SYNTHESIS AND CHARACTERIZATION OF CUO NANOPARTICLES BY ALOE BARBADENSIS LEAVES

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Abstract. Nanotechnology becomes the most innovative and interesting field for researchers in last decade. Nanotechnology deals with particles of size 1-100 nm. Recent advances in science and nanotechnology leads to the wide applications of metal and metal oxide nanoparticles in several fields. Copper oxide (CuO) nanoparticles have a large potential due to its distinct properties and environment friendly behaviour. There are several methods to synthesize CuO nanoparticles but due to environment benign process, availability of plants, non-toxicity and cheap process we prefer green approach. In this paper we synthesize CuO nanoparticles via green route in which Aloevera leaf (Aloe Barbadensis) extract used as reducing and capping agent. The formation of CuO can be found with naked eyes. The phytochemicals present in leaf extract plays an important role in synthesis. In synthesis process copper sulphate penta hydrate ($Cu(So_4)_2.5H_2O$) use as metal precursor salt and NaOH used to maintain the pH of the solution. The synthesis process has been carried out at temperature 50 ^{0}C. The synthesized nanoparticles thereby obtained characterized by various analytical techniques like FESEM with EDX, XRD, FTIR and UV-Visible. The surface morphology of spherical shape revealed by Field Emission Scanning Electron Microscope (FESEM), elemental analysis by Energy Dispersive analysis (EDX), Fourier Transform Infrared (FTIR) revealed the different groups attached with CuO nps, the structure and average particle size of 5-6 nm estimated by XRD and the Optical properties revealed by UV-Vis spectroscopy.
Keywords: *copper oxide, nanoparticles, green synthesis, plant extract*

Introduction

Green approach becomes the most pronounced and effective method for synthesizing nanoparticles due to its non-toxic features, environment friendly behavior, recyclability and cost effective methods as compared to other physical and chemical methods. The Green route involves different biological agents like bacteria, fungi, algae but plant extract method proved himself more effective than other biological agent by various studies (Waris et al., 2021; Bandeira et al., 2020; Yashni et al., 2020; Gour and Jain, 2019; Lai et al., 2019; Reddy, 2017). In green approach plant extract serves as reducing and capping agent for synthesization of nanoparticles of desired shape and morphology. The concentration of phytochemicals present in plant extract, pH of solution ,Reaction temperature are the different parameters which affect the synthesis process of metal or metal oxide nanoparticles (Akintelu and Folorunsi, 2020). Metal and Metal oxides have a large scope for various fields like Physics, Chemistry, Environment science ,material science and many others. Copper (Cu) is very important element for humans, plants, and animals, and involved in the functions of human immune cells which are responsible for the killing of pathogens (Latorre et al., 2019).

Due to the abundance and vast properties of copper we report CuO nanoparticles via Green method by using Aloevera plant leaves extract. The phytochemicals works as reducing and stabilizing agent. The phytochemicals which are present in aloevera are protein, carbohydrates, phenols, tannin, steroids, terpenoids and glycosides which perform the reduction mechanism of nanoparticles (Bista et al., 2020; Chakraborty et al., 2020). The property of nanoparticles varies with their size and morphology. Copper oxide nanoparticles proved their importance as antibacterial activity, in magnetic phase transitions, gas sensors, as a catalyst and also in the field of superconductivity (Anantharaman et al., 2016; Ahamed et al., 2014; Sutradhar et al., 2014; Srivastava, 2013; Durán et al., 2011; Ren et al., 2009). It had been also reported that the copper oxide nanoparticles had more thermodynamic stability than pure copper and also have high dielectric constant (Ahamed et al., 2014). The synthesis of CuO nanoparticles also reported by green synthesis of CuO NPs by Arjuna bark extract (Yallappa et al., 2013), Malva sylvestris leaf extract (Awwad et al., 2015), carica papaya (Sankar et al., 2014), Lxiro Coccinea plant leaves (Vishveshvaret al., 2018), Moringa Oleifera leaves (Galan et al., 2018), Juglans regia leaf extract (Asemani and Anarjan 2019) and many more. In this paper plant extract mediated synthesized CuO nanoparticles were reported and characterized by different techniques.

