Corrosion resistance of three orthodontic brackets: a comparative study of three fluoride mouthwashes

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SUMMARY In the present study, three types of orthodontic brackets were investigated: cobalt-chromium (CoCr), iron-chromium-nickel (FeCrNi) and titanium (Ti) based. Their corrosion resistance was compared with that of platinum (Pt), which was chosen as the reference material because of its excellent electrochemical properties. The test solutions were Elmex®, Meridol® and Acorea® fluoride mouthwashes. Fusayama Meyer artificial saliva was used as the reference solution. The corrosion resistance of the different brackets in the three mouthwashes was assessed electrochemically to determine the corrosion potential and corrosion current density, and polarization resistance values were then calculated. A scanning electron microscopic (SEM) study and an analysis of released metal ions confirmed the electrochemical studies.

The results showed that the bracket materials could be divided into two groups: Ti and FeCrNi in one, and CoCr, which has properties close to those of Pt, in the other. Similarly, two groups of electrolytes were identified: Elmex® and Acorea® mouthwashes in one group, and Meridol® mouthwash in the second group. The results indicate that because of the risk of corrosion Meridol® mouthwash should not be prescribed for patients wearing Ti or FeCrNi-based orthodontic brackets.

Introduction

Fixed appliance mechanics involves stages during which the teeth are moved by sliding brackets along an archwire. These brackets are exposed to the oral cavity, which is a potentially hostile environment where electrochemical corrosion phenomena can occur.

Orthodontic brackets are made from a variety of materials including metal alloys. Titanium (Ti), iron-chromium-nickel (FeCrNi) and cobalt-chromium (CoCr) alloys are known for their good electrochemical properties (Metwally et al., 1995; Platt et al., 1997; Nakagawa et al., 1999). However, it is important not to ignore the role that may be played by certain minor alloys such as silicium or molybdenum.

The regular use of fluoride-containing products such as toothpaste (Pröbster et al., 1992, Toumelin-Chemla et al., 1996) and fluoride mouthwashes is recommended to reduce the risk of dental caries. However, numerous studies have shown that in an acidic environment and in the presence of fluoride ions (fluoride mouthwashes), the corrosion resistance of certain materials, in particular, Ti, can deteriorate (Boere, 1995; Reclaru and Meyer, 1998; Nakagawa et al., 2002a; Schiff et al., 2002; Kaneko et al., 2003). Ti is electronegative and a highly protective oxide film, TiO2, forms on its surface, acting as a seal: the presence of fluoride ions in an acidic environment can cause a breach of this layer.

No previous studies have been performed to assess the influence and clinical implications of fluoride mouthwashes on orthodontic brackets. The aim of the present study was to compare the corrosion resistance of three different bracket materials to that of platinum (Pt), in a reference solution of Fusayama Meyer artificial saliva and three fluoride mouthwashes.

Materials and methods

Specimens

Platinum was chosen as the reference material because it is resistant to corrosion. Three bracket materials were selected from those currently available on the market (FeCrNi, CoCr and Ti) and each bracket comprised (Hamula et al., 1996): the bracket itself, the bracket base, and the metal mesh base to which the adhesive is applied. X-ray microanalysis was used to determine the composition, by percentage weight, of the three different brackets and the results of these analyses are given in Table 1.

Four specimens of each type of material (three bracket materials and Pt, a total of 16 specimens) were placed into a cylinder in order to constitute the cap of a rotating disk electrode. The metal part was inserted in a polytetrafluoroethylene (PTFE) ring, adaptable to the working electrode. In order to prevent infiltration of the electrolyte, the space between the metal and the PTFE was
filled with a resin with inert electrochemical properties (Figure 1). The exposed surface of the metal disk constituted the working electrode (the areas concerned were 0.02 cm² for each bracket and 0.1 cm² for Pt). The working electrodes were mechanically polished with wet abrasive paper (BF03, Escil, Lyon, France) of increasingly finer grit: 120, 240, 620, 800 and 1200. They were polished, degreased in acetone and alcohol and then washed in demineralized water before corrosion testing.

