

Study of metal release from stainless steels in simulated food contact by means of total reflection X-ray fluorescence

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1. Introduction

The release of metals from materials used in food contact is an interesting field of application for elemental chemical analysis of water based solutions. Metals and alloy based materials are used for food contact mainly in processing equipment, containers and household utensils. Very often they are covered with a surface coating, to reduce metal transfer in foodstuffs. Indeed, when they are not covered, leaching of metal ions into food may occur, with possible negative effects on human health if the total content exceeds the sanitary recommended exposure limits (CoE, 2002).

Stainless steels are widely used in food contact applications, such as cooking utensils and in cutlery, due to their high corrosion resistance and superior mechanical properties. Steel is an alloy of iron and carbon (less than 2% carbon), which may contain other elements such as Mn, Si, S, Ni, Cr, and Mo to modify or improve its properties. The most frequently used steel grades for food applications are the austenitic ones, such as AISI 304 and AISI 316, both

containing 16–20 wt% Cr and 8–14 wt% Ni. Alloying with nickel enhances the corrosion resistance leading to slower kinetics of metal release (Herting et al., 2008a; Wallinder et al., 2006).

Several papers about the release of alloy constituents into different food and food simulants have been published (Accominotti et al., 1998; Chiavari et al., 2014; Herting et al., 2009, 2008a; Jellesen et al., 2006; Kamerud et al., 2013; Kumar et al., 1994).

The Italian Ministerial Decree of March the 21st 1973 (Text, 1973) is one of the most detailed regulation text on global migration. According to this text the most relevant constituents of stainless steel, whose migration in food contact must be considered, were Cr and Ni. A more recent update of the Italian regulation have included Mn to the list of elements to verify its specific migration (Health, 2013).

Chromium is an essential element required for sugar and fat metabolism (Anderson, 1997). The estimated safe daily dietary intake of Cr(III) is 50–200 µg for humans, and there are no documented toxic effects in nutritional studies at levels up to 1 mg per day (Anderson, 1997). On the contrary, Cr(VI) is carcinogenic and the World Health Organization (WHO) has set its maximum value at 0.05 mg/L in drinking water (WHO, 2008).

Nickel is a fundamental component of stainless steels whose

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effects on human health are mainly related to allergic reactions (Jellesen et al., 2006). Food is the major source of Ni in the non-smoking and non-occupationally exposed population. The average daily intake of Ni is estimated about 0.2 mg/day. Stainless steel cooking utensils significantly contributed to the Ni content in cooked food that sometimes may exceed 1 mg/kg in meat. Water contribution is generally about 0.005–0.025 mg per day (WHO, 2007).

Manganese is another essential element widely distributed in the environment. Adverse health effects can be caused by inadequate Mn intake or over exposure (O'Neal and Zheng, 2015). Average daily intake of Mn in adults is about 0.7–10.9 mg from diet. The daily contribution of drinking water is usually lower, about 20 µg for an adult, assuming a daily water intake of 2 L (US-EPA, 2004). It is known that the exposure to high Mn levels via inhalation and ingestion may cause neurological problems (Borgese et al., 2011; US-EPA, 2004). For this reason, United States Environmental Protection Agency (US-EPA) has reduced the recommended limit to 0.050 mg/L (US-EPA, 2004).

Up to now there are no universal guidelines on the composition of stainless steels to be used in food contact applications. The Italian law provides a list of stainless steels allowed for food contact, without any further indication for the choice of material. However, several regulations report guidelines for release test on stainless steels in simulated food contact, and set the maximum levels of released metals, to avoid adverse effects on human health, at 0.1 mg/L for Cr, Ni (Text, 1973), and Mn (Health, 2013).

According to Italian text, 3% vol. acetic acid (pH 2.4) should be used in release tests to simulate the contact with foodstuffs. Two kinds of tests are reported, continuous and discontinuous contact, in different times and temperatures. A value about 0.5–2 is recommended for the ratio between the surface area of the material to be tested and the volume of the solution where it is immersed. Metal release is evaluated analyzing the concentration of Cr, Ni, and Mn in the contact solution at the end of the test (Text, 1973). However, guideline conditions are quite broad leading to poor reproducibility of results and impossibility to compare different materials.

