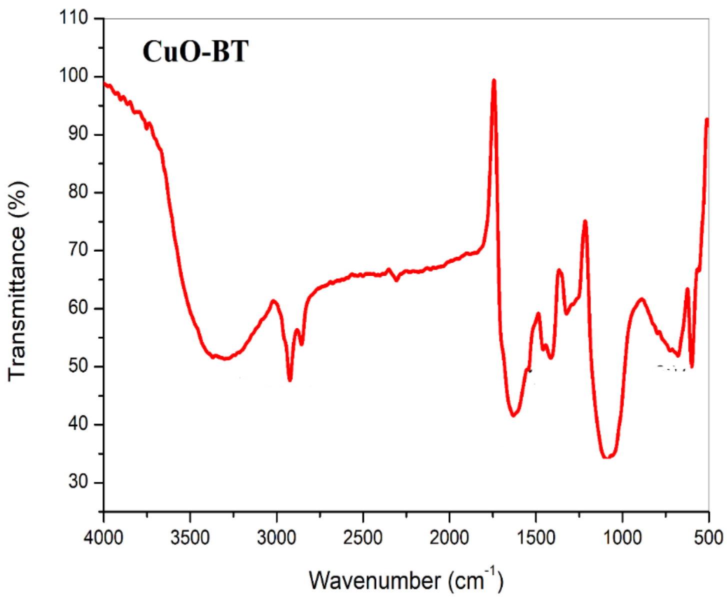
Metal nanoparticles have widespread popularity because of their application in biomedical sciences, chemical industry, electronics, drug gene delivery, and biosensor (Hedieh Malekzad et al. 2017). CuO nanoparticles were one of the most important catalysts used to eliminate industrial effluents in the environment. Earlier studies indicate that the catalytic reaction was apparently a structure sensitive process, due to the involvement of the oxygen surface lattice of CuO nanoparticles. CuO nanoparticles play an important role in the field of medicine in which they act as antioxidant (Das et al.2012), antimicrobial agents (Azam et al.2012), and pesticide formulations. A part from, it has numerous applications like gas sensors, solar energy conversion (Xu etal.2015), and catalyst (Huang et al.2006). Due to the versatile properties and diverse applications, various kinds of CuO nanostructures, namely nanorods, nano sheets, and nanodendrites, honeycomb, urchin, and dumbbell-like structures have been synthesized and studied (Kechun Li et al. 2019). Presently, material scientists and researchers focused on the development of novel routes to synthesized nanoparticles through green route as which is an eco-friendly, inexpensive, cheap, without use of harsh chemicals. Recently, green approached synthesis of metal oxide nanoparticles by plants, namely Gloriosa superb (Suresh et al.2015), Carica papaya (Sankar et al.2014), Aloe barbadensis Miller (Gunalan et al. 2012), Taber- naemontana divaricate (Sivaraj et al.2014), Emblica officinalis (Ankamwar et al.2005), and Cassia fistula (Suresh et al.2015). Copper oxide is a compound from two elements copper and oxygen, which are block d and block p elements in periodic table respectively. In a crystal copper ion is coordinated by four oxygen ions. Copper oxide (Cu2O) nanoparticles have attracted considerable attention because copper is one of the most important in modern technologies and is readily available. The most important feature is the size of the nanoparticles because it allows the tailored modelling of their optical, catalytic, electrical, and biological properties. These properties make them useful for multiple applications such as the development of cosmetics, pharmacological alternatives, paints and coatings. Green synthesis of nanoparticles using plant extracts is an emerging area of research and is potentially advantageous over chemical or microbial synthesis as it eliminates the elaborate process and can also meet large-scale production (Khan et al. 2013). CuO is a p-type semiconductor material with a narrow band gap of 1.2 eV. In recent years, they are receiving lot of attention for their versatile properties and potential use as gas sensors, solar cells, lithium-ion batteries, heterogeneous catalysts and antibacterial agents. Besides, CuO nanoparticles are stable, robust and have a longer shelf-life com- pared to organic, antimicrobial agents (Rakhshani, A. E. 1986). By considering the importance of medicinal plants, it is aimed to synthesis CuO NPs using Black tea, Peppermint leaves and Psidium guajava leaves extract and study their antibacterial activity.