Test solutions

The reference electrolyte used was Fusayama Meyer artificial saliva (Venugopalan and Lucas, 1998; Gal et al., 2001; Huang et al., 2003). All reagents were supplied by Sigma Chemical Company, St Louis, Missouri, USA. The composition of the artificial saliva, which closely resembles natural saliva, was: KCl (0.4 g/l), NaCl (0.4 g/l), CaCl₂·2H₂O (0.906 g/l), NaH₂PO₄·2H₂O (0.690 g/l), Na₂S·9H₂O (0.005 g/l) and urea (1 g/l). The pH was measured with an X6061 glass electrode (Radiometer Analytical, Villeurbanne, France) connected to a PHM 220 pH meter (Radiometer Analytical) and was found to be 5.3.

The active ingredients in the second test solution (Elmex® ready-to-use mouthwash, Gaba, Boulogne, France) were amine fluoride Olafluor (100 ppm) and sodium fluoride (150 ppm). The pH was 4.3.

The third test solution (Meridol® ready-to-use mouthwash, Gaba, Boulogne, France) had a pH of 4.2 and contained the active ingredients: amine fluoride Olafluor (125 ppm) and stannous fluoride (125 ppm).

The last solution (Acorea® ready-to-use mouthwash, Medicaler, Orchies, France) had a pH of 4.5 and contained the active ingredient sodium monofluorophosphate (65.9 ppm).

Electrochemical set-up

A glass electrochemical cell (Metrohm, Courtabeuf, France) was used with the thermostat (Bioblock, Illkirch, France) set at 37 ± 0.1°C. Three openings in the cover of this cell was used with the thermostat (Bioblock, Illkirch, France) connected to a PHM 220 pH meter (Radiometer Analytical, Villeurbanne, France) to limit noise disturbance and then connected to an EG&G PAR 273A computer controlled potentiostat (Perkin Elmer, Evry, France) (Figure 2).

The rotating speed of the disk electrode was 500 rpm.

Corrosion resistance analysis

Corrosion potential (Ecorr) was measured over a 24 hour period. Polarization curves (current in function of potential, i = f(E)) (Holland, 1992) were plotted once the potential had stabilized in order to determine the corrosion resistance of the materials in the different test solutions. Voltammetry was then performed at a scanning rate of 0.25 mV/s in a potential range of –800 to 1200 mV/SCE.

The polarization curves and Stern–Geary relationship (Lorenz and Mansfeld, 1981) were used to determine the corrosion current density (icorr) and the polarization resistance (Rp) which was equal to the potential variation against current variation (ΔE/Δi) at the end of corrosion potential exposure (Horasawa et al., 1996; Nakagawa et al., 1999). The Rp values are quoted in kohm.cm² and the icorr values in A.cm⁻². The corrosion rate (Vcorr) of the materials in the test solutions was calculated from icorr (Lorenz and Mansfeld, 1981) and the values given in µm/year.

The experiments were performed four times for each orthodontic bracket and Pt in each test solution. Each curve represented one of the four experiments for each material in each solution and was obtained from reproducible individual measurements. The Ecorr, icorr, Vcorr and Rp values are the mean values of four experiments with a maximum error of 10 to 15 per cent.

Surface analysis and measurement of released ions

One specimen of each type of bracket was observed by scanning electron microscopy (SEM; Leica, Cambridge, UK). Corrosion tests were then performed by immersing these specimens in the test solutions for two months and re-examining the surfaces using SEM. A comparison was thus obtained of their surface state before and after corrosion testing. The SEM photomicrographs showed the average surface state of the specimens.
The different test solutions recovered at the end of the two-month immersion period were analysed with Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-MS) (Jobin-Yvon, Ultima 2000, Longjumeau, France). The ICP-MS parameters included 1000W applied power, and a 0.05 ppm detectable limit. The reagents were standard solutions for atomic absorption spectrometry diluted to 1 ppm. The results of these released ion measurements are given in µg/l with a maximum error dosage of 10 to 15 per cent.