Elemental analysis of release test solutions is usually performed by atomic absorption spectroscopy (AAS) (Herting et al., 2009, 2008a; Kumar et al., 1994), graphite furnace atomic absorption spectroscopy (GF-AAS) (Hedberg et al., 2011, 2013; Herting et al., 2007; Wallinder et al., 2006), inductively-coupled plasma mass spectroscopy (ICP-MS) (Herting et al., 2006, 2007; Pataro et al., 2014) or ICP optical emission spectroscopy (ICP-OES) (Hedberg and Midander, 2014). In AAS and ICP analyses the elements of interest are selected before the measurements. While, X-ray fluorescence (XRF) provides immediate and simultaneous multielemental analysis. In particular, total reflection X-ray fluorescence technique (TXRF) is a well established analytical technique for multielemental analysis of liquids, which are usually deposited on a reflective carrier and dried before measurements (Borgese et al., 2014). Very small volumes are required (5–50 µL) and detection limits in the range of µg/L may be achieved (Margui et al., 2013).

In this paper we present a well defined procedure to perform release tests in simulated food contact, according to the Italian law, which provides useful information for the selection of the best material to use. The comparison of results obtained for 6 stainless steels accepted for food contact is presented and the contribution of material composition is considered.

TXRF is used to analyze the composition of release test solutions. The comparison of results obtained in three different laboratories for the same sample are presented.

2. Materials and methods

2.1. Sample preparation

The following stainless steels, which are the most widely used for the manufacture of kitchen utensils, pots and pans and components of machinery for the food industry are considered: AISI 420 (similar to X30Cr13) – martensitic stainless steel; AISI 430 (similar to X6Cr17) – ferritic stainless steel; AISI 202 – austenitic stainless steel; AISI 303 (similar to X8CrNi18-9) – austenitic stainless steel; AISI 304 (similar to X5CrNi18-10) – austenitic stainless steel; AISI 316 (similar to X5CrNi17-12-2) – austenitic stainless steel.

Sample composition was measured according to the Standard Test Method for Analysis of Austenitic Stainless Steel (ASTM E1086 – 14) by Spark Atomic Emission Spectrometry (SPECTRO – SPECTRAMAX™ LMX06).

The Italian Law (Text, 1973) does not describe shape, size and coating of samples. They were defined in order to obtain the ratio between immersed sample surface (cm²) and solution volume (cm³) equal to 0.5 cm⁻¹ ± 0.1 cm⁻¹.

Disk-shaped samples (10 mm thick) for release tests were obtained cutting (Struers Discotom-100/-10) stainless steel samples annealed bars having diameter of 25 mm. To avoid overheating, cutting operations were performed with high coolant amount. Cylindrical disc surfaces were coated with 2.5 mm phenolic resin layer. Resin layers were applied with hot mounting press (Struers CitoPress-1) leaving uncoated upper and lower surfaces of the disk-shaped samples. Then flat surfaces were mechanically grinded and polished (Struers LaboPol-5 with variable speed from 50 to 500 rpm) using silicon carbide papers (120–2500 grit). In the end, the average surface roughness was about 0.5 µm. After mechanical polishing each disc was rinsed in distilled water, cleaned with ultrasonic unit (Elma Elmasonic One) and then air-dried. The experimental setup to perform release test is shown in Fig. 1.

2.2. Release tests

Glass containers were cleaned with 10% hydrochloric acid solution, rinsed with distilled water and then with 3% acetic acid solution. Containers were filled with 59.5 mL acid acetic solution. The solution amount was estimated in order to obtain a ratio between the geometrical area of the immersed sample (cm²) and the solution volume (cm³) equal to 0.5 cm⁻¹ ± 0.1 cm⁻¹.

