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Biosynthesis of Copper Nanoparticles from *Indigofera tinctoria* Leaves

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Abstract

Leaf extracts are known to be rich in phytoconstituent biomolecules, making them a valuable source of medicinal compounds. They also serve as both capping and reducing agents in nanoparticle fabrication. A reaction between $CuSO_4.5H_2O$ aqueous solution and the *Indigofera tinctoria* leaf extract results in the formation of stable copper nanoparticles. Phytochemical screening of the *Indigofera tinctoria* leaf extract revealed the presence of various compounds including carbohydrates, terpenoids, phenol, tannins, flavonoids, saponins, and glycosides in the sample. The biosynthesized copper nanoparticles (CuNPs) were subsequently subjected to various forms of analysis. Techniques used included UV–visible (UV–vis) spectroscopy, Fourier transform infrared spectroscopy (FTIR), and scanning electron microscopy. An observable change in the color of solutions from pale to thick brown indicated the formation of CuNPs. Further confirmation came from UV–vis spectroscopy, which established the production of CuNPs at 500 nm. FTIR analysis revealed that the CuNPs were covered by organic residues. The particles ranged from 210 nm to 260 nm as indicated from the SEM analysis.

Keywords: extract, fourier transform infrared, nanoparticles, scanning electron microscopy, synthesis

Introduction

The Indigofera plant, with its many species, is widespread across the southwestern regions of Nigeria. The most commonly found species are *I. arrecta and I. tinctoria*. These plants serve as a primary source for indigo dye and are known for their antitoxic, hemostatic, and sedative properties. They have been utilized in the treatment of various ailments, including piles, ulcers, and dropsy. The plant leaves, roots, and stems are also beneficial for promoting hair growth and treating conditions such as gastropathy, splenomegaly, cepholagia, cardiopathy, chronic bronchitis, asthma, and ulcers [1, 2]. Different parts of the plant, such as the stem, bark, tubers, fruits, leaves, seeds, and roots contain natural chemical substances, which can be extracted [3]. These natural chemicals, rich in phytochemicals, act as stabilizing and reducing agents in the nanoparticle fabrication [4]. This assists in nanoparticle production under controlled temperature and pressure. Consequently, plant extracts play a crucial role in the synthesis of nanoparticles, which have numerous applications, especially in catalytic and biological usages [5]. Nanotechnology has enabled material production at the nanoscale level, giving rise to nanoparticles. These are classes of materials that comprise particulate substances, which at least one dimension measuring less than 100 nm [6]. There are two main methods for producing nanoparticles: bottom-up and top-down techniques. The top-down approach involves breaking down larger molecules into smaller units that are then transformed into suitable nanoparticles. Examples of this approach include grinding/milling, chemical vapor deposition, and physical vapor deposition [7]. Conversely, the bottom-up approach forms complex structures from simpler substances, with examples including electrolysis [8], atomization [9], pyrolysis [10], chemical reduction methods [11–15], and biosynthesis [3]. Copper nanoparticles (CuNPs) are currently garnering significant research interest owing to their wide-ranging applications in heat transfer systems, powder metallurgical materials, electronic circuits, casting [16, 17], antimicrobial materials [18, 19], super strong materials (SSM) [20, 21], sensors [22–24], and catalysts. Previous studies conducted by Kayode *et al*. [3] and Ajeboriogbon *et al*. [4] have explored the use of *J. curcas* aqueous leaf extract as a reducing and capping agent for the production of silver nanoparticles (AgNPs). Their results showed an average crystal size of 36.4 nm with a face-centered cubic structure, concluding that successful nanoparticle fabrication can be achieved with the *J. curcas* leaf extract.

Materials and Methods

Indigofera tinctoria (Figure 1) leaves were obtained from Ilawe–Ekiti, Nigeria (7° 35' 60 N and 5° 5' 60 E). These specimens were thoroughly cleaned with distilled water until free of any contamination, then dried in an oven at 50 °C for 5 h. The dried leaves were subsequently ground into a fine powder using a pulverizer (model number ES-1731F, 300 W power, 50 Hz frequency, and 220 V AC voltage. A 50-g sample of the pulverized *Indigofera tinctoria* leaves was measured and placed in a beaker with 500 mL of distilled water. To expedite the extraction process, this mixture was placed in a water bath rotary shaker at 50 °C for 2 h. After the extraction, the solution, which now contained the *Indigofera tinctoria* leaf extract (Figure 2), was permitted to cool at room temperature before being filtered using Whatman filter paper [3].