Results

Corrosion potential measurements

The corrosion potential of the different specimens in the four solutions was measured after 24 hours and the results are shown in Table 2.

Pt (Figure 3a): The corrosion potential in artificial saliva stabilized at +200 mV/SCE, suggesting good corrosion resistance in this solution, as was also found for Meridol® mouthwash, where the potential stabilized at +120 mV/SCE.

For Acorea® and Elmex® mouthwashes, a reproducible decline in the curves was observed during the first 10 hours, indicating a decrease in the corrosion resistance of this material. The corrosion potential stabilized at –50 mV/SCE after 10 hours.

FeCrNi alloy (Figure 3b): The corrosion potential for the artificial saliva was close to 0 mV/SCE, with the shape of the curve indicating passivation of the metal.

For Elmex® and Acorea®, the corrosion potentials were +40 and +80 mV/SCE, respectively, with curves similar to those obtained in artificial saliva.

For Meridol®, the corrosion potential dropped after 12 hours, reaching –170 mV/SCE at 24 hours, indicating corrosion of the metal.

CoCr alloy (Figure 3c): The corrosion potential values were relatively stable for this material in all test solutions.

In artificial saliva and in Meridol®, the material was rapidly passivated and the potential values were high, of the order of +20 and +40 mV/SCE, respectively.

For Elmex® and Acorea®, although the potential values stabilized, they were considerably lower, reaching –200 and –170 mV/SCE, respectively, indicating poorer resistance to corrosion.

Ti (Figure 3d): For artificial saliva, the corrosion potential stabilized rapidly at +50 mV/SCE. This value suggests that the material was passivated. For Acorea® the corrosion potential was similar to that of the reference medium, reaching +90 mV/SCE, and with a similar curve.

Although the potential for Elmex® only reached 0 mV/SCE, the steady rise in potential suggested good corrosion resistance. Thus, corrosion potential measurements for the Ti specimens in these three solutions indicated passivation of the material.

For Meridol®, the potential obtained was much lower, reaching –220 mV/SCE and the curve declined after 10 hours, thus indicating a risk of corrosion in this solution.

Table 2 Corrosion potential ($E_{corr}$) of the specimens in the different experimental solutions after 24 hours.

<table>
<thead>
<tr>
<th>Material</th>
<th>$E_{corr}$ (mV/SCE)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fusayama Meyer</td>
<td></td>
</tr>
<tr>
<td>artificial saliva</td>
<td>200 ± 22</td>
</tr>
<tr>
<td>Elmex®</td>
<td>–50 ± 6</td>
</tr>
<tr>
<td>Meridol®</td>
<td>120 ± 14</td>
</tr>
<tr>
<td>Acorea®</td>
<td>–50 ± 5</td>
</tr>
<tr>
<td>FeCrNi</td>
<td>0 ± 15</td>
</tr>
<tr>
<td>CoCr</td>
<td>20 ± 32</td>
</tr>
<tr>
<td>Ti</td>
<td>–170 ± 20</td>
</tr>
<tr>
<td></td>
<td>40 ± 5</td>
</tr>
<tr>
<td></td>
<td>–170 ± 20</td>
</tr>
<tr>
<td></td>
<td>50 ± 6</td>
</tr>
<tr>
<td></td>
<td>–220 ± 24</td>
</tr>
<tr>
<td></td>
<td>90 ± 10</td>
</tr>
</tbody>
</table>
Corrosion current density \( (i_{\text{corr}}) \), corrosion rate \( (V_{\text{corr}}) \) and polarization resistance \( (R_p) \) measurements

To assess corrosion resistance of the materials and the stability of the fluoride solutions, cyclic voltammograms were plotted for the specimens in the different test solutions and the corrosion current, corrosion rate and polarization resistance were determined.

The polarization curves were plotted in the potential range of \(-800\,\text{mV/SCE}\) to \(+1200\,\text{mV/SCE}\) at a scanning rate of \(0.25\,\text{mV/s}\).

The \(i_{\text{corr}}, V_{\text{corr}}\) and \(R_p\) values obtained are shown in Tables 3 and 4.