Materials and Methods

Materials used are: (1) Copper Sulphate pentahydrate $Cu(SO_4)_2.5H_2O$ as precursor salt; (2) Aloevera leaf extract as reducing and capping agent; (3) NaOH Pallets to maintain pH; (4) Whatmann filter paper 12 for filtration; and (5) DI water as synthesis medium.

Preparation of Aloevera leaf extract

Fresh leaves of Aloevera collected from local region wash thoroughly with tap water and again washed thoroughly with Double distilled water to remove the contaminating particles then after removing its thorns Leaves cut in to fine pieces and leave them to dry for 15 days after that using Grinder found a fine powder. 200 m.l. D.I. mixed with 25 gm aloevera leaves powder and stirrer for 45 minutes at 60 $^{\circ}$C and after filtering we get the aloevera leaf extract.

Synthesis of CuO nanoparticles

For synthesis of CuO nanoparticles $Cu(So_4)_2.5H_2O$ (2.5 gm) mixed in 100 ml D.I. (Solution A) and 4 gm NaOH Pallets dissolved in 50 ml DI (Solution B) then mixed solution A and Solution B with 20 ml aloevera leaf extract. The whole mixture was stirred at 50 $^{\circ}$C for 3 hours in Glove box to avoid the environment contamination. The solution becomes blackish in Colour after 3 Hours which shows the formation of nanoparticles. Leave it for 10 days. After thoroughly washing with DI water, filtering and drying in oven overnight we found CuO nanoparticles in powder form.

Analysis of CuO nanoparticles

The CuO nanoparticles thereby obtained were characterized by different analytical techniques. X-Ray Diffractometer (XRD) reveals the average crystalline size, Field Emission Scanning Electron Microscope (FESEM) reveals the morphology of nanoparticles, detection of elemental composition by Energy dispersive spectroscopy (EDS), Optical properties by UV-Visible spectroscopy (UV-Vis), and the presence of different functional groups revealed by Fourier Transform Infrared (FTIR) spectroscopy.

Results and Discussion

X-Ray Diffraction (XRD)

The crystalline size and structure can be revealed by X-ray diffraction technique. The XRD pattern recorded for a wide range of angles from 10^0 to 80^0 which is shown in *Figure 1*.The XRD pattern shows the diffraction peaks at 32.63, 35.60, 38.65, 49.08, 53.36, 57.96, 61.52, 66.47, and 67.58 which indexed to diffracting planes to (-110), (111), (200), (-202), (020), (202), (-113), (-311), and (113) as reported by Sharma et al. by JCPDS file no.45-0937 (Sharma and Kumar, 2020).The observations are in good agreement with data files. The average particle size was estimated by Debye Scherrer formula as $D=0.9\lambda/\beta\cos\theta$, where $\lambda=1.5406$ Å wavelength of incident X rays, β defines the Full width at half maximum (FWHM) in radians and θ shows the diffracting angle in radians. The crystalline size determined with respect to two intense peaks found between the ranges of 30^0 to 40^0 which provides the crystalline size 5-6 nm.