Release tests were carried out at controlled temperature using a stainless steel tank with heating circulator (Julabo EH-39) with a heating capacity of 2 kW, pump capacity flow rate of 15 L/min and pump pressure of 0.035 MPa. The working temperature range was between 20 °C and 150 °C, with a temperature stability about 0.03 °C. Glass containers were equipped with cap to prevent solution evaporation. A digital thermometer (Thomas Scientific 1235C68) was used, with a temperature range from –50° to 260 °C and 1 °C accuracy.

Discontinuous contact tests were performed at 100 °C. Triplicate samples were inserted in test solutions and maintained at constant temperature for 30 min. Heating was performed by partial immersion of glass containers inside heating tanks. During heating, caps were put on glass containers and temperature was monitored with digital thermometers. After 30 min the solution was prelevated and a new amount of 59.5 mL of acetic acid was added. This was repeated three times for a total of 3 half hours of contact.

Continuous contact tests were performed at 40 °C for 10 days. Heating was performed by partial immersion of glass containers inside stainless steel tank with heating circulator filled with distilled water. During heating, caps were put on glass containers

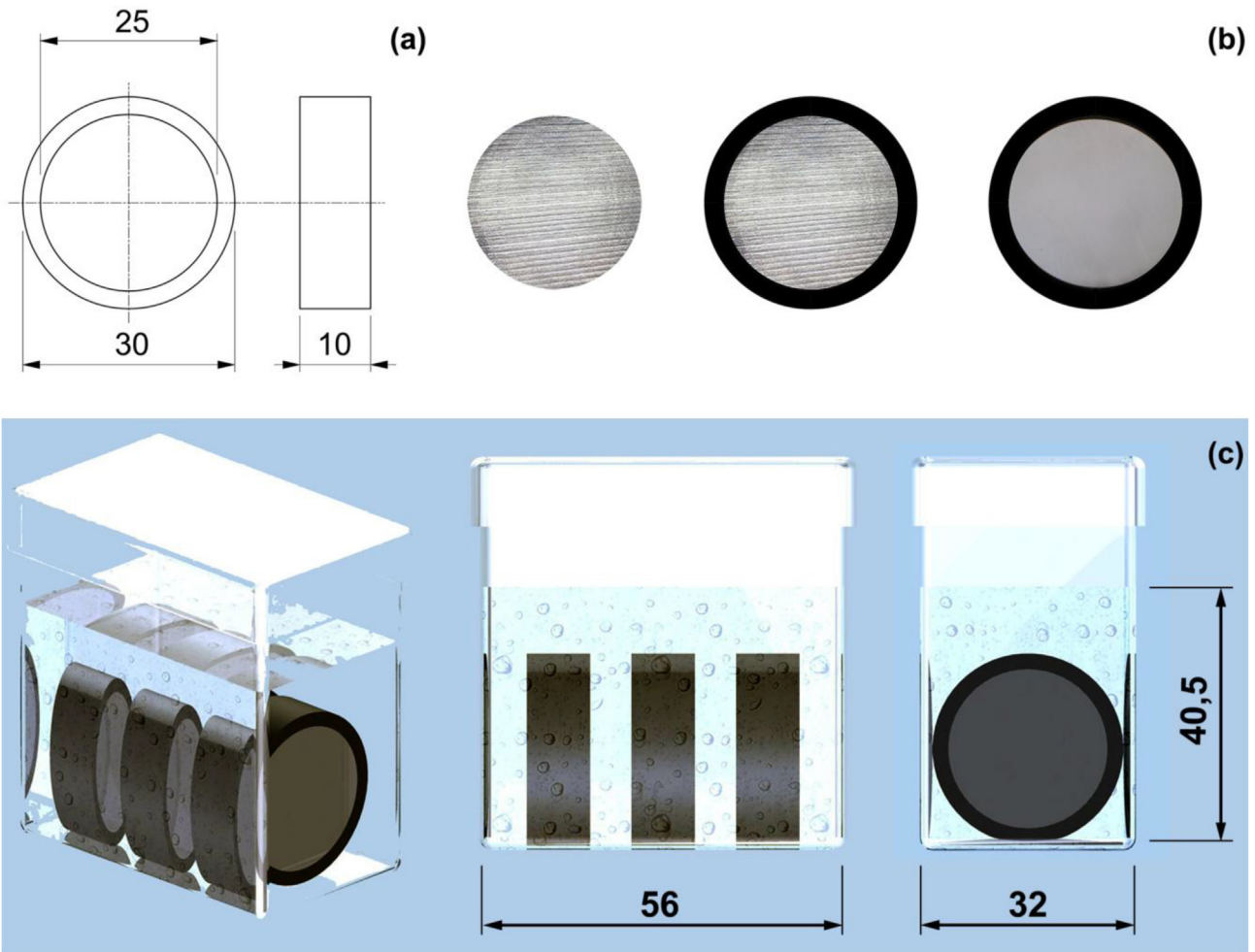


Fig. 1. Experimental setup for release tests. a) Disk-shaped sample (dimensions in mm), b) sample preparation, from left: sample cut, coating with phenolic resin and polishing, c) Graphical representation of the release test configuration (glass container dimensions in mm).

and temperature was monitored with digital thermometers. At the end of the test, solutions were removed.

2.3. TXRF analysis