Phytochemical screening of *Indigofera tinctoria* plant extract. The phytochemical screening of the *Indigofera tinctoria* plant extract was carried out to identify the presence of the chemical constituents following a standard method [21].

Biosynthesis of CuNPs. The process began with dissolving copper sulfate pentahydrate ($CuSO_4.\ 5H_2O$) powder in distilled water to prepare a 0.2 M $CuSO_4.5H_2O$ solution. This solution (Figure 3) solution was then combined with the *Indigofera tinctoria* leaf extract in a 1:4 ratio, totaling 100 ml–400 ml volume in a flask. The leaf extract contains phytochemicals that act as reducing and capping agents for the green fabricated *Indigofera tinctoria* CuNPs. These phytochemicals are responsible for reducing Cu^{+2} to Cu^0. The mechanism responsible for the fabrication of the *Indigofera tinctoria* CuNPs is shown in Figure 4. The successful formation of copper nanoparticles was confirmed visually through a color change (Figure 5) in the colloidal solution, shifting from pale brown to bluish-brown. The mixture was heated at 50 °C for 15 min and stirred continuously for 4 h at 40 °C using a magnetic stirrer. The resulting copper nanoparticles were separated by centrifugation at 4000 rpm for 40 min. After being washed with distilled water and ethanol, the nanoparticles were dried in the oven to obtain dried CuNPs (Figure 6).

Analytical characterization of CuNPs. The UV–visible (UV–vis) spectra of CuNPs were monitored using a state-of-the-art Shimadzu (UV2450) spectrophotometer. To examine the morphological analysis of dried CuNPs, we employed a Tescan VEGA 3 LMH scanning electron microscope. Fourier transform infrared (FTIR) spectroscopy was performed on the powdered CuNPs to detect the biomolecules present on their surface.

