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Novel Synthesis and Characterization of Copper Nanoparticles from Copper Tailings/Wastes

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ABSTRACT

In recent time, copper nanoparticles (Cu-NPs) are versatile nanomaterials that find wide array of utilizations in biomedicine, molecular biology, dentistry, dye degradation, catalysis, among others. The aim of this study is to assess the quality of Cu-NPssynthesized from copper tailings and reagent grade copper salt. Chemical reduction method was used to synthesize Cu NPs from copper tailings and reagent grade copper salt using sodium tetrahydroborate ($NaBH_4$) as the reducing agent. The synthesized copper nanoparticles were characterized using different analytical techniques such as: quantitative X-ray Diffraction (XRD),scanning electron microscopy (SEM), X-Fluorescence spectroscopy (XRF), Brunauer-Emmett-Teller (BET), Fourier Transform Infrared Spectroscopy (FTIR) and Thermogravimetric analysis (TGA). The results of XRD identified three major mineral phases tenoriteSyn, garnet and quartz in tailing(87.3%, 7.8%, and 4.9%) and reagent grade salt (90%, 6%, and 4%) respectively and confirmed the formation of face-centered cubic (FCC) metallic copper. SEM revealed irregular shape particle morphology with rough surface and particle of 31.3 nm for Cu tailings and 37.6 nm for Reagent Cu nanoparticles. XRFrevealed high elemental compositionof copper inboth synthesized nanoparticles from tailings and reagent salt with 85.15% and 85.98% respectively, BET surface area are 342.11 m^2/g and 464.95 m^2/g forCu tailings and reagent salts respectively, and FTIR revealed the specific functional groups O-H, C=O, C-O and Cu-O stretching for both tailings and reagent grade Cu-NPs. However, this type of studyhas not been reported in an accredited journal. Thermogravimetric analysis (TGA) confirms the thermal stability of these Cu-NPs up to 325 °C. In conclusion, synthesized TCu-NPs and andRCu-NPs were pure, highly crystalline, nano-sized and the quality of the nanoparticles from tailings was slightly lesser than the reagent grade saltdue to the presence of impurities.

1.0 Introduction

The recent advances in nanotechnology have made the field of nanoscience to be an important area of research, which has made it to be one of the most researched areas of science over the past two decades. Nanoparticles (NPs) can be described as particles having diameter sizes less than or equal to 100 nm with specific properties that depend purely on their sizes as they differ from the bulk material (1, 2, 3). Cu-NPs have wide range of applications in different fields and some of these properties include catalytic (4, 5, 6), thermal, electrical conductivity, optical (7) and biological applications (8). Their properties are influenced by their high surface energy with a high surface area to volume ratio and relatively small sizes. Their properties can be influenced by their high surface energy with a high surface area to volume ratio with relatively small sizes (9). However, different metallic nanomaterials are being produced using copper, zinc, titanium, magnesium, gold, alginate and silver. Different morphologies and particle sizes of copper nanoparticles have been synthesized using different routes (10). Nanoparticles can be synthesized chemically or biologically. Several adverse effects have been found to be associated with chemical synthesis methods due to the presence of some toxic chemical absorbed on the surface. Some reducing agents are toxic and expensive, but some of the reductants are have high costs, with poor reducing ability, some and some are easy to introduce other impurities to the process (11). Eco friendly alternatives to Chemical and physical methods are Biological ways of nanoparticles synthesis using microorganisms (3, 4) enzymes (5), fungus (6), and plants or plant extracts. Of all the various methods of synthesizing copper nanoparticles, a simple reduction process has been well developed (12, 13). The development of these eco-friendly methods for the synthesis of nanoparticles is evolving into an important branch of nanotechnology especially silver nanoparticles, which have many applications (9, 14, 15). Synthesis of NPs in polymer media has been promising due to their ease of processing, solubility, less toxicity and also because of the possibility of controlling the growth of the resulting nanoparticles. Synthesis Cu-NPs has attracted more interest compared to synthesis of other NPs because of the usefulness of their properties have made it to be achievable at much less cost than silver and gold (14). Copper like other noble metals, exhibits thermal and electrical conductivity which makes it to be a material in electronic systems and conductive inks (15). However, Cu-NPs have some antimicrobial properties and it is readily available. These

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properties have made Cu-NP synthesis to be an attractive area. There are different methods of synthesis of Cu-NPs which include: laser ablation, thermal reduction, micro-emulsion techniques, chemical reduction, polyol processes, vacuum vapor deposition, radiation methods, and metal-vapor synthesis, etc. Reducing agent is often used in present method such as sodium borohydride and hydrazine hydrate (16, 17). Some reducing agents are toxic and expensive as some have poor reducing ability, and some are costly, they are easy to introduce other impurities to the process. In this research, we present the synthesis of copper NPs using chemical reduction method involving aqueous solutions of copper tailings and copper sulphate with sodium borohydride as reductant.

