

Ni, Cr, Co Detected in Some Materials Tested According to European Standards

LAURA-CRISTINA RUSU*

² Victor Babes University of Medicine and Pharmacy of Timisoara, 2 Piata Eftimie Murgu, 300041, Timisoara, Romania

This study was designed to determine the composition of three representatives of the class of materials widely used in dentistry through the cation analysis method in spectrometry by atomic absorption. We performed two measurements: the cations extraction in Aqua Regia and cation extraction in artificial sweat. Our purpose is to analyze the nominal composition in Ni, Cr, Co of the materials used in these tests according to ISO standards[7].

Keywords: materials, cations, Aqua Regia, artificial sweat

It is interesting to have investigated the impact of dental materials through their components upon the immune system both in patients and medical staff.

This article aims to identify the chemical composition of three dental summarized in Ni, Cr, Co, being already known the allergenic potential of these components.

We studied representatives of the main material classes used in dentistry, being tested *in vitro*. Thus, we worked with: dental alloys, of which the most used in the clinic are alloys of Ni-Cr and Co-Cr: **M1** Ni-Cr alloy for crowns, **M2** Cr-Co alloy for framed prostheses and **M3** self-adhesive resin-cement for fixation.

Data from the literature show that concerns for cation analysis[2, 4-6] method in spectrometry through atomic absorption were there too, but in this article, we compare the results of extraction in Aqua Regia and artificial sweat, reporting the combined results of the two methods to the European rules for the admitted concentration of these materials that come into contact with the human body. European standard for Ni provides a maximum concentration of this element accepted by the European Union as harmless to humans[1, 5, 7].

Experimental part

Materials and methods

Field trials were conducted on 3 different materials: **M1** Ni-Cr alloy for crowns; **M2** Cr-Co alloy for framed prostheses and **M3** self-adhesive resin-cement for fixation.

The experiment was a randomized complete block design. To determine the chemical composition of the dental materials considered, we used first the cation extraction method in Aqua Regia. The method [6] is based on measurement by atomic absorption spectrometry of an element concentration in a sample extract in Aqua Regia prepared according to ISO 11466. Reagents used are[6]: hydrochloric acid, 37%, 1.18 g/mL, nitric acid 65%, 1.42 g/mL, nitric acid diluted 1+3 (V/V), sulphuric acid, 98%, 1.84 g/mL, sulphuric acid diluted 1+9 (V/V), lanthanum chloride solution 37 g/L lanthanum, calibration blank solution without lanthanum, calibration blank solution with lanthanum, acetone [19].

Sample preparation is done according to ISO 11464 "Pre-treatment of samples for physico-chemical analysis": Extraction of trace elements (microelements) according

Material	Chemical composition (% m/m)
M1#	Ni 64,5; Cr 22,0; Mo 10,0; Si 2,1; Nb <1,0; Mn <1,0; B <1,0
M3#	BIS-GMA 1565-922, UDMA 74389-53-0, TEGDMA 109-10-6, HEMA 868-77-9, Amorphous Silice 69012-64-2, additives in variable amount, BiOCl 7787-59-9, variable system for curing, Ca-Al-F-silicate variable.
M2#	Co63.0; Cr30.0; Mo5.0; Si1.1; Mn0.5; C 0.4.

Table 1
CHEMICAL COMPOSITION OF THE
STUDIED MATERIALS INDICATED BY THE
SUPPLIER

Sample		Ni µg/L	Cr µg/L	Co µg/L
Metal Alloys	1 M2	19.3	3.86	0.76
	2 M2	19.92	4.12	0.95
	3 M2	18.56	3.75	0.91
	4 M1	24.92	4.35	0.87
	5 M1	26.23	4.63	0.72
	6 M1	24.67	4.71	0.74
Self-adhesive resin cement	1 M3	14.85	3.47	0.63
	2 M3	13.45	3.67	0.51
	3 M3	14.78	2.97	0.34
	4 M3	13.41	3.68	0.37

Table 2
EXTRACTION RESULTS IN AQUA
REGIA

* email: laura.rusu@umft.ro

Sample		Ni µg/L	Cr µg/L	Co µg/L
Metal alloys	1 M2	18.5	3.46	0.56
	2 M2	19.7	3.72	0.45
	3 M2	18.0	3.03	0.49
	4 M1	24.7	4.05	0.65
	5 M1	25.8	4.16	0.58
	6 M1	24.1	4.23	0.44
Self-adhesive resin cement	1 M3	14.6	3.01	0.31
	2 M3	13.8	2.89	0.35
	3 M3	14.0	2.91	0.29
	4 M3	12.9	2.97	0.31

Table 3
ANALYSIS RESULTS FOR ALL 10
SAMPLES

	No. samples	Average	Standard deviation	Average standard error
Ni	3	18.7333	0.87369	0.50442
Cr	3	3.4033	0.34847	0.20119
Co	3	0.5000	0.05568	0.03215

Table 4
STATISTICAL STUDY OF THE
VALUES OBTAINED FOR Ni-Cr
ALLOY

to ISO 11466:1999 "Extraction of soluble trace elements in Aqua Regia"[18].