Figure 1. *Indigofera tinctoria* Plant Figure

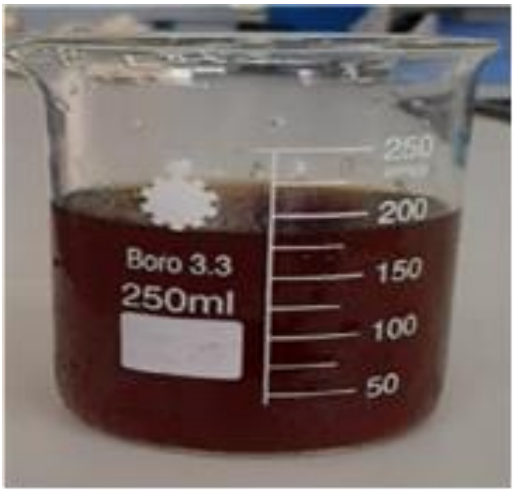

Figure 2. *Indigofera tinctoria* Leaf Extract

Figure 3. 0.2M $CuSO_4.5H_2O$ Solution

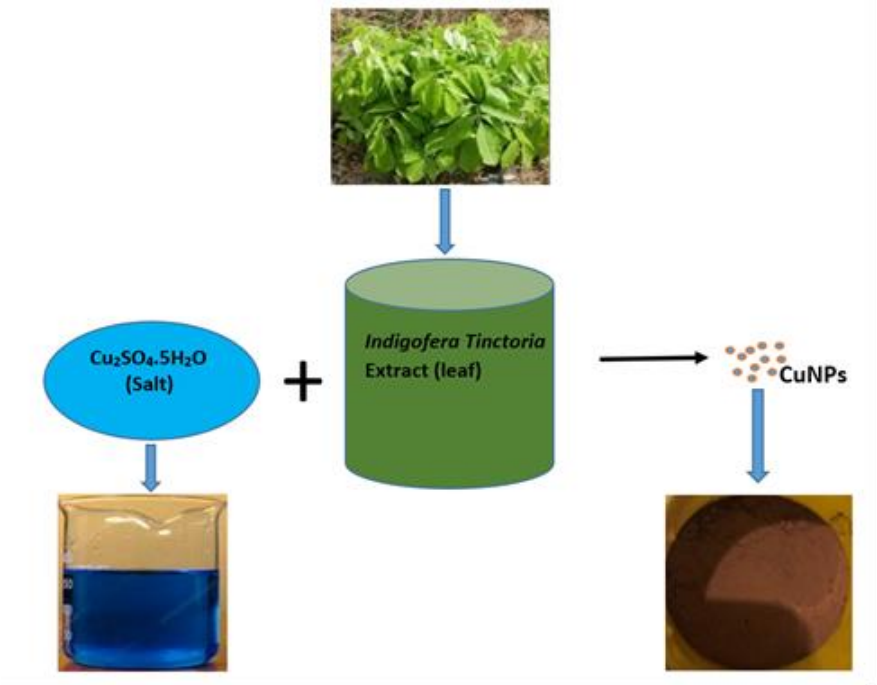

Figure 4. Mechanism for the Synthesis of *Indigofera tinctoria* CuNPs

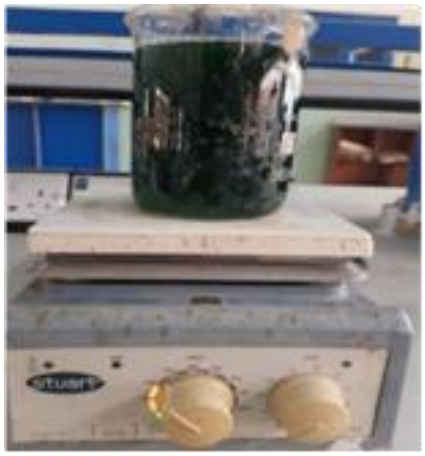

Figure 5. Colloidal Solution

Figure 6. Dried CuNPs

Results and Discussion

Phytochemical screening of *Indigofera tinctoria* leaf aqueous extract. The screened extract of *Indigofera tinctorial* leaf contains various biomolecules. These include carbohydrates, terpenoids, tannins, alkaloids, phenol, flavonoids, saponins, glycosides, protein, and cardiac glycosides as presented in Table 1. These compounds play a significant role in reducing the precursor salt.

UV–vis spectra. CuNP formation was confirmed using UV–vis absorption spectroscopy (Figure 7). Surface plasmon resonance (SPR), conducted via UV–vis spectrophotometry, proved to be instrumental in examining

Table 1. Phytochemical Screening of *Indigofera tinctorial* Extract

S/N	Phytochemical	Qualitative
1	Carbohydrates	+
2	Terpenoids	+
3	Tannins	+
4	Alkaloids	−
5	Phenol	+
6	Flavonoids	+
7	Saponins	+
8	Glycosides	+
9	Protein	−
10	Cardiac glycosides	−

Where (+) means the presence of the chemical constituent and (−) means the absence of chemical constituent

