



Spectroanalytical method for evaluating the technological elements composition of magnets from computer hard disks

Jeyne Pricylla Castro, Edenir Rodrigues Pereira-Filho*

Group of Applied Instrumental Analysis, Chemistry Department, Federal University of São Carlos, P. O. Box 676, 13565-905 São Carlos, São Paulo State, Brazil



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ABSTRACT

In this study, inductively coupled plasma optical emission spectrometry (ICP OES) was used for the development of an analytical procedure for elemental analysis of hard disks (HDs). More than 50 samples were acquired and separated according to the manufacturers. Both magnets were used, namely, the actuator (a) and the spindle (s) motor. The samples were heated to remove magnetism, ground in a knife mill and sieved. Different digestion procedures were performed with a block digester and microwave, and three acid concentrations were employed. Due to the lack of certified materials or references from HDs, tests of standard addition and recovery were performed to verify the accuracy of the proposed method. Hyperspectral image and principal component analysis were also used to assist in the data treatment related to the Ni layer. For both magnets, the best analytical procedure was 100 mg of sample, 7 mol L⁻¹ nitric acid and a digester block. The elements observed in the highest concentrations were Fe followed by Nd and Pr. This procedure was simpler than others were and prioritized the principles of green chemistry.

1. Introduction

Environmental contamination by incorrect disposal of electronic equipment and its components is one of the largest problems derived from modern human activities [1]. These materials are named e-waste or WEEE and are the residue type with the highest growth on a global scale. The term “e-waste” is used for all electrical and electronic equipment (EEE) discarded without the intention of reuse [2].

EEE include toxic elements in their composition, which can contaminate the environment and affect human health when improperly discarded [3–5]. Conversely, EEE contain several noble and rare earth elements (REEs) with industrial, technological and strategic importance [6]. Therefore, e-waste has the potential to become an important source of resources using methods or strategies for the recovery of specific elements, minimizing the environmental impact and improving the economy in several countries [7].

Computer hard disks (HDs) are a component where data are stored. The HD is a highly complex computer component with several parts composed of plastic, metals, printed circuit boards (PCBs) and two types of magnets: (1) actuator and (2) spindle motor. In this study, both magnets were analytically interrogated due to the rich and strategic REE composition [8]. Actuator magnets move the reading arm, and spindle motor magnets are responsible for maintaining a constant rotation of the disk. These magnets are composed of a Nd-Fe-B alloy,

which includes a large amount of REEs [8]. REE, due their electronic configurations, have several technological characteristics, such as magnetism, luminescence, optical, electrical and thermal properties [9,10].

The scientific literature regarding this type of sample is notably scarce, and only a small number of examples were found and are presented in the following. Ueberschaar and Rotter [11], for instance, presented a sample preparation procedure for further determination of 14 analytes using inductively coupled plasma optical emission spectrometry (ICP OES). In the mineralization protocol, nitric acid and aqua regia were used for the actuator and spindle motor magnets, respectively, and the process was accomplished using microwave-assisted dissolution. The authors used a concentrated acid mixture (aqua regia) with a large amount of sample for more than 10 h, and only microwave-assisted dissolution was tested. The whole procedure makes the sample preparation process longer and limited to a small number of laboratories. In addition, the authors did not report important details regarding magnet characterization, such as the method's limits of detection and evaluation of its efficiency with certified reference material or standard addition and recovery tests.

Habib et al. [8] described a method for the direct analysis of actuator magnet composition using wavelength dispersive X-ray fluorescence (WD XRF). The authors found approximately 30% Nd, 5% Pr, and 2% Dy, and the balance was mainly Fe. Low-concentration elements in

* Corresponding author.

E-mail address: erpf@ufscar.br (E.R. Pereira-Filho).

the range of mg/kg were not determined.

Munchen et al. [12] published a study where a thermal demagnetization process was described for the magnets magnetic field elimination. The samples were ground in a knife mill, and the digestion used the 3051 A method as described by the United States Environmental Protection Agency (US-EPA). Nd, Fe and B were determined by ICP OES, and the Ni coating layer composition was investigated by WD XRF. As can be noted, the scientific literature regarding this type of important sample is limited, and a systematic investigation could shed light on this problem, such as the effects of acid composition, the use of different and inexpensive sample preparation instruments and the chemical concentration of minor elements.

Currently, analytical chemistry represents an important role by providing qualitative, quantitative, temporal and spatial results on the chemical composition of matter [13]. Several applications have already been published targeting polymers, printed circuit board (PCB), mobile phone classifications, and chemometric application in this field [14–20]. The goal of the present study is the development of a reliable analytical procedure for the characterization of both HD magnets, considering the principles of green chemistry and minimum sample preparation. The results presented can help recycling companies improve their HD and e-waste recovery processes. In addition, more than 50 HD samples from different countries were collected, permitting a global chemical view of this material. The experimental part was mainly devoted to REE determination, and Nd alone represents a market of approximately 100 dollars/metric ton for its oxide [21].

