ANNALS OF “DUNAREA DE JOS” UNIVERSITY OF GALATI
MATHEMATICS, PHYSICS, THEORETICAL MECHANICS
FASCICLE II, YEAR XIV (XLV) 2022, No. 2
DOI: https://doi.org/10.35219/ann-ugal-math-phys-mec.2022.2.09

Scanning Electron Microscopy and Energy Dispersive X-ray Spectrometry (SEM/EDX) - a semiquantitative eco-friendly method for identifying the metals in e-waste multi circuits boards

Dorina Simedru*, Anca Becze

INCDO-INOE2000, Subsidiary Research Institute for Analytical Instrumentation, ICIA, 67 Donath Street, 400293, Cluj-Napoca, Romania
**Corresponding author: dorina.simedru@icia.ro*

Abstract
Fast technological growth, the lack of civic responsibility regarding e-waste recycling, storage, and disposal, the lack of many years of specific legislation, and the lack of solutions to manage e-waste have led to an accelerated increase in environmental pollution. Besides pollution, other aspects, such as the depletion of natural resources, must be considered regarding e-waste, especially minerals and metals. Efficient waste management programs aim to recover the essential components of e-waste and to dispose of the others safely. A case study was applied on several boards from computer hard drives that were intended for testing if Scanning Electron Microscopy and Energy Dispersive X-ray Spectrometry (SEM/EDX) can be used to identify in a quick and eco-friendly manner the metals from e-waste multi-circuit boards. The results confirm that SEM/EDX can successfully replace the traditional method for metal determination, the first step in establishing the recovery methods.
Keywords: SEM/EDX, metals, e-waste, XRF, recovery

1. INTRODUCTION

In its most basic sense, e-waste refers to electric and electronic equipment (all household and business items with power or battery supply [1]) which has reached or is near the end of its useful life and must be disposed of. Many factors play a role in the amount of e-waste generated annually, but the major factors include rapid technological development, poor specific legislation, and socioeconomic factors. As a result of all these factors, 53.6 million tons of e-waste were produced globally in 2019 (only 17% is considered to be recycled appropriately), and it is estimated that the global e-waste will reach 110 Mt in 2050 [1]. Besides recycling, another critical issue when talking about e-waste is related to the recovery of metals, especially precious metals.

It is known that the quantity of metals from e-waste is approximately 60% [2] and is due to rare and/or precious metals (Cu, Au, Ag, Pd, and Pt) [3] and also to toxic metals (Pb, Hg, As, Cd, Se, and Cr) with a significant negative impact on the environment [2]. Their quantities in e-waste vary for different reasons [4, 5], such as the equipment’s age, the testing time [5], the producer, and the production year. Identifying their quantities will allow specific recovery methods to be applied [3, 6-8], reducing metal production and the environmental impacts of e-waste [3]. The most common method to determine the metals from e-waste involves the dissembling and granulation of e-waste followed by the treatment of the samples with an acid mixture and processing the samples using atomic absorption spectrometry, atomic emission spectrometry, and mass spectrometry techniques [4, 5]. This method is time and electrical energy-consuming and needs a high quantity of acids for its development. Therefore, it is not necessarily eco-friendly.

Scanning electron microscopy (SEM) and Energy Dispersive X-ray Spectrometry (SEM/EDX) is a quick, non-destructive technique that provides detailed visual images of a particle with high-quality and spatial resolution [9] and also a semi-quantitative analysis of elemental composition. Its potential to be used for metal determination in e-waste is poorly explored in several articles where the e-waste sample was previously shredded or incinerated [10, 11].

