Fluorinated Agents Effects on Orthodontic Alloys: An In Vitro Study

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Abstract

Background

In fixed orthodontics, fluoride-based mouthwashes and gels are effective preventive oral devices in counteracting demineralisations and dental caries onset but, modifying environmental acidity, they can reduce the resistance to wet corrosion of orthodontic alloys.

Methods

To evaluate the effective chemical stability of these products, a series of in vitro experiments were performed on thirty-two preformed rectangular orthodontic wires, (twenty-four in stainless steel and eight in nickel-titanium), cut in sections, obtaining ninety-six samples. They were weighed before and after immersion in five mouthwashes and two fluorinated dental gel, at three time-points, to measure weight variations and metal ions elution by acid corrosion phenomena. Elution samples, including control group of normal saline, were analysed by inductively coupled plasma mass spectrometry, to detect metal ions residual concentration.

Results

Minimal variations in loss from initial weight were recorded. Metallic ions release was quantified, at all time-points, for all wires and it was considered in correlation with alloys composition, pH and Fluor concentration of mouthwashes and gels, and time-exposition.

Conclusions

Acidic pH substances, like fluorinated agents and normal saline considered, cause metal ions dissolution from orthodontic wires. Dissolution depended by chemical nature of the elements, alloys composition, and Fluor concentration in mouthwashes and gels. This study helps in proposing timing and methods for the usage of fluorinated agents to guarantee the prophylactic action without damaging orthodontic alloys superficial structure.

Background

Although fixed orthodontic appliances are principally consisted of metals, materials considered non-cytotoxic in most conditions, during clinical use alterations may occur thus changing their chemical properties which can lead to the manifestation of adverse effects, including release of constituent elements, such as ions from alloys [1].

The biodegradation of metal orthodontic appliances is a well-known adverse phenomenon that is first and foremost the primary cause of local and systemic reactions of hypersensitivity, due precisely to the release of certain metal ions such as nickel (Ni) and chromium (Cr). It has been observed that the metal ions released can be incorporated into the hard dental structures, staining them or they can even cause the structural or functional
damage to the appliance. For this reason, a primary requirement of any metal alloy refers to its ability to avoid the production of corrosion products they can result harmful to the body [2].

However, it has been shown that there is a continuous reaction of orthodontic wires and brackets with the hostile environment of the oral cavity, in which metal components are continually released. It has been also demonstrated how alloy composition can influence the corrosion resistance phenomenon [3]. Indeed, several conditions like masticatory forces, orthodontic loads, fluctuations in temperature and pH contribute to trigger these corrosion phenomena. Additionally, the electrochemical processes play a crucial role in corrosion when there are two alloys and a medium such as electrolyte. Indeed, the alloy with lower corrosion resistance acts as an anode and dissolves in the electrolyte and the ions are then released. On the surface of orthodontic wires, electrochemical corrosion, or wet corrosion, can therefore occur, even in the presence of a fluid electrolyte such as saliva, and this determines the elution of metal ions or the formation of chemical compounds produced by electrochemical reactions to which orthodontic alloys have been submitted [4].

Orthodontic alloys used for the manufacture of arches must be highly reactive base metal, with a good biocompatibility and have a high resistance to corrosion. This latter property is ensured by the presence, in the metal mixture of chromium, a greatly reactive base metal that is able to spontaneously form a passive and protective surface film [5, 6].

Oxygen is necessary for forming and maintaining the film, while some microorganisms, such as *Bacteroides corrodens* and *Streptococcus mutans*, can also cause degradation of metal orthodontic alloys during long-term exposure [7].

During the fixed orthodontic treatment, in addition to periodontal implications [8] phenomena of demineralization of the dental enamel may frequently appear, such as white spots or even caries, for this reason, the regular use of oral products containing fluorides is essential since the fluoride ion, favouring the formation of calcium fluoride globules, can promote tooth remineralisation process. Oral care products containing fluoride have a variable concentration of fluoride ions (250 – 10,000 mg / L) with pH values ranging from 3.5 to 7. Fluoride-based gels with a low pH have proven to be more effective in increasing the formation of calcium fluoride (CaF$_2$) [9]. Some studies have investigated how fluorinated agents, depending on their acidity, are able to modify some of the main mechanical properties of orthodontic alloys. For instance, S Alavi, have evaluated the *in vitro* effects of fluoride solutions with different acidities on load-deflection characteristics of nickel-titanium (Ni-Ti) orthodontic wires, demonstrating that daily mouthwash with a fluoride solution with more acidic pH of 4 affected the Ni-Ti wires load-deflection characteristics during the unloading phase [10].