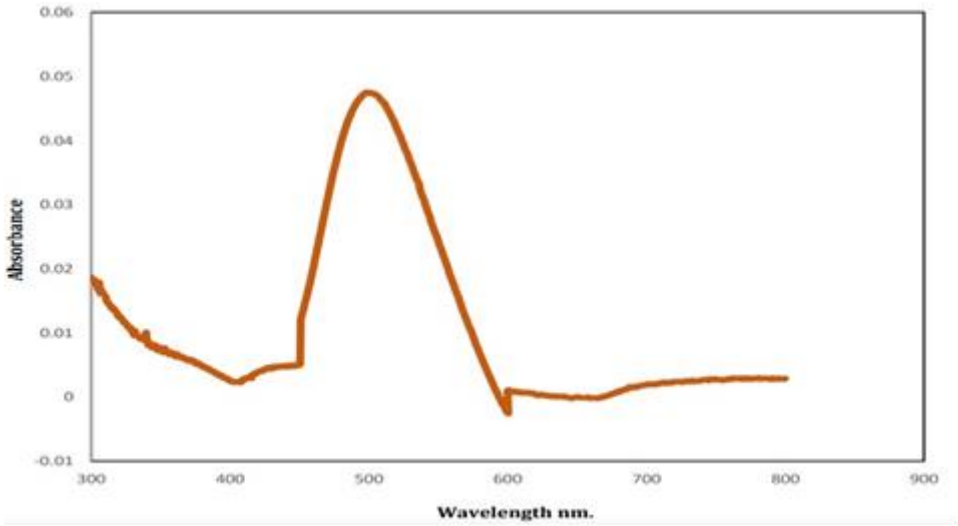

Figure 7. UV–vis Spectra of CuNPs

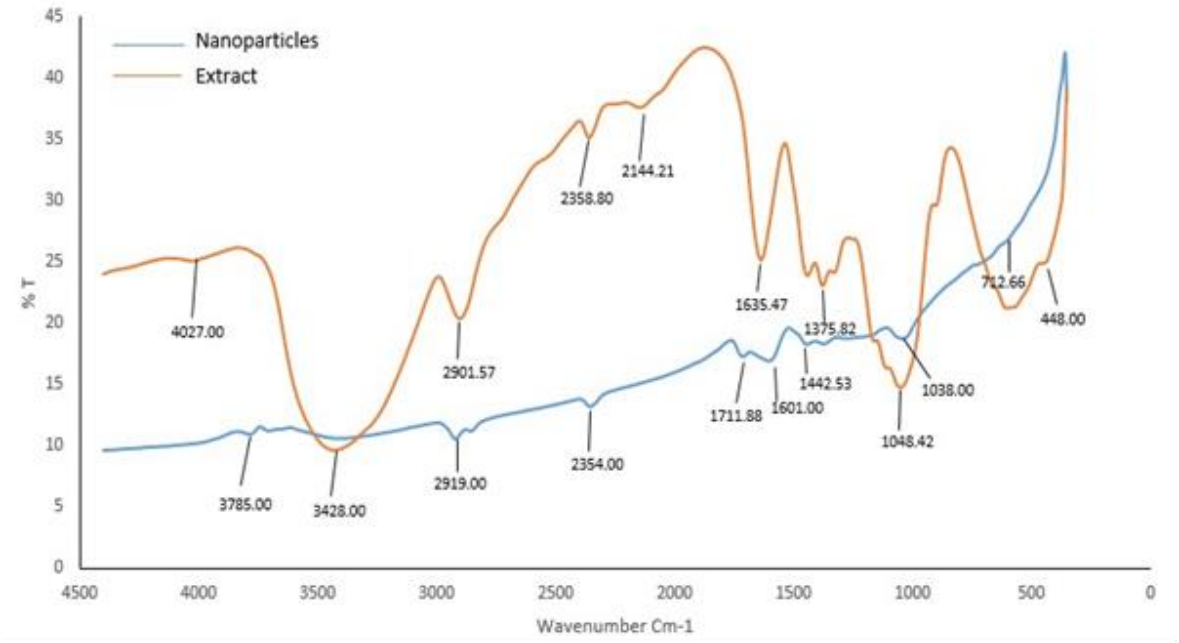

Figure 8. FTIR Spectrum of CuNPs Synthesized using the *Indigofera tinctoria* Leaves Extract

Table 2. FTIR Spectra Analysis and Functional Groups *Indigofera tinctoria* CuNPs

FTIR spectrum peak, v (cm^{-1})	Functional group	Compound class
3428	O-H stretching	Alcohol
2901	C-H stretching	Alkane
2358	C≡N stretching	Nitrile
2144	C≡C stretching	Alkyne
1635	C=C stretching	Alkene
1375	C-H bending	Alkane
1048	C-O stretching	Ether
600	C-Br stretching	Alkyl halide

CuNP formation and stability within the *Indigofera tinctoria* leaves. Key parameters for characterizing the synthesized CuNPs included variations in bandwidth and shifts in resonance. The UV–vis absorption spectrum of the synthesized CuNPs revealed a strong absorption peak at 500 nm [25]. This peak is likely attributed to SPR, which results from oscillations of surface electrons and their excitation caused by incident electromagnetic radiation [26].

