

Recovery of high added-value materials from waste printed circuit boards



**Thesis submitted in partial fulfillment
for the Award of Degree**

Doctor of Philosophy

By

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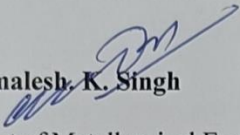
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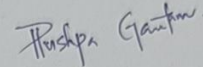
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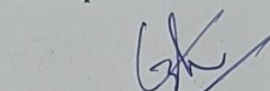
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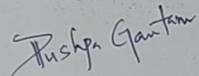
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Pushpa Gautam.

Pushpa Gautam

Dedicated
To
My Parents

List of Figures

Fig. 1.1	Different components of electronic waste	4
Fig. 1.2	Global e-waste generation from 2014 to 2019 and projection from 2020 to 2030 (modified from (Forti et al., 2020; Panda et al., 2021)) (Effect of COVID 19 is not included in the projected data)	6
Fig. 1.3	Total e-waste generated and recycled continent-wise in 2019	6
Fig. 1.4	Composition of printed circuit boards (Kaya, 2016)	9
Fig. 1.5	Overview of stages involved in metal recycling from electronic waste (Rao et al., 2020)	11
Fig. 1.6	Integrated process for the recycling of PCBs (based on the work of (Gautam et al., 2022a; Nayak et al., 2019; Wang and Xu, 2017; Wang et al., 2016))	20
Fig. 2.1	Pre-processing treatment of waste printed circuit boards	41
Fig. 2.2	XRD analysis of metal clad	44
Fig. 2.3	Graphical representation of copper dissolution from WPCBs and synthesis of CuO nanoparticles	47
Fig. 2.4	Graphical representation of all the samples obtained by this synthesis route	48
Fig. 2.5	Process of obtaining copper strip solution from mobile phone PCBs (Rao et al., 2021)	51
Fig. 2.6	Graphical illustration of the synthesis of CuO-based nanostructures from Cu strip solution of PCBs	53
Fig. 3.1	(a) Low magnification image showing cross-section of PCB, (b,c) zoomed-in image to show the distinct layers present in the PCB	66
Fig. 3.2	EDX of the different layers of the cross-section of WPCB-motherboard (a) top resin layer (b) fiberglass (c) metallic layer (d) fiberglass layer	68
Fig. 3.3	Elemental mapping of the cross-section of PCB	69

Fig. 3.4	Effect of temperature on nitric acid leaching of (a) copper, (b) zinc [Concentration of nitric acid 3 M; time 3 h; stirring speed 500 rpm; pulp density 50 g/L]	74
Fig. 3.5	Effect of temperature on nitric acid leaching of (a) copper, (b) zinc [Concentration of nitric acid 3 M; time 3 h; stirring speed 500 rpm; pulp density 50 g/L]	74
Fig. 3.6	Effect of pulp density on nitric acid leaching of (a) copper, (b) zinc [Concentration of nitric acid 3 M; temp. 30°C; time 2 h; stirring speed 500 rpm]	75
Fig. 3.7	Effect of pulp density on nitric acid leaching of (a) copper, (b) zinc [Concentration of nitric acid 3 M; temp. 30°C; time 2 h; pulp density 50 g/L]	75
Fig. 3.8	Leaching efficiency of various metals in nitric acid leaching [Concentration of nitric acid 3 M; temperature 30°C; time 2 h; stirring speed 500 rpm; pulp density 50 g/L]	77
Fig. 3.9	Chemical analysis of nitric acid leached solution obtained at optimized leaching parameters	77
Fig. 3.10	SEM-EDX of leach residue obtained in nitric acid leaching	78
Fig. 3.11	Kinetics plots for leaching of copper for (a) diffusion-controlled model and (b) reaction-controlled model and leaching of zinc for (c) diffusion-controlled model and (d) reaction-controlled model	80
Fig. 3.12	Arrhenius plot for leaching of copper for (a) diffusion-controlled model and (b) reaction-controlled model and leaching of zinc for (c) diffusion-controlled model and (d) reaction-controlled model	82
Fig. 4.1	X-ray diffraction pattern of calcined N1, N2, N3, and N4 samples	93
Fig. 4.2	FTIR spectra of calcined N1, N2, N3, and N4 powders	94
Fig. 4.3	SEM-EDX analysis of N1 (a,b), N2 (c,d), N3 (e,f), and N4 (g, h)	96
Fig. 4.4	UV-vis diffuse reflectance spectrum and the energy band gap (inset) of (a) N1, (b) N2, (c) N3, and (d) N4 samples	98-99

