

Fingerprint of Precious Metals Ornaments to Post-Mortem Inventory by Energy Dispersive X-Ray Fluorescence Spectrometry

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Abstract: *Quantitative analysis of metal alloys in ornaments can reveal relevant aspects about the substrates used in the manufacturing process. Fifty-three female ornaments (chains, rings, and earrings) were identified as jewelry and semi-jewelry using EDXRF. Twenty-three samples were identified and evaluated as jewelry because they had a substrate composed of different concentrations of gold (40-83%) ranging from 10 to 21 carats. This study can be considered the first to use EDXRF to analyze jewelry and semi-jewelry for post-mortem inventory, and substrate composition was decisive for fingerprinting the valuation of each artifact.*

Keywords: jewelry, semi-jewelry, post-mortem inventory, fingerprint

1. Introduction

The post-mortem inventory in succession law is used to make a detailed list of inheritance author (*cujus* or deceased) assets. According to Brazilian Civil Code Law 10406 [1], an inventory person in possession can leave real estate or movable property such as jewelry as an inheritance [31].

Jewelry is an eccentric and expensive piece that can be disputed between heirs as it has economic [2], [3], historical, social, and cultural value [4]. Metal alloys of noble metals such as gold (Au), palladium (Pd), platinum (Pt), silver (Ag), and titanium (Ti) are used in jewelry making [4]. These alloys combine two or more metal characteristics: malleability, toughness, change in concentration, and shade [5]. The alloys are according to evaluated to concentration Au in carats (K), 24 K (100% Au); 22 K (91.6% Au); 18 K (75% Au); 14 K (58.5% Au); 9 K (37.5% Au) [6].

The most common ternary alloys have shades and concentrations [4], [6-8]: Yellow (Au 18 K) composition 75% Au + 16.66% Ag + 8.33% Cu; White (Au 18 K) with 75% Au + 12.50% Ag + 12.50% Pd; Red (Au 14 K) 75% Au + 8.33% Ag + 16.66% Cu; Yellow (Au 14 K) 58.33% Au + 27.78% Ag + 13.89% Cu; Red (Au 14 K) 58.33% + 13.89% Ag + 27.78% Cu; White (Au 14 K) 58.33% Au + 20.80% Ag + 20.80% Cu; Yellow (Au 12 K) 50% Au + 25% Ag + 25% Cu; Red (Au 12 K) 50% Au + 50% Cu; White (Au 12 K) 50% + 25% Ag + 25% Pd. Every piece of jewelry has alloys made from Au (75%) Cu and Ag, available commercially in wide jewelry and semi-jewelry varieties [8]. Semi-jewelry is electroplated [4] depending on its purpose and according to the manufacturer [8]. Semi-jewelry is

produced from metal alloys, stainless steel, zamak, brass, and alpaca and coated by deposition or electroplating noble metals Ag and Au [9]. After deposition, there is no visual distinction between semi-jewelry and jewelry.

Jewelry appraisal consists of identifying, classifying, and pricing the piece. The pieces are identified using physical tests such as checking for welds, oxidation, the manufacturer's signature, defects, adornments, plagiarism, and determining mass, size, and density. Chemical tests that damage pieces use royal water and touchstones (basalt or slate) [10]. In addition, the chemical constitution of jewelry-making alloys can contain elements in minor or trace concentrations that can be difficult to detect and dramatically affect economic value. Therefore, thorough analysis for post-mortem inventory is essential, and selecting analytical techniques is very important since analysis must be non-destructive, fast, highly sensitive, and quantitative [11], such as energy dispersive X-ray fluorescence spectrometry (EDXRF).

EDXRF is an instrumental technique with a sufficient spectral resolution to identify and quantify various elements in different matrices [12]-[27]. In addition, analytical methods involving EDXRF do not usually require the sample to be subjected to chemical treatment with concentrated acids, heating, or dilution in organic solvents [17], [20]. It has excellent sensitivity and measurement accuracy in solid samples, making it extremely important for its application in identifying precious metal artifacts and jewelry for post-mortem inventory. Therefore, this work aims to evaluate fingerprint precious metals in jewelry and semi-jewelry for post-mortem inventory by EDXRF.

2. Methodology

Two lot (AM and CR) jewelry artifacts were identified, including rings, earrings (Figure 1), pendants, and neck chains.