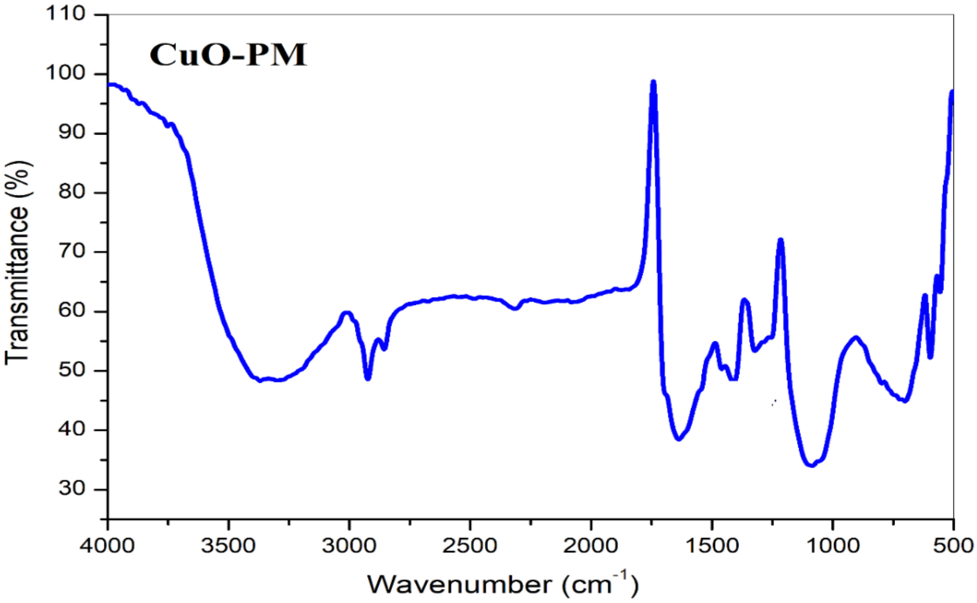
2. MATERIALS AND METHODS

Copper acetate and Ammonium hydroxide NH_4OH were purchased from Lobachemie Ltd. Mumbai, India. All the reagent were used without further purification. Tea dust, Peppermint leaves and *Psidium guajava* leaves were collected in Tiruvannamalai. Tea leaves, Peppermint leaves and *Psidium guajava* were dried in shade and powdered. All the three materials were (50 g each) was boiled with distilled water to produce extract. The filtrate was collected separately. The extract was filtrate through muslin cloth to remove insoluble particles. Further the extract was filtered through muddy filter paper. The insoluble material was completely removed and extract are used as stabilizing agent for the production of copper oxide nanoparticles. CuO nanoparticles were prepared by precipitation method. In this method, the reaction mixture was prepared by adding black tea extract, Psidium guajava leave extract and Peppermint leaves extract and copper acetate (0.1M) as a source of copper. Small amount of double distilled water was added to get homogenous mixture under constant stirring for ~10 min. The reaction mixture taken in a beaker and put on magnetic stirrer. Ammonium hydroxide was dropwise added to the copper acetate solution with continuous stirring. The light green precipitate was obtained as product. The precipitate was removed by careful centrifugation and thoroughly washed with double distilled water. Then the precipitate was thoroughly washed with ethanol to remove organic impurities. The solid was dried at 80°C for 3 hours. The precipitate was annealed at 500°C for 4 hours to convert copper hydroxide (Cu $(OH)_2$ into copper oxide (CuO). The obtained products of CuO NPs were stored in air tight container for further analysis. CuO NPs synthesized using *Tea*, *Psidium guajava* and *Pippermint* extract were named CuO-BT, CuO-PM and CuO-PG, respectively. Fourier transform infrared (FT-IR) spectra were recorded on a Bruker Tensor 27 spectrometer in the frequency range of 4000–400 cm^{-1}. The XRD patterns of copper oxide nanoparticles were obtained using X-ray diffractometer (X PERTPRO, PANalitical, Germany) equipped Cu Ka (l = 1.541A°) radiation source in the range of 20°–80°, operating at 40 kV and 40 mA. Morphological features were studied by using Hitachi-7000 Scanning Electron Microscopy (SEM) coupled with EDSX. The antibacterial activity of phytosynthesized CuO NPs was done by well diffusion method. The micro-organisms such as *Bacillus subtilis and Escherichia Coli* were cultured on nutrient agar plates. The CuO NPs 20 µl of sterile nutrient agar medium was poured to petriplates and overnight grown bacterial cultures were swabbed on the agar surface. Four wells (5 mm) were made in each plate using a cork borer and 50 µL of CuO NPs (1 mg/ml), antibiotic disc and water were added to each plate to determine the antibacterial activity of CuO NPs. The zone of inhibition was calculated for each tested organism after 24 h of incubation at 37°C.

