Determination of inorganic contaminants in polyamide textiles used for manufacturing sport T-shirts

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ABSTRACT

An acid microwave closed vessel digestion method was used for the determination of inorganic contaminants (Sb, As, Pb, Cd, Cr, Co, Cu, Ni and Hg) in polyamide raw materials (pellets) and textiles by inductively coupled plasma optical emission spectrometry (ICP OES). The initial tests were carried out with samples of polyamide pellets, which is the main raw material used to manufacture sport textiles. The recovery factors obtained were 94.4–105.7% with relative standard deviation (RSD) of 0.5–2.2%. The proposed method was evaluated by addition and recovery tests and also using certified reference materials (ERM-BCR680 and ERM-BCR681) showing good accuracy. The residual acidity was about 4% HNO_3 (w/w) and the quantification limits were from 0.1 to 6.6 mg kg^{-1}. After the development of these parameters for the raw material, the method was applied to textile samples from different sport fabrics obtained from three different brands. The residual carbon after sample digestion was 0.2% (w/w) and the most significant result was obtained for chromium, 901 mg kg^{-1}, in black fabric. Lixiviation tests using synthetic sweat and temperature were carried out on two black samples, showing that only 0.3% of the initial concentration migrated to the solution.

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1. Introduction

Textile fibers can be divided into two groups: natural and synthetic fibers. The main natural fibers are wool, silk, cotton and flax. The most common synthetic fibers are viscose, cellulose acetate, polyester, polyamide and polyacrylic [1,2].

Polyamides represent linear polymers resulting from a polycondensation reaction of amino acids, lactams or dicarboxylic acids with diamines and are called nylon when coming from linear, unsubstituted aliphatic monomers [3].

There is interest in trace inorganic constituent levels in both natural and synthetic fibers and fabrics, but mainly in the synthetic ones because they may contain residues of catalysts, treatments or stabilizing agents [4]. The determination of the contaminant content is very important not only for the safety of consumers, but also for the textile industry, because the presence of certain elements may cause manufacturing problems and less quality in the dying process. The use of dyes with chromium on nylon is essential for the fast black dyeing and is generally used. Copper (applied to enhance the light fastness), antimony (improves wash fastness) nickel and cobalt are also present in commonly used dyes, but the last one is found in selected applications. Mercury, cadmium and arsenic are found sometimes as impurities from intermediates [5].

Considering the interaction of the textiles with the human skin, due to daily contact with clothes and bed linen, the chemical substance contents in these materials can cause allergic and toxic effects that represent a health hazard to users [5,6], such as the allergic skin reaction (dermatitis) caused by Ni [7], for instance. Other elements come from the wet processing in the textile industry, such as cobalt, chromium, copper and nickel that are part of the dyes. Additionally, cationic dyes contain zinc, as well as trace levels of mercury, cadmium and arsenic, which may also cause damage [8]. The presence of chromium is a big problem because Cr(VI) is extremely toxic and can lead to liver damage, pulmonary congestion, skin irritation and carcinogenesis [9]. The fast black dyeing on wool and nylon is carried out using a chromium based dye [6], making the determination of this element very important. Some commercially available metal complex dyes containing chromium are C.I. Acid Orange 173 and C.I. Acid Black 60 [10]. The most widely used dye in the world is the chromium dye "C.I. Mordant Black 11" (commercial name) [6].

In sports, clothes are usually exposed to perspiration which may cause metal extraction from textiles and absorption to skin [2,6]. In some of them, like marathons, the sweaty athlete remains in contact with the textile for several hours and often under the sun, causing a temperature elevation that may raise the possibility of contamination.

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An important material for manufacturing sport clothes is the polyamide fibers and there are some papers that show metal determination in granules of this polymer. Resano et al. described the determination of silicon and manganese by means of solid sampling electrothermal vaporization inductively coupled plasma mass spectrometry (ETV-ICP-MS), comparing the results with inductively coupled plasma optical emission spectrometry (ICP OES) determination in ashes after alkaline fusion with $Na_2B_4O_7$ [11]. De Schrijver et al. developed a method for aluminum, silicon and manganese determination by graphite furnace atomic absorption spectrometry (GFAAS) and ICP-MS after a dissolution procedure using formic acid and compared the results with solid sampling approaches [12].