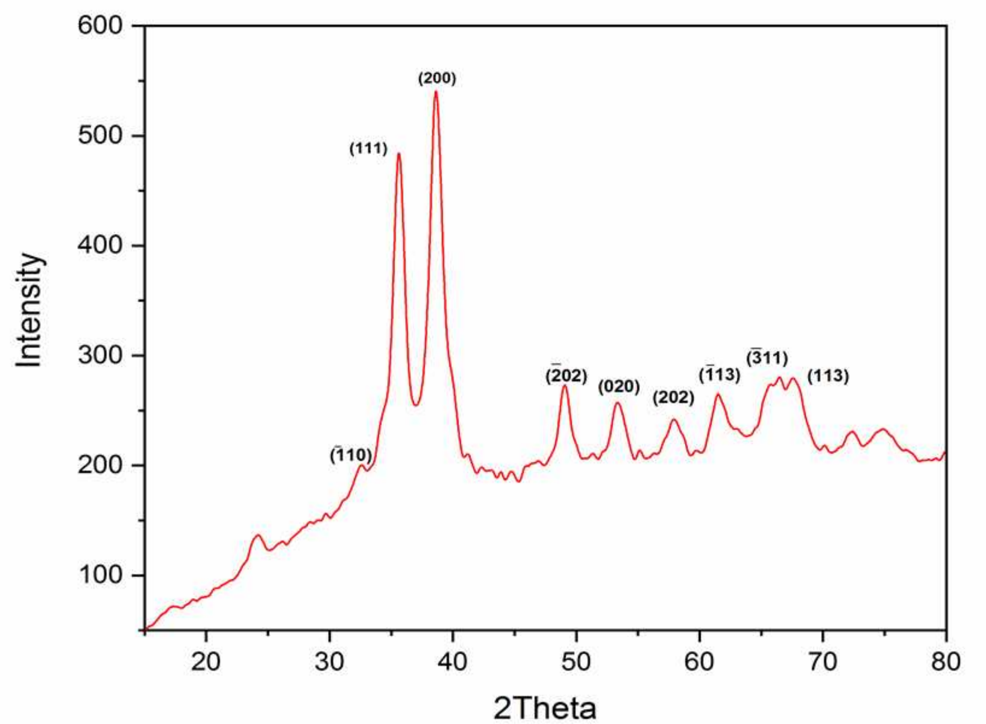

Figure 1. *XRD pattern of CuO np's.*

Scanning Electron Microscope (SEM)

The morphology and structure of synthesized nanoparticles were revealed by FESEM images as shown in *Figure 2* and *Figure 3*.The SEM images reveals that the synthesized nanoparticles are of spherical shape with aggregation due to large surface area and high surface energy (Pakzad et al., 2019). The elemental analysis revealed by

EDS pattern (*Figure 4*). The signals appeared in EDS pattern revealed the existence of Cu and O in our sample with atomic weight percent of 75.73 % and 24.27% respectively as shown in *Table 1*.

Table 1. *Atomic weight percentage of EDAX spectrum.*

Spectrum Number	Element	Series	Weight (%)	Atomic (%)
1	Cu	K	92.53	75.73
2	O	K	7.47	24.27
	Total		100	100