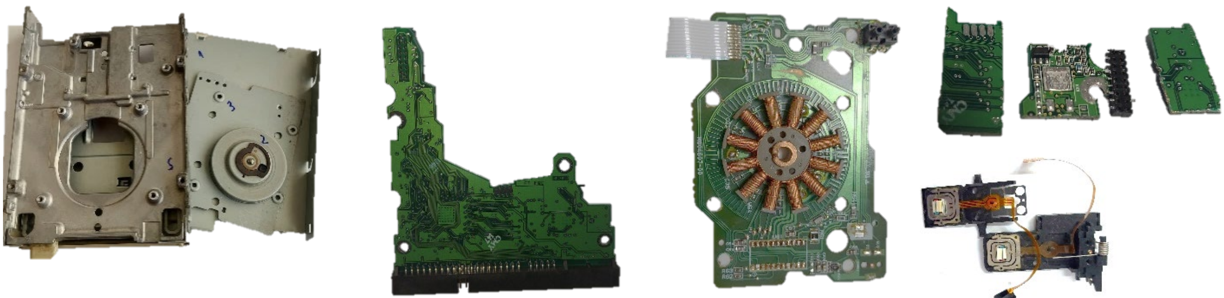
Having in view all the above and considering that the e-waste recycling and recovery strategies must be elaborated to comply with the measures of National Recovery and Resilience Plans (NRRPs), especially to the principle DNSH (do no significant harm to the environment), the purpose of this study is to verify if Scanning Electron Microscopy and Energy Dispersive X-ray Spectrometry (SEM/EDX) can be used as a semiquantitative eco-friendly method for identifying the metals in e-waste multi circuits boards.

2. EXPERIMENTAL

To verify the statement of this study, 3 similar hard drives from 2003 were analyzed to test SEM-EDX's capability to identify metals in e-waste multi-circuit boards.

Sample preparation

The hard drives were disassembled (Fig. 1). Hard drive metal covers were examined using X-ray Fluorescence (XRF). The electronic boards were cut into smaller pieces, up to 2 mm^2, and taped on the microscope stub with carbon tape for Scanning Electron Microscopy and Energy Dispersive X-ray Spectrometry (SEM/EDX) analysis.



a) b)

Fig. 1. Components of investigated hard drive: a) hard drive metal covers; b) electronic boards

X-ray Fluorescence (XRF)

The elemental composition of hard drive covers was determined using a handheld XRF elemental analyzer system, Bruker Tracker 5i (Fig 2 a).

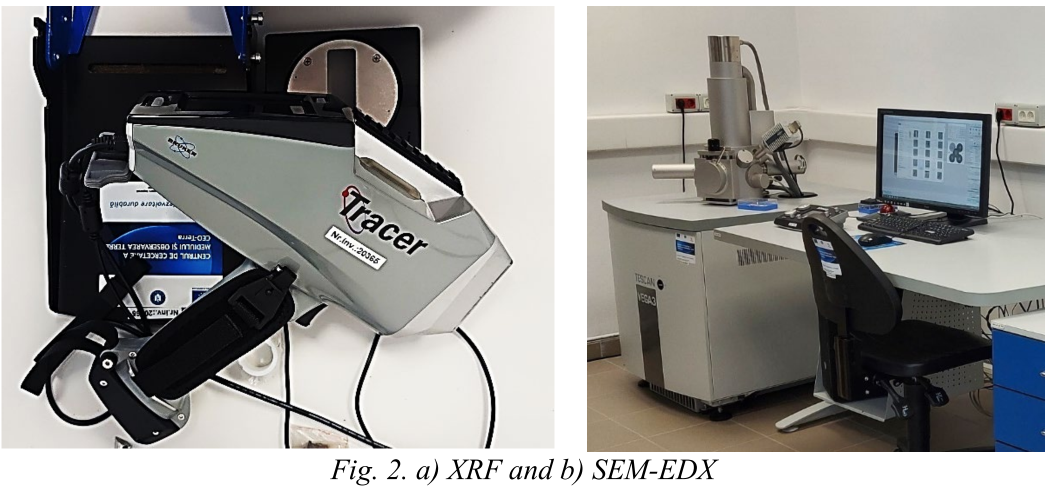


Fig. 2. a) XRF and b) SEM-EDX

Scanning Electron Microscopy and Energy Dispersive X-ray Spectrometry (SEM-EDX)

SEM TESCAN VEGA 3 SBU microscope with a QUANTAX EDS Xflash detector (Fig. 2b) was used to obtain information regarding the surface morphology and elemental composition of several parts of hard drives’ electronic boards. Secondary Electron (SE) and Backscattered Electron Detectors (BSE) were used. EDX data were processed using Bruker Esprit software that allows single-point Easy EDX analysis and sample mapping.