TH Lee verified in vitro that Ni-Ti wires have dissimilar corrosion resistance in an oral environment containing fluoride and this resistance does not correspond to the appreciated variations in the surface topography of the archwires. In particular, they have demonstrated that the presence of fluorine to 0.5% concentration of Sodium fluoride (NaF) in the artificial saliva is normally detrimental to the corrosion resistance offered from these alloys [11].

The fluoride ions are very aggressive on the protective film of titanium dioxide present on the Ni-Ti wires and can cause greater corrosion of the orthodontic arch. Furthermore, depending on the concentration, the fluoride ions are able to penetrate the narrow gaps between the orthodontic arch and orthodontic bracket by attacking the interface. This in turn leads to an increase in surface roughness as well as greater friction [12, 13].
A Jamilian, observed that the amount of ions released by Ni-Ti wires is always higher than that released by SS wires and, HH Huanga showed that the release of ions from orthodontic arches in Ni-Ti increases with decrease the pH of the immersion solution [14, 15].

The null hypothesis of the present *in vitro* study, has assumed that the corrosion phenomena that occur following the immersion of orthodontic alloys in fluoride solutions are no greater than those that happen using a normal saline solution as eluent. Established this, the aim of the study was to evaluate and quantify the release of metal ions from stainless steel (SS) and Ni-Ti orthodontic wires after their exposure, in vitro, to 5 topical mouthwashes and 2 professional gels based on different fluoride agents, Sodium fluoride (NaF) or Amine fluoride (AmF) with specific concentrations of fluoride and pH, at predetermined time intervals. In addition, the possible weight variations, through these exposure protocols, are recorded to evaluate a larger scale potential harmful corrosion phenomena.

**Methods**

Using a Distal End Cut plier in SS (Leone Spa, Sesto Fiorentino, Fl, Italy), 32 preformed rectangular orthodontic archwires, with the equal dimensions of 0.017×0.025 inch, have been manually sectioned by the same operator: 24 in SS and 8 in Ni-Ti. All wires were placed in acetone solution for two minutes for surface cleansing. A total of 96 arch sections (3 cm in length, each) have been obtained. At first \((t_0)\), sections have been weighed \((w_0)\) using a precision balance Mettler, Toledo XS204 scales (Cole-Parmer, IL, USA) with 0.1 mg accuracy [Table 1] and then placed into a sterile 15 ml Falcon tube.

Each tube has been filled with 10 ml of one of the topical fluoride mouthwashes and gels taken in exam [Table 2] in order to completely submerge every section.

Following this protocol, 96 specimens have been obtained and divided in three subgroups which, until the *in vitro* tests, have been stored at a controlled temperature of 37° C, for three different time storage intervals: 1 \((t_1)\), 24 \((t_2)\) and 168 \((t_3)\) hours.

A control group was added, whose arch sections were instead immersed in 10 ml of normal saline (H).

At the end of each established storage time, each section has been extracted from the test tube, immersed in deionized water for 30 minutes and then dried with a jet of pure nitrogen and weighed \((w_1, w_2, w_3)\) again.

Each eluate, residual from the immersion of the arch sections at predetermined time intervals, was analysed by Mass Spectrometry (ICP - MS of AGILENT Mod. 7800) in order to search for the possible presence of metal ions, such as Ni, Cr and Iron (Fe) resulting from the possible acid corrosion of surfaces exposed to the fluorinated agents contained in mouthwashes and gels.

The analysis has been carried out by diluting the liquid samples of mouthwash in acidified water, with ultrapure nitric acid, and then sonicated for 30 seconds. The gel samples, on the other hand, were weighed, mineralized in a microwave mineralizer with the addition of ultra-pure nitric acid and reported to a known volume.

Blank samples (Bs) of the 8 pure products, or reagents, analysed, have been also realized.
All samples, including Bs, have been subsequently analysed using an inductively coupled plasma mass spectrometer. This sensitive instrumental technique is able to determine the presence of various metallic and non-metallic inorganic substances, present in concentrations of about one part per billion (ppb).

**Statistical Analysis**

Univariate ANOVA served to assess the effect of mouthwashes and gels both on the weigh variations and the release of and Chromium, Iron and Nickel ions from the orthodontic wires taken in exam. Since the interaction of the variables was significant, one-way ANOVA and Tukey's HSD test were used to assess the effect of each variable.