Results for each material

Pt (Figure 4a): For Pt, similar curves were obtained in artificial saliva and Meridol®: the relatively low corrosion current values and high polarization resistance values were of the same order of magnitude in these test solutions. These results confirm the good corrosion resistance of this material.

For Elmex® and Acorea® a different behaviour was observed: corrosion resistance was lower, with higher \(i_{\text{corr}}\) and \(V_{\text{corr}}\) values, and lower \(R_p\) values.

The oxidation currents at \(500\,\text{mV/SCE}\) showed that Elmex® and Acorea® were the most aggressive solutions for Pt, confirming the first \(E_{\text{corr}}\) measurements.

FeCrNi alloy (Figure 4b): For Acorea®, the corrosion current and polarization resistance values were similar to those obtained in the artificial saliva. The curves were characteristic of a passivated material.

For Elmex®, the \(i_{\text{corr}}\) and \(R_p\) values were also similar to those obtained in artificial saliva, whilst for Meridol®, the \(i_{\text{corr}}\) value was higher \((10.10^{-6}\,\text{A.cm}^{-2})\) than that obtained in artificial saliva and the \(R_p\) value was lower \((160\,\text{kohm.cm}^2)\). These results indicate that FeCrNi was less resistant to corrosion in Meridol®, thus confirming the \(V_{\text{corr}}\) values.

The oxidation currents for this specimen in the different mouthwashes showed that at \(500\,\text{mV/SCE}\) Acorea® was the least aggressive of the test solutions.

CoCr alloy (Figure 4c): In artificial saliva and in Meridol®, the polarization curves were similar. The low \(i_{\text{corr}}\) values and high \(R_p\) values were also similar. These results suggest good corrosion resistance of this specimen in the two solutions. For Elmex® and Acorea® the values obtained were considerably different. The \(i_{\text{corr}}\) values were high while the \(R_p\) values were much lower, indicating a risk of corrosion of this material in the two solutions. This was also confirmed by the high \(V_{\text{corr}}\) values.

It can be seen from the oxidation currents at \(500\,\text{mV/SCE}\) that Elmex® and Acorea® mouthwashes appeared to be less aggressive than Meridol®.

Ti (Figure 4d): The \(i_{\text{corr}}\) and \(R_p\) values for Acorea® and Elmex® were of the same order of magnitude as those
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In Meridol®, Ti appeared to lose its corrosion resistance. In fact, the corrosion current value obtained was distinctly higher and the Rp value much lower.

The oxidation currents for Ti at 500 mV/SCE, in the different mouthwashes, suggested that Acorea® was the least aggressive solution for this material.

Surface analysis and measurement of released ions

On the basis of the electrochemical parameters obtained, surface analyses of the specimens presenting the highest risk of corrosion were carried out. Analyses were also performed to determine the concentrations of ions released by the materials into the mouthwashes concerned, i.e. CoCr in Elmex® and Acorea®, and FeCrNi and Ti in Meridol®.

The ICP results are given in Table 5. For FeCrNi and Ti the released ion concentrations in Meridol® were very high. For CoCr, the released ion concentration values in Elmex® and Acorea® were fairly low.

The SEM photomicrographs confirmed these ICP results. For CoCr only slight localized corrosion was noted: very few physical changes were observed in the appearance of the surface, whether in Elmex® or Acorea® (Figures 5a,b,c). In contrast, FeCrNi presented localized intergranular corrosion in Meridol® (Figures 6a,b). The Ti specimen also presented localized corrosion in Meridol® (Figures 7a,b).

Discussion

In artificial saliva, the corrosion resistance of the Pt specimen was high, thus confirming its suitability as a reference material for examining electrochemical behaviour.