FTIR analysis. To identify the capping agent, the functional groups of biomolecules from the *Indigofera tinctoria* leaf extract on the surface of copper nanoparticles, we employed FTIR analysis [27]. The metabolites found in the *Indigofera tinctoria* leaf extract, which contained polyphenols compounds such as terpenoids, tannins, phenol, flavonoids, and saponins, play a crucial role in the bioreduction and chelation of copper ions into CuNPs. The tautomeric change of flavonoids, which involves the release of a responsive hydrogen particle transitioning from enol to keto form, acts as a reducing agent in this transformation of copper ions into CuNPs [28]. Meanwhile, the functional groups, as reported in Table 2, serve as a capping agent for the synthesized CuNPs. The FTIR spectra of the *Indigofera tinctoria* leaf extract and CuNPs are presented in Figure 8. Table 2 details the functional groups present, their corresponding peaks, and their interpretation. The absorption bands for the *Indigofera tinctoria* CuNPs were detected at 3428, 2901, 2358, 2144, 1635, 1375, 1048, and 600 cm^{-1}, corresponding to the functional groups of O–H, C–H, C≡N, C≡C, C=C, C–H, C–O, C–Br, and C–O, respectively. These groups are believed to contribute to the bioreduction of Cu^{+2} to *Indigofera tinctoria* CuNPs [29]. In the IR spectrum of the nanoparticles, shifts in band peaks were observed from 2901 cm^{-1} to 2919 cm^{-1}, 2358 cm^{-1} to 2354 cm^{-1}, 1635 cm^{-1} to 1601 cm^{-1}, and 1048 cm^{-1} to 1048 cm^{-1}. These shifts corresponded to C–H, C≡N, C≡C, C=C, and C–O functional groups of the *Indigofera tinctoria* leaf extract, indicating their involvement in CuNP synthesis [30].

The functional groups observed in the *Indigofera tinctoria* leaf extracts, as presented in Table 2, likely indicate the presence of carbohydrates, terpenoids, tannins, phenol, flavonoids, saponins, and glycosides, as detailed in Table 1. Therefore, the FTIR analysis results align with the qualitative assessment of these phytochemicals.

SEM Analysis. The surface size and shape of the developed CuNPs were determined using SEM (Figure 9). The image reveals that the CuNPs, biosynthesized with *Indigofera tinctoria* leaf extract, were well dispersed and homogeneous. According to the SEM study conducted in this research, the size of the formed CuNPs ranged from 210 nm to 260 nm [31].

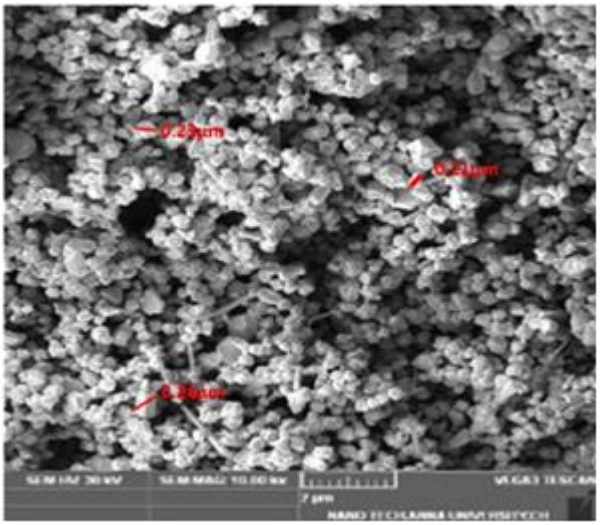

Figure 9. SEM Image of Developed CuNPs

Conclusion

In this study, we investigated CuNP synthesis using *Indigofera tinctoria* plant extract, an innovative approach that forms the crux of our research. The use of *Indigofera tinctoria* plant extract for the synthesis of CuNPs is the novelty of this research. The reaction resulted in a color change from pale brown to dark brown, indicative of CuNP formation. This was further validated by UV–vis spectroscopy, which showed an absorption peak at 500 nm. The *Indigofera tinctoria* plant extract acted as a capping and a reducing agent. FTIR analysis showed that the nanoparticles were covered with various functional groups. The sizes of CuNPs synthesized in the research ranged from 210 nm to 260 nm, as indicated from the SEM analysis.

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References

[1] Mishra, D.N., Gomare, K.S., Sheelwant, S.V. 2020. GC-MS Analysis and phytochemical screening of *Indigofera tinctoria* (Linn.) leaf extract characterizing its medicinal use. Int. J. Ayurvedic Med. 11(2): 289–299, https://doi.org/10.47552/ija m.v11i2.1540.

[2] Ullah, Z., Ali, S., Hussain, A., Öztürk, M., Ertaş, A., Alamzeb, *et al*. *In vitro* antioxidant, anticholinesterase, tyrosinase activity studies, and LC-MS/MS simultaneous determination of 37 bioactive compounds in *Indigofera heterantha*. S. Afr. J. Bot. 148: 537–545, https://doi.org/10.1016/ j.s ajb.2022.05.012.