2.0 Materials and Methods

2.1Study Area

Ojo is a Local Government Area and town in Lagos State, Nigeria. Lagos State University,Ojo is located on the eastern section of the Trans–West African Coastal Highway, about 37 km west of Lagos State. It is part of the Lagos Metropolitan Area. The coordinates of the area is 6°28′N 3°11′E, with a total population census ofOjo was609,173 (2006 census), and estimate of 838,900 (2016 census) with a density of 8,700/sq mi (3,300/km^2). Ojo is primarily a residential township and it has some major markets such asAlaba International Market, and International Trade Fair complex. It also houses the divisional headquarters of 81 division, Nigerian Army and Navy Town. South of the town (across Badagry creek), the rest of the local government is sparsely populated and consists of mangrove swamps and sandy beaches. Some of these beaches are holiday resorts during the festive season. Wildlife mostly consists of reptiles, rodents and birds including crocodiles, iguanas, monitor lizards and squirrels. Whales and dolphins have been known to visit the coastal areas.Some towns there are Iba, Igando, and Okokomaiko, e.t.c.

2.2. Chemical Reagents

Copper sulphatepentahydrate reagent grade salt, nitric acid, hydrochloric acidwas purchased from Merck, and sodium borohydride from Tunnex chemicals, Nigeria. The copper tailing salt sample was purchased from a local market stores at Ojo local government in area, Lagos State. Analytical reagent grade chemicals were used in the experiment without further purification. Distilled water was used throughout the experiment for preparing solutions and washing purposes.

2.3 Synthesis of Copper Nanoparticles

10 g of each copper II sulphatefrom tailings and reagent salt were weighed separately and dissolved in 200 mL of distilled water in separate 500 mL volumetric flask. 22 g of Sodium tetrahydroborate ($NaBH_4$) was weighed and dissolved in 500 mL of distilled water in a beaker. 100 mL of the copper II sulphate tailings and reagent grade $CuSO_4$ solution were measured in two separate 250 mL conical flasks. 100 mL copper tailings solution was subjected to constant stirring on a magnetic stirrer and 50 mL $NaBH_4$ solution was added gradually with the formation of black precipitate and the addition continues until a clear solution was formed. The precipitate formed was filtered, washed three times with 50 mL distilled water and dried in an oven regulated at 100^o C for one hour. The procedure was repeated for the remaining solution. The same synthesis procedure was carried out for the reagent grade $CuSO_4$ solution. The synthesized copper nanoparticle precipitates from both copper tailings and reagent grade copper salt solutions were sent out for characterization such as XRD, XRF, SEM, FTIR, BET and TGA.

$$Cu^{2+} + 2BH_4^- + 6H_2O \longrightarrow Cu^0 + 2B(OH)_3 + 7H_2 \quad (1)$$

2.4. Characterization

The black synthesized Cu-NPs were separately identified with an quantification x-ray powder diffraction patterns using on a RigakuminiFlex 600 X-ray Diffractometer with Cu Kα radiation (40kV, 15 mA, λ = 1.542Å). Scan was conducted from 10^0 to 80^0 (2θ). The particle size of nanoparticles was measured using scherrers equation. This technique is used to identify mineral phase, crystallinity, and Cu-NP size. The X-ray fluorescence (XRF) technique was used to measure the elemental composition of major oxides which includes major and minor heavy metals present in the synthesized Cu-NP samples. This technique was used for qualitative and quantitative elemental analysis of solid samples using Genius IF by Xenemetrix. The Phillips PW 1480 X-ray spectrometer instrument was set at 40 kV and 50 mA tube operating conditions required for the sample analysis. High Resolution Scanning Electron Microscopy (HRSEM) PhenomProX was used to determine the morphology of the Cu-NPs. FTIR spectra of Cu-NPs were recorded using Nicolet Magna-IR Spectrometer model 550. BET surface area of the Cu-NP samples obtained from different sources were measured at a temperature of 77.35 K using nitrogen adsorption method with a quantachrome NOVA 2000e Made in USA analyzer. The FTIR of the sample was taken in the region between 400-4000cm^{-1} on a Thermo-Nicolet Avatar 370 model FTIR. Thermalgravimetric analysis (TGA) of Cu-NP samples were analyzed using TGA 4000 by perkin Elmer analysis to determine the thermal stability of solid materials in the temperature range of 50–800 °C at a heating rate of 10 °C/min in nitrogen atmosphere.