Sample preparation for TXRF analysis was performed according to the protocol already described for water samples having

elemental concentration in the range 0.01–50 mg/L (Borgese et al., 2014). 1 mL of release tests solution was added with Ga, used as internal standard, to obtain a final concentration of 1 mg/L in the specimen. From each specimen, three independent trials were prepared by depositing 10 μ L of solution on the reflector resulting in a total number of 9 measurements, Fig. 2. The prepared specimens of AISI 304 sample were delivered to the three participant

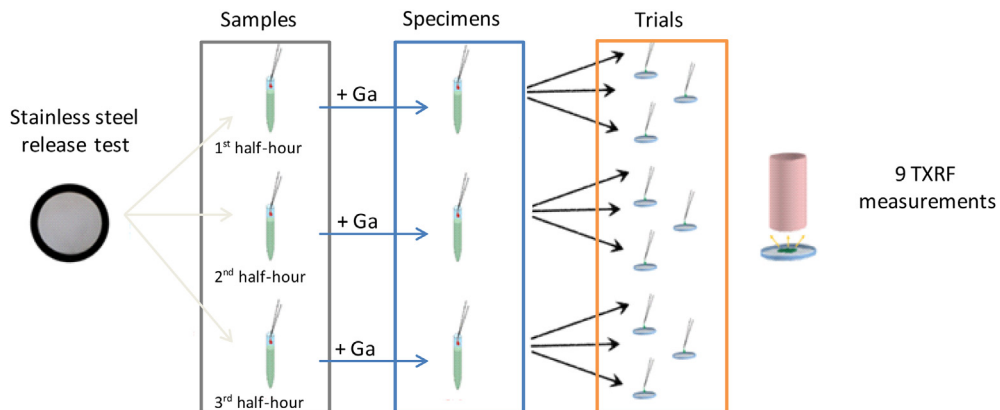


Fig. 2. Scheme of TXRF measurements.

Table 1
Nominal and actual chemical composition (wt%) of the six stainless steels, subjected to the release tests, measured according to ASTM E1086 – 14 by Spark Atomic Emission Spectrometry (SPECTRO – SPECTRAMAX™ LMX06) subjected to the release test.

	C	Mn	Si	P	S	Cr	Ni	Mo
AISI 420 (nominal)	0.26–0.35	≤1.5	≤1.0	≤0.040	≤0.030	12–14	–	–
AISI 420 (actual)	0.303	0.723	0.623	0.012	0.0141	12.415	0.327	0.053
AISI 430 (nominal)	≤0.08	≤1.0	≤1.0	≤0.040	≤0.030	16–18	–	–
AISI 430 (actual)	0.054	0.504	0.165	0.020	0.003	16.033	0.149	0.016
AISI 202 (nominal)	≤0.15	7.5–10	≤1.0	≤0.040	≤0.030	17–19	4–6	–
AISI 202 (actual)	0.044	8.212	0.396	0.021	0.0003	17.307	5.197	0.212
AISI 303 (nominal)	≤0.10	≤2.0	≤1.0	≤0.045	0.15–0.35	17–19	8–10	–
AISI 303 (actual)	0.053	1.806	0.349	0.019	0.200	17.107	8.442	0.338
AISI 304 (nominal)	≤0.07	≤2.0	≤1.0	≤0.045	≤0.030	17.5–19.5	8–10.5	–
AISI 304 (actual)	0.069	1.502	0.402	0.015	0.023	17.577	8.001	0.353
AISI 316 (nominal)	≤0.07	≤2.0	≤1.0	≤0.045	≤0.030	16.5–18.5	10–13	2–2.5
AISI 316 (actual)	0.020	1.464	0.361	0.015	0.0217	16.951	10.739	1.937