3.RESULTS AND DISCUSSION

In this chapter, CuO NPs were synthesized using *Black tea*, *Psidium guajava* and *Pippermint* leaves extract as reducing agent and the structure of CuO NPs were confirmed by using Fourier transform infrared spectrometer (FTIR), X-ray diffraction pattern (XRD), Scanning electron microscopy (SEM) and Energy dispersive X-ray diffraction study. Appropriate concentration of copper acetate (0.1 M) was used precursor for the synthesis of CuO nanoparticles. The copper acetate solution was taken in a 100 ml beaker, ammonium hydroxide was dropwise added which produced blue colour precipitate. The precipitate was thoroughly washed, characterized, and used as antibacterial agent against pathogenic bacteria. The functional groups present in the synthesized CuO were determined by IR analysis recorded in the range of 400-4000 cm^{-1} as shown in Figure 1. A broad band at 3347 cm^{-1} corresponds to the O-H group which may present due to the presence of phenolic compounds. The peaks 2925 cm^{-1} is due to impurities. The bands 1624 and 1399 cm^{-1} are attributed to (C=O) and (C-O) functional groups of carboxylic acids. The peak at 1079 and 694 cm^{-1} confirms the presence of Cu-O vibrations [Sankar et al 2014]. IR analysis revealed that the functional groups of both bioactive compounds and CuO were present in the extract derived CuO NPs.





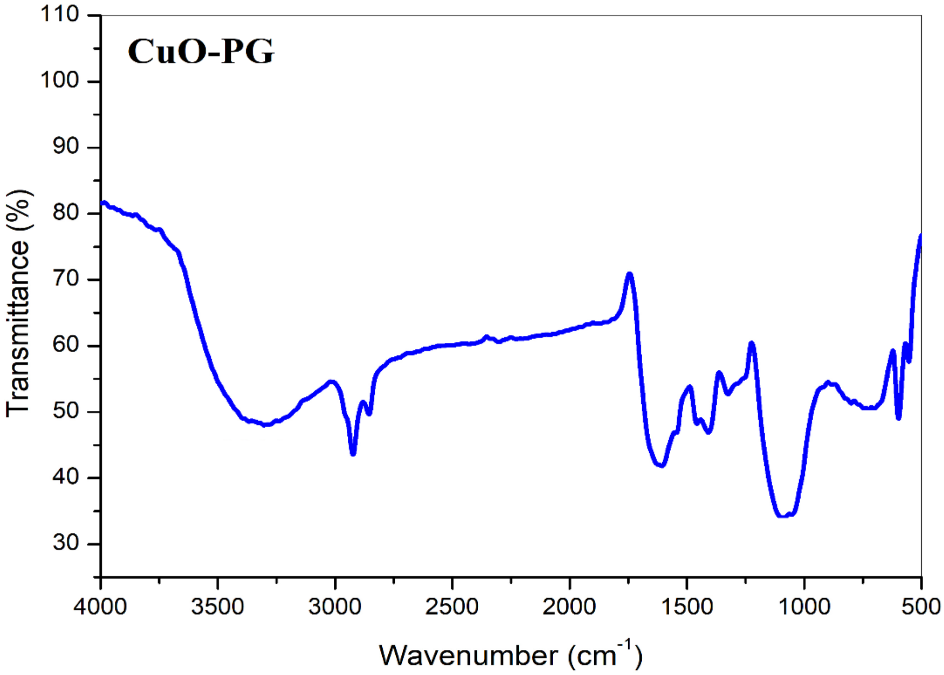
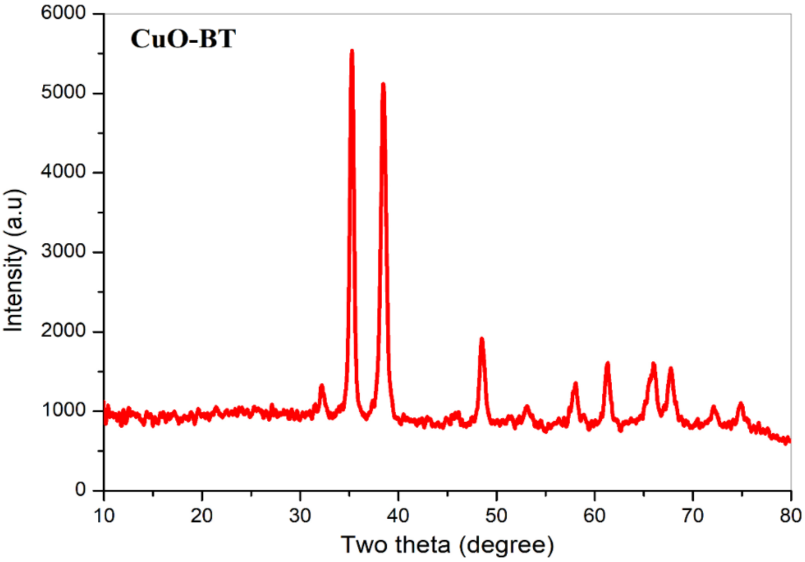