Regarding total metal determination in textile materials Menezes et al. [7] evaluated the mineral profile in Brazilian and Chinese textiles after microwave assisted digestion using ICP OES and thermospray flame furnace atomic absorption spectrometry (TS-FF-AAS). Rezic et al. [13,14] described an analytical procedure applied to cotton, flax, hemp and historical textile threads using ICP OES. Some workers [15,16] have applied microwave digestion with metal determination by FAAS, GFAAS and ICP-MS and others [6–8,17] have applied lixiviation and ultrasonic extraction to determine the extractable concentrations.

The widespread use of nanoparticles such as Ag, Cu and Zn [18–22] in commercial products, especially textiles, due to their antimicrobial, antifungal and antiviral properties, has pointed to an important line of research in the determination of these elements in textiles, liberated by lixiviation due to sweat or by washing.

In Europe different textile ecological standards are used for monitoring contaminants present in textiles: Markenzeichen achadstoffgeprüfter Textilien, Oeko-Tex Standard, Clean fashion, Steilman and Ecomarc Scheme [5,8,13]. The Oeko-Tex Standard 100 [23] label was applied to protect consumers and to limit the use of some chemical substances that are dangerous to human safety [6]. According to this requirement there are limits to heavy metals in digested samples (Pb and Cd) and in extractable ones (Sb, As, Pb, Cd, Cr, Co, Cu, Ni and Hg) [24].

The aim of this work was the development of an analytical method to determine total inorganic contaminants (Sb, As, Pb, Cd, Cr, Co, Cu, Ni and Hg) in polyamide (raw material and textile used in sport T-shirts) by inductively coupled plasma optical emission spectrometry. A lixiviation test using synthetic sweat solution was carried out for the most important contaminant element found.

2. Experimental

2.1. Instrumentation

All the measurements were made with a Varian 720 ES (axial view) inductively coupled plasma optical emission spectrometer (ICP OES), equipped with a solid-state charge-coupled device detector (CCD), a peristaltic pump and a "Seaspray" nebulizer coupled to a cyclonic nebulization chamber. A CCI (Cooled Cone Interface) was set with an argon flow rate of $3 \, L \, min^{-1}$. The conditions of operation (optimized as previously described [30]) of the spectrometer are presented in Table 1.

For treatment of the samples a microwave oven (Milestone ETHOS TC), equipped with pressure sensor (50 bar) and temperature sensor was used with TFM bottles with maximum temperature of 260 °C and pressure of 100 bar.

The determination of the elements in the sample of residual solid (obtained from white textile) was performed using a scanning electron microscope coupled with an energy dispersive X-ray spectrometer Jeol JSM-5800LV. The Dubnoff thermostatic bath used during the migration test was from Quimis and the potentiometric

Table 1
Instrument operating conditions (Varian ES-720).

Radio frequency power (kW)	1.2
Plasma gas flow rate ($L \, min^{-1}$)	15
Auxiliary argon flow rate ($L \, min^{-1}$)	1.5
Nebulizer pressure (kPa)	210[a]
Replicate read time (s)	5
Pump rate (rpm)	15[b]
Replicates	3
Sample uptake delay (s)	40
Pump rate (rpm)	15
Rinse time (s)	10
Replicates	3
Sampler	SPS3
Chamber	Cyclonic
Nebulizer	Seaspray
Torch	Highsolids (injector tube 2.4 mm)

[a] This instrument is not fitted with a mass flow controller, the setting nebulizer is set by pressure. A pressure of 210 kPa is about $0.73 \, mL \, min^{-1}$.
[b] The sample uptake rate is set by adjusting the peristaltic pump speed.

titrator used in acidity tests was a Metrohm 682 with combined glass electrode (Metrohm 6.0233.100) using a titrant – solution of $0.1 \, mol \, L^{-1}$ NaOH.