Fig. 4.5	TEM images at various magnifications (a,b), SAED pattern (c), High resolution-TEM image with interplanar d-spacing (d), particle size distribution plot (e) of as-synthesized	101
Fig. 4.6	TEM images at various magnifications (a–c), SAED pattern (d), High resolution-TEM image with interplanar d-spacing (e), particle size distribution plot (f) of as-synthesized N2 nanoparticles	102
Fig. 4.7	TEM images at various magnifications (a–c), SAED pattern (d), High resolution-TEM image with interplanar d-spacing (e), particle size distribution plot (f) of as-synthesized	103
Fig. 4.8	TEM images at various magnifications (a–c), SAED pattern (d), particle size distribution plot (e) of synthesized N4 microspheres	104
Fig. 4.9	XPS widescan spectra of N1 and N2 sample	105
Fig. 4.10	Deconvoluted XPS spectra of Cu 2p (a) and O 1s (b) for the as-prepared N1 sample	107
Fig. 4.11	Deconvoluted XPS spectra of Cu 2p (a), O 1s (b) and Zn 2p (c) for the as-prepared N2 sample	107
Fig. 4.12	(a, c) N2 adsorption–desorption isotherm and (b, d) corresponding BJH pore size distribution curve of N1 and N2	110
Fig. 5.1	Wastewater generation, its toxicity and treatment approaches in textile industry (modified from Azanaw et al., 2022)	117
Fig. 5.2	Photocatalytic mechanism of organic pollutant degradation in presence of photocatalyst	119
Fig. 5.3	Chemical structure of Congo red	120
Fig. 5.4	UV-visible spectra study of CR dye degradation recorded in the presence of N1, N2, N3, N4 catalyst upto 100 min visible light radiation	122
Fig. 5.5	Percentage degradation of CR in the presence of N1, N2, N3 and N4 upto 100 min irradiation time	122
Fig. 5.6	UV-visible spectra of CR dye recorded at different time points in the absence of catalyst	123
Fig. 5.7	UV-visible spectra of CR dye recorded at different time points in the presence of N1 catalyst	124
Fig. 5.8	Percentage degradation of CR with N1 NPs	125

Fig. 5.9	Kinetic study of photocatalytic performance of catalyst N1, N2, N3 and N4 with respect to CR dye degradation up to 50 min visible light irradiation	125
Fig. 5.10	Chemical structure of Methylene Blue (El-Sayed et al., 2022)	127
Fig. 5.11	UV-visible spectra study of MB dye degradation recorded in the presence of (a) N1, (b) N2, (c) N3, and (d) N4 catalysts upto 120 min	130
Fig. 5.12	Percentage degradation of MB in the presence of N1, N2, N3, and N4 upto 120 min time period	131
Fig. 5.13	UV-visible spectra of MB dye recorded at different time points in the absence of catalyst	131
Fig. 5.14	UV-visible spectra of MB dye recorded at different time points in the presence of N1 catalyst	132
Fig. 5.15	Percentage degradation of MB with N1 NPs	132
Fig. 5.16	Kinetic study of photocatalytic performance of N1, N2, N3, and N4 catalysts with respect to MB degradation upto 50 min	133
Fig. 5.17	Schematic diagram of the plausible photocatalytic mechanism	135
Fig. 6.1	Process flowsheet for synthesis of nanoparticle from strip solution of mobile phone PCBs	145
Fig. 6.2	XRD analysis of P1 and P2 samples	146
Fig. 6.3	FTIR analysis of P1 and P2 samples	148
Fig. 6.4	FESEM-EDX image of P1 (a,b) and P2 (c,d)	149
Fig. 6.5	Direct bandgap of P1 and P2 found from Tauc's plots	151
Fig. 6.6	XPS spectra for samples P1 and P2	153
Fig. 6.7	High resolution deconvoluted XPS spectra for (a,b) Cu 2p (samples P1 and P2) and (c,d) O1s (samples P1 and P2)	154
Fig. 6.8	TEM micrographs of P1 at varying magnifications (a-c), SAED pattern (d), interplanar spacing in high resolution image (e), zoom-in view of distinct planes (f)	155
Fig. 6.9	Particle width distribution plot	156