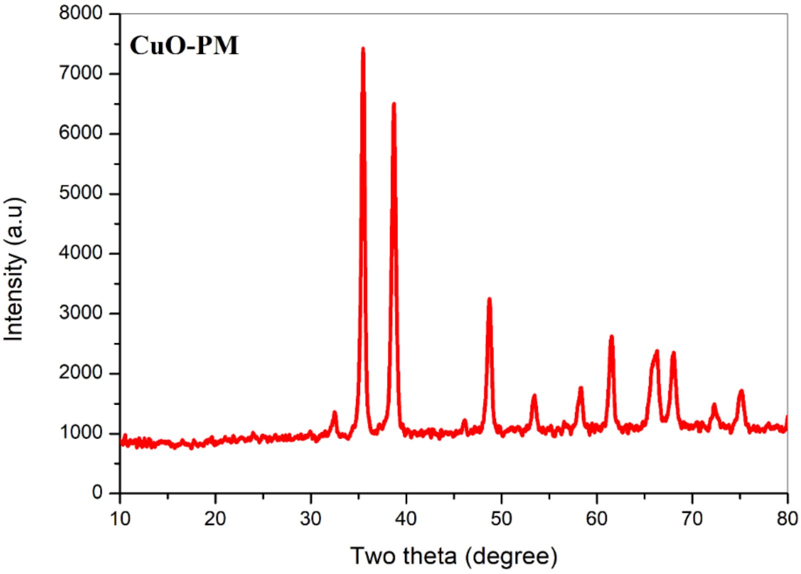
FIGURE 1. FTIR spectra of CuO NPs synthesized using Black tea (CuO-BT; Peppermint leaves extract (CuO-PM and Psidium guajava leaves extract (CuO-PG)

The existence of the apparent and sharp peaks with different 2θ values obviously confirmed the crystallinity of CuO NPs. The diffraction pattern of CuO NPs is shown in Figure 2. The XRD pattern showed diffraction peaks at 2θ of 33.07, 35.25, 38.59, 48.62, 53.27, 58.08, 61.57, 66.23, 67.97, 72.18 and 75.24^{o} which can be designated (110), (-111), (111), (-202), (020), (202), (-113), (-311), (220), (311) and (004) planes, respectively. The analysis of given Bragg reflection of quite prominent peaks can be indexed to the monoclinic phase of CuO (No.96-410-5686). Similarly, XRD of CuO NPs prepared using starch solution and banana peel extract showed that the nanoparticles are monoclinic structure. The XRD pattern showed no additional peaks, revealing that the synthesized CuO is highly pure in nature. The average crystallite size of the biosynthesized copper oxide nanoparticles has been estimated using Scherer formula (Patterson 1939):

$$D = K\lambda/\beta_{1/2} \cos \theta$$

where D is the crystallite size, k is the wavelength of the X-ray source (l.154l6 nm), b is the FWHM and h is the angle of diffraction. According to the formula, average size of the copper oxide nanoparticles has been calculated. The average crystallite size of pure CuO and CuO synthesized using black tea extract, peppermint extract and *Psidium guajava* leaves extract found to be 104, 80 and 40 nm, respectively.