**Results**

*Weight loss tests.* In Table 3 are shown the values, expressed in difference of weight percentage, of the final weighs ($w_1$, $w_2$, $w_3$) of all the specimens under examination. The specimens have been maintained in the specific solution and, at fixed times, they have been dried and weighted. The average values have been used as reference data for the calculations of the weight lost percentage at different times ($t_1$, $t_2$, $t_3$). Weight variations corresponding to the systematic error of the precision balance (± 50 ug) are not considered significant. Additionally, variations above or below 1% are not considered significant.

*Mass Spectrometry tests.* The collected data on the concentration of Cr, Fe and Ni ions present in the eluates, resituated from the immersion of arch sections in the different solutions at predetermined time intervals, are shown in Table 4.

**Discussion**

Fluoride-based mouthwashes and gels are very effective preventive oral devices to counteract the onset of dental caries and for this reason they have always been widely used in fixed orthodontics. On the other hand, it has also been observed that they are able to modify the environmental acidity, thus reducing the capacity of resistance to wet corrosion offered by pure titanium and its alloys due to the breaking of the protective layers of surface oxide and thereby corroding stainless steel dental appliance. It is known that the corrosion resistance of titanium is strongly dependent on the concentration and pH value of the fluoride contained in the various formulations for home and professional topical use [16].

Corrosion phenomena of orthodontic wires are able to release heavy metals into the oral cavity such as Cobalt, Cr, Ni and others ions [17].

In austenitic steels that contain Ni as a primary austenite stabilizer, Ni atoms are not strongly bonded to form an intermetallic compound so the likelihood of slow release of Ni ions, *in vivo*, from the alloy surface is higher, which can have biocompatibility consequences [18–21].

*In vivo,* it was demonstrated that Ni concentrations in saliva increased after the placement of SS bands and brackets but decreased to the starting levels two weeks after the placement of appliances. Subsequently, the placement of a Ni-Ti wire also determines an elongation of this effect which however decreases within 10 weeks [22].
Ni is the most common cause of metal-induced allergic contact dermatitis in humans and produces more allergic reactions than all other metals combined. Cr is second in frequency. In fact, extra oral adverse reactions are much more common than intraoral ones and allergic skin reactions are also difficult to distinguish from irritative lesions [23].

There is an evident abundance of evidence in scientific literature to support the fact that Ni has carcinogenic, mutagenic and cytotoxic actions in cell cultures. These findings should be interpreted with caution, because documented toxicities generally apply to the soluble forms of these elements. Currently, any association between the release of metal and metabolic, immunologic, or carcinogenic toxicity is conjectural because a cause and effect relationship has not been demonstrated in humans.

In an in vitro immersion study performed by T Eliades in 2004, ions released from both SS and Ni-Ti brackets and wires did not report any measurable effect on the vitality and physiology of the periodontal ligament and gingival fibroblasts [18].

However, studies have investigated on the release of metal ions from orthodontic alloys following contact, more or less prolonged, with fluorinated agents. In environments containing fluoride dissociated into ions, a reduction in corrosion resistance of pure titanium and titanium alloys has been demonstrated [24, 25].

Additionally, the exposure of Ni-Ti orthodontic wires to NaF-acidulated topical products causes a production of fluoridric acid, which quickly dissolves the titanium, resulting in the corrosion of the metal alloy [26].

In 2002, N Schiff et al compared the electrochemical properties of different titanium alloys with respect to fluoride ion content and salivary pH. Although Ni-Ti and Ni-Ti-Co were found to be less affected by corrosion phenomena, fluoride ions had negative effects on all materials [27]. Furthermore, in 2004 Schiff et al, measuring corrosion related to the use of fluorinated mouthwashes on titanium orthodontic alloys, observed that NiTi-based alloys are strongly corroded in the presence of monofluorophosphate, TMA wires are intensely corroded with Stannous Fluoride while, the TiNb archwires demonstrate the greatest resistance to corrosion. According to the authors, it is therefore necessary, based on the orthodontic alloy used, to advise the patient which is the most suitable fluorinated agent to perform oral rinses [9].