3. RESULTS AND DISCUSSION

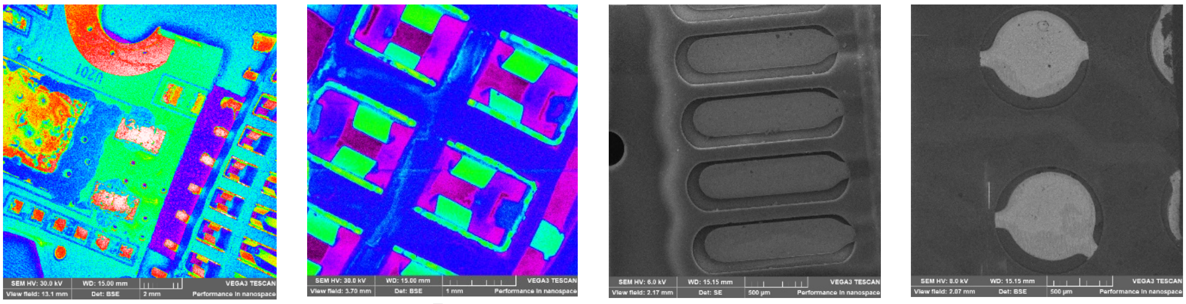
XRF analysis was performed on the metal covers of the hard drive (Fig. 1 a)) to have a complete view of their elemental composition. The results are presented in Table 1. The major components in metal covers are Fe, Zn, Si, and Mg. Their concentration varies in small percent.

Table 1. Elemental composition of metal covers from investigated hard drives

Sample	Fe (%)	Zn (%)	Si (%)	Mg (%)
1	75.93	21.15	1.55	<LOD*
2	82.76	7.71	4.15	1.94
3	77.13	20.16	1.52	<LOD*
4	777.24	20.49	<LOD*	1.21

*Limit of Detection (LOD)< 1%

SEM microscopy was used to study the morphology (shape and size) of several components from the electronic board (Fig. 3) of the investigated hard drives. Their elemental composition and the elemental mapping were obtained using an EDX detector.



A B C D

Fig. 3. A – part of hard drives’ electronic board; B – Integrated Circuit (IC); C– HDMI port; D – connector

Component A (Fig. 3) was investigated using single-point Easy EDX analysis. Several resistors, ports and interconnections pads were analyzed (Fig. 4) . In all analyzed components, the major component Sn's value ranges from 50.2 to 63.5% and Pb's value ranges from 24.9 to 34.0%.

A part of an integrated circuit (component B – IC) can be observed in the right corner of component A (Fig. 4). The Easy EDX analysis on several small plates within IC shows the presence of 54.3% Au. The dimensions of the plates were measured, and a map showing the distribution of elements on component B was recorded to confirm the presence of Au (Fig. 5). The results obtained are presented in Tables 2 and 3. The measurements of Au plates show that area of the plates ranges from 46915.43 to 134293.61 μm^2, while the elemental map distribution shows that IC major components are Al, Au, Ni, O, Cu, Sn and Pb where Al, Au and Ni have the highest concentrations.

The components of an HDMI port (component C) and a regular connector (component D) were measured (Fig. 6). Their EDXs map elemental distribution was recorded (Fig. 7). The map results show that the major elements of both components are Cu, Sn, and Pb, their results are being presented in Table 4.

