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Analysis of ISO 5832-1 Stainless Steel Alloys by Nuclear Techniques

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1. Introduction

The biomaterials market is experiencing a particularly sharp growth, mainly due to factors such as global aging of the population, higher purchasing power, increased living standard, and technological progress improvements in treating diseases previously viewed as untreatable [1]. This increase in life expectancy among the population drives up the demand for these devices, in order to mitigate the impacts of accidents and age-related injuries [2].

The improvement of biomedical devices has enhanced the quality of life for millions of individuals [3]. The austenitic stainless steel produced according to the ISO 5832-1 standard is widely used to meet the high demand for applications in orthopedic prostheses in Brazil. The advantages of using the ISO 5832-1 stainless steel alloy include its biocompatibility, low cost, good mechanical performance, and corrosion resistance [2,4-5] compared to titanium alloys and of Cr-Co [6]. Stainless steels are frequently used as biomaterials since they exhibit high corrosion resistance due to their elevated levels of chromium, molybdenum, and nitrogen [4].

Chemical analyses of biomaterials play a crucial role in ensuring fair treatment between producers and consumers in both raw materials and finished products. Furthermore, these analyses are indispensable for quality control in its production, as well as for evaluating performance and ensuring the reliability of materials in various applications [7]. Consequently, biomaterial analysis holds significance not only in the realm of healthcare but also for the industries responsible for their production.

Several analytical techniques are applied in elemental analyses of metallic alloys, such as atomic absorption spectrometry (AAS) [8], inductively coupled plasma atomic emission spectrometry (ICP-AES) [9], wavelength dispersive X-ray fluorescence spectrometry (WD XRFS) [10], UV-Visible spectrophotometry [11] and neutron activation analysis (NAA) [10-14].

The objective of this study was to evaluate the performance of wavelength dispersive X-ray fluorescence spectrometry (WD-XRFS) and neutron activation analysis (NAA) in the analysis of ISO 5832-1 stainless steel. The use of these two techniques was based on their advantageous capability to conduct multi-element analysis across a broad spectrum of element mass fractions without requiring sample dissolution.

Neutron activation analysis (NAA) provides more accurate results for both major and trace elements compared to those obtained by atomic absorption spectroscopy (AAS) [15]. Alternatively, The WD XRFS is also regarded as a suitable analytical instrument for determining elemental compositions because of its robust standardless quantification capability [16].

2. Methodology

A sample of ISO 5832-1 austenitic stainless steel was purchased in the form of a bar from Villares Metals S/A for analysis by NAA and XRFS. Sample preparation for NAA of ISO 5832-1 stainless steel as well as results of quality control in the NAA are in a previous publication [17]. In short, this preparation consisted of obtaining the alloy in small fragments followed by cleaning and drying for analysis.

For NAA, about 50 mg of this sample were weighed in a polyethylene envelope for irradiation. The synthetic standard of the elements was prepared for NAA analysis by the comparative method as described by Braguin et al. [17]. Epithermal neutron activation analysis (ENAA) was utilized to determine the silicon (Si) content by measuring ^{29}Al, formed through the nuclear reaction ^{29}Si (n,p) ^{29}Al. For silicon determination, 20 mg of metallic silicon in powder form with 99.9999% purity was used as a standard. For this determination ^{29}Al with half live of 6.56 min and gamma ray energy of 273.36 keV was measured.

The analyses were carried out at the Neutron Activation Analysis Laboratory of the Research Reactor Center (CERPq) IPEN-CNEN/SP by irradiation at the IEA-R1 nuclear reactor. The ENAA process involved irradiating a ISO 5832-1 alloy along with a standard, both placed in polyethylene envelopes, for short irradiation period of 60 seconds. The sample along with the Si standard, was placed in a cadmium capsule and exposed to the neutron flux of a nuclear reactor, which comprised of thermal and epithermal neutrons, with flux rates of approximately 1.9×10^{12} and 5.43×10^{10} n^{-} cm-$2s^{-1}$, respectively.

Sample preparation for stainless steel by WD XRFS was the same applied for sample used in corrosion test described by Braguin et al. [18] The elemental analysis of ISO 5832-1 by WD XRFS was performed using spectrometer model RIX 3000 of Rigaku Co. of the Laboratório de Espectrometria de Fluorescencia de Raios X, at IPEN

3. Results and Discussion

Table I presents mass fractions of elements in the ISO 5832-1 stainless steel alloy quantified by NAA and WD XRFS and specification values.