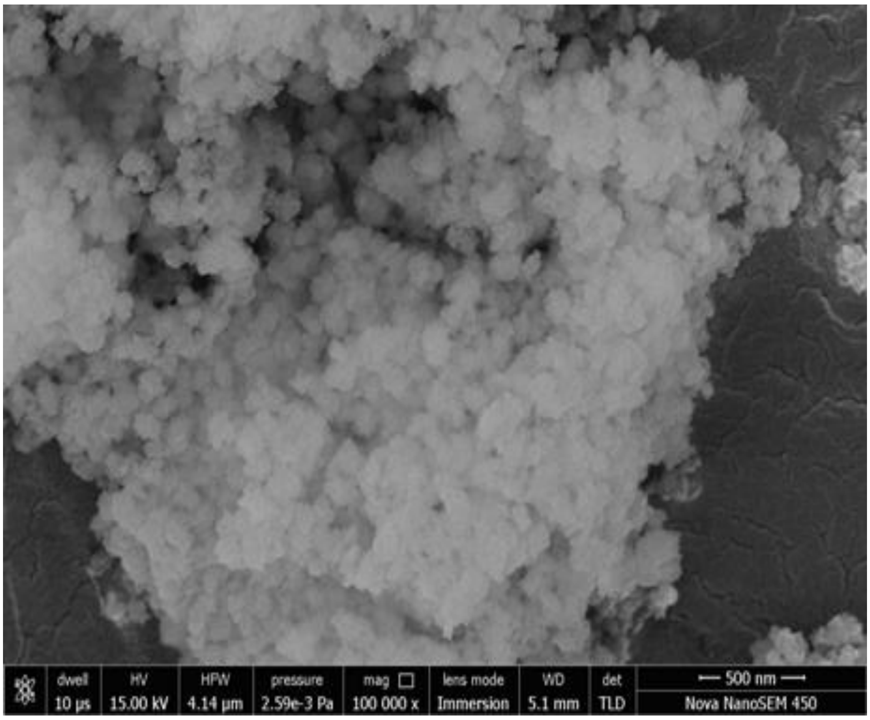

Figure 2. *SEM micrograph of CuO np's.*

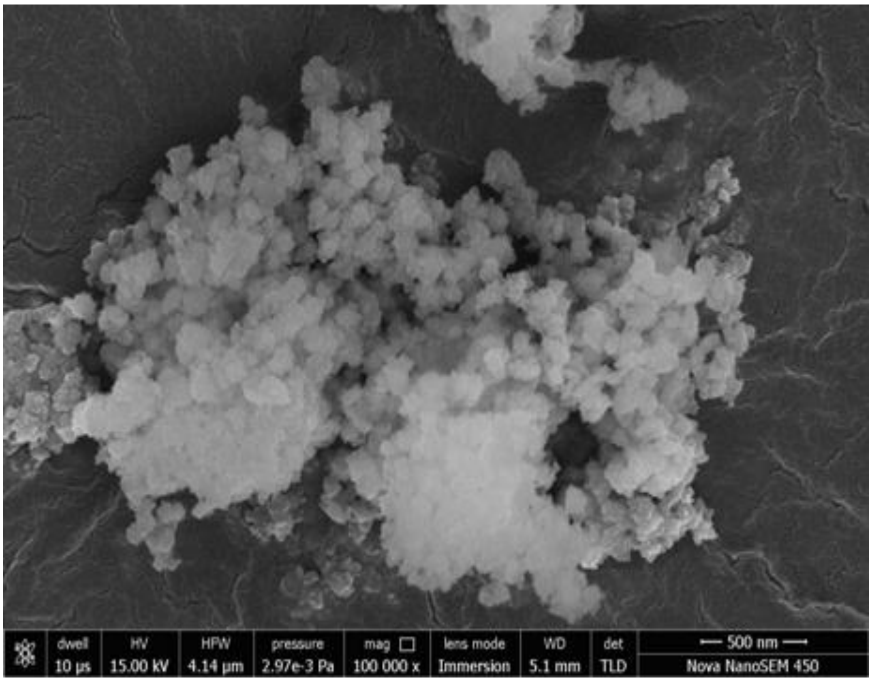

Figure 3. *SEM micrograph of CuO np's.*

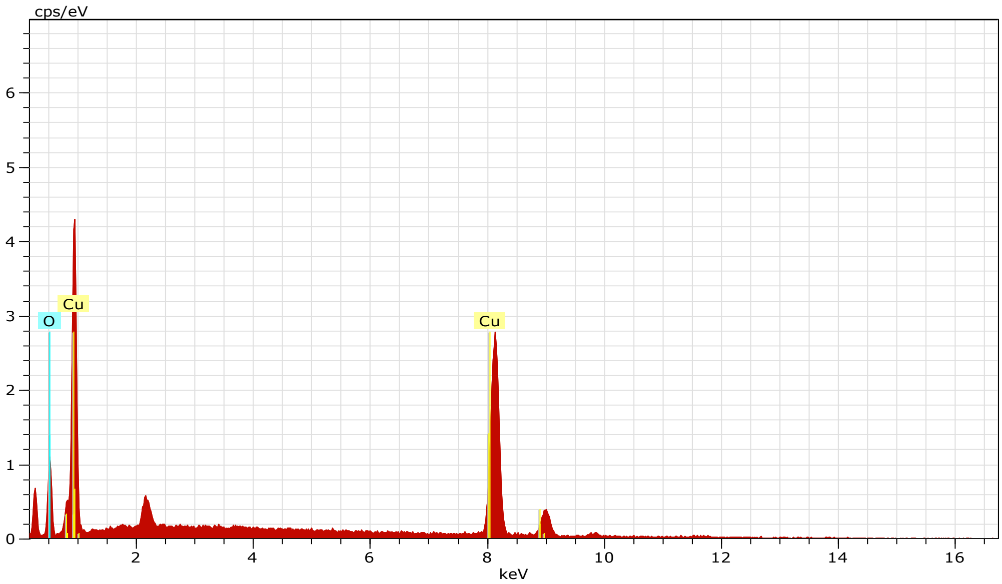

Figure 4. *EDS spectra of CuO np's.*

UV-Visible Spectroscopy

Ultraviolet and visible spectroscopy is used for optical measurements and further quantitative analysis of the samples for band gap. UV-Visible spectroscopy is based on Beer-Lambart law to calculate the Plasmon resonance and oscillation of conduction electrons with electromagnetic waves. In this spectroscopy incident beam get splitted in two parts half travels with sample and remaining half travels through reference material. The light absorbed by the sample explores the surface Plasmon resonance (Akintelu et al., 2020). UV-Visible spectra revealed the absorbance peaks in absorbance spectra at 220 nm (Renuga et al., 2020) and 388 nm (*Figure 5*) which is in good agreement with the results reported by the different researchers (Gebremedhn et al., 2019). The SPR band at 388 nm may due to the cause of smaller size (Velsankar et al., 2020). It also revealed the red shift in wavelength which explains the newly formed nanoparticles of different size and shape (Gebremedhn et al., 2019).