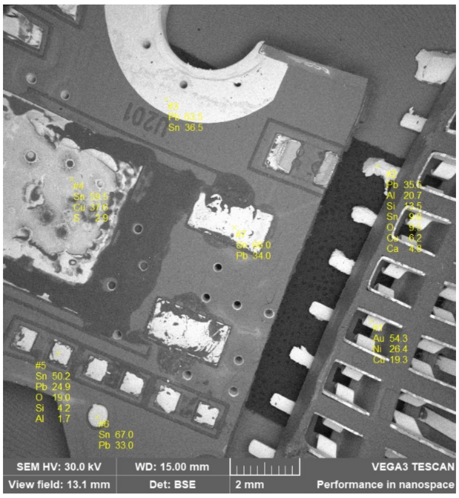
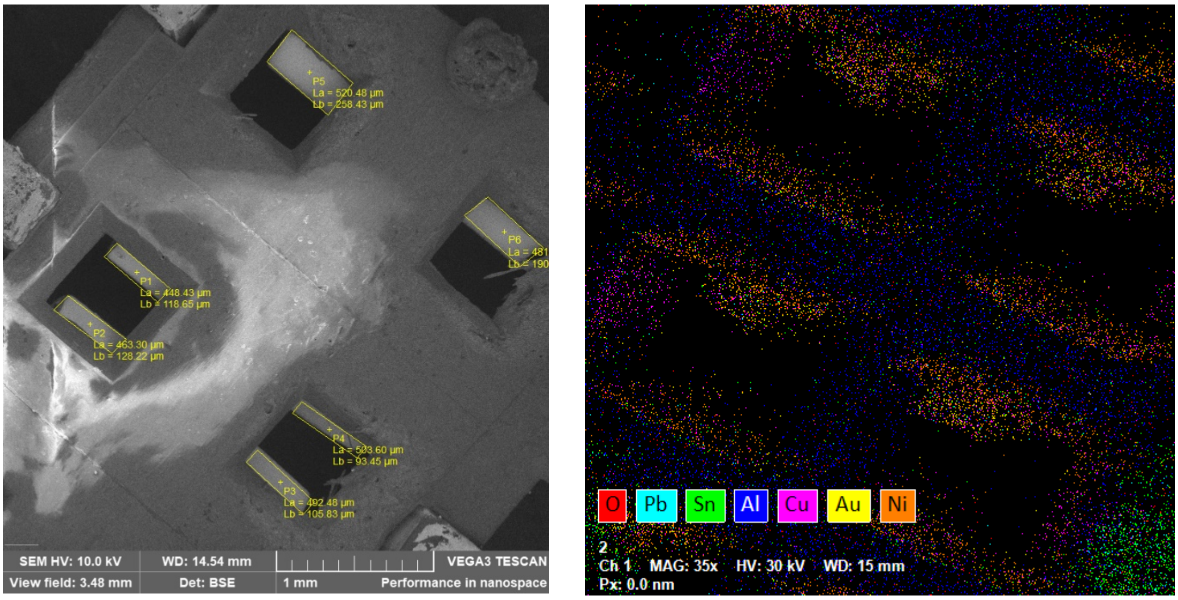


Fig. 4. Easy EDX analyses of component A



a) b)

Fig. 5 a) measurement of IC gold plates and b) map of the elemental distribution of IC (component B)