Fig. 6.10	Chemical structure of Rhodamine blue	157
Fig. 6.11	UV-visible spectra of RhB degradation without catalyst (a), with P1 (b) and P2 (c) at different time intervals under visible light, (d) percentage degradation of RhB upon reacting with P1 and P2, and its Kinetic study (e,f)	160
Fig. 6.12	A diagrammatic illustration of the possible photocatalytic mechanism	164

List of Tables

Table 1.1	Broad use of recycled components of different parts of WPCBs	17
Table 1.2	Nanostructured alloys recovered from waste PCBs	28
Table 2.1	Chemical analysis of powder PCBs of computer motherboards	43
Table 2.2	Chemical analysis of metallic fraction obtained from WPCBs of computer motherboards (after pretreatment)	45
Table 2.3	List of characterization methods involved in the study of value-added products	62
Table 4.1	List of different samples obtained from the synthesis route	90
Table 4.2	Average crystallite size of as-synthesized N1, N2, N3, and N4 samples	93
Table 4.3	Atomic weight% of elements present in N1 and N2	108
Table 5.1	Comparative assessment of Congo red degradation efficiency using CuO photocatalysts reported in different literatures	126
Table 5.2	Comparative assessment of Methylene blue degradation efficiency using CuO photocatalysts reported in different studies	134
Table 5.3	Characterization results summarized for N1, N2, N3, and N4 samples	137
Table 6.1	Chemical analysis of delaminated metallic fractions during leaching and after solvent extraction purification step.	144
Table 6.2	Atomic weight% of elements present in P1 and P2	153
Table 6.3	A comparison of the reported efficiency of RhB degradation using photocatalysts in various light sources	162

List of Abbreviations

Abbreviation	Description
WEEE	Waste electrical and electronic equipments
Mt	Million tonnes
PCBs	Printed circuit boards
wt%	Weight percent
WPCB	Waste-printed circuit board
CuO, Cu(II)O	Cupric oxide, copper oxide
ZnO	Zinc oxide
NiO	Nickel oxide
eV	Electron volt
NPs	Nanoparticles
Cu(OH) ₂ /CuO	Copper hydroxide/copper oxide
EEEs	Electrical and electronic equipments
UNU	United nations university
USD	United States dollar
e-waste	Electronic waste
kg	Kilogram
Cu	Copper
Ni	Nickel
Zn	Zinc
Al	Aluminium
Pb	Lead
Fe	Iron
Sn	Tin
Au	Gold
Ag	Silver
Pt	Platinum
Pd	Palladium
Cd	Cadmium
Hg	Mercury
SiO ₂	Silicon dioxide / silica

