In vitro evaluation of fluoride release of orthodontic bonding adhesives

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Aim: To assess the fluoride release from adhesives used for bonding in orthodontics. Methods: Six bonding agents were evaluated: five adhesives (Biofix, Fill Magic Orthodontic, EagleBond, Orthobond, and Transbond XT) and one resin-reinforced glass-ionomer cement (Fuji Ortho LC), the latter serving as a control. Ten samples of each adhesive were stored in containers with 5 mL of deionized distilled water at 37°C. Fluoride release was measured with an ion-specific electrode. Readings were taken periodically for a total of 33 days. On day 28, all samples were immersed in a 0.221% sodium fluoride solution for 5 minutes to assess their recharge potential. Descriptive statistics were calculated for the fluoride release rates of each material. ANOVA with multiple comparisons and the Kruskal-Wallis tests (P < .05) were used to assess differences between the groups.

Results: All adhesives released significant amounts of fluoride (P < .05). During the first day, Biofix released the highest amount of fluoride, followed by Fuji Ortho LC, which also showed the highest fluoride release from day 7 to day 33. The fluoride release from Biofix and Fuji Ortho LC decreased by the third day to 40% of its original value. All other adhesives maintained their fluoride release with only small fluctuations. Conclusion: All the materials released significant amounts of fluoride, with Biofix having the highest release in the first 3 days and Fuji Ortho LC over the total length of the experiment. ORTHODONTICS (CHIC) 2011;12:290–295.

Key words: adhesive, bonding agents, fluoride release, glass-ionomer cement, recharge

The demineralization of tooth surfaces adjacent to orthodontic brackets is a common adverse effect of orthodontic treatment.¹ Patients often have difficulties in maintaining adequate oral hygiene with fixed appliances. Increased plaque accumulation with concomitant bacterial acid production results in demineralization, which is caused by a diffusion of calcium and phosphate ions from the enamel.²⁻⁵ This loss results in clinically detectable white spots, which are most pronounced at the gingival margin because it demonstrates the highest plaque accumulation.⁶⁻⁸
This early form of caries can occur as soon as 4 weeks after bonding. Within certain limits, teeth can remineralize in the presence of fluoride. Protective measures such as oral hygiene instructions, plaque removal, and application of topical fluorides depend on patient cooperation and have proven to be of limited clinical value in reducing demineralization. However, low doses of free fluoride ions released over a long period of time promote regrowth of depleted fluorapatite crystals.

At present, the most commonly used bonding agents are composite resins. Their popularity is based on their easy application and clinically acceptable bond strength. However, they cannot prevent demineralization around a bracket if they do not contain fluoride. In an attempt to minimize demineralization, various manufacturers have added fluoride formulations to their adhesives.

The objective of the present study was to assess the fluoride release of five orthodontic adhesives over 33 days.

METHODS

The following light-curing adhesives for bracket bonding were investigated: Biofix, Fill Magic Orthodontic, EagleBond, Orthobond, and Transbond XT. The resin-modified glass-ionomer cement Fuji Ortho LC served as a control.

Ten disks of each adhesive 4 mm in diameter and 4-mm thick were produced with an acrylic resin template compressed between two glass slabs covered with polyethylene sheets to prevent fluoride contamination. All materials were handled according to the manufacturers’ instructions. They were light cured for 40 seconds (20 seconds from each side) through the glass plates with a Light Curing 2500 unit (3M Dental Products) emanating a light intensity of 550 mW/cm². After curing, all specimens were immersed in a sealed container with 5.0 mL of deionized distilled water; this container was stored in an incubator at 37°C. The containers were randomly numbered, ensuring the fluoride testing was blindly carried out.

The fluoride levels of the container solution were measured with a fluoride electrode (model 96-09BN, Orion Research), a solid-state device that measures fluoride ion concentrations in water and some organic solvents. The sensor of the electrode consists of a single lanthanum fluoride crystal bonded onto an epoxy body. When the sensor contacts the solution, a potential is developed across the crystal that depends on the level of the fluoride ions. The potential is then measured on a millivolt meter (model 720A plus, Orion Research).

Since the fluoride electrode is sensitive to pH changes, the pH level was maintained between 5.0 and 5.5 with a total ionic strength adjustment buffer (TISAB). TISAB eliminates hydroxyl ion interferences and enables an accurate measurement of the total fluoride content.

The fluoride electrode was also calibrated using a series of standard solutions of sodium fluoride at the following concentrations (in ppm): 1,000, 100, 10, 1, and 0.1. These solutions were diluted in stages with deionized water to produce standard solutions of 10 and 1 ppm fluoride.

After 1 hour and on