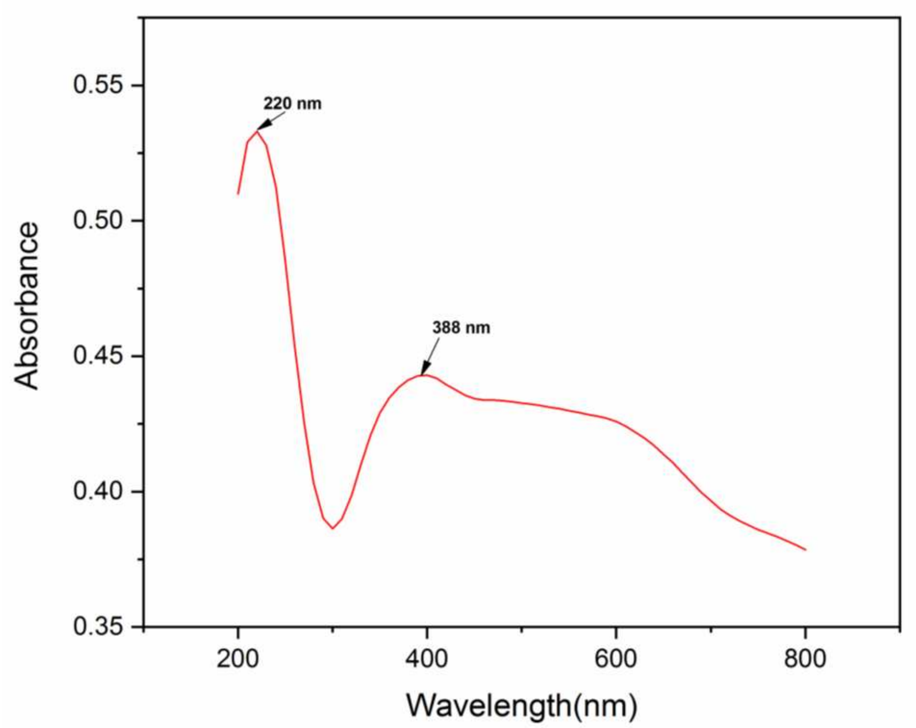

Figure 5. *UV-Visible spectra of CuO np's.*

FTIR Spectroscopy

Figure 6 shows the FTIR spectra of synthesized CuO nanoparticles in the range 4000-400cm^{-1}. FTIR spectra revealed the functional groups which are attached with the surface of nanoparticles. The broad peaks in the range of 3300-3600 cm^{-1} corresponds to the presence of stretching vibrations of hydroxyl groups (O-H). The peak at 1613.66 cm^{-1} reveals the presence of C-C-vibrations. The peak at 932.80 cm^{-1} shows the C-H bending. The interesting result provided by the peaks found at 701.48 cm^{-1}, 594.92 cm^{-1}, 515.12 cm^{-1} and 483 cm^{-1}.These peaks shows the presence of Cu-O vibrations and also verifies the purity of CuO nanoparticles (Siddiqi and Husen, 2020; Velsankar et al., 2020; Pakzad et al., 2019; Rajendaran et al., 2019).

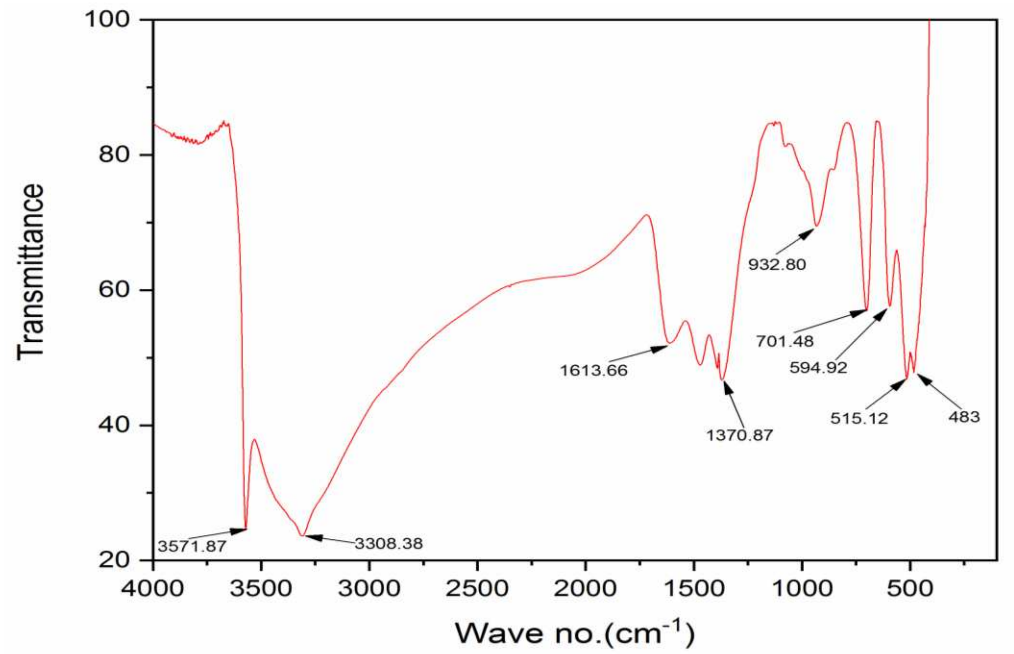

Figure 6. *FTIR spectra of CuO np's.*

Conclusion

In this paper, CuO nanoparticles were successfully synthesized by an ecofriendly and cost effective approach (Green synthesis) by using Copper sulphate penta hydrate ($Cu(So_4)_2.5H_2O$) as precursor salt and aloevera leaf extract as reducing and capping agent. The crystalline size of 5-6 nm revealed by XRD analysis SEM reveals the morphology of nanoparticles of spherical shape. EDS pattern reveals the elemental composition of sample which is in good agreement with other results. The optical characteristics revealed by UV-Visible spectroscopy which shows absorption peaks at 220 nm and 388 nm. FTIR spectra reveals the functional groups which are attached with the surface of nanoparticles FTIR peaks at 483 cm^{-1}, 515 cm^{-1} strongly prove the existence of CuO stretching in our sample. Due to cost effectiveness and environment friendly nature the Green route for synthesization have a remarkable potential in the field of nanotechnology.