As regards weight loss tests performed in the present in vitro study, minimal changes were recorded, of the order of 1%, in defect or in excess for all the stain steel samples. We did not consider these variations significant, according to the specific measurement procedure. For the Ni-Ti arches, we observed higher weight loss in case of gels, especially after 1 week (t3) of immersion (samples F3 and G3) [Tab 3]. Considering these observations, all the orthodontic materials reveal a good chemical stability to corrosion. In fact, we can estimate the immersion time of 1 hour (t1) of the metal sections to be approximately considered as about one month of oral exposure of an orthodontic wire during a fixed orthodontic treatment, for an orthodontic patient, who constantly brushes his teeth with a fluoridated toothpaste 3 times a day, uses at least once a day an oral rinse with a fluoride mouthwash and, at least every 6 months, receives a topical application of professional fluorinated gel. Moreover, we observed significant weight loss only for prolonged immersion times (t3) in fluorinated gels, especially for Ni-Ti orthodontic arches.

These findings can be confirmed by mass spectroscopy tests, where consistent amount of metal ions (Ni) have been collected in the samples F3 and G3.
The tests carried out on the weight loss of orthodontic archwires exposed to fluorinated agents with different concentrations of fluorine have shown minimal and not significant weight variations. These changes, even if marginal, have affected the external layer of the wires and have negatively influenced the shiny appearance and the metallic luster of their surface. The SS AISI 302 Twist wire samples (2), among all, show the best dimensional stability for all fluorinated solutions at 1 (t₁) and 24 (t₂) hours, although they have the largest exposed surface since, it is made up of eight thin rectangular wires intertwined with each other. Samples in SS AISI 304 Extrahard (1) were found to be less resistant to corrosion than SS AISI 304 (4) wires, after immersion at 1 hour (t₁) and 24 (t₂) hours, despite the alloy with which they are both made has the same AISI classification, while at 1 week their behaviour looks similar.

Ni-Ti (3) samples, after an initial weight increase at 1 hour of immersion (t₁), present a certain stability even after a week of immersion (t₃) in AmF (0.025%) and NaF (0.02%) mouthwashes.

In all alloys, at different time points, after immersion in the fluorinated agents but also in saline solution, some slight increases in weight were recorded. This phenomenon is predictable and is linked to the cleaning phase of the sample previously exposed to fluorinated agent which is performed before the new weighing: the wire section is in fact immersed for 30 minutes in deionized water and then dried. This concurrent process, which can occur when these measurements are carried out, increases the initial weight also due to further deposits of salts from mouthwashes and gels or even of organic material from gels which, according to the nature and composition of the fluorinated solution (gels are more viscous), they can chemically adhere to the most superficial layer of the metal alloy section, effectively increasing its weight.

The results obtained by Mass Spectrometry tests [Tab.] show that in normal saline solution, at 1 hour (t₁) of immersion there is no release of Cr from the alloys examined, it is present in minimal quantities only in the residual eluate after 24 hours (t₂) of immersion of the SS AISI 304 (4) wires, but at 168 hours (t₃) Cr is released in minimal quantities (9.27 ug / kg) from all orthodontic archwires, in particular from SS AISI 302 Twist (2) wires. In normal saline solution, on the other hand, Fe ion is always present: from the first hour (t₁) of immersion it is released from all alloys (+ - 10 ug / kg), at 24 hours (t₂) its value tends to triple (33.2 ug / kg) for SS AISI 304 (4) wires and at 168 hours (t₃) it increases (69.4 ug / kg) for all orthodontic wires, especially for SS AISI 302 Twist (2) in which the eight thin metallic wires, each other intertwined, offer a greater exposed surface. Ni, on the other hand, is not released by SS AISI 304 Extra-hard (1) and SS AISI 304 (4) wires at no immersion time, in the orthodontic archwires SS AISI 302 Twist (2) slightly increases from t₁ to t₂ and then its value quadruples to t₃ while in the wires 0.017 "× 0.025" Ni-Ti (3) increases, doubling from t₁ to t₂ and multiplying nine times from t₂ to t₃. From these results it emerged that not even in normal saline solution the alloys seem to show a neutral or inert behaviour, they do not seem at all free from corrosion. These phenomena can be explained by the presence in the saline solution of components that can form chloridric acids acting as etching agent for the alloy.

There are many published studies on the corrosion resistance of Ni-Ti alloys in physiological solutions [7, 17, 23, 24].

With regard to exposure to different fluorinated mouthwashes, it is observed that in general, as the immersion time increases, orthodontic alloys elute gradually greater quantities of metal ions. This general increase is recorded to be more constant and linear for the Cr ion and more variable and inconstant for both Fe and Ni ions which, at 168 hours (t₃), seem to show the most significant increases. At one hour (t₁) of exposure, Cr is not
present in the various eluates in a very significant way, in fact its concentration appears almost superimposable
to the Blank samples (B); a maximum value (40.8 ug / kg) is recorded for it only at 168 hours (t₃) of immersion
of the threads SS AISI 302 Twist (2) in NaF (0.02%) (E) mouthwash. In general, Cr is the ion that was most
released by orthodontic arches SS AISI 302 Twist (2) in contact with all the mouthwashes under examination for
all the times considered.