2.2. Reagents and solutions

For measurements by ICP OES 99.996% argon was used (White Martins). All glassware was cleaned with 10% (v/v) nitric acid solution and deionized water (Milli-Q System, 18.2 MΩ cm). It were also used 65% (w/w) nitric acid, 37% (w/w) hydrochloric acid and 30% (w/w) hydrogen peroxide solution, all from Merck. The stock ICP metal solutions ($1000 \, mg \, L^{-1}$) were from Quimis and Teclab. All other reagents used were p.a. grade.

2.3. Treatment of samples

Sample treatment was carried out according to an earlier work [25] using microwave assisted digestion (12 min–120 °C–600 W; 5 min–180 °C–900 W; 20 min–220 °C–900 W). Initially samples of polyamide pellets (obtained directed from producer factory), which are the main raw material used to manufacture sport textiles, were tested. The samples were enriched with the studied element (0.10 mg) and its recovery was evaluated after the preparation procedure. The analyte addition was made from standards of each element ($100 \, mg \, kg^{-1}$, in aqueous solution) directly into the microwave digestor bottle.

After obtaining quantitative recovery factors for the raw material, the method was applied to sport fabrics from three different brands, as described in Table 2.

The samples were treated as follows: about 0.25 g of polyamide pellets or fabric samples were directly weighed into the microwave digestor bottle. Then 3 mL of water and 5 mL of nitric acid were added for the first experiments. Later the volumes were optimized

Table 2
Samples of sport fabrics.

Identification	Description
Sample 1	White T-shirt – brand 1
Sample 2	Dark blue fabric – brand 2
Sample 3	Wine fabric – brand 2
Sample 4	Beige fabric – brand 2
Sample 5	Black fabric – brand 2
Sample 6	Light blue fabric – brand 2
Sample 7	Gray fabric – brand 2
Sample 8	Black fabric – brand 1
Sample 9	Black fabric – new sample – brand 2
Sample 10	Black fabric type 1 – brand 3
Sample 11	Black fabric type 2 – brand 3

to 2 mL of HNO_3 and 6 mL of deionized water. After finishing the evolution of gases the microwave digestion was initiated. The final solution was diluted to 25 mL in a volumetric flask, after filtration through black filter paper. A blank with the same amount of reagents, without the addition of the sample, was also prepared.

The recovery was evaluated by adding a standard solution of analytes to be investigated (0.1 mg each) directly into the bottle of microwave oven. The calibration curve was prepared in HNO_3, initially 10% (w/v) and later 5% (w/v), at concentrations of 0, 0.5, 1, 2 and 5 mg kg^{-1}.

2.4. Determination of residual acidity

The residual acidity of polyamide pellets obtained after sample preparation (2 mL HNO_3 + 6 mL deionized water) was measured by potentiometric titration and the result was expressed as total acidity in nitric acid.

2.5. Determination of residual carbon content

After the optimization of sample preparation for polyamide pellets the procedure was applied to textiles and the residual carbon content was determined by ICP OES using a potassium biphthalate calibration curve with the following standard concentrations: 50, 100, 300, 500, 800 and 1000 mg kg^{-1}.

2.6. Migration test

After analyzing the total element content in all samples, it was verified that only black fabric presented high concentration level of chromium. Considering this fact migration test of this element in this kind of sample (black fabric) was carried out.

In order to verify the migration of chromium, the fabrics were immersed in artificial sweat solutions for 24 h, at 37 °C, under shaking using a Dubnoff thermostatic bath. The samples (0.2 g) were cut and placed into contact with 10 mL of synthetic sweat (ratio 1:50, w/v) in plastic tubes. For this experiment, two samples of black fabric (Sample 8: black fabric brand 1 and Sample 9: black fabric – new sample – brand 2) and three different synthetic sweat solutions were used.