3.0 Results and Discussion

3.1Moisture

The moisture of the synthesized Cu-NPs was determined to be 4.25% for TCu-NPs while 7.68 % for RCu-NPs. Moisture content presented in Figure 2 revealed that the synthesized copper tailings nanoparticles has lower moisture than the reagent grade sample which could be due to the drying process of the nanoparticle sample.

3.2 Quantification XRD Analysis

Quantification XRD analysis was used to identify thequalitative and quantitative major mineral phases (see Figures 3 and 4)in the synthesized Cu-NPsexhibit similar diffraction pattern for both Cu-NP samples and all the peaks obtained from the XRD pattern and the samples demonstrated high level of crystallinity with diffraction angles of spectral peaks indexed at angle 2θ diffraction at 32.2◦, 35.72◦, 38.8◦, 48.76◦, 58.13◦, 61.56◦, and 65.94◦ which correspond to the characteristic face centered cubic (fcc) of metallic copper lines (18).The diffraction pattern of the RCu-NPs (A) and TCu-NPs (B) at their reflection anglesidentified quartz, garnet and tenoriteSyn mineral phases The JCPDS file was used to identify the diffraction of the synthesized Copper nanoparticles are composed of metallic copper (tenoriteSyn) and the surface shell is formed as copper II oxides (CuO) which is the protective shell layer and it agrees with previous report of Radhakrishnan, and Beena, (19). Mineral phases of tenoriteSyn, quartz, and garnet consistent with the JCPDS card (card no) is presented in Table 1. The percentage mineral compositions in the synthesized copper nanoparticles in Figure 4 areTCu-NPs (87.3%, 7. 8%, and 4.9%) and RCu-NPs (90%, 6%, and 4%). The XRD mineral composition showed that the purity of RCu-NPs was higher than the TCu-NPs presented in Figures 4. The size of the NPs obtained were estimated to be 31.3 nm for TCu-NP and 37.6 nm for RCu-NP respectively using Debye-Scherrer Equation,which may indicate a high

surface area, and surface area to volume ratio of the nano-crystals. The equation is written below

The crystallite particle size is calculated by using Debye Scherrer equation

$$D=0.9\lambda/\beta Cos\theta \quad (2)$$

Where λ is the X-ray wave length, β is the line broadening at half the maximum intensity in radians, θ is the Bragg angle.From the calculations the average crystallite size of the synthesized CuOnano particles are 31.3 nm and 37.6 nm for tailings and reagent grade respectively.

3.2 XRF Elemental Composition of Synthesized Cu Nanoparticle

The XRF elemental composition of copper nanoparticles (Cu-NPs) in both samples are expressed in percent composition of TCu-NPs (85.154%)and RCu-NPs (85.978%), however,the percentage composition of copper in the synthesized nanoparticle from reagent grade copper salt was slightly higher than that of tailing copper. The XRF elemental composition result of the mineral phases is in agreement with XRD mineral composition that RCu-NP is purer than TCu-NPsin Figure 4. The XRF elemental composition of copper in synthesized nanoparticles from tailings (TCu-NPs) and reagent grade Cu salt (RCu-NPs) is presented in Table 1.

3.3 SEM Analysis

SEM surface morphology results of the synthesized Cu-NPs in Figure 5 revealed that both samples have rough surface withirregular particle sizes and some agglomerations which can be attributed to the presence of moisture.

3.4 BET Analysis

The BET results of the synthesized nanoparticles presented in Table 2 revealed the pore diameter, micropore volume, surface, pore size distribution, and pore width. BET analysis of the synthesize nanoparticles is presented in Figures 6, 7 and 8 revealed the surface area, pore size distribution, multiple point BET.