Figure 1: Lot AM artifacts considered precious jewels.

The individual artifacts were inserted into the support for analysis with special care (cotton insertion to protect the pieces) (Figure 2).



Figure 2: Sample AM-14 upper face (a) and lower face (b).

Data acquisition was carried out using a Shimadzu Co. Energy Dispersive X-ray Fluorescence Spectrometer (EDXRF-model 720), cooled with liquid N₂ and 1-10 mm collimator, configured with X-ray tube Rh (250W), Si (Li) semiconductor detector, instrumental conditions: voltage 15-50 kV, self-adjusting current of max. 1 mA, air atmosphere. For each sample, 2,048 points were measured at energies ranging from 0.00 to 40.96 keV with a 0.02 keV interval. The exposure time samples in the chamber were 20 s, and the "dead time" detector (DT) was 40%.

The layers' thicknesses are calculated after determining the heaviest elements using software coupled to the EDXRF system.

2.1. Interpreting data

The data are interpreted by identifying the elements using the intensity/count rate (cps μA⁻¹) obtained by the fundamental parameter (FP) algorithm (Eq 1).

$$I_L = I_0 \omega_A g_L \frac{r_{A-1} \frac{d\Omega}{4\pi} \mu_M(\lambda_{prim}) \cos \sec \varphi}{r_A \mu_M(\lambda_{prim}) \cos \sec \varphi + \mu_M(\lambda_L) \cos \sec \psi} C_A \mu_A(\lambda_{prim}) \cos \sec \varphi \quad (1)$$

where:

IL - the intensity of element A; I₀ - the intensity of the primary beam in dispersive energy λ_{prim}; λ_{prim} - wavelength the primary X-ray beam; λ_L - line energy of element A; ω_A - fluorescence yield element A; g - value the fraction in line L of the element in its series; r_A - ratio

absorption edge element A; dΩ/4π - value fluorescent X-ray beam directed at the detector; C_A - concentration element A; μ_A (λ_{prim}) - mass absorption coefficient of element A for λ_{prim}; μ_M (λ_{prim}) - mass absorption coefficient the matrix for λ_{prim}; μ_M (λ_L) - mass absorption coefficient the matrix for λ_L; φ - incidence angle the primary beam; ψ - exit angle the fluorescent beam [28].

The jewelry samples were evaluated by determining the carat (Eq 2).

$$Carat (K) = \frac{Au (\%)}{\frac{100}{24}} \quad (2)$$

where:

Au (%) - concentration gold in the sample; 24 - 100% concentration Au in the Certified Reference Material [29].

3. Results and Discussion

Each sample has a very heterogeneous composition, as they are made from different alloys. This allowed us to calculate the concentration (%) composition of each piece of gold, whose Cu, Zn, Ni, Au, Ag, and Sn contents are shown in Table 1. We can see the variety in the composition of alloys used in the jewelry evaluated, ranging from high-purity gold alloys (86.8% Au + 2.5% Ag + 9.9% Cu) to copper-rich gold alloys (30.4% Au + 65.5% Cu + 1.9% Zn + 2.2% Ni). Cu was identified in all the samples, with concentrations varying from 7.7 to 65.5%, because when it is added to metal alloys, there is a considered increase in various physical-chemical parameters such as mechanical strength, anti-corrosion, malleability, and ductility [10], [30].

The elements spectra are shown in Figure 3 of the samples evaluated as jewelry; Au 18 K, compared to Au 24 K reference standard (CRM).

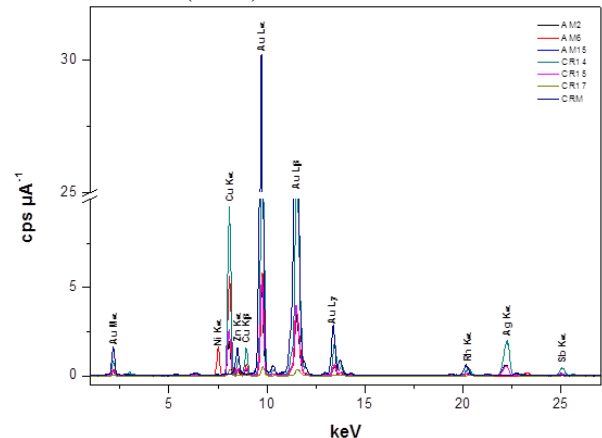


Figure 3: Elements spectra in the samples evaluated in jewelry (18 K).