The artificial sweat solutions were prepared according to the International Standard Organization (ISO105-E04-2008E), the American Association of Textile Chemists and Colorists (AATCC Test Method 15-2002) and the British Standard (BS EN1811-1999), as described below [22]:

- *AATCC solution*: 0.025 g L-histidine, 1 g NaCl, 0.25 g $Na_2HPO_4 \cdot 12H_2O$ and 0.1 g 85% lactic acid were dissolved in 100 mL of deionized water. 0.1 mol L^{-1} NaOH was used to adjust the solution to pH 4.3.
- *Solution ISO105*: 0.05 g of L-histidine, 0.5 g NaCl and 0.5 g $Na_2HPO_4 \cdot 12H_2O$ dissolved in 100 mL of deionized water was adjusted to pH 8.0 with 0.1 mol L^{-1} NaOH.
- *EN solution*: 1.08 g NaCl, 0.12 g 85% lactic acid and 0.13 g of urea, dissolved in 100 mL of deionized water, adjusted to pH 6.5 with 0.1 mol L^{-1} NaOH.

After 24 h, the solutions were filtered through black label paper and transferred to a 25 mL volumetric flask. The volume was completed with deionized water.

The calibration curve was prepared in 0.4% (w/v) NaCl at concentrations of 0, 0.1, 0.2 and 0.5 mg kg^{-1} of chromium.

Table 3
Recovery test for polyamide pellets ($n = 3$).

Elements	Recovery (%)	RSD (%)
Cd 226.502	103.5	1.8
Cd 228.802	105.5	2.2
Cd 214.439	104.8	1.8
As 188.980	104.2	1.7
As 193.696	104.2	1.5
Sb 217.582	102.8	1.7
Sb 206.834	104.6	1.4
Cr 267.716	103.1	1.2
Cr 205.560	103.1	1.6
Cr 206.158	102.3	1.2
Cu 324.754	94.5	0.8
Cu 327.395	94.4	0.5
Cu 213.598	96.0	1.6
Ni 231.604	103.9	1.5
Ni 230.299	104.8	1.8
Ni 216.555	105.7	1.5
Pb 220.353	102.9	1.2
Pb 182.143	104.0	1.4
Pb 217.000	102.9	1.7
Hg 184.887	101.2	1.2
Hg 194.164	101.4	1.6
Hg 253.652	101.3	0.6
Co 230.786	103.5	1.5
Co 237.863	103.6	1.3
Co 228.615	104.4	1.1
Co 238.892	104.1	1.4

3. Results

3.1. Microwave digestion and addition and recovery tests

Polyamide pellet samples were spiked with the analytes and then treated by the microwave program. After complete solubilization the elements Sb, As, Pb, Cd, Cr, Co, Cu, Ni e Hg were determined by ICP OES. The results for addition and recovery tests (spiked tests) were from 94.4 to 105.7% and the RSD was in the range 0.5–2.2%, as shown in Table 3.

3.2. Residual acidity

Acid presence, in matrices analyzed by ICP OES, may cause interference that Brenner and Zander [26] assigned to the following sources: the aerosol generation and transport system due to decrease solution uptake as a result of increased viscosity, changes in the primary and tertiary aerosol drop size distribution and changes in atomization and excitation conditions. Concentrations of HCl and HNO_3 up to 10% (w/w), when using axial viewing, may require internal standard to improve recoveries [26].

Sample solutions were prepared by polyamide pellet microwave digestion, mixing 0.25 g of sample, 2 mL of conc. HNO_3 and 6 mL of deionized water. The digested solution was diluted to a final volume of 25 mL. The residual acidity was determined by potentiometric titration with 0.1 mol L^{-1} NaOH. The results obtained for two solutions were 4.43% (w/w) and 4.22% (w/w), expressed in HNO_3, lower than 10% (w/w).

In a previous work (polymeric matrix, similar residual carbon content and acidity higher than 10%, w/w) [30] tests using internal standards were carried out. The results showed no significant difference (95% confidence level) in the analytical signals in the presence or absence of these standards.

3.3. Detection and quantification limits

The limits of detection (LOD) and quantification (LOQ) were calculated as three times and ten times the blank standard deviation, respectively [27,28]. The results showed that LOD were from

Table 4
Limits of detection and quantification in polyamide pellets (mg kg^{-1}).