3.5 FTIR Analysis

FTIR analysis resultof Cu-NPs in Figure 7revealed the various stretching vibrations of some functional groups present in the synthesizednanoparticles from tailings and reagent grade salt. The absorption broad and sharp peaks at 3400-3224 cm^{-1} relates to OH stretching vibration (20), absorption at 2046-1532 cm^{-1}relates C=O stretching bands which revealed that the nanoparticles have strong affinity for CO2 gas (20) (21), The spectral band at 1640-1532- cm^{-1} indicates that the copper (II) oxide (CuO) bonds stretching, and the result agrees with the report of previous studies(22, 23, 24), at 1252-1070 cm^{-1}indicate C-O stretch confirming the O-H stretch identified at high wave number, and peaks observed at 760-640 cm^{-1}, indicates the presence of Cu-O stretch.The FTIR spectrum in Figure.7 shows sharp peak bands observed at around 764 cm^{-1} in the spectrum of CuO nanoparticles is the characteristics of Cu-O bond formation(25). The broad band of O-H stretch for RCu-NP confirms the high moisture content presented in Figure 2 while the TCu-NP have sharp peak.

3.6 Thermogravimetricanalysis

The results of TGA effect of Cu-NPs on the thermal stability of temperature of the synthesized nanoparticle samples were examined. The derivative thermograms of the Cu-NPs generated is presented in Figure 9 and it was found that the degradation for both Cu-NPs occurred gradually with weight loss which indicates loss of water and any other volatile substances present. The loss of weight TCu-NPs fell 100 g to less than 20g as temperature **rise from 300 ^{o}C to 420** ^{o}C while the same weight of RCu-NPs fell from 100g to less than 5.0 g as the temperature rise from 300 ^{o}Cto 560^{o}C.The thermal stability of the TCu-NPs was found to be lower than that of the RCu-NPs because they attain stability in weight loss. The lowering of thermal stability in the case of both Cu-NPs can be attributed to the catalytic nature of the Cu-NPs in lowering the degradation temperature(26).

4. Conclusion

The assessment of the quality of nanoparticles synthesized from copper tailings and reagent grade copper salt in this study helps to identify the sample that gave a better quality. The XRD revealed pure crystalline copper nanoparticles consisting of three mineral phases (tenoriteSyn, garnet and quartz) with particle sized of 31.3 nm for TCu-NPs and 37.6 nm for RCu-NPs. XRF showed that RCu-NPs (88.978%) contained slightly more pure copper than TCu-NPs (88.154%). The moisture of TCu-NP (0.49%) is higher than RCu-NPs (0.36%) which been attributed to the agglomeration of the former to be more than the latter with rough morphology. BET surface area of the nanoparticle samples are TCu-NPs (342.11 m^2/g) and RCu-NPs (464.95 m^2/g) and the FTIR revealed some functional groups in both samples O-H, C=O, C-O, Cu-O stretching vibration peaks. The TGA analysis examine the effect of temperature on the weight of Cu-NPs and the thermal stability of the synthesized nanoparticle samples revealed that the TCu-NPs was lower than RCu-NPs because they attain stability with weight loss. Lowering of the thermal stability of both samples can be attributed to the catalytic nature of the Cu-NPs in lowering the degradation temperature.The quality of the nanoparticles synthesized from copper tailings is slightly lower than the reagent grade copper salt. This study has not been reported in any accredited journal.In conclusion, the quality of the synthesized Cu NPs from Cu tailings is slightly lower than the Cu from reagent grade salt which can be attributed to the presence of impurities.

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Figure1. Location of Ojo in the Lagos Metropolitan Area.