Twenty-two jewelry compositions were added to the ternary diagram in Figure 4, containing Au 30.4-86.8% and Cu 7.7-65.5% about K.

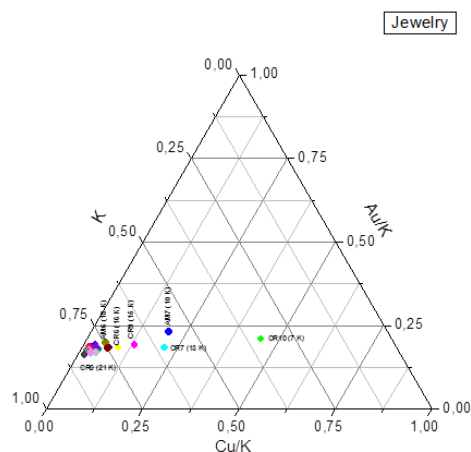


Figure 4: Fingerprint the jewelry about a carat.

The composition of gold alloys indicates low Cu/Au ratios (0.1-2.2), which correspond to malleability and ductility [10], [30]. The ratio Au/K and Cu/K concentrations show the fingerprint of the jewelry about the carat. The higher-carat jewel has a more significant shift to the left in Figure 4. For example, sample CR0 (21 K) is to the left, and CR10 (7 K) is more to the right. Gold fingerprints on jewelry are crucial for post-mortem inventory, criminal forensics, and jewelry theft.

The pieces considered semi-jewelry varied in the substrate used to give. The substrates identified in this work were alpaca alloys (Table 2), stainless steel, brass, and indeterminate (Table 3), and a layer Au used in electrochemical/electroplating processes. The Au electrodeposition layers showed different layer thicknesses, ranging from 0.2 to 966.5 nm, for the substrates evaluated. This variation in Au thickness may be due to wear and tear or the quality of the semi-jewelry.

The alpaca alloy used in producing semi-jewelry is made up of Cu, Zn, and Ni, which has ductility and resistance to oxidation [4], [24]. In this study, a wide range of Cu, Zn, and Ni content was in the samples evaluated (Table 2). The percentages can vary according to the manufacturer, and the most common contents are 65% Cu, 23% Zn, and 12% Ni [4], [24].

The semi-jewelry made with brass substrate had a chemical composition of Cu ranging from 99.0 to 79.2% and Zn from 0.9 to 11.9% (Table 3). Brass is an alloy of Cu and Zn, with up to 36% Zn used in producing semi-jewelry and ornamental objects, among others [4].

Stainless steel is used in semi-jewelry as an alternative substrate for enhancing ornamental pieces with an Au or Cu layer. There was slight variation in Ni and Cr in pieces identified with stainless steel substrate, while Fe varied from 57 to 76%.

In the other pieces, there was variation in element concentration, and we could not precisely identify the alloy substrate, and they were considered indeterminate.

4. Conclusion

This study can be considered the first to use EDRFX to analyze jewelry and semi-jewelry for post-mortem inventory. Quantitative analysis of metal alloys in jewelry and semi-jewelry can reveal exciting aspects of the manufacturing process. Based on practical knowledge, non-destructive methodology and matrices properties with different metal concentrations samples considered jewelry with different carats can be evaluated. As with any quantitative chemical analysis experiment, non-destructive reiteration is of fundamental importance for developing and testing hypotheses and acquiring and collecting knowledge through manipulation matrices. The analyses in this work serve as a reference for confirming, modifying, or suggesting financial hypotheses for post-death inventories.

Author contributions

Conceptualization, M.C.T.-Z. and M.A.S.; methodology, M.A.S.; validation, M.C.T.-Z. and M.A.S.; formal analysis, M.A.S.; investigation, M.A.S. resources, M.C.t.-Z.; data analysis, M.C.T.-Z. and M.A.S.; writing - original draft preparation, M.C.T.-Z. and M.A.S.; writing-review and editing, M.C.T.-Z.; supervision, M.E.B.C.

Conflicts of interest

The authors declare that they are unaware of any financial interests or personal relationships that could influence the work reported in this document.

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Table 1: Metal concentration in the samples considered jewelry.