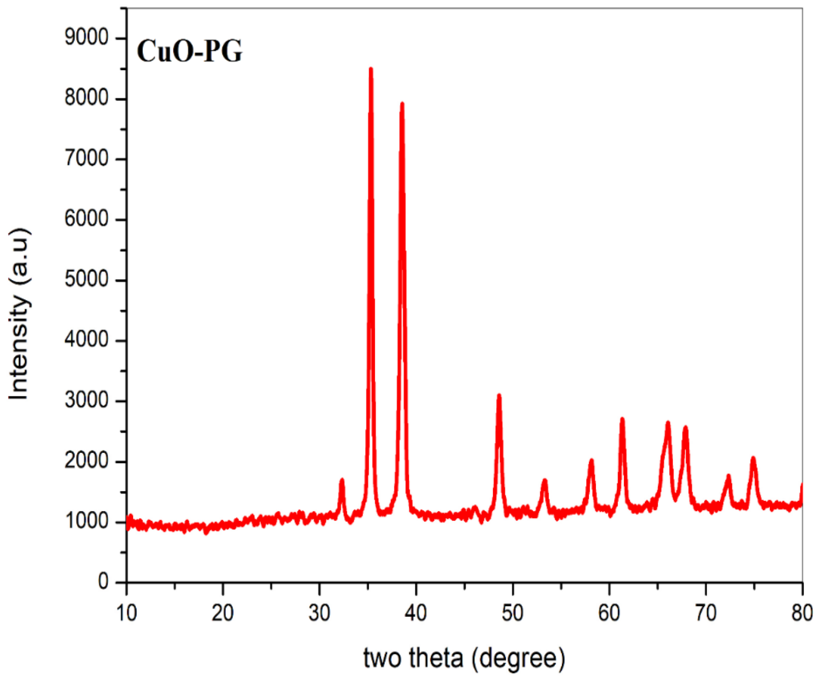
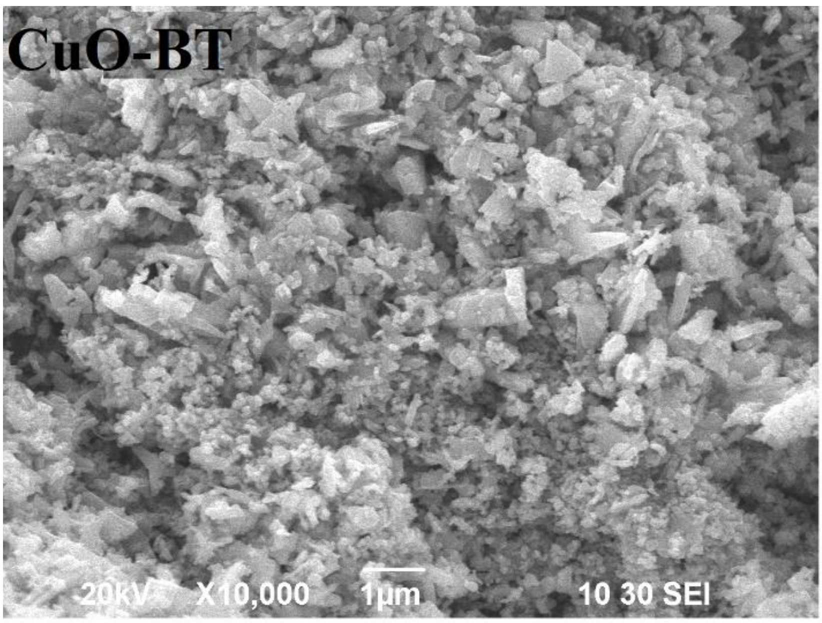
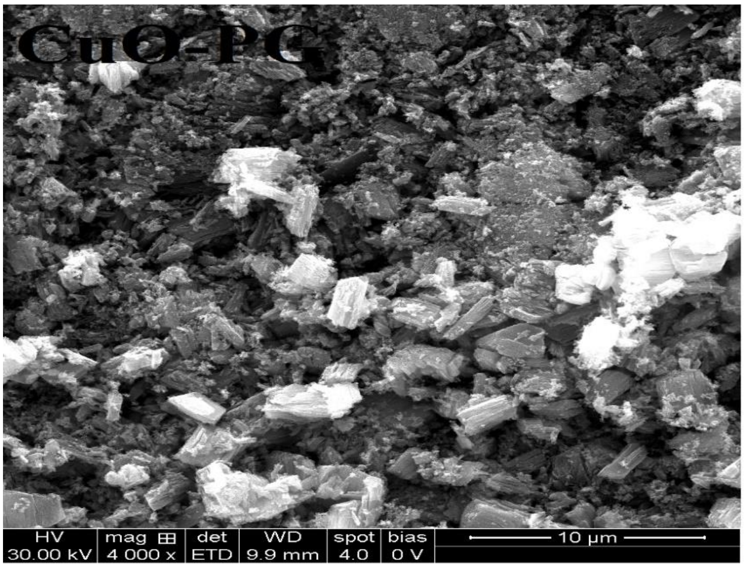