Table 2
Instrumental characteristics and setup of the three TXRF systems used.

TXRF instrumental characteristics	Laboratory A	Laboratory B	Laboratory C
Anode	Mo	Mo	Mo
X-ray tube	Air-cooled metal ceramic	Air cooled Micro-focus	Air-cooled
Maximum power	40 W	50 W	50 W
Optics	Multilayer monochromator (17.5 keV)	Curved multilayer monochromator (17.5 keV)	Multilayer monochromator
Detector	Silicon drift detector, Area: 30 mm ² , FWHM: 139.43 eV (Mn K α)	Silicon drift detector, Area: 30 mm ² , FWHM: 139.43 eV (Mn K α)	Silicon drift detector, Effective area: 7 mm ² , FWHM: 165 eV (Mn K α)
Filter	Mo 10.00 μ m	–	–
Sample changer	Manual version for single samples	Manual version for single samples	Automatic version for 16 sample turret
Atmosphere	Air	Air	Air
Voltage	50 kV	50 kV	50 kV
Current	750 μ A	750 μ A	600 μ A
Measuring time	600 s	500 s	600 s
Sample carrier	Plexiglass	Quartz	Soda lime glass

laboratories, named Lab A, B and C, to perform parallel TXRF measurements. Measurements were performed with three different TXRF instruments and experimental setups, reported in Table 2. For comparison, ICP-MS measurements and analysis were performed according to the EPA 200.8 method (Creed et al., 1994).

3. Results and discussion

3.1. “Stainless steel” sample composition

Nominal and measured chemical composition of the six stainless steels used in this study, are shown in Table 1. Values are in agreement with the standards set by AISI, even though the presence of Ni in AISI 420 and 430, and Mo in AISI 420, 430, 202, 303 and 304, was not expected.

3.2. Discontinuous and continuous contact release tests of all stainless steel grades

All the discontinuous contact tests solutions were analyzed by TXRF and the results obtained for Ni, Cr and Mn in the first, the second and third half-hour are reported in Fig. 3.

Fig. 3 clearly shows as the amount of metals transferred in solution is lowered from the first to the third half hour of release test, except the content of Ni in series 202. Such behavior is common for all the tested materials and this phenomenon for stainless steel was already observed (Herting et al., 2008a). This decrease is a consequence of the time-related formation of a more corrosion resistant surface film, characterized by gradual chromium enrichment during exposure in the stimulant (Chiavari et al., 2014; Herting et al., 2009, 2008a, 2008b). According to the Italian law only the concentration of the third half hour solution is relevant for comparison