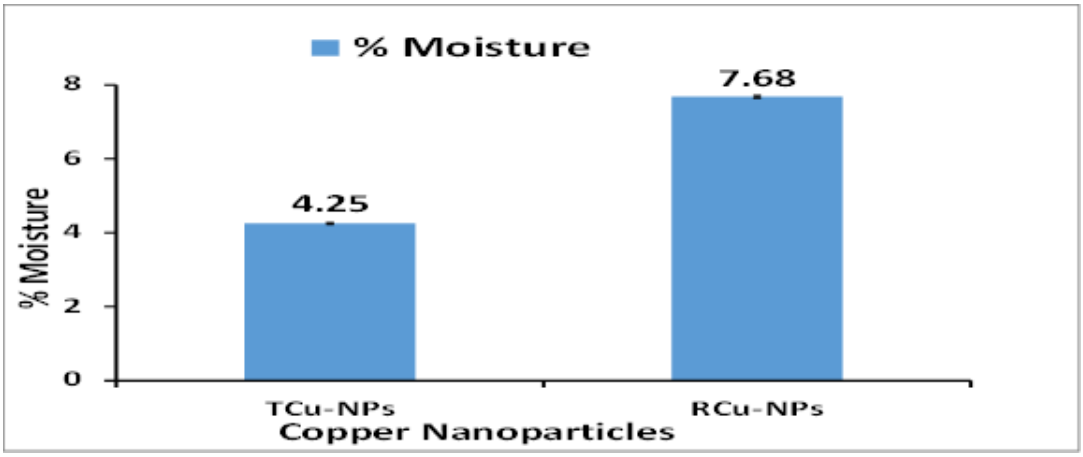

Figure 2. Moisture content of synthesized TCu-NPs and RCu-NPs.TCu-NPs = Tailings copper nanoparticles, RCu-NPs = Reagent salt copper nanoparticles

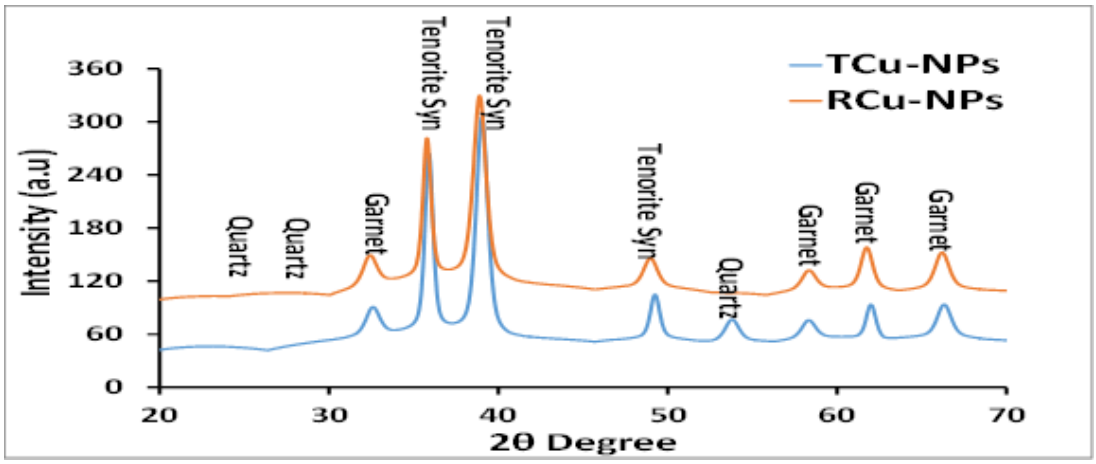

Figure 3. Quantification XRD mineral phases of synthesized copper nanoparticles from Cu tailings and reagent grade Cu salt

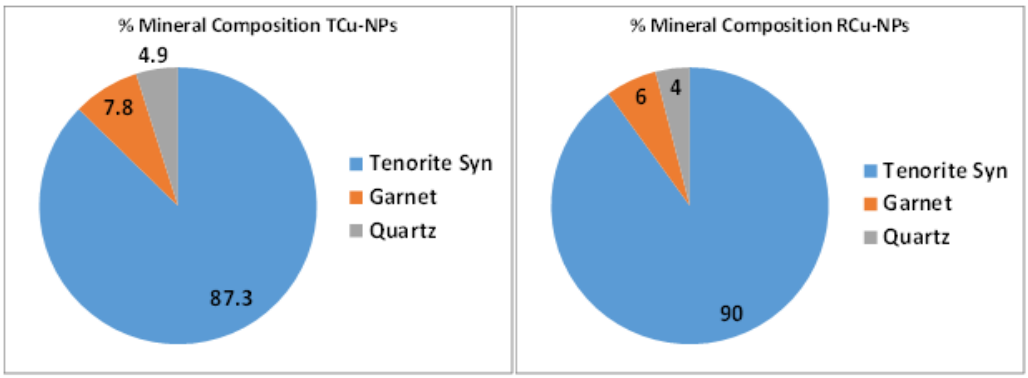

Figure 4. Quantification XRD mineral composition ofsynthesized copper nanoparticles from tailings (TCu-NPs) and reagent grade Cu salt (RCu-NPs)