Elements	Limit of detection (mg kg^{-1})	Limit of quantification (mg kg^{-1})	LOQ × DF (100) (mg kg^{-1})
Sb 217.582	0.009	0.029	2.9
Sb 206.834	0.006	0.020	2.0
As 188.980	0.003	0.010	1.0
As 193.696	0.007	0.024	2.4
Cd 228.802	0.001	0.003	0.3
Cd 214.439	0.000	0.001	0.1
Cd 226.502	0.000	0.001	0.1
Co 237.863	0.001	0.002	0.2
Co 230.786	0.001	0.002	0.2
Co 228.615	0.001	0.003	0.3
Co 238.892	0.000	0.002	0.2
Ni 216.555	0.004	0.012	1.2
Ni 230.299	0.004	0.014	1.4
Ni 231.604	0.004	0.012	1.2
Cr 205.560	0.001	0.004	0.4
Cr 267.716	0.001	0.004	0.4
Cr 206.158	0.002	0.007	0.7
Cu 213.598	0.005	0.017	1.7
Cu 324.754	0.004	0.014	1.4
Cu 327.395	0.005	0.016	1.6
Hg 253.652	0.013	0.044	4.4
Hg 184.887	0.019	0.063	6.3
Hg 194.164	0.020	0.066	6.6
Pb 182.143	0.006	0.021	2.1
Pb 217.000	0.008	0.028	2.8
Pb 220.353	0.001	0.004	0.4

LOQ × DF (dilution factor of 100) = 0.25 g diluted to 25 mL.

0.001 to 0.020 mg kg^{-1} and LOQ were from 0.001 to 0.066 mg kg^{-1}. Considering the dilution factor of 100 times (0.25 g diluted to 25 mL), the LOQ multiplied by this factor were in the range of 0.1–6.6 mg kg^{-1}. All the results are shown in Table 4.

3.4. Analysis of certified reference materials (CRM)

Although the addition and recovery tests (Table 3) showed quantitative recoveries, the method was also evaluated using two certified reference materials. Because of the lack of certified materials for polyamide, similar matrix (polymeric materials) was used.

It was carried out the analysis of two certified reference materials, ERM-BCR 680 and ERM-BCR 681, which are high and low density polymers, respectively. The materials analyzed are from the Institute for Reference Materials and Measurements – ERM (European Reference Materials) and the results are described in previous work [30].

The determination of As, Cd, Cr, Hg, Pb and S in certified reference materials of low density polyethylene showed recovery values in the range 79–107%, except for mercury in ERM-BCR 680, in which the concentration obtained was 4.64 mg kg^{-1}, a value close to the limit of quantification. Considering that Sb is present in the reference sample as Sb_2O_3, that is not soluble in nitric acid [25], determination of this element was not possible.

3.5. Industrial samples

After the initial experiments with the raw material (polyamide pellets), samples 1–7 were analyzed. Sample 1 (white T-shirt) was washed with soap powder, rinsed 3 times with distilled water and dried under sun light. This sample was washed because it is from a T-shirt bought directed from self store. Table 5 shows the results for these samples.

Only samples 1 and 2, "white shirt" and "dark blue fabric", presented a white residue after microwave digestion assisted (all the other samples were completely digested), which was filtered, dried and analyzed by SEM EDS (scanning electron microscopy coupled with energy dispersive X-ray spectroscopy) (Fig. 1) which shows that the main component is titanium oxide, a common substance used in fabrics, and some other minor elements, which are not part of the contaminants of interest in this work, as described by Oeko-Tex 100. Thus, the digested solution for these two samples was filtered and the measurements performed by ICP OES on the filtrate.

3.6. Determination of residual carbon content

The increase of carbon concentration could produce non-specific emission and deteriorate the power of detection due to the increase of background radiation (BEC) [29]. In order to evaluate if the digestion procedure is effective to degrade the organic content, until acceptable levels ([29] showed that until 10,000 mg L^{-1} the variations in BEC were not significant) the residual carbon content of the microwave digested solution of polyamide textile was determined by ICP OES using a calibration curve prepared with potassium biphthalate in 5% (w/v) nitric acid.

The measurements were carried out in duplicate and the highest value obtained was 0.2% (w/w), which is below the concentration of 10,000 mg L^{-1} and thus will not cause interference in BEC.