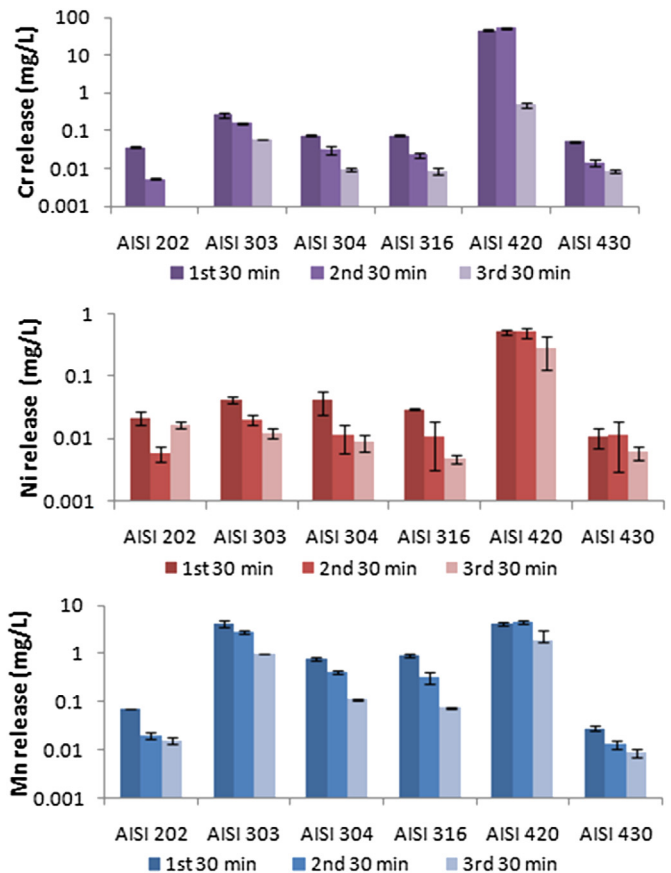


Fig. 3. Concentration (mg/L) of Cr, Ni and Mn at the end of the first, second and third half-hour of discontinuous contact release test. Error bars represent standard deviations.

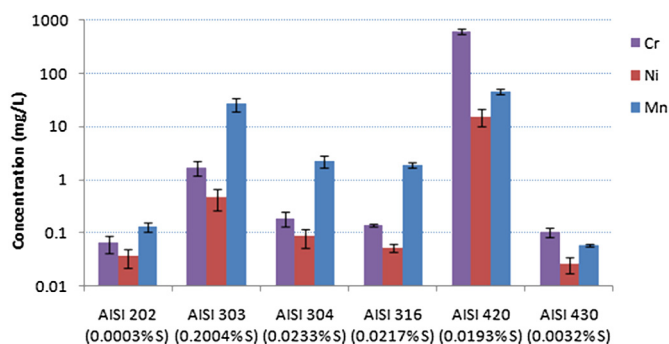


Fig. 4. Release of Cr, Ni and Mn after 10 days of continuous contact at 40 °C. Error bars represent standard deviations.

with the limit value of 0.1 mg/L for Mn, Cr and Ni.

AISI 420 releases the highest amount of Cr, Ni and Mn with respect to the other stainless steel samples. The lower Cr content and the negligible content of Ni and Mn make the passive film of chromium oxide: thinner, less stable and easily removed from weakly acid solutions. However recent analysis of the passive film made with X-ray photoelectron spectroscopy (XPS), highlighted the presence of Fe and Mo, in the form of oxides and hydroxides and showed that Ni and Mn tend to accumulate at the interface between the passive film and the steel substrate (Casaroli, 2014).

The austenitic stainless steel AISI 303 releases higher amount of Mn compared with AISI 420. This is probably due to the higher content of S, specially added to improve the workability of AISI 303. Sulfur may cause the formation of Mn sulphides which, are easily dissolved by the acetic solution. Sulfur has a strong influence on the release of Mn even when the steel is not re-sulfurized and its content is in agreement with the expected chemical composition of the nominal grade material.

The comparison of the third half hour composition highlights that AISI 202 and AISI 430 release less Mn than AISI 303, AISI 304 and AISI 316, even though the first two materials are nominally less resistant to corrosion. This is probably due to the extremely low S content of AISI 202 and 430. AISI 303 and AISI 420 exceed the limit required by the Italian law for Mn.

The role played by sulfur on the release of Mn is even more evident in the results of continuous contact tests (see Fig. 4). Indeed, in this case the release of Mn exceeds the limit of 0.1 mg/L for all the tested materials except AISI 430. These results suggest that sulfur, lowering the corrosion resistance of steel, is able to influence the release of Ni and Cr less than Mn. This may be due to a

significant amount of Mn in the outermost surface oxide of stainless steels (Chiavari et al., 2014; Hedberg et al., 2011; Hedberg and Midander, 2014; Hedberg et al., 2013; Herting et al., 2008b). It should also be considered that the continuous contact test solutions include the total amount of metal released.