2. Experimental

2.1. Sample collection, organization and preliminary preparation steps

In this study, 56 hard disk samples and a group of 6 actuators were collected from old laptop and desktop computers. These samples were manufactured in Brazil, China, Malaysia, Korea, Singapore and Thailand from 2001 to 2014. Nine different manufacturers (M1 – M9) were identified. The sample preparation procedure was divided into three steps. The first step was the HD disassembly. The main HD parts identified are presented in Fig. 1: original (a) and open (b) HD, platters (c), magnets (d), heads (e) and logic PCB (f).

Both magnet types (Fig. 1, part d) were used in this study. The second step was the elimination of the sample magnetic field. In this step, the samples were heated in a furnace at 500 °C for approximately two hours. These magnets have high resistance to demagnetization, losing the magnetic field if they are heated above the Curie temperature (~ 312 °C) [12].

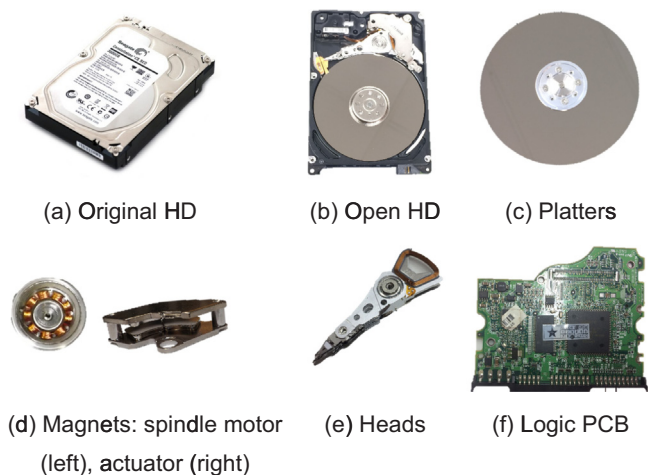


Fig. 1. Computer HD sample disassembly (a and b) with the presentation of main components: platters (c), magnets (d), heads (e) and logic PCB (f).

Table 1

Sample characteristics (country origin and manufacturer codification, M1-M9 and unidentified) for each magnet studied: actuator (a) and spindle motor (s).

Sample identification	Quantity of sub-samples	Country origin	Manufacturer codification
1a ^a and 1s ^b	12	Brazil	M1
2a and 2s	6	Korea and two unknown	M1
3a and 3s	13	China	M2
4a and 4s	7	Brazil, Singapore and Thailand	M2
5a and 5s	7	China, Singapore and Thailand	M3
6a and 6s	5	Malaysia and Thailand	M4
7a and 7s	6	China, Singapore and Thailand,	M5 – M9
8a	6	Unidentified	Unidentified

^a Magnets from the actuator.

^b Magnets from the spindle motor.

The third step was sample milling. In this case, the magnets were combined in groups from 5 to 13 individual sub-samples in order to have sufficient volume for the milling process. The criteria used for sample categorization was the magnet's manufacturer. Table 1 shows the samples for each group of magnets.

The sub-samples were ground in a knife mill (IKA, A11), and the particle size was estimated through the use of a 500-µm sieve.

2.2. Reagents and sample digestion procedure

The literature is scarce in analytical procedures for HD analysis. Therefore, some digestion tests with concentrated or diluted HNO₃ (Synth, Diadema, São Paulo State, Brazil) were evaluated. The acid used was purified in a sub-boiling distillation system, Distillacid™ BSB-939-IR (Berghof, Eningen, Germany). In all experiments, 100 mg of sample was weighed, and 10 mL of acid mixture was used. Three different concentrations of acid were tested: 7 mol L⁻¹ (one part of acid + one part of deionized water), 9 mol L⁻¹ (two parts of acid + one part of deionized water) and 14 mol L⁻¹ (only the concentrated nitric acid). The digestions were performed in a microwave system (Speedwave four, Berghof, Eningen, BW, Germany) and a digester block (Marconi, Piracicaba, São Paulo State, Brazil) to compare both digestion methods. The heating program used in the microwave is described in Table 1S in the Supplementary material and was accomplished with three steps. For the digester block, perfluoroalkoxy (PFA) closed tubes with an inner volume of 50 mL (Savillex, MN, USA) were used, and the heating program constituted two hours at 100 °C.

The final volume was made 50 mL with deionized water (Milli-Q®, Millipore, Bedford, MA, USA), and a number of consecutive dilutions were performed. ICP OES (Thermo Scientific, iCAP 6000) with the axial view was used to determine Al, B, Co, Cu, Dy, Fe, Gd, Nd, Ni, Pr, Sm, Sn, Tb and Zn. The maximum sample and standard acidity was 10% to avoid compromising the physical integrity of the ICP OES components. These experiments were made only for sample 3 of the actuator (3a) magnet (see Fig. 1 and Table 1 for details).

The limits of detection (LOD) and quantification (LOQ) for both sample preparation procedures were calculated using equations from (1) to (4) [22].

$$SBr = \frac{(I_{\text{standard}} - I_{\text{blank}})}{I_{\text{blank}}} \quad (1)$$

$$BEC = \frac{C_{\text{standard}}}{SBr} \quad (2)$$

$$LOD = \frac{3 \times BEC \times RSD}{100} \quad (3)$$

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