The second phase of the study is the cations extraction in artificial sweat.

Its purpose is to know the quantities of cations (Ni, Cr, Co) in artificial biological media (artificial sweat).

For the samples tested, the difference toward the extraction in Aqua Regia is that the extraction was done this time in artificial sweat (5/g NaCl, 1g/L urea 940 microliter lactic acid) at 37°C for 4 days, 1 mL volume of sweat per cm² of sample. We worked with 3 samples from each alloy and 4 samples of resin-cement, round, flat, with a diameter of 5 mm. We obtained the concentration in Cr and Ni in micrograms per liter. The final result will be in microgram/ liter and cm².

	No. samples	Average	Standard deviation	Average standard error
Ni	3	24.8667	0.86217	0.49777
Cr	3	4.1467	0.09074	0.05239
Co	3	0.5567	0.10693	0.06173

Table 5
STATISTICAL STUDY OF THE
VALUES OBTAINED FOR Cr-Co
ALLOY

	No. samples	Average	Standard deviation	Average standard error
Ni	4	13.8250	0.70415	0.35208
Cr	4	2.9450	0.05508	0.02754
Co	4	0.3150	0.02517	0.01258

TABLE 6
STATISTICAL STUDY OF THE
VALUES OBTAINED FOR RESIN-
CEMENT

	Ni µg/L	µg/cm ² /week	Cr µg/L	µg/cm ² /week	Co µg/L	µg/cm ² /week
1 M4	18.5	0.28	3.46	0.053	0.56	0.009
2 M4	19.7	0.30	3.72	0.057	0.45	0.007
3 M4	18	0.28	3.03	0.046	0.49	0.007
4 M1	24.7	0.38	4.05	0.062	0.65	0.010
5 M1	25.8	0.39	4.16	0.064	0.58	0.009
6 M1	24.1	0.37	4.23	0.065	0.44	0.007
1 M3	14.6	0.22	3.01	0.046	0.31	0.005
2 M3	13.8	0.21	2.89	0.044	0.35	0.005
3 M3	14	0.21	2.91	0.044	0.29	0.004
4 M3	12.9	0.20	2.97	0.045	0.31	0.005

Table 7
ALLERGENS VALUES EXPRESSED
IN µg/cm²/WEEK

Results and discussions

The compositions declared by the manufacturer for the three materials is shown in table 1. This composition will be compared with our results.

The above table 3 comprises the results of all samples expressed in micrograms/liter, being shown for each of the two alloys and resin-cement.

We transformed the results obtained through extraction from mg/L in µg/cm²/week. In the case of Ni, the European law requires that the expressing of disposed quantities of Ni to be done in µg/cm²/week; in contact with skin is limited to 0.5 µg/cm²/week, while in piercing to 0.2µg/cm²/week. We obtained the following values:

- comparing the results obtained through Aqua Regia extraction with the results indicated by the provider for M3, we found Ni, Cr, Co in relatively small quantities;

- regarding the analysis in artificial sweat, the results can be correlated with the data in literature [8-10, 12] that incriminate the ions of Ni, Cr, Co as allergens [13-15]. The obtained values do not exceed the limits set by the European Directives of nickel.

Regarding Cr and Co we have no information about a limit at level of European legislation [11, 16, 17], therefore we consider the limits of nickel, meaning that the values we obtained are relatively small.

Conclusions

Cations concentration with allergenic potential (Ni, Cr, Co) in the three tested dental materials does not reach the maximum value admitted by the European law (0.5µg/cm²/week). Regarding the material M1, the results approach this upper limit.

The chemical composition indicated by the manufacturer for M2 does not contain Ni. M2. After the tests have been done, we detected traces of Ni in the composition of this material.

With no European standard for Cr and Co, we compared to the Ni. The transferred cation amounts are of the same order.

The test results in artificial saliva are significantly lower than those in Aqua Regia. We tend to compare them to those in the artificial saliva because it is about the materials used in dentistry.

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