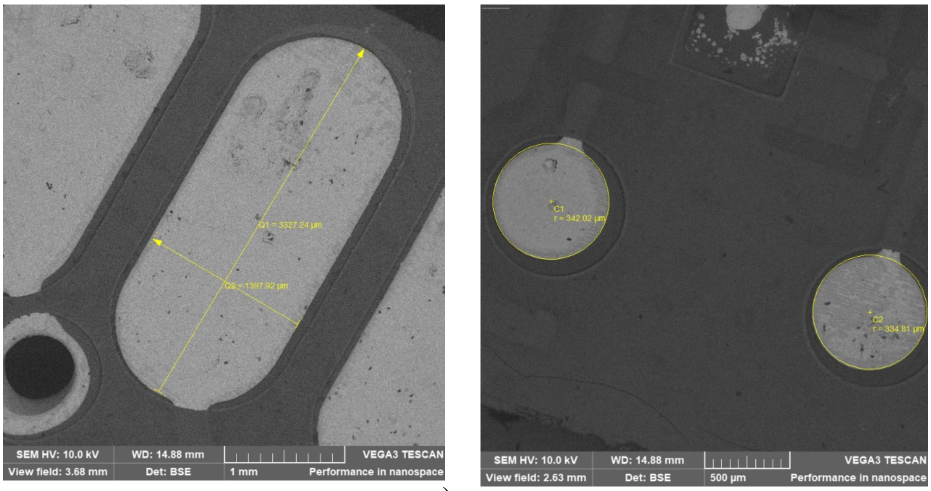
Table 2. Measurement data for Au plates

No.	ID	La (µm)	La (µm)	A (µm²)
1.	P1	448.43	118.65	53142.05
2.	P2	463.30	128.22	58772.72
3.	P3	492.48	105.83	52114.55
4.	P4	503.60	93.45	46915.43
5.	P5	520.48	258.43	134293.61
6.	P6	481.20	190.45	90501.77

Table 3. Values obtained for major constituents of component B, as a result of map elemental distribution analysis

No.	Element	Mass (%)
1.	Al	27.68
2.	Au	19.86
3.	Ni	19.30
4.	O	14.51
5.	Cu	8.96
6.	Sn	7.84
7.	Pb	1.84

While only one set of the results obtained for the electronic board of investigated hard drives is presented, it was noticed during the research that the values are closely similar for all three investigated electronic boards, with a deviation of ±2% for major elements and ±0.4% for minor elements.

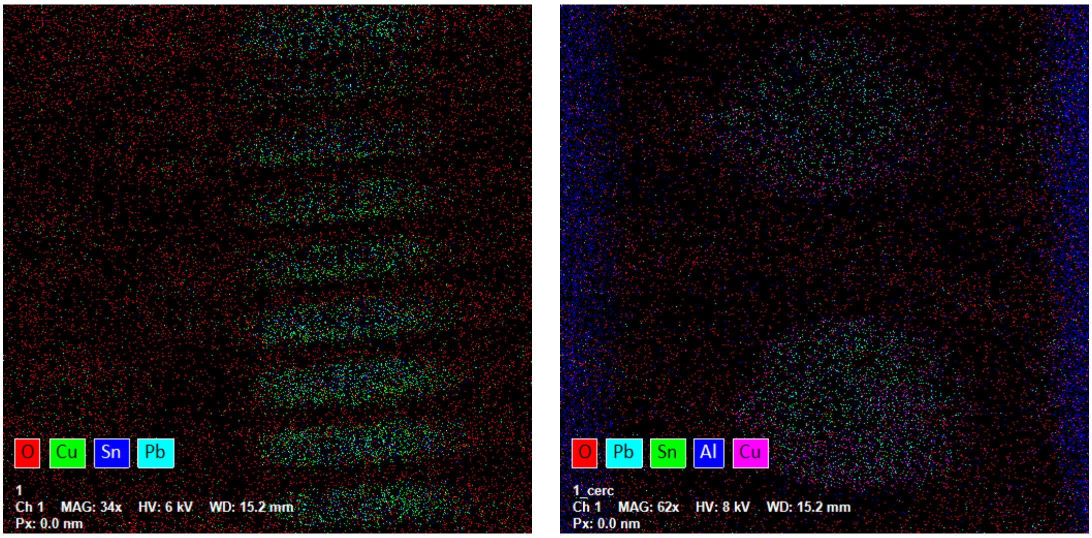


a) b)

Fig. 6. Measurements of components from a) HDMI port (component C) and b) a regular connector (component D)

Table 4. Values obtained for major constituents of components C and D as a result of map elemental distribution analysis

No.	Element	Mass (%)	
		Component C	Component D
1.	O	43.19	33.46
2.	Sn	15.30	22.78
3.	Al	-	18.85
4.	Pb	22.40	15.23
5.	Cu	19.12	9.68



a) b)

Fig. 7. a) HDMI port (component C) and b) a regular connector (component D) map elemental distribution

4. CONCLUSIONS

Scanning Electron Microscopy and Energy Dispersive X-ray Spectrometry (SEM-EDX) were used to investigate the surface morphology and the elemental composition of several parts from the electronic boards of three similar hard drives from 2003. The elemental mapping on investigated surfaces showed the distribution of the elements on the surface and their concentration. The measurements allow estimating the dimensions of the components. The results obtained for all investigated hard drives were almost similar (max. variation of ±2% for major elements and ±0.4% for minor elements), showing the accuracy of the analysis. These results are a first step in replacing the traditional methods to determine the metals from e-waste with non-destructive eco-friendly methods and can be further used for developing prediction models.