As can be seen in Table I, by WD XRFS, Co and V, that did not present its specification values in reference {19} were quantified. By comparing results obtained by WD XRFS and NAA, good agreement was obtained for the elements Co, Cr, Cu, Mn, and Mo. Using NAA, it was possible to determine As, Fe, and W which were not quantified by WD XRFS. On the other hand, using WD XRFS, it was possible to determine P and Si. It should be noted that the P mass fraction is slightly higher, in contrast with Si within the alloy specification.

In the analysis of the ISO 5832-1 stainless steel, the elements Cr, Cu, Mn, Mo, and Ni were quantified and their obtained mass fractions indicated that they are within the ranges of values of ABNT NBR ISO 5832-1. For the elements As, Co, Fe, V and W, its specification values are not presented.

Table I: Mass fractions of elements obtained for ISO 5832-1 alloy by WD XRFS and NAA and specification values.

Elements	WD XRFS	NAA	Specification values
	F ± U	$\underline{X}$ ± SD	[19]
As, µg g^{-1}	-	15.0 ± 1.5	A
Co, µg g^{-1}	300 ± 10	213.8 ± 4.4	A
Cr, %	16.9 ± 0.2	17.06 ± 0.61	17.0 – 19.0
Cu, %	0.06 ± 0.01	0.0427 ± 0.0025	0.5 max
Fe, %	-	62.6 ± 2.1	A
Mn, %	1.9 ± 0.1	1.764 ± 0.025	2.0 max
Mo, %	2.7 ± 0.1	2.49 ± 0.33	2.25 – 3.00
Ni, %	15.5 ± 0.1	13.3 ± 1.3	13.0 – 15.0
P, %	0.026 ± 0.003	-	0.02 max
Si, %	0.30 ± 0.02	-	1.0 max
V, %	0.06 ± 0.01	0.03522 ± 0.00074	A
W, µg g^{-1}	-	110 ± 15	A

F= mass fraction of the element; U: uncertainty calculated for a 95% confidence level (i.e. coverage factor k = 2); $\underline{X}$= arithmetic mean of the mass fractions, SD= standard deviation, - = indicates that the element was not determined, A= indicates that the value is not presented in reference {19}.

Using XD XRF, mass fractions of Co, Cr, Cu, Mn, Mo, Ni, P, Si e V in ISO 5832-1 alloy. Cr, Cu, Mn, Mo, and Si mass fractions obtained are within the specification [19]. Ni and P mass fractions obtained by WD XRFS are slightly above the specification. As it has been mentioned, Ni quantification is of great interest, because it is toxic for humans and may cause mutagenic, cytotoxic [1], carcinogenic [20] effects and other allergies [21].

4. Conclusions

By chemical characterization of elements in ISO 5832-1 stainless steel sample by NAA and WD-XRFS it was possible to conclude that both techniques allowed the determination of several elements of interest for the researchers concerning its application as biomaterials. These techniques due to its elemental character can be applied in the quantification of main elements as well the impurities present in low mass fractions.

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References

[1] A. L. R. Pires, A. C. K. Bierhalz, A. M. Moraes, “Biomateriais: tipos, aplicações e mercado,” *Química Nova*, vol. 38, pp. 957–971 (2015).
[2] R. Risco-Alfonso, R. Pérez-Rodríguez, P. C. Z. Robledo, M. R. Santana, R. Quiza, “Optimization of the cutting regime in the turning of the AISI 316L steel for biomedical purposes based on the initial progression of tool wear,” *Metals*, vol. 11, pp. 1–15 (2021).
[3] A. Nouri, C. Wen, *Stainless steels in orthopedics*, Woodhead, Cambridge & United Kingdom (2021).
[4] J. A. Disegi, L. Eschbach, “Stainless steel in bone surgery,” *Injury*, vol. 31, pp. S-D2-6 (2000).
[5] R. F. Reis, R. L. Villanova, K. D. Costa, G. C. Durante, “Nitrogen surface enrichment of austenitic stainless steel ISO 5832-1: SHTPN vs Low-temperature plasma nitriding,” *Materials Research*, vol. 18, pp. 575–580 (2015).