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Conflict of interest

There are no conflict of interest involve with any parties in this research study.

REFERENCES

[1] Ahamed, M., Alhadlaq, H.A., Khan, M.A., Karuppiah, P., Al-Dhabi, N.A. (2014): Synthesis, characterization, and antimicrobial activity of copper oxide nanoparticles. – Journal of Nanomaterials 4p.

[2] Akintelu, S.A., Folorunso, A.S. (2020): A review on green synthesis of zinc oxide nanoparticles using plant extracts and its biomedical applications. – BioNanoScience 10(4): 848-863.

[3] Akintelu, S.A., Folorunso, A.S., Folorunso, F.A., Oyebamiji, A.K. (2020): Green synthesis of copper oxide nanoparticles for biomedical application and environmental remediation. – Heliyon 6(7): 12p.

[4] Anantharaman, A., George, L., George, M. (2016): Green synthesis of copper oxide nanoparticles and its applications. – International Journal of Advance Research in Science 5(9): 522-529.

[5] Asemani, M., Anarjan, N. (2019): Green synthesis of copper oxide nanoparticles using Juglans regia leaf extract and assessment of their physico-chemical and biological properties. – Green Processing and Synthesis 8(1): 557-567.

[6] Awwad, A.M., Albiss, B.A., Salem, N.M. (2015): Antibacterial activity of synthesized copper oxide nanoparticles using Malva sylvestris leaf extract. – SMU Medical Journal 2(1): 91-101.

[7] Bandeira, M., Giovanela, M., Roesch-Ely, M., Devine, D.M., da Silva Crespo, J. (2020): Green synthesis of zinc oxide nanoparticles: A review of the synthesis methodology and mechanism of formation. – Sustainable Chemistry and Pharmacy 15: 10p.

[8] Bista, R., Ghimire, A., Subedi, S. (2020): Phytochemicals and Antioxidant Activities of Aloe Vera (Aloe Barbadensis). – Journal of Nutritional Science and Healthy Diet 1(1): 25-36.

[9] Chakraborty, S., Farida, J.J., Simon, R., Kasthuri, S., Mary, N.L. (2020): Averrhoe carrambola fruit extract assisted green synthesis of ZnO nanoparticles for the photodegradation of congo red dye. – Surfaces and Interfaces 19: 6p.

[10] Durán, N., Marcato, P.D., Durán, M., Yadav, A., Gade, A., Rai, M. (2011): Mechanistic aspects in the biogenic synthesis of extracellular metal nanoparticles by peptides, bacteria, fungi, and plants. – Applied Microbiology and Biotechnology 90(5): 1609-1624.

[11] Galan, C.R., Silva, M.F., Mantovani, D., Bergamasco, R., Vieira, M.F. (2018): Green synthesis of copper oxide nanoparticles impregnated on activated carbon using Moringa oleifera leaves extract for the removal of nitrates from water. – The Canadian Journal of Chemical Engineering 96(11): 2378-2386.

[12] Gebremedhn, K., Kahsay, M.H., Aklilu, M. (2019): Green synthesis of CuO nanoparticles using leaf extract of catha edulis and its antibacterial activity. – Journal of Pharmacy and Pharmacology 7: 327-342.

[13] Gour, A., Jain, N.K. (2019): Advances in green synthesis of nanoparticles. – Artificial Cells, Nanomedicine, and Biotechnology 47(1): 844-851.

[14] Lai, X., Guo, R., Xiao, H., Lan, J., Jiang, S., Cui, C., Ren, E. (2019): Rapid microwave-assisted bio-synthesized silver/Dandelion catalyst with superior catalytic performance for dyes degradation. – Journal of Hazardous Materials 371: 506-512.

[15] Latorre, M., Troncoso, R., Uauy, R. (2019): Biological aspects of copper. – In Clinical and Translational Perspectives on Wilson Disease, Academic Press 7p.