Fe ion, on the other hand, is almost always present in eluates in greater quantities than chromium. These values,
in fact, are more significant when the orthodontic alloys are exposed to the mouthwash based on NaF 0.022%
(D), while with that based on NaF 0.02% (E) at 168 hours (t₃) it undergoes an abrupt halving. Fe is eluted mainly
from wire SS AISI 304 Extra-hard (1) after immersion in mouthwashes based on NaF 0.04% (A), NaF 0.0226% (C)
and NaF 0.022% (D) and orthodontic archwires SS AISI 302 Twist (2) exposed to fluorinated agents AmF 0.025%
(B), NaF 0.0226% (C) and NaF 0.022% (D).

Finally, Ni is the metal ion present in general in lower quantities than Fe ion and is always released by the alloys
of Ni-Ti (3) wires exposed to all fluorinated mouthwashes and especially those based on AmF 0.025% (B) and
NaF 0.02% (E) at the maximum exposure times. In literature, it has been shown that the amount of ions released
by the Ni-Ti (3) wires is higher than that released by the steel wires and that this phenomenon is closely related
to the increase in pH in the oral cavity [14].

The mouthwashes that prove to have caused a greater release of ions in general are those based on NaF 0.022%
and NaF 0.02%. Cr was released to a similar extent especially after exposure to NaF 0.04% (A) and AmF 0.025%
(B), Fe especially after immersion in NaF 0.04% (A) and equally from AmF 0.025% (B), while NaF 0.0226% (C)
was the least aggressive also towards Ni. AmF 0.025% (B) and NaF 0.02% (E) show the greatest peak of increase
after 1 week of contact thus demonstrating the greatest release of nickel from Ni-Ti wires at 1 week (t₃). Also
Barret et al., evaluated the release of Cr and Ni from Ni-Ti and SS wires at 1, 7, 14, 21 and 28 days showing that
Ni release reaches a maximum after approximately 1 week [28].

The data included in Table 4 allow to state that the different fluoride agents have had a negative effect on all the
orthodontic archwires under examination, causing, in general, a reduction in their ability to resist metallic
corrosion as the exposure time increased.

Corrosion is known to have significant consequences on the mechanical properties of orthodontic alloys such
as: increase in surface roughness, weakening of their resistance which can lead to mechanical failure or even
wire fracture [29]. On the other hand, it was also the cause of the release of variable quantities of metal ions
from the surface layers of the arches in exam [Tab. 5].

The elution of metal ions from the SS wires, in all the tested fluorinated mouthwashes and for all the time
intervals considered, was found to be within the biological safety threshold, also with regard to the relative
toxicity of Ni. Castro SM et al. reported that in SS alloys which have a Ni content of 8.00%, the crystal lattice
binds the nickel ions, making them unavailable to react. Therefore, these low Ni alloys are not capable of
causing Ni hypersensitivity, being tolerated by Ni sensitive patients [30].

In the safety data, tables relating to the composition of the archwires alloys in SS, list the following values
relating to Ni content: 8.00–10.50% for SS AISI 304 Extra-hard (1), 8.00–10.00% for SS AISI 302 (2) and 9.00%
for SS AISI 304 (4). From Table 6 it can be seen that as regards the release of Ni ions after immersion in all five
fluorine mouthwashes, the three alloys in SS assume a rather similar behaviour, which can be represented by the following equation:

$$A = C < E < D < B$$

where NaF (0.04%) (A) and NaF (0.0226%) (C) mouthwashes determine the lowest release of Ni, therefore among all mouthwashes they were found to be the least corrosive towards orthodontic alloys in SS. On the contrary, the AmF (0.025%) (B) mouthwash is the most aggressive towards SS alloys in exam.

In the scientific literature, it has been shown that the amount of ions released by the Ni-Ti wires is higher than that released by the SS wires and this phenomenon is closely related to an increase in oral pH [31].