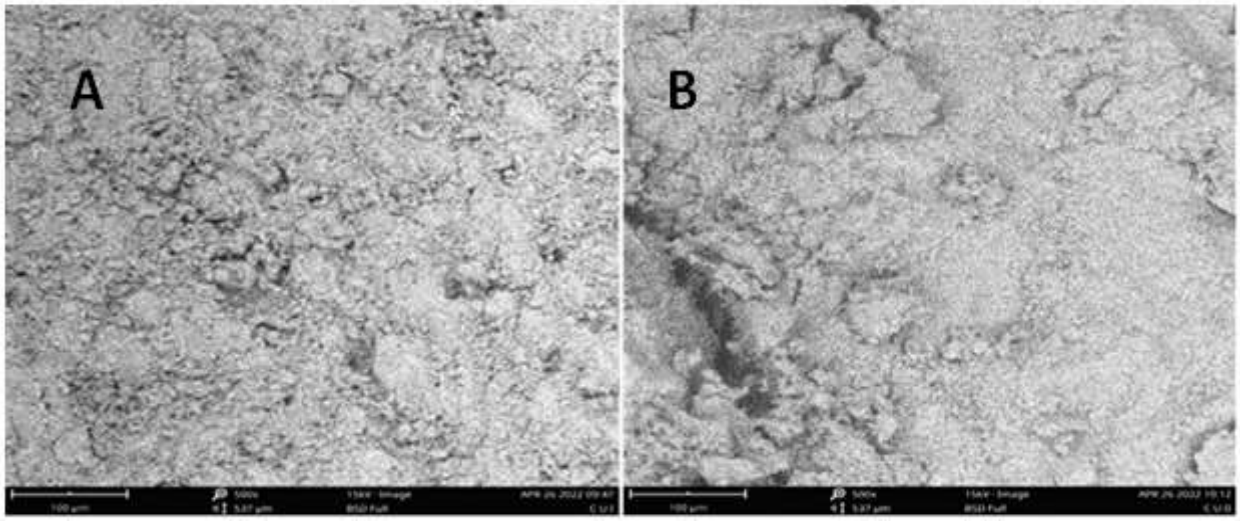

Figure 5. SEM morphology of synthesized nanoparticles from Cu tailings (A) and reagent grade Cu salt (B)

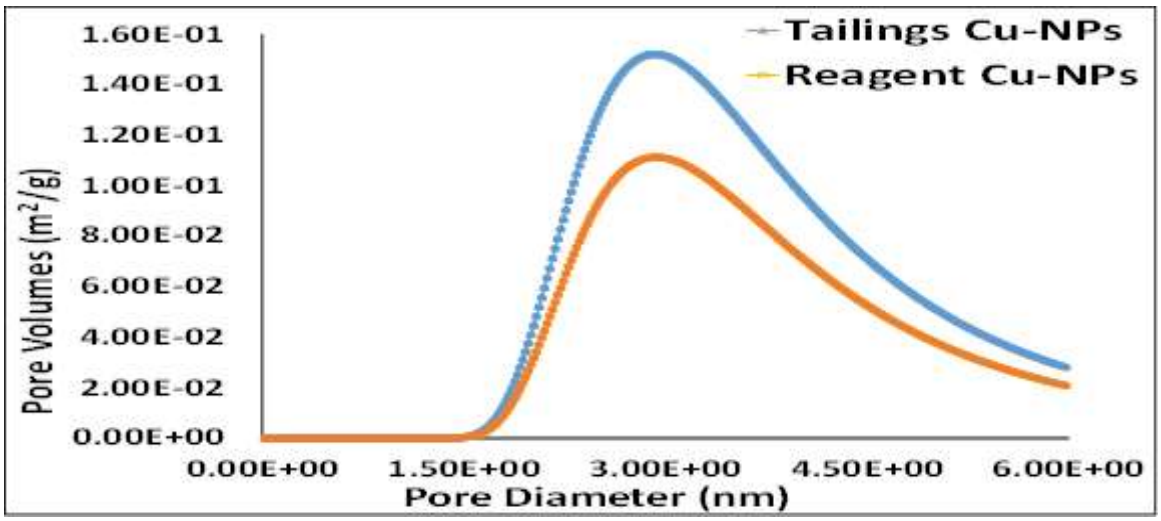

Figure 6. BET microporevolume analysis ofsynthesized nanoparticles from Cu tailings and reagent grade Cu salt