A number of authors have investigated corrosion potential with varying findings. For example, when using artificial saliva, Kedici et al. (1998) obtained a value of –140 mV/SCE for FeCrNi alloy, –63 mV/SCE for CoCr alloy and –425 mv/SCE for Ti. In contrast, Schiff et al. (2002) reported lower values of +25 mV/SCE for Ti in similar conditions, and Reclaru and Meyer (1998) obtained similar values at +20 mV/SCE. In the current study, the value obtained for Ti in artificial saliva (+50 mV/CSE) was similar to that found by both Reclaru and Meyer (1998) and Schiff et al. (2002). However, higher E_{corr} values were obtained for CoCr in artificial saliva (+20 mV/SCE) and for FeCrNi (0 mV/SCE) than those found by Kedici et al. (1998). However, the different composition of the artificial saliva used must also be considered.

In the present study there was variation between the materials and between the different test solutions. For example, the corrosion potential of the CoCr brackets was found to be unsatisfactory in Elmex®. When these findings are compared with other studies, Kedici et al. (1998) noted values of –260 mV/SCE in a fluoridated medium, with slightly lower values from Reclaru and Meyer (1998) at –170 mV/SCE. For FeCrNi alloys, Kedici et al. (1998) reported values in the region of –250 mV/SCE in a fluoridated medium. The values in the present study were slightly lower, with E_{corr} values of –170 mv/SCE for FeCrNi in Meridol®. For Ti, Schiff et al. (2002) obtained –300 mV/SCE in a fluoridated medium, while Reclaru and Meyer (1998) obtained –60 mV/SCE and Kedici et al. (1998) –460 mV/SCE. The current study showed values close to the those of Schiff et al. (2002), with an E_{corr} value of –220 mV/SCE for Ti in Meridol®. It is important to note, however, that these findings can not be compared with the current study as the composition of the fluoridated medium was very different to the mouthwashes being used.

When corrosion current density is considered, Platt et al. (1997) and Kedici et al. (1998) reported similar i_{corr} values.

Table 3 Corrosion current densities (i_{corr}) (10^{-6} A.cm^{-2}) and corrosion rates (V_{corr}) (μm/year) of the specimens in the different experimental solutions.

<table>
<thead>
<tr>
<th></th>
<th>Pt</th>
<th>FeCrNi</th>
<th>CoCr</th>
<th>Ti</th>
</tr>
</thead>
<tbody>
<tr>
<td>i_{corr}</td>
<td>V_{corr}</td>
<td>i_{corr}</td>
<td>V_{corr}</td>
<td>i_{corr}</td>
</tr>
<tr>
<td>Fusayama Meyer</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>artificial saliva</td>
<td>0.5 ± 0.05</td>
<td>4 ± 0.5</td>
<td>3 ± 0.4</td>
<td>32 ± 3</td>
</tr>
<tr>
<td>Elmex®</td>
<td>7 ± 0.5</td>
<td>52 ± 5</td>
<td>3 ± 0.4</td>
<td>32 ± 3</td>
</tr>
<tr>
<td>Meridol®</td>
<td>1 ± 0.1</td>
<td>7 ± 0.8</td>
<td>10 ± 1</td>
<td>108 ± 10</td>
</tr>
<tr>
<td>Acorea®</td>
<td>6 ± 0.5</td>
<td>45 ± 5</td>
<td>2 ± 0.3</td>
<td>22 ± 2</td>
</tr>
</tbody>
</table>

Table 4 Polarization resistance (Rp) of the specimens in the different experimental solutions.

<table>
<thead>
<tr>
<th>Rp (kohm.cm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pt</td>
</tr>
<tr>
<td>Fusayama Meyer</td>
</tr>
<tr>
<td>saliva</td>
</tr>
<tr>
<td>Elmex®</td>
</tr>
<tr>
<td>Meridol®</td>
</tr>
<tr>
<td>Acorea®</td>
</tr>
</tbody>
</table>

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Table 5  Concentrations of released ions.