[6] M. M. Dewidar, H. Yoon, J. K. Lim, "Mechanical properties of metals for biomedical applications using powder metallurgy process: a review," *Metals and Materials International*, vol. 12, pp. 193-206 (2006).
[7] G. A. Uriano, C. C. Gravatt, G. H. Morrison, "The role of reference materials and reference methods in chemical analysis," *Critical Reviews in Analytical Chemistry*, vol. 6, pp. 361-412 (2008).
[8] T. Sipola, T. Alatarvas, T. Fabritius, P. Peramaki, "Determination of alloying and impurity elements from matrix and inclusions from a process sample of a double stabilized stainless steel.," *ISIJ International*, vol. 56, pp. 1445-1451 (2016).
[9] C. Yonga, "ICP-AES determination of 15 kind of impurity elements in the vanadium-aluminum alloy," *Procedia Engineering*, vol. 24, pp. 447-453 (2011).
[10] C. A. J. Silva, L. N. M. Braguin, M. A. Scapin, I. Costa, M. Saiki, "Analyses of magnesium-based alloys by nuclear techniques," *Brazilian Journal of Radiation Sciences*, vol. 10, pp. 1-12 (2022).
[11] R. Acharya, S. Kolay, A. V. R. Reddy, "Determination of nickel in finished product alloys by instrumental neutron activation analysis and spectrophotometry," *Journal of Radioanalytical and Nuclear Chemistry*, vol. 297, pp. 309-313 (2012).
[12] M. Saiki, S. O. Rogero, I. Costa, O. V. Correa, O. Z. Higa, "Characterization of ear-piercing studs and their corrosion products by neutron activation analysis," *Journal of Radioanalytical and Nuclear Chemistry*, vol. 248, pp. 133-136 (2001).
[13] E. Cincu, I. Manea, V. Manu, D. Barbos, O. Sima, I. Gustavsson, P. Vermaercke, N. Vajda, Z. Molnar, H. Polowska-Motreko, "Comparative performance of INAA and other spectroscopy techniques in the elemental analysis of stainless steel materials," *Journal of Radioanalytical and Nuclear Chemistry*, vol. 274, pp. 199-205 (2007).
[14] A. D. Shinde, R. Acharya, A. V. R. Reddy, "Analysis of zirconium oxides by relative and internal monostandard neutron activation analysis methods," *Nuclear Engineering and Technology*, vol. 49, pp. 562-568 (2017).
[15] E. Cincu, L. Cracuyn, I. Maenea-Grigore, I. L. Cazan, V. Manu, D. Barbos, A. Cocis, "Application of the INAA technique for elemental analysis of metallic biomaterials used in dentistry," *Applied Radiation and Isotopes*, vol. 67, pp. 2133-2136 (2009).
[16] "Alloy Characterization using Wavelength Dispersive X-Ray Fluorescence Spectroscopy," https://www.eag.com/app-note/alloy-characterization-using-wavelength-dispersive-x-ray-fluorescence-spectroscopy/ .
[17] L. N. M. Braguin, C. A. Justino, I. Costa, M. Saiki, "Elemental determination of austenitic stainless steel alloy used as biomaterial by neutron activation analysis," *Brazilian Journal of Radiation Sciences*, vol. 9, pp. 1-16 (2021).
[18] L. N. M. Braguin, C. A. J. Silva, L. Berbel, B. V. G. Viveiros, J. Rossi, I. Costa, M. Saiki, "A study on corrosion resistance of ISO 5832-1 austenitic stainless steel used as orthopedic implant," *International Journal of Advances in Medical Biotechnology*, vol. 3, pp. 23-28 (2020).
[19] ABNT -Associação Brasileira de Normas Técnicas. Implantes para cirurgia –Materiais metálicos –Parte 1: Aço inoxidável conformado.ABNT NBR ISO 5832-1: 2017, Brazil (2017).
[20] I. A. R. C. International Agency for Research on Cancer, "IARC monographs on the evaluation of carcinogenic risks to human," *International Agency for Research on Cancer*, vol. 49, pp. 1-687 (1990).
[21] L. S. Morais, G. S. Guimarães, C. N. Elias, "Liberação de íons por biomateriais metálicos.," *Brazilian Revista Dental Press Ortodon Facial*, vol. 12, pp. 48-53 (2007).