3.3. Repeatability and reproducibility of TXRF measurements

A more detailed evaluation of TXRF measurements reproducibility and repeatability is performed with the solutions of AISI 304 release tests. Fig. 5 shows the raw spectra of three trials of Lab A highlighting the high repeatability of the raw measurements, as well as the presence of other potentially interesting elements: Fe and Zn.

The three considered instrumental settings are very similar (see Table 2). All TXRF systems are equipped with low power Mo X-ray tubes and they operate in the same energy range. Elements identification is performed by means of the same spectral lines. The main difference is the material used for sample carrier reflectors. Fig. 6 shows the results obtained by the three participant laboratories for Cr, Ni and Mn to evaluate the reproducibility and possible differences due to the experimental setup. The trend of results obtained by the three laboratories is clear and reproducible. All the data, obtained by TXRF and ICP-MS, fall in the 95% confidence range. It can be noted that relative standard deviations (RSD) are higher, and comparable, for Laboratory A and C with respect to Laboratory B. This can be due to the reflector material. Indeed, it is known that background is higher for plexiglass and soda lime glass with respect to quartz glass carriers (Klockenkämper and V. Bohlen, 2015).

To check if there are significant differences between the three laboratories performing TXRF analysis, for the first and second half hour, and the reference technique ICP-MS, for the third half hour, analysis of variance (ANOVA) was performed. Results of ANOVA show that significant differences ($P < 0.05$ and $F > F$ critical) occur for Ni in the second and third half hour. In both cases this can be due to the low Ni concentration, very near to the detection limits (see Table 3).

4. Conclusion

In this paper we propose a procedure to study metal release from stainless steel in simulated food contact. Among the six tested stainless steel samples, AISI 202 and 430 showed the lowest release of Mn, Cr and Ni even in continuous contact, not exceeding the maximum limit value of 0.1 mg/L. As it was expected, AISI 420

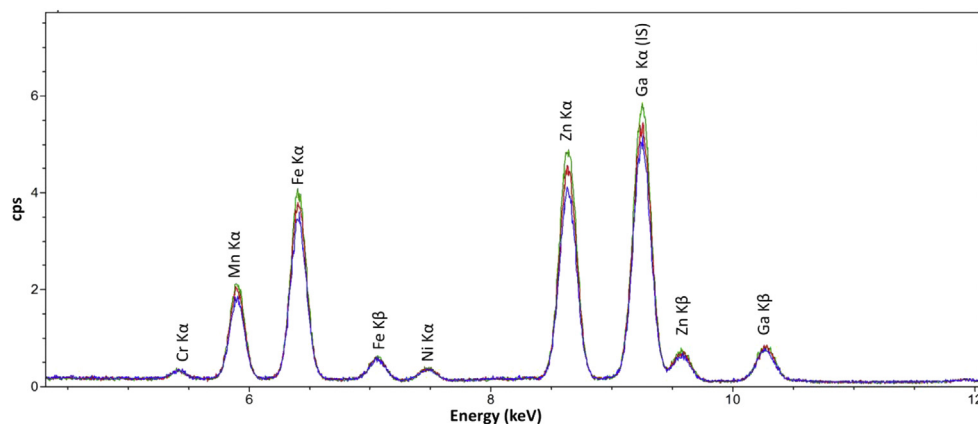


Fig. 5. Raw spectra of three measurements of the first half hour release test of AISI 304.