<table>
<thead>
<tr>
<th>Ions</th>
<th>CoCr in Elmex®</th>
<th>CoCr in Acorea®</th>
<th>FeCrNi in Meridol®</th>
<th>Ti in Meridol®</th>
</tr>
</thead>
<tbody>
<tr>
<td>analysed</td>
<td>Co: 81 ± 10</td>
<td>Co: 43 ± 5</td>
<td>Cr: 1060 ± 100</td>
<td>Ti: 15000 ± 2000</td>
</tr>
<tr>
<td></td>
<td>Cr: 17 ± 2</td>
<td>Cr: 11 ± 1</td>
<td>Ni: 14800 ± 2000</td>
<td></td>
</tr>
</tbody>
</table>

When the results for the ion release are considered, for FeCrNi and Ti, the released ion concentrations in
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Meridol® were high, confirming the risk of corrosion of these materials in this particular test solution (Nakagawa et al., 1999; Huang et al., 2001; Kao et al., 2002; Mockers et al., 2002). For CoCr, the released ion concentration values in Elmex® and Acorea® were fairly low, suggesting a much lower risk of corrosion. It should also be noted that Pt presented a corrosion risk in these two test solutions (compare $i_{\text{corr}}$ and $R_p$ values), even though this material is known for its non-corrosive properties. It was thus confirmed that Elmex® and Acorea® were not aggressive solutions for CoCr, with no surface denaturation observed for either CoCr or Pt, but that they did contain species subject to oxidation–reduction (minor additives) which reacted and created a current.

The surface analysis study also produced interesting findings, with FeCrNi exhibiting localized intergranular corrosion in Meridol®. This type of selective attack at
the grain boundaries can result from local heterogeneity or the depletion of a component. It is characterized by a dissolution of the anodic areas and is characteristic of chromium. Metwally et al. (1995) noted similar results in their study using CoCr and FeCrNi.

In contrast, pitting corrosion was seen on the Ti specimen in Meridol® (Reclaru and Meyer, 1998; Zavanelli et al., 2000). This type of corrosion is caused by anions such as chloride and fluoride ions which attack the protective oxide layer.

In general, the results showed that the electrochemical properties of the CoCr bracket were similar to those of Pt. In contrast, the electrochemical properties of the Ti and FeCrNi brackets were poorer in Meridol® than in the other experimental solutions.

From a clinical point of view, corrosion of brackets may affect how the bracket slides on the archwire (Kao et al., 2002; Hamula et al., 1996) and the final result of orthodontic treatment may be compromised. Furthermore, if the bonding surface corrodes, permanent stains can appear on the dental enamel. Electrochemical phenomena also lead to the release of metal ions as corrosion products. It is now recognized that certain ions, such as Ni^{2+} and Cr^{3+}, can result in symptoms of toxicity and allergic reactions in humans (Mockers et al., 2002): these symptoms may be short-lived and intense, or longer lasting and moderate, and some may be completely resolved while others become a chronic problem. As far as the toxic effects of nickel are concerned, as long as the natural capacity to eliminate the nickel exceeds the accumulation capacity, the risks are minimal, although contact dermatitis is undoubtedly an increasing problem (Staffolani et al., 1999; Schmalz and Garhammer, 2002).

Therefore, although orthodontic devices cause major side-effects, corrosion does constitute a risk factor.

Conclusions

Based on the results of this study, the experimental materials can be divided into two groups according to their electrochemical behaviour: Ti and FeCrNi in one and CoCr in the other. The electrolytes can also be divided into two groups: Meridol® in one, and Elmex® and Acorea® in the other.

In all three mouthwashes, the electrochemical properties of CoCr were found to be satisfactory and similar to those of Pt. Meridol® had no influence on the electrochemical behaviour of the brackets made from this alloy. For Elmex® and Acorea®, no degradation of the materials was observed, but rather a degradation of the solutions, with the oxidation–reduction of the species present in these solutions. All three mouthwashes can therefore be prescribed for patients wearing CoCr brackets.

However, when using Ti or FeCrNi brackets, the results of the present study indicate that it is preferable to prescribe Elmex® or Acorea®. The study showed that the electrochemical properties of Ti and FeCrNi in these two mouthwashes were practically the same as those observed in the reference solution.

The stannous fluoride found in Meridol® mouthwash caused considerable corrosion of the FeCrNi and Ti (Boere, 1995; Nakagawa et al., 1999) specimens by destruction of the passive layer. These results were confirmed by SEM and analysis of released ions.

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