As for the orthodontic wires in Ni-Ti (3), they have shown to be strongly corroded only if immersed in AmF (0.025%) and NaF (0.02%) mouthwashes at the maximum exposure times. The release of Ni ions, consequent to this corrosive phenomenon, was however significant since the concentration of Ni found in the eluates with the AmF (0.025%) (B) and NaF (0.02%) (E) mouthwashes was found to be greater than the biocompatibility threshold values in fact, the daily intake of Ni from food and drinks is about 300–500 µg (Chromium is 5 to 100 µg per day) and it is confirmed that if this absorption exceeds 2.5 µg / kg, allergic symptoms may appear, in fact, the concentrated dose required for allergic reactions is 600–2500 µg [32, 33].

This data is to be considered significant only for long and continuous exposure times because we observed that the amount of released metal ions by Ni-Ti wires increased with immersion time but, it should also be considered that the length of an orthodontic archwire is generally greater than 3 cm considered in this study to obtain metal sections useful for carrying out the investigation in vitro, therefore the total release of Ni, as well as that of the other metal ions is certainly greater and can be estimated at about 3–4 times as much.

The results obtained after immersion of the wires in the fluoride gels have revealed that both at t1 and at t2 the Cr ion was released more following exposure to AmF (1.25%) (F) while at t3 was found to be eluted more by coupling the gel of NaF (1.23%) (G) with SS AISI 302 Twist (2) wire. With fluorinated gels, the Fe ion is eluted from the alloys in a very similar way to Cr while, Ni is released in general much more and in particular from the Ni-Ti wires after their exposure to the AmF gel (1.25%) (F) but above all to NaF (1.23%) (G) reaching toxicity threshold values even at intermediate immersion points. This phenomenon is certainly linked to the higher concentration of fluorides present in the formulations of dental gels.

A recent study conducted in vitro on fixed orthodontic appliance exposed to soft drinks, shows how Coca Cola® causes an increase in the release of Ni, while orange juice does not intensify the release of metal ions so much [34]. Therefore, the acidity presence in the oral environment can be damaging for orthodontic alloys and in reality, any substance with an acidic pH, as well as mouthwashes and gels considered in our study, when placed in contact with orthodontic metal alloys, can cause a dissolution of metal ions which will be greater the more acidic the pH of these solutions. This is why it is essential to faithfully respect the indications established by the manufacturer, both in terms of timing and methods of use, of these oral prophylaxis agents.

The regular use of fluoride-containing products during the course of orthodontic treatment is essential to promote and stimulate re-mineralization of tooth enamel. Fluoride mouthwashes, available in 0.05% and 0.2%
fluoride concentrations, are often prescribed by orthodontists for weekly and even daily use to prevent dental caries [35].

Regarding professional topical application, compared to gels, fluorinated varnishes offer a definite advantage, represented by the possibility of selecting the site of clinical application. This allows to avoid the direct contact of the fluorinated agent with the alloys of arches and orthodontic brackets, thus avoiding all those damages related to the acid dissolution of metal ions that occurs following the generalized application and therefore also on these structures, of fluorides, as happens when using fluoride dental gels.

**Conclusions**

In general, SS arches are more stable than Ni-Ti arches when immersed in fluorinated mouthwashes, except in gels. In fact, SS orthodontic wires do not undergo significant erosion when exposed to most of the fluorinated mouthwashes for home use, while it has been observed that, for prolonged exposure, Ni-Ti arches are more sensitive to some of them. This is also confirmed by the scientific literature. The elution of metal ions results to change significantly in terms of metal erosion especially for the Ni-Ti wires when exposed to the two fluorinated gels, for which sensitive negative values have been obtained, i.e. above 1% until reaching for the gel G maximum loss of 2.7% at 1 week of exposure.

All the variations of 0.4 and 0.5% were not considered as significant as they were within the measurement error that was calculated for each measured for both positive (increasing) and negative (loss) values. Negative values higher than 1% are instead considered significant because they all represented real losses not dependent on the measurement procedure.

Therefore, although the initial hypothesis has been demonstrated, the results obtained in this in vitro study suggest that the concentrations of eluted metal ions are in any case negligible because they are minimal, not uniform in all the time intervals of the tests and in any case more significant only at very long exposure intervals, the last condition that is difficult to verify clinically. Therefore, even if the phenomena of corrosion of metals by fluorinated products exist, the orthodontic wires examined in this in vitro study showed good chemical stability and low toxicity. The warning message is for fluoride gels which, although they are electively intended for professional use, in reality at least in one case, are available at the same concentration of fluoride also as over-the-counter drugs and recommended for home use.