3.7. Determination of chromium in black fabrics

As the concentration of chromium obtained in the analysis of black fabric was two orders of magnitude higher than the other elements, new samples from the same company and from three other companies were analyzed. The results obtained are shown in Table 6. The international standards Oeko-Tex® Standard [23,24] for class II fabric (in direct contact with skin) does not set limits for total chromium content (after digestion) but the acceptable limit after extraction is 2 mg kg^{-1}. As the total concentration obtained was about 900 mg kg^{-1}, on average, a migration test was carried out, in order to verify how much of this element could be released from the fabric.

Table 5
Contaminants in fabric samples used in sports clothes (mg kg^{-1}).

Element and wavelength	Sample 1: white T-shirt	Sample 2: dark blue fabric	Sample 3: wine fabric	Sample 4: beige fabric	Sample 5: black fabric	Sample 6: light blue fabric	Sample 7: gray fabric	LOQ × DF (100)
Cd 214.439	0.2 ± 0.1	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.1
As 188.980	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	1.0
Cr 267.716	1.9 ± 0.1	2.3 ± 0.1	0.6 ± 0.1	1.0 ± 0.3	897 ± 12	1.2 ± 0.1	0.3 ± 0.1	0.4
Cr 205.560	1.9 ± 0.1	2.3 ± 0.1	0.6 ± 0.1	0.9 ± 0.3	901 ± 11	1.1 ± 0.1	0.3 ± 0.1	0.4
Cu 327.395	3.5 ± 0.1	4.0 ± 0.1	2.3 ± 0.4	2.2 ± 0.1	2.3 ± 0.2	2.8 ± 0.1	2.3 ± 0.2	1.6
Ni 231.604	3.3 ± 0.2	2.0 ± 0.2	1.1 ± 0.2	2.7 ± 1.2	1.5 ± 0.2	2.1 ± 0.7	0.9 ± 0.2	1.2
Ni 216.555	3.3 ± 0.2	2.1 ± 0.7	1.1 ± 0.1	2.7 ± 0.7	1.6 ± 0.3	1.9 ± 0.7	1.0 ± 0.2	1.2
Pb 220.353	1.4 ± 0.1	<LOQ	0.4 ± 0.1	0.4 ± 0.1	<LOQ	<LOQ	<LOQ	0.4
Hg 184.887	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	6.3
Hg 194.164	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	6.6
Co 238.892	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	0.2
Sb 206.834	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	2.0

<LOQ: lower than limit of quantification.
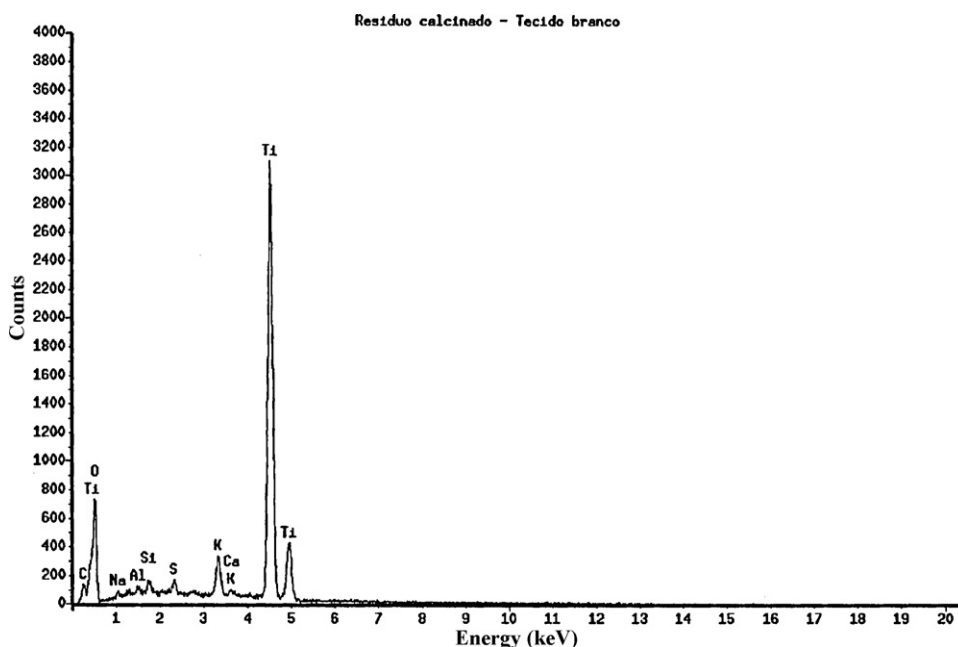
Fig. 1. SEM/EDS spectrum for the residue of samples 1 and 2.