[3] Kayode, T.H., Yetunde, T.J., Omotayo, A.B. 2022. Silver nanoparticles' biosynthesis and characterization with the extract of *Jatropha curcas* leaf: Analysis of corrosion inhibition activity. Makara J. Sci. 26(2): 137–143, https://doi.org/ 10.7454/mss.v26i2.1276.

[4] Ajeboriogbon, A.F., Adewuyi, B.O., Talabi, H.K. 2019. Synthesis of silver nanoparticles from selected plants extract. Acta Tech. Corviniensis-Bull. Eng. 12(2): 55–58.

[5] Dewan, A., Sarmah, M., Thakur, A.J., Bharali, P., Bora, U. 2018. Greener biogenic approach for the synthesis of palladium nanoparticles using papaya peel: An eco-friendly catalyst for C–C coupling reaction. ACS Omega. 3(5): 5327–5335, https://doi.org/10.1021/acsomega.8b00039.

[6] Shabatina, T.I., Vernaya, O.I., Shabatin, V.P., Melnikov, M.Y. 2020. Magnetic nanoparticles for biomedical purposes: Modern trends and prospects. Magnetochemistry. 6(3): 30, https://doi.org/10.3390/magnetochemistry6030030.

[7] Khandel, P., Yadaw, R.K., Soni, D.K., Kanwar, L., Shahi, S.K. 2018. Biogenesis of metal nanoparticles and their pharmacological applications: present status and application prospects. J. Nanostruct. Chem. 8: 217–254, https://doi.org/10.1007/s40097-018-0267-4.

[8] Li, G., Wang, N., Wang, Y., Yan, Y. 2023. Synthesis and performance characterization of nano-copper by electrochemical deposition method based on coaxial electrode structure. J. Iranian Chem. Soc. 20(4): 839–848, https://doi.org/10.1007/s13738-022-02722-5.

[9] Zhang, A., Zhou, M., Liu, S., Chai, M., Jiang, S. 2021. Synthesis of single-atom catalysts through top-down atomization approaches. Front. Catal. 1: 754167, https://doi.org/10.3389/fctls.2021.754167.

[10] Majerič, P., Rudolf, R. 2020. Advances in ultrasonic spray pyrolysis processing of noble metal nanoparticles. Materials. 13(16): 3485, https://doi.org/10.3390/ma13163485.

[11] Khatoon, U.T., Velidandi, A., Rao, G.N. 2023. Copper oxide nanoparticles: Synthesis via chemical reduction, characterization, antibacterial activity, and possible mechanism involved. Inorg. Chem. Comm. 149: 110372, https://doi.org/10.1016/j.inoch e.2022.110372.

[12] Ahoba-Sam, C., Olsbye, U., Jens, K.J. 2018. Low temperature methanol synthesis catalyzed by copper nanoparticles. Catal. Today. 299: 112–119, https://doi.org/10.1016/j.cattod.2017.06.038.

[13] Rajamohan, R., Raorane, C.J., Kim, S.C., Ashokkumar, S., Lee, Y.R. 2023. Novel microwave synthesis of copper oxide nanoparticles and appraisal of the antibacterial application. Micromachines. 14(2): 456, https://doi.org/10.3390/mi14020456.

[14] Zhang, Y., Epshteyn, Y. and Chromik, R.R. 2018. Dry sliding wear behaviour of cold-sprayed Cu-MoS_2 and Cu-MoS_2-WC composite coatings: The influence of WC. Tribol. Int. 123: 296–306, https://doi.org/10.1016/j.triboint.2017.12.015.

[15] Pesesse, A., Carenco, S. 2021. Influence of the copper precursor on the catalytic transformation of oleylamine during Cu nanoparticle synthesis. Catal. Sci. Technol. 11(15): 5310–5320, https://doi.org/10.1039/D1CY00639H.

[16] Sarkar, J., Nilanjan, C., Arindam, C., Avisek, B., Disha, D., Krishnendu, A. 2020. Green synthesized copper oxide nanoparticles ameliorate defence and antioxidant enzymes in *Lens culinaris*. Nanomaterials-Basel. 10(2): 312, https://doi.org/10.3390/nano10020312.

[17] Talabi, H.K., Adewuyi, B.O., Olaniran, O., Ayekusibe, P.O. 2021. Reinforcement efficiency of copper nanoparticles on epoxy matrix bactericide composite. J. Chem. Technol. Metall., 56(2): 321–326.