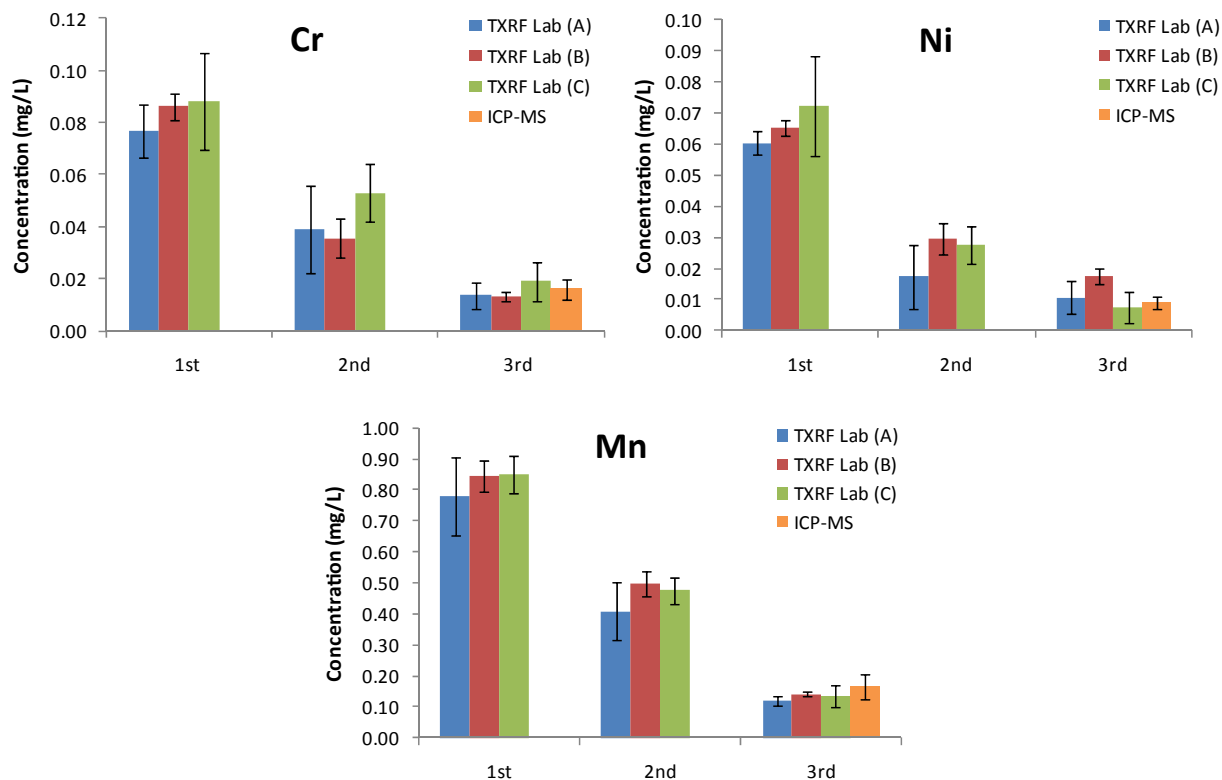


Fig. 6. Concentration values (mg/L) of Cr, Ni and Mn in the three half hours of contact measured by TXRF in laboratories A, B and C and third half hour measured also by ICP-MS. Error bars represent the 95% confidence range.

Table 3
Concentration range (mg/L) of Cr, Ni and Mn in three half hours of contact and maximum RSD (%) and average LOD (mg/L) values obtained by the involved laboratories.

Element	Concentration range (mg/L)	RSD (%)		LOD (mg/L)		RSD (%)		LOD (mg/L)	
		Laboratory A	Laboratory B	Laboratory A	Laboratory B	Laboratory C	Laboratory C		
Cr	0.014–0.105	19	13	0.008	0.002	21	0.011		
Ni	0.012–0.066	15	10	0.004	0.001	16	0.005		
Mn	0.129–0.894	7	4	0.007	0.002	10	0.010		

having low corrosion resistance (low Cr and Ni content), released the highest amount of Mn, Cr and Ni exceeding this limit. The presence of S has an important role in Mn release, and its presence in AISI 303 suggests the formation of Mn sulphides that are easily dissolved by acetic solution.

The optimized method, to perform release tests, allows selecting the most suitable material to be used in food contact. TXRF analysis is suitable to perform the analysis of release test solutions and compare the concentration values of Cr, Mn and Ni with the guidelines limits recommended by the Italian regulation, satisfying the requirements of this field and analysis.

Our results show that the proposed procedure for release tests followed by TXRF analysis is reliable and it may be useful to prescribe the preferential use of a material for food utensils.

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