FIGURE 2. XRD pattern of CuO nanoparticles synthesized using Black tea (CuO-BT; Peppermint leaves extract (CuO-PM and Psidium guajava leaves extract (CuO-PG)

The SEM images of pure CuO nanoparticles synthesized by chemical precipitation method are shown in Figure 3. Particles found to be mixed with each other. Some square shape, spherical and irregular shape particles are seen in the SEM images confirmed the formation of CuO NPs. However, particles are uniformly distributed. From the SEM image, it is confirmed the formation CuO NPs in this reaction. However, the exact shape of the particles may not be identified. Elemental compositions of CuO NPs were determined using energy dispersive X-ray diffraction pattern (EDAX).





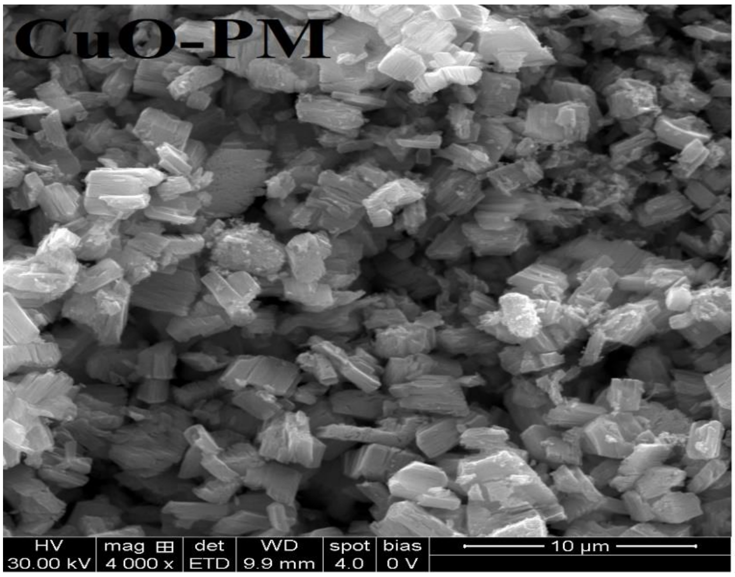
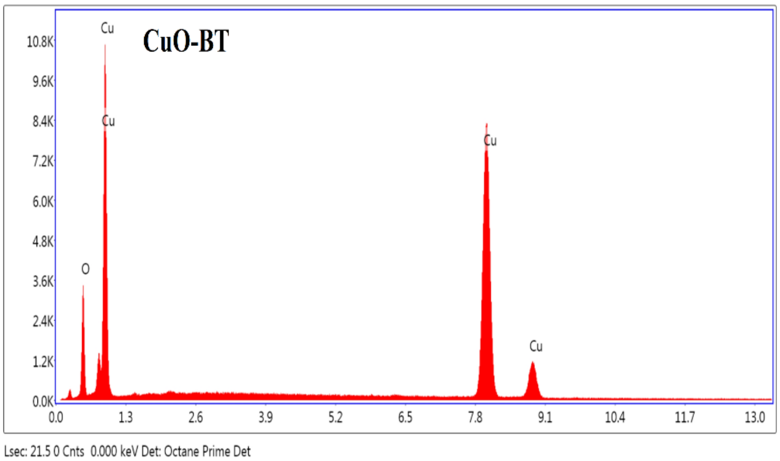
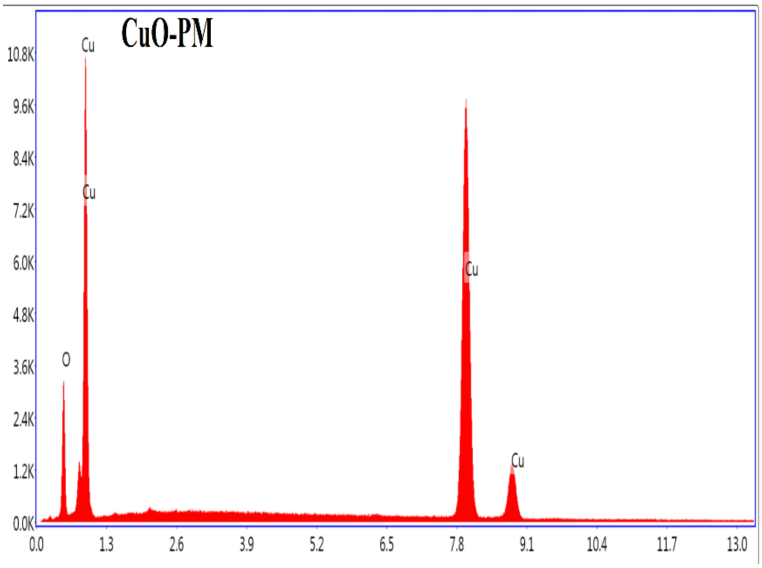