Sample	Cu (%)	Zn (%)	Ni (%)	Au (%)	Ag (%)	Sn (%)	Evaluation (K)
AM2	9.8	-	-	75.8	14.4	-	18
AM3	28.1	-	-	71.1	0.3	-	17
AM6	19.1	-	6.2	74.7	-	-	18
AM7	35.7	4.3	1.7	40.9	-	-	10
AM10	11.6	-	16.9	71.5	-	-	17
AM15	11.3	-	-	76.8	11.9	-	18
AM17	16.7	-	-	83.0	-	-	20
CR0	9.9	-	-	86.8	2.5	0.8	21
CR1	17.1	1.6	-	81.4	-	-	20
CR2	9.9	-	-	79.1	8.7	2.3	19
CR3	26.5	2.8	-	70.0	0.5	0.3	17
CR6	33.8	1.4	-	64.8	-	-	16
CR7	46.4	5.2	8.8	39.7	-	-	10
CR8	19.1	5.4	-	67.7	-	7.8	16
CR9	38.4	6.5	-	55.1	-	-	13
CR10	65.5	1.9	2.2	30.4	-	-	7
CR11	20.5	0.5	-	77.5	-	1.6	19
CR12	32.0	3.5	-	61.8	2.7	-	15
CR13	8.2	0.4	-	78.1	13.2	-	19
CR14	13.6	-	-	73.7	12.7	-	18
CR15	15.3	3.5	4.6	76.7	-	-	18
CR16	16.1	0.5	-	83.1	0.3	-	20
CR17	7.7	-	-	75.1	17.3	-	18

- Undetermined.

Table 2: Semi-jewelry with Alpaca substrate.

Sample	Cu (%)	Zn (%)	Ni (%)	Au (%)	Thickness Au (nm)
AM4	76.2	18.7	5.1	24.2	848.8
AM5	76.1	16.6	7.3	13.5	343.4
AM8	71.0	26.4	2.6	10.7	277.9
AM12	65.7	6.4	27.8	27.6	966.5
AM16	80.8	10.6	8.6	12.2	716.6
AM18	60.4	11.7	27.9	5.3	237.1
AM20	74.5	9.8	15.7	9.6	248.8
AM24	83.1	1.2	11.6	4.0	5.9
AM27	57.8	5.3	27.8	8.9	20.2
AM28	58.1	5.4	27.6	8.9	20.4
AM29	43.9	20.3	33.1	2.6	52.0
AM30	51.9	26.0	19.8	2.3	50.7
AM33	89.7	2.2	6.4	1.1	35.8
AM35	86.8	4.0	0.02	9.2	13.5
AM36	89.6	6.0	0.1	4.3	55.2

Table 3: Semi-jewelry with stainless steel, brass, and indeterminate substrates.

Sample	Substrate	Cu (%)	Zn (%)	Ni (%)	Fe (%)	Cr (%)	Mn (%)	Au (%)	Thickness Au (nm) Layer 1	Thickness Cu (nm) Layer 2
AM25	Stainless steel	0.3	-	8.3	73.6	15.6	2.1	-	1.1	11.4
AM26	Stainless steel	0.3	-	8.4	74.5	16.4	-	-	-	1.1
CR5	Stainless steel	14.9	-	4.2	57.3	15.1	-	8.5	1.7	4.5
AM1	Brass	95.1	1.7	-	-	-	-	3.1	17.0	-
AM19	Brass	97.5	1.8	-	-	-	-	0.7	12.3	-
AM21	Brass	99.0	0.9	-	-	-	-	0.1	0.3	-
AM22	Brass	79.2	11.9	5.5	-	-	-	3.3	7.5	*58.8
AM23	Brass	98.1	1.9	-	-	-	-	-	0.2	-
AM9	Indeterminate	52.7	-	45.7	-	-	-	1.6	34.8	-
AM13	Indeterminate	89.2	-	2.6	-	-	-	8.2	20.4	-
AM14	Indeterminate	100	-	-	-	-	-	-	-	-
AM31	Indeterminate	-	-	98.3	0.5	0.1	0.1	-	188.8	-
AM32	Indeterminate	59.7	-	37.3	2.7	-	0.3	**0.1	**83.9	9,435.3
AM37	Indeterminate	0.6	-	-	-	0.01	-	3.8	24.6	-
AM38	Indeterminate	0.8	-	99.1	-	-	-	-	3.4	-

- Undetermined; * Ni (%); **Ag (%).

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