[18] Wu, F., Zhou, X., Yu, X. 2018. Reaction mechanism, cure behavior and properties of a multifunctional epoxy resin, TGDDM, with latent curing agent dicyandiamide. RSC Adv. 8(15): 8248–8258, https://doi.org/10.1039/c7ra13233f.

[19] Asadikia, A., Mirjalily, S.A.A., Nasirizadeh, N., Kargarsharifabad, H. 2020. Hybrid nanofluid based on CuO nanoparticles and singlewalled Carbon nanotubes: Optimization, thermal, and electrical properties. Inter. J. Nano Dimens. 11(3): 277–289.

[20] Jiang, Y., Hu, J., Jiang, Z., Lian, J., Wen, C. 2018. Strain rate dependence of tensile strength and ductility of nano and ultrafine grained coppers. Mater. Sci. Eng. A. 712: 341–349, https://doi.org/10.1016/j.msea.2017.11.083.

[21] Zhou, X., Feng, Z., Zhu, L., Xu, J., Miyagi, L., Dong, H., *et al*. 2020. High-pressure strengthening in ultrafine-grained metals. Nature. 579: 67–72.

[22] Sridara, T., Upan, J., Saianand, G., Tuantranont, A., Karuwan, C., Jakmunee, J. 2020. Non-enzymatic amperometric glucose sensor based on carbon nanodots and copper oxide nanocomposites electrode. Sensors. 20(3): 808, https://doi.org/10.3390/s20030808.

[23] Zhao, J., Wei, L., Peng, C., Su, Y., Yang, Z., Zhang, L., *et al*. 2013. A non-enzymatic glucose sensor based on the composite of cubic Cu nanoparticles and arc-synthesized multi-walled carbon nanotubes. Biosens. Bioelectron. 47: 86–91, https://doi.org/10.1016/j.bios.2013.02.032.

[24] Jagadeesan, M.S., Movlaee, K., Krishnakumar, T., Leonardi, S.G., Neri, G. 2019. One-step microwave-assisted synthesis and characterization of novel CuO nanodisks for non-enzymatic glucose sensing. J. Electroanal. Chem. 835: 161–168, https://doi.org/10.1016/j.jelechem.2019.01.024.

[25] Talabi, H.K., Adewuyi, B.O., Olaniran, O., Oladele, I.O., Oladotun, J.A. 2022. Microwave accelerated chemical reduction method for the production of

copper and copper oxide nanoparticles as nanometal lubricant additives. J. Chem. Technol. Metall. 57(3): 598–606.

[26] Liu, J., Jalali, M., Mahshid, S., Wachsmann-Hogiu, S. 2020. Are plasmonic optical biosensors ready for use in point-of-need applications? Analyst. 145(2): 364–384, https://doi.org/10.1039/c9an02149c.

[27] Vijayakumar, G., Kesavan, H., Kannan, A., Arulanandam, D., Kim, J.H., Kim, K.J., *et al*. 2021. Phytosynthesis of copper nanoparticles using extracts of spices and their antibacterial properties. Processes. 9(8): 1341, https://doi.org/10.3390/pr9081341.

[28] Jain, S., Mehata, M.S. 2017. Medicinal plant leaf extract and pure flavonoid mediated green synthesis of silver nanoparticles and their enhanced antibacterial property. Sci. Rep.-UK. 7: 15867, https://doi.org/10.1038/s41598-017-15724-8.

[29] Ramadhan, V.B., Ni'mah, Y.L., Yanuar, E., Suprapto, S. 2019. Synthesis of copper nanoparticles using Ocimum tenuiflorum leaf extract as capping Agent. AIP Conf. Proc. 2202: 020067, https://doi.org/10.1063/1.5141680.

[30] Vijayan, R., Joseph, S.,Mathew, B. 2018. *Indigofera tinctoria* leaf extract mediated green synthesis of silver and gold nanoparticles and assessment of their anticancer, antimicrobial, antioxidant and catalytic properties. Art. Cells Nanomed. Biotech. 46(4): 861–871, https://doi.org/10.1080/21691401.2017.1345930.

[31] Vijayakumar, G., Kesavan, H., Kannan, A., Arulanandam, D., Kim, J.H., Kim, K.J., *et al*. 2021. Phytosynthesis of copper nanoparticles using extracts of spices and their antibacterial properties. Processes. 9(8): 1341, https://doi.org/10.3390/pr9081341.