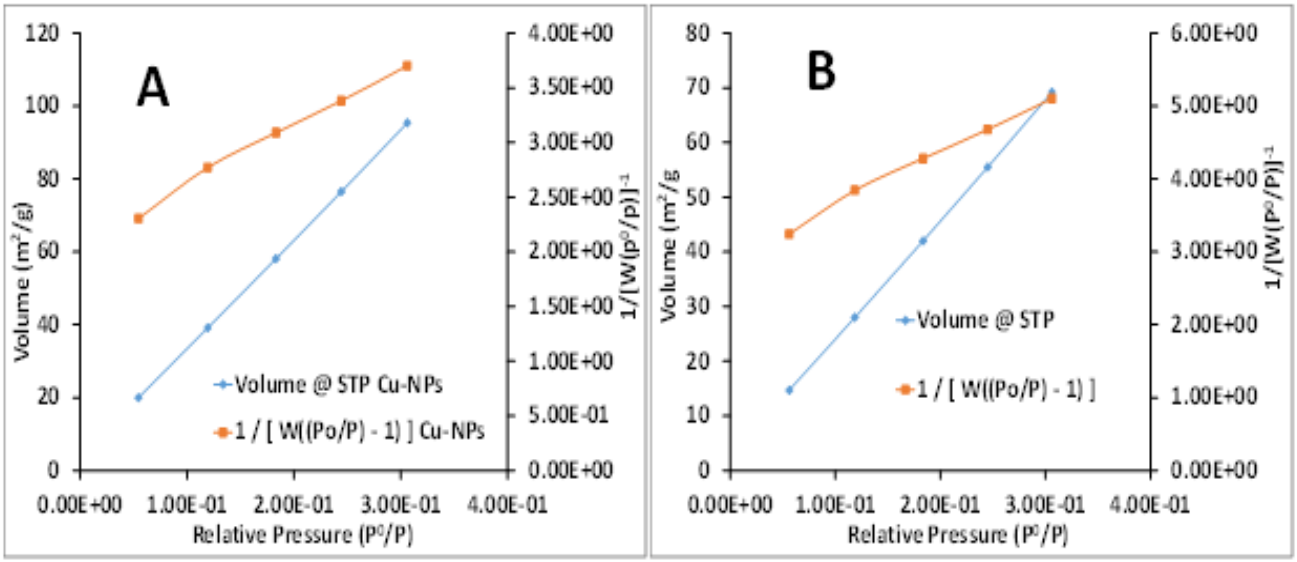

Figure 7. Multi-Point BET plot of Cu nanoparticle synthesized from Cu tailings (A) and reagent grade (B) salt

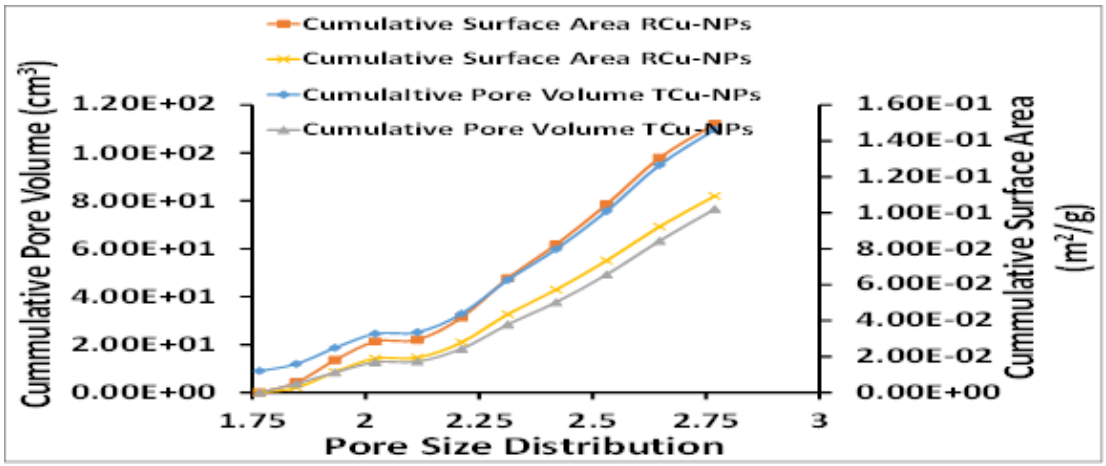

Figure 8 . Pore size distributionof copper nanoparticles synthesized from Cu tailings and reagent grade salt