Al ₂ O ₃	Aluminium oxide
CaO	Calcium oxide
Kt	Kilo ton
ppm	Parts per million
ICSG	International copper study group
IBM	Indian bureau of mines
NMF	Non-metallic fraction
°C	degree Celsius
LNK carbonates	Li ₂ CO ₃ (Lithium carbonate), Na ₂ CO ₃ (Sodium carbonate), K ₂ CO ₃ (Potassium carbonate)
K	Kelvin
CO ₂	Carbon dioxide
Sb	Antimony
Bi	Bismuth
HCl	Hydrochloric acid
H ₂ SO ₄	Sulphuric acid
H ₂ O ₂	Hydrogen peroxide
HNO ₃	Nitric acid
HClO ₄	Perchloric acid
EDTA	Ethylene diamine tetraacetic acid
DTPA	Diethylene triamine penta acetate
Cr	Chromium
FeCl ₃	Ferric chloride
CuCl ₂	Copper(II) chloride
EoL	End-of-life
TV	Television
LTCC	Low-temperature co-fired ceramic
THSJs	through-hole solder joints
HAVP	High added-value products
EC	Electronic components
IC	Integrated circuit
Ta	Tantalum
CRT	Cathode ray tube

nm	nanometer
PVP	Polyvinylpyrrolidone
HEPES	4-(2-hydroxyethyl)-1-piperazine ethane sulfonic acid
DOPC	1,2-dioleoyl-Sn-glycero-3-phosphocholine
AC	Alternate current
ESM	Electronic scrap material
MLCCs	Multilayer ceramic capacitors
SnO ₂	Tin oxide
Si	Silicon
RSCWs	Rejected Solar cell wafers
AgCl	Silver chloride
μm	micrometer
HF	Fluoric acid
RSM	Response surface methodology
MEMs	Micro-electromechanical system
PEG	Polyethylene glycol
RAM	Random access memory
BER	Brominated epoxy resin
DMF	Dimethyl formamide
β-NCD	β-native cyclodextrins
CTAB	Cetyl trimethyl ammonium bromide
NaBH ₄	Sodium borohydride
PN-PCBs	Polymetallic nanoparticles from Printed Circuit Boards
RB4	Reactive blue 4
NaOH	Sodium hydroxide
NiMH	Nickel-metal hydride
SCM	Supercritical methanol
Cu ₂ O	Cuprous oxide
SCW	Supercritical water
EK	Electrokinetic
SIM	Subscriber Identity Module
NaOH	Sodium hydroxide

UV-vis	Ultra violet-visible
TiO ₂	Titanium oxide
SCWO	Super Critical Water Oxidation
MB	Methylene Blue
P25	Mixed-phase titania commercial photocatalyst
MO	Methyl Orange
H ₂ O ₂	Hydrogen peroxide
AAS	Atomic absorption spectroscopy
P-XRD	Powder X-ray diffraction
FTIR	Fourier transform infrared spectroscopy
ATR	Attenuated total reflectance
SEM	Scanning electron microscopy
EDS	Energy dispersive X-ray spectroscopy
EBSD	Electron backscatter diffraction
HR-SEM	High-resolution scanning electron microscopy
TEM	Transmission electron microscopy
SAED	Selected area electron diffraction
XPS	X-ray photoelectron spectroscopy
BET	Brunauer-Emmett-Teller
BJH	Barrett-Joyner-Halenda
DMA	n,n-Dimethyl acetamide
PTFE	Polytetrafluoroethylene
JCPDS	Joint Committee on Powder Diffraction Standards
v/v	Volume per volume
rpm	Revolutions per minute
M	Molar
CR	Congo Red
MB	Methylene Blue
LED	Light-emitting diode
ACORGA M5640	5-nonyl salicylaldoxine
RhB	Rhodamine blue
HAVPs	High added-value products

Al_2O_3	Aluminum Oxide
SiO_2	Silicon Dioxide
MgO	Magnesium Oxide
CaO	Calcium Oxide
BaO	Barium Oxide
NO_x	Nitrogen Oxides
$\text{Cu}(\text{NO}_3)_2$	Copper Nitrate
$\text{Cu}(\text{OH})_2$	Copper Hydroxide
FWHM	Full Width at Half Maximum
$\text{O}_2^{\cdot -}$	Superoxide radical
OH^{\cdot}	Hydroxyl radical
h^+_{VB}	Hole in valence band
e^-_{CB}	Electron in conduction band