3.8. Migration test

The chromium present in the polyamide may originate from the dye [1,2], which cannot be fully incorporated into the fiber, affecting mainly consumers who maintain close proximity between the fabric and the areas of skin subject to perspiration. Skin exposure to these substances can be a dangerous route of absorption, promoting dermatitis [2].

In order to verify if the chromium present in the black fabrics could migrate to the skin in contact with sweat and temperature, a migration test using synthetic sweat solutions was carried out. More than one solution was used because the composition of sweat varies not only between individuals but also in the same individual,

depending on body region, age, season, climate, diet, presence of infection and activity level [22]. The results are in Table 7.

Considering the average concentration of total chromium in samples 8 (926 mg kg^{-1}) and 9 (847 mg kg^{-1}), only about 0.08% and 0.3%, respectively, migrate to the solution. The migration was slightly more pronounced in basic solution (pH = 8.0).

Comparing the results in Table 7 with the limit of 2 mg kg^{-1}, set for the concentration of chromium after extraction by the Oeko-Tex® Standard [23], it seems that the sample of black fabric from brand 2 does not meet specifications in basic solution (ISO-pH = 8.0). The standard gives a limit for an acid extraction, which is made with the AATCC solution (pH 4.3). In this case, the average value obtained (1.7 mg kg^{-1}) is below the acceptable limit.

Table 6
Determination of chromium in black fabrics used in sport T-shirts.

Sample	Cr 205.560 (mg kg^{-1})	Cr 267.716 (mg kg^{-1})
Sample 8: black fabric – brand 1	928 ± 13	924 ± 12
Sample 9: black fabric – brand 2[a]	850 ± 6	844 ± 6
Sample10: black fabric – type 1 – brand 3	805 ± 10	798 ± 11
Sample 11: black fabric – type 2 – brand 3	965 ± 16	958 ± 15

[a] New sample from the same brand.

Table 7
Determination of chromium from black fabrics, after extraction with synthetic sweat.

Synthetic sweat solutions	Sample 8 black fabric – brand 1		Sample 9 black fabric – brand 2	
	Cr 205.560 (mg kg^{-1})	Cr 267.716 (mg kg^{-1})	Cr 205.560 (mg kg^{-1})	Cr 267.716 (mg kg^{-1})
AATCC – pH 4.3	<LOQ	<LOQ	1.6 ± 0.5	1.8 ± 0.5
ISO – pH 8.0	0.8 ± 0.1	0.5 ± 0.1	3.2 ± 0.1	2.9 ± 0.1
EN – pH 6.5	<LOQ	<LOQ	2.1 ± 0.1	2.2 ± 0.2

<LOQ: lower than limit of quantification 0.5 mg kg^{-1}.
LOQ $\times$ DF (125) = quantification limit (0.004) $\times$ dilution factor of 125 times (25/0.2).

4. Conclusions

The results for the application of the microwave assisted digestion method for the determination of inorganic contaminants (Sb, As, Pb, Cd, Cr, Co, Cu, Ni and Hg) in polyamide (raw material) showed accurate results (addition and recovery tests) with low residual acidity. The limits of quantification obtained, considering the dilution factor of 100 times, were in the range of 0.1–6.6 mg kg^{-1}.

This method was applied to polyamide textiles used in sport T-shirts and the major inorganic contaminant found was chromium in black fabrics. However, a migration test for this element, with synthetic sweat solutions, showed chromium transfer of 0.3% (maximum) when using a basic solution (pH 8.0). The extractable content using acid solution was lower than the limits values suggested by Oeko-Tex Standard 100.

Acknowledgements

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