A fluorinated solution with an important corrosive power, such as that demonstrated by fluorinated gels, if arbitrarily used for incorrect times, is undoubtedly capable of damaging orthodontic alloys and in particular those of Ni-Ti, modifying their mechanical properties. For this reason, their home use should always be accompanied by careful prescription and supervision by the clinician.

**Abbreviations**

Ni
nickel

Cr
chromium

mg
Declarations

Ethics approval and consent to participate

Not applicable

Consent for publication

Not applicable.
Availability of data and materials

The datasets used and/or analysed during the current study are available from the corresponding author on reasonable request.

Competing interests

The authors declare that they have no competing interests.

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Conflict of interest statement

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Authors’ Contributions section:

All authors made substantial contributions to the conception of the work.
R.C. wrote the entire manuscript, performed the conceptualization of the work, the investigation and she is the project administrator.

G.M. provided the acquisition of bibliographic resources, performed the statistical analysis of the data and prepared the tables.

A.C. (Alessandro Cioffi) provided the acquisition of bibliographic resources, performed the statistical analysis and the interpretation of the data.

M.E.C. provided the bibliographic resources and performed the statistical analysis of the data.

M.D. provided the bibliography resources.

a.c. (Adriano Casaglia) provided the interpretation of bibliographic resources.

A.G. provided the bibliography resources.

G.P. provided the acquisition of bibliography resources and substantively revised the work.

F.M. carried out the investigation and prepared the tables.

L.M. performed the conceptualization, investigation and supervision of the work and prepared the tables.

All authors have read, reviewed and approved the manuscript.

All authors have agreed both to be personally accountable for the author's own contributions and to ensure that questions related to the accuracy or integrity of any part of the work, even ones in which the author was not personally involved, are appropriately investigated, resolved, and the resolution documented in the literature.

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Tables

| Group | Orthodontic archwires                                    | Number of archwires | Number of sections | Length of sections |
|-------|----------------------------------------------------------|---------------------|--------------------|--------------------|
| 1     | SS AISI 304 Extra-hard (Leone Spa, Italy)                | 8                   | 24                 | 3 cm               |
| 2     | SS AISI 302 Twist (Leone Spa, Italy)                     | 8                   | 24                 | 3 cm               |
| 3     | Ni-Ti (Leone Spa, Italy)                                | 8                   | 24                 | 3 cm               |
| 4     | SS AISI 304 (G&H Wire Company, USA)                      | 8                   | 24                 | 3 cm               |
Table 2
Fluoride mouthwashes and gels taken in exam in the *in vitro* study.

| Group | Formulation | Commercial name | Fluorinated agent | pH  |
|-------|-------------|-----------------|-------------------|-----|
| A     | mouthwash   | GUM® Ortho Rinse (SUNSTAR GUM) | NaF (0.04%) | 6.5 |
| B     | mouthwash   | Elmex® JUNIOR dental rinse (Colgate-Palmolive) | AmF (0.025%) | 4.7 |
| C     | mouthwash   | GUM® JUNIOR (SUNSTAR GUM) | NaF (0.0226%) | 5.5 |
| D     | mouthwash   | Listerine® HEALTHY WHITE™ (Johnson & Johnson) | NaF (0.022%) | 5.6 |
| E     | mouthwash   | Forhans medico (Uragme) | NaF (0.02%) | 4.5 |
| F     | dental gel  | Elmex® dental gel (Colgate-Palmolive) | AmF (1.25%) | 4.5 |
| G     | dental gel  | Miradent mirafluor® (Hanger & Werken) | NaF (1.23%) | 5.1 |

Due to technical limitations, table 3 is only available as a download in the Supplemental Files section.
Table 4
Concentration (ug/L) of metallic ions present in the eluates, resituated from the immersion of arch sections in the different solutions at predetermined time intervals.