PREFACE

Due to the rapid development in electronic industries and high consumer demand for upgrades in functionality and design, electrical and electronic equipment have a shorter lifetime in developed and developing countries markets, leading to tonnes of electronic waste. According to global transboundary e-waste flows monitor 2022, the worldwide generation of waste electrical and electronic equipment (WEEE) reached 41.8 million tonnes (Mt) in 2014, which increased to 44.7 Mt in 2016 and 53.6 Mt in 2019, and is expected to reach 74.7 Mt by 2030 and 110 Mt by 2050. Researchers have found that only 20% of the e-waste produced is being recycled every year and 80% is informally recycled, dumped, or incinerated, causing loss of valuable resources and environmental hazards. Printed circuit boards (PCBs), a valuable part of any e-gadget, contain up to 60 different elements and a significant metal content (40 wt%), should be viewed as valuable secondary resources. Their treatment not only reduces environmental impact compared to mining but also offers economic opportunities and promotes circular economies. Recycling with the only objective of waste management is not a striking scenario in emergent nations where economic interests oust environmental liabilities. Hence, it is essential to consider remanufacturing products in the process of WPCB recycling to re-utilize the recovered metals as starting material for production of commercially valuable products, e.g. nanomaterials. This would help acquire the goal of circular economy by closing the materials loop, decreasing inputs, reusing, minimizing waste, and efficiently recycling huge quantity of scrap electronics, creating wealth and reducing pollution. In this direction, nanotechnology has also been recently used to recover base metals, toxic metals, and precious metals in different sizes and morphologies. Oxides of transition metals, such as CuO, ZnO, NiO exhibit typical photocatalytic and catalytic attributes. Monoclinic cupric oxide, Cu(II)O, a p-type semiconductor, has a bandgap range of 1.2–1.8 eV. CuO offers diverse potential applications in fields of catalysis due to its thermal

stability, superconductivity, and photocatalytic behaviour.

In view of this, we present an eco-friendly and integrated process for the recovery of valuable metals from discarded computer motherboard PCBs via a hydrometallurgical route. Chemical pre-treatment liberates metallic fractions from PCB layers, followed by nitric acid leaching to obtain a copper-rich solution. We synthesize copper oxide nanoparticles (CuO NPs) from the reclaimed copper, adding value to the recovered material. Furthermore, the recovered value-added material is explored as a potential photocatalyst for degradation of textile dyes in the presence of visible light.

The thesis is structured into seven chapters:

- Chapter I: Reviews e-waste generation, composition, and recycling techniques, emphasizing high-value materials recovered from electronic waste.
- Chapter II: Details the experimental methodology used for recovery steps.
- Chapter III: Discusses the characterization and leaching of bare PCBs from computer motherboards, achieving high leaching efficiency for copper and zinc.
- Chapter IV: Illustrates the recovery of copper as copper oxide nanoparticles from the leach liquor of waste PCBs of computer motherboards by precipitation followed by calcination route and details the structural analysis.
- Chapter V: Focuses on evaluating the photocatalytic properties of the synthesized nanoparticles for degrading textile dyes Congo Red and Methylene Blue.
- Chapter VI: Explores the recovery of $\text{Cu}(\text{OH})_2/\text{CuO}$ and CuO nanostructures, via alkaline precipitation and low-temperature aging methods using the strip solution originated from laboratory-scale spent mobile phone printed circuit board recovery process and demonstrated their photocatalytic activity by evaluating Rhodamine Blue degradation.
- Chapter VII: Summarizes conclusions, outlines future work, and provides a

bibliography and publication list.

This integrated approach not only extracts valuable metals but also explores their applications, offering a sustainable solution to electronic waste management while contributing to environmental and economic benefits.