FIGURE 3. SEM image of CuO nanoparticles synthesized using *Black tea* (CuO-BT; *Peppermint* leaves extract (CuO-PM and *Psidium guajava* leaves extract (CuO-PG)





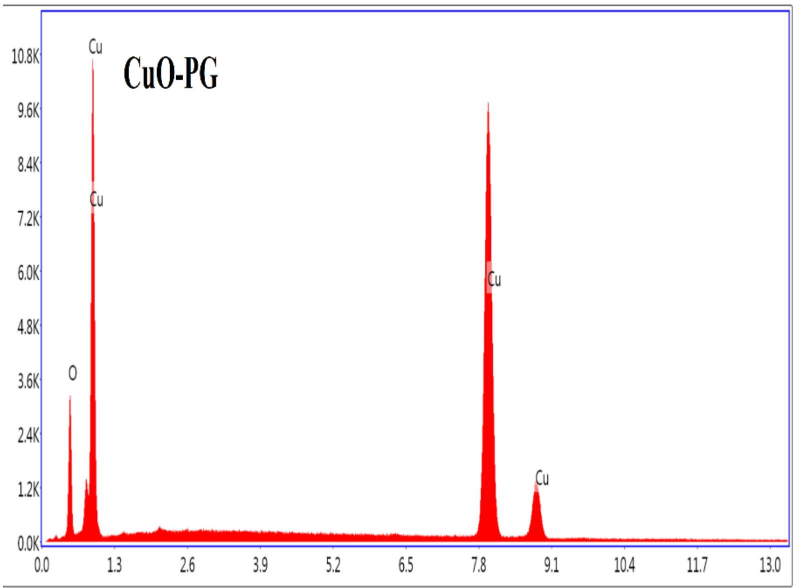
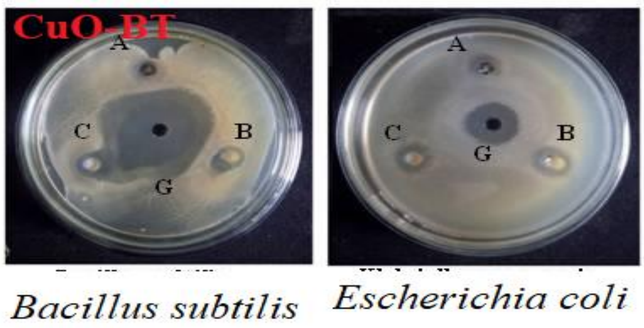


FIGURE 4. EDAX analysis of CuO NPs (Black tea (CuO-BT; Peppermint leaves extract (CuO-PM and Psidium guajava leaves extract (CuO-PG). EDAX spectrum of CuO-BT, CuO-PM and CuO-PG are shown in Figure 4. The existence of Copper and Oxygen in each spectrum confirmed the elemental compositions.