|        | Cr ug/L |    |    | Fe ug/L |    |    | Ni ug/L |    |    |
|--------|---------|----|----|---------|----|----|---------|----|----|
|        | Bs      | t₁ | t₂ | t₃     | t₁ | t₂ | t₃     | t₁ | t₂ | t₃ |
| A      | 1       | 3,76 | 3,81 | 5,11 | 8,74 | 43,4 | 46,0 | 90,4 | 95,7 | 2,5 | 2,54 | 4,34 | 7,16 |
|        | 2       | 4,16 | 4,52 | 7,76 |     |     | 43,9 | 52,7 | 81,0 | 3,74 | 6,46 | 22,6 |
|        | 3       | 3,85 | 4,13 | 4,25 |     |     | 37,5 | 43,7 | 43,8 | 7,24 | 16,1 | 28,1 |
|        | 4       | 4,16 | 5,17 | 5,79 |     |     | 46,8 | 60,5 | 79,9 | 7,09 | 22,1 | 31,6 |
| B      | 1       | 9,61 | 10,8 | 15,2 | 16,3 | 40,3 | 68,3 | 115 | 131 | 10,3 | 10,5 | 16,1 | 17,4 |
|        | 2       | 17,8 | 41,6 | 79,7 |     |     | 381 | 743 | 971 | 40,6 | 68,2 | 85,2 |
|        | 3       | 9,68 | 9,70 | 13,4 |     |     | 41,6 | 42,3 | 62,1 | 53,2 | 838 | 4071 |
|        | 4       | 11,2 | 15,1 | 17,7 |     |     | 57,7 | 130 | 132 | 12,5 | 22,1 | 31,6 |
| C      | 1       | 2,75 | 2,86 | 4,19 | 7,50 | 19,2 | 28,3 | 49,3 | 127 | <1 | <1 | 1,40 | 2,08 |
|        | 2       | 2,93 | 5,21 | 9,93 |     |     | 31,0 | 51,9 | 96,7 | 3,20 | 6,43 | 6,94 |
|        | 3       | 2,76 | 3,71 | 4,72 |     |     | 19,7 | 19,8 | 30,6 | 4,38 | 24,4 | 121 |
|        | 4       | 3,39 | 5,60 | 6,11 |     |     | 31,5 | 39,0 | 56,9 | <1 | 1,84 | 2,84 |
| D      | 1       | 17,3 | 17,6 | 30,1 | 31,4 | 290 | 293 | 341 | 396 | 6,1 | 6,20 | 15,4 | 17,6 |
|        | 2       | 19,6 | 29,6 | 29,8 |     |     | 311 | 316 | 334 | 10,4 | 18,4 | 20,4 |
|        | 3       | 18,3 | 29,7 | 29,9 |     |     | 292 | 302 | 310 | 15,4 | 113 | 156 |
|        | 4       | 17,4 | 25,3 | 28,1 |     |     | 298 | 321 | 325 | 6,29 | 13,1 | 14,4 |
| E      | 1       | 7,04 | 7,25 | 12,1 | 12,3 | 25,1 | 166 | 136 | 71 | 4,13 | 4,44 | 8,87 | 9,19 |
|        | 2       | 7,10 | 28,2 | 40,8 |     |     | 163 | 541 | 705 | 8,60 | 61,9 | 63,14 |
|        | 3       | 7,56 | 7,61 | 7,84 |     |     | 29 | 26 | 34,7 | 117 | 1061 | 3772 |
|        | 4       | 8,24 | 11,5 | 12,3 |     |     | 47 | 94 | 95,1 | 4,94 | 9,02 | 36,7 |
| H      | 1       | <1 | <1 | <1 | 1,24 | 3,04 | 9,15 | 10,5 | 18,6 | <1 | <1 | <1 | <1 |
|        | 2       | <1 | <1 | <1 | 9,27 |     | 10,4 | 13,7 | 69,4 | 1,82 | 2,90 | 13,1 |
|        | 3       | <1 | <1 | <1 | 1,29 |     | 9,54 | 11,30 | 19,9 | 4,86 | 17 | 146 |
|        | 4       | <1 | 1,27 | 1,60 |     |     | 12,1 | 33,2 | 44,1 | <1 | <1 | <1 |

|        | Cr ug/kg |    |    | Fe ug/kg |    |    | Ni ug/kg |    |    |
|--------|----------|----|----|----------|----|----|----------|----|----|
|        | Bs       | t₁ | t₂ | t₃     | t₁ | t₂ | t₃     | t₁ | t₂ | t₃ |
| F      | 1        | 99 | 120 | 183 | 185 | 458 | 607 | 653 | 1100 | 437 | 464 | 1325 | 1605 |
| Cr ug/L | Fe ug/L | Ni ug/L |
|--------|--------|--------|
| 2      | 205    | 636    | 567 |
| 3      | 174    | 570    | 1748 |
| 4      | 127    | 201    | 1355 |
| G      | 11,2   | 46,4   | < 12 |
| 2      | 11,8   | 85     | 17,9 |
| 3      | 21,3   | 49,4   | 8707 |
| 4      | 24,2   | 122    | < 12 |

### Supplementary Files

This is a list of supplementary files associated with this preprint. Click to download.

- Table3.jpg