[16] Pakzad, K., Alinezhad, H., Nasrollahzadeh, M. (2019): Green synthesis of Ni@ Fe3O4 and CuO nanoparticles using Euphorbia maculata extract as photocatalysts for the degradation of organic pollutants under UV-irradiation. – Ceramics International 45(14): 17173-17182.

[17] Rajendaran, K., Muthuramalingam, R., Ayyadurai, S. (2019): Green synthesis of Ag-Mo/CuO nanoparticles using Azadirachta indica leaf extracts to study its solar photocatalytic and antimicrobial activities. – Materials Science in Semiconductor Processing 91: 230-238.

[18] Reddy, K.R. (2017): Green synthesis, morphological and optical studies of CuO nanoparticles. – Journal of Molecular Structure 1150: 553-557.
[19] Ren, G., Hu, D., Cheng, E.W., Vargas-Reus, M.A., Reip, P., Allaker, R.P. (2009): Characterisation of copper oxide nanoparticles for antimicrobial applications. – International Journal of Antimicrobial Agents 33(6): 587-590.
[20] Renuga, D., Jeyasundari, J., Athithan, A.S., Jacob, Y.B.A. (2020): Synthesis and characterization of copper oxide nanoparticles using Brassica oleracea var. italic extract for its antifungal application. – Materials Research Express 7(4): 7p.
[21] Sankar, R., Manikandan, P., Malarvizhi, V., Fathima, T., Shivashangari, K.S., Ravikumar, V. (2014): Green synthesis of colloidal copper oxide nanoparticles using Carica papaya and its application in photocatalytic dye degradation. – Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy 121: 746-750.
[22] Sharma, S., Kumar, K. (2020): Aloe-vera leaf extract as a green agent for the synthesis of CuO nanoparticles inactivating bacterial pathogens and dye. – Journal of Dispersion Science and Technology 14p.
[23] Siddiqi, K.S., Husen, A. (2020): Current status of plant metabolite-based fabrication of copper/copper oxide nanoparticles and their applications: a review. – Biomaterials Research 24: 1-15.
[24] Srivastava, S. (2013): Synthesis and characterisation of copper oxide nanoparticles. – IOSR Journal of Applied Physics 5: 61-65.
[25] Sutradhar, P., Saha, M., Maiti, D. (2014): Microwave synthesis of copper oxide nanoparticles using tea leaf and coffee powder extracts and its antibacterial activity. – Journal of Nanostructure in Chemistry 4(1): 6p.
[26] Velsankar, K., RM, A.K., Preethi, R., Muthulakshmi, V., Sudhahar, S. (2020): Green synthesis of CuO nanoparticles via Allium sativum extract and its characterizations on antimicrobial, antioxidant, antilarvicidal activities. – Journal of Environmental Chemical Engineering 8(5): 13p.
[27] Vishveshvar, K., Krishnan, M.A., Haribabu, K., Vishnuprasad, S. (2018): Green synthesis of copper oxide nanoparticles using Ixiro coccinea plant leaves and its characterization. – BioNanoScience 8(2): 554-558.
[28] Waris, A., Din, M., Ali, A., Ali, M., Afridi, S., Baset, A., Khan, A.U. (2021): A comprehensive review of green synthesis of copper oxide nanoparticles and their diverse biomedical applications. – Inorganic Chemistry Communications 123: 13p.
[29] Yallappa, S., Manjanna, J., Sindhe, M.A., Satyanarayan, N.D., Pramod, S.N., Nagaraja, K. (2013): Microwave assisted rapid synthesis and biological evaluation of stable copper nanoparticles using T. arjuna bark extract. – Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy 110: 108-115.
[30] Yashni, G., Willy, K.B., Al-Gheethi, A.A., Mohamed, R.M.S.R., Salleh, S.M., Hashim, M.A. (2020): A Review on Green Synthesis of ZnO Nanoparticles Using Coriandrum Sativum Leaf Extract For Degrading Dyes in Textile Wastewater: A Prospect Towards Green Chemistry. – In IOP Conference Series: Materials Science and Engineering 736(4): 8p.