The antibacterial activity of CuO NPs and zone of inhibition is presented in Figure 5. CuO NPs showed significant inhibition on gram negative bacteria such as *Bacillus subtilis and Escherichia Coli.*



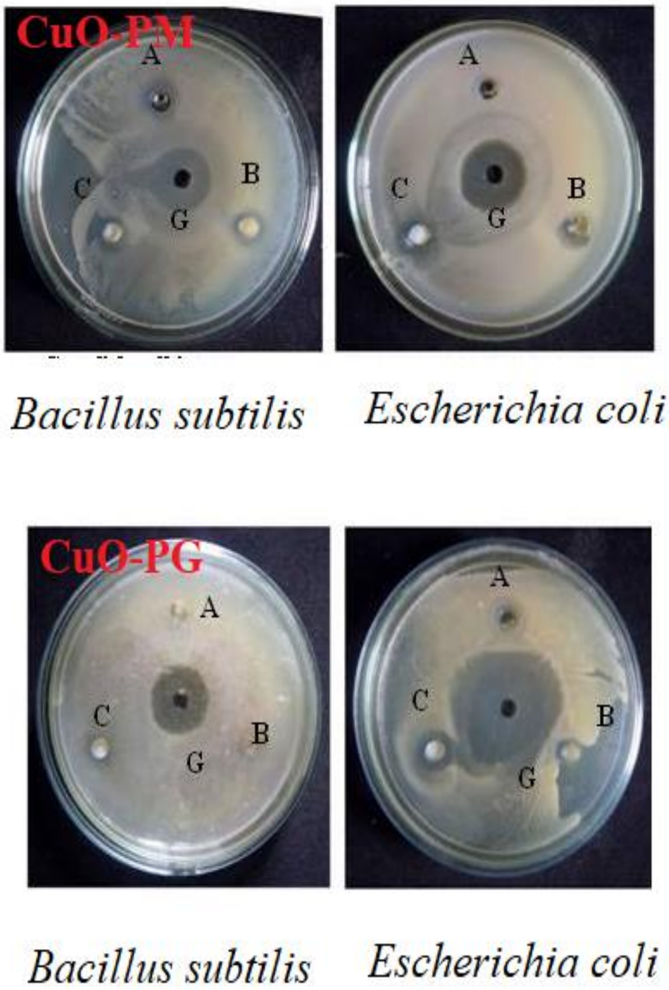


FIGURE 5. Zone of inhibition shown by CuO NPs bacteria against bacteria; Bacillus subtilis and Escherichia Coli.

Where, A, B and C reveals the 25, 50 and 75 µg/mL and, G reveals the Antibiotic used as a reference compound. As seen in the Figure 5 the CuO NPs exhibit less zone of inhibition against bacteria. The antibacterial activity of CuO NPs may be attributed by its binding on the bacterial cell membrane and inhibit the active transport process which results in cell lysis [Katwal et al 2014]. The larger surface area of nanoscale CuO NPs has better bactericidal activity than the larger particles. Antibacterial activity results showed that CuO NPs possess antibacterial potential. However, the zone of inhibition shown by the *Bacillus subtilis and Escherichia Coli* are 8 mm and 10 mm, respectively reveals the miderate antibacterial activity of CuO nanoparticles.

4. CONCLUSION

CuO NPs was successfully synthesized by chemical precipitation method using black tea extract, Peppermint leaves extract and Psidium guajava leaves extract as natural reducing agent. The sample was characterized using FTIR, XRD and SEM techniques. The XRD pattern reveals the crystalline nature of the CuO NPs. These extracts are significantly impact on physico-chemical properties of CuO NPs. The SEM image reveals the shape and morphology the CuO NPs. The synthesized CuO NPs can be used antibacterial agent to kill the Bacillus subtilis and Escherichia Coli.

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