Acknowledgments. This project is financed by the Ministry of Research, Innovation and Digitization through Program 1- Development of the national research &development system, Subprogram 1.2 - Institutional performance - Projects that finance the RDI excellence, Contract no. 18PFE / 30.12.2021.

References

1. Baldé C.P., D'Angelo E., Luda V., Deubzer O., Kuehr R. (2022), Global Transboundary E-waste Flows Monitor - 2022, United Nations Institute for Training and Research (UNITAR), Bonn, Germany.
2. Chakraborty S.C., Qamruzzaman M., Zaman M.W.U., A. Masruck, H. Delowar, Pramanik B.K., Nguyen L.N., Nghiem L.D., Ahmed M.F., Zhou J.L., H. Mondal Ibrahim, Hossain M.A., Johir M.A.H., Ahmed M.B., Sithia J.A., Zargar M., Mohammad Ali Moni, Metals in e-waste: Occurrence, fate, impacts and remediation technologies, Process Safety and Environmental Protection 162 (2022) 230-252.
3. Kumar A., Holuszko M., Espinosa D.C.R., E-waste: An overview on generation, collection, legislation and recycling practices, Resources, Conservation and Recycling 122 (2917) 32-42.
4. Gorewoda T., Eschen M., Charasińska J., Knapik M., Kozłowicz S., Anyszkiewicz J., Jadwiński M., Potempa M., Gawliczek M., Chmielarz A., Kurylak W., Determination of Metals' Content in Components Mounted on Printed Circuit Boards from End-of-Life Mobile Phones, Recycling 5(3) (2020) 1-20.

5. Anić-Vučinić A., Bedeković G., Šarc R., Premur V., Determining Metal Content in Waste Printed Circuit Boards and their Electronic Components, Journal of Sustainable Development of Energy, Water and Environment Systems 8(3) (2020) 590-602.
6. Łukomska A., Wisniewska A., Dabrowski Z., Lach J., Wróbel K., Kolasa D., Domanska U., Recovery of Metals from Electronic Waste-Printed Circuit Boards by Ionic Liquids, DESs and Organophosphorous-Based Acid Extraction, Molecules 27 (2022) 1-21.
7. Islam A., Ahmed T., Awual R., Rahman A., Sultana M., Aziz A.A., Monir U.M., Teo S.H., Hasan M., Advances in sustainable approaches to recover metals from e-waste-A review, Journal of Cleaner Production 244 (2020) 1-12.
8. Debnath B., Chowdhury R., Ghosh S.K., Sustainability of metal recovery from E-waste, Frontiers of Environmental Science & Engineering 12 (6) (2018) 1-13.
9. Akhtar K., Khan S.A., Khan S.B., Asiri A.M., Scanning Electron Microscopy: Principle and Applications in Nanomaterials Characterization, Handbook of Materials Characterization (2018) 113–145.
10. Sun Z.H.I., Xiao Y. Sietsma J., Agterhuis H., Visser G., Yang Y., Characterization of metals in the electronic waste of complex mixtures of end-of-life ICT products for development of cleaner recovery technology, Waste Management 35 (2015) 227-235.
11. López C.C., Reyes Cruz V. E., Veloz Rodríguez M. A., Hernández Ávila J., Flores Badillo J., Cobos Murcia J.A., Speciation and Characterization of E-Waste, Using Analytical Techniques: TMS/Characterization, in: Characterization of Minerals, Metals, and Materials (Shadia Jamil Ikhmayies S.J., Li B., Carpenter J.S., Hwang J.Y, Monteiro S.N., Li J., Firrao D., Zhang M., Peng Z., Escobedo-Diaz J.P., Bai C. Editors), Chapter 79, Wiley Online Library, pp. 629-636, 2016.