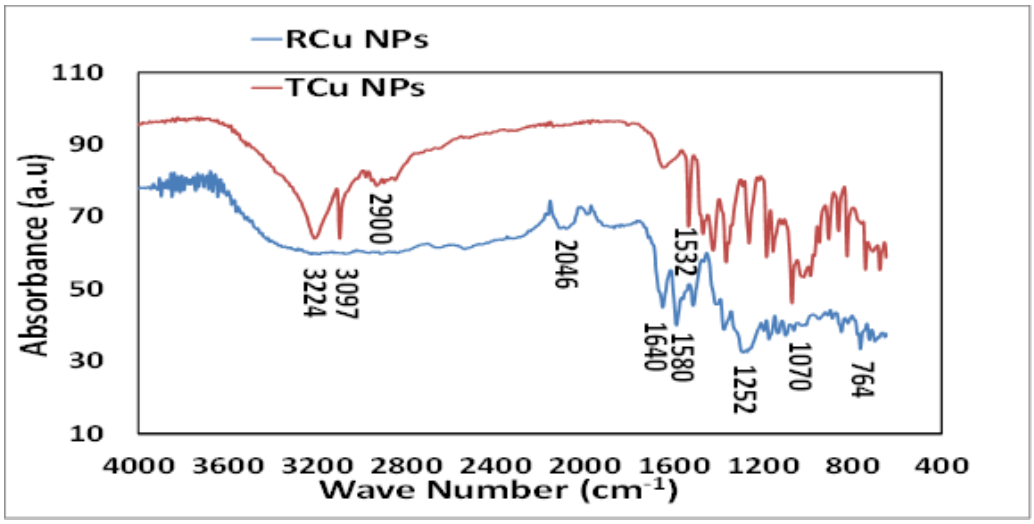

Figure 9. FTIR graph showing the functional groups in copper nanoparticles synthesized from copper tailings and reagent grade copper salt

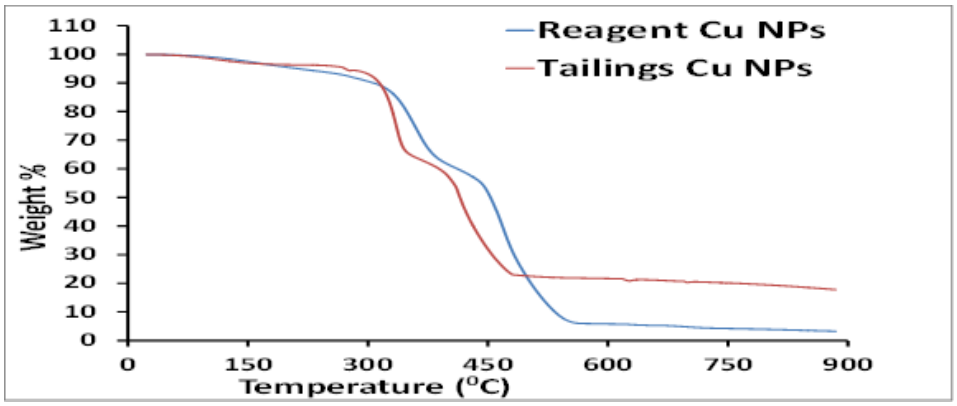

Figure 10 . TGA analysis of copper nanoparticles synthesized from reagent grade copper salt and copper tailings

Table 1. XRF elemental composition of Cu present in Cu nanoparticles synthesized from tailings (TCu-NPs) and reagent grade Copper salt (RCu-NPs).

Metal Oxide	% mass (% w/w) TCu-NPs (A)	% mass (% w/w) RCu-NPs (B)
SiO_2	**1.864**	**1.058**
Fe_2O_2	**0.351**	**0.118**
CuO	**85.154**	**85.978**
CaO	**0.383**	**0.224**

Table 2. BET Analysis of parameters

BET ANALYSIS	TCu-NPs	RCu-NPs
Pore Diameter	2.940e 00 nm	2.940e 00 nm
Micropore Volume	0.422e 00 nm	0.31e 00 nm
Surface Area	464.948 m2/g	342.107 m2/g
Pore Size Distribution	0.134 cc/g	0.099 cc/g
Pore Width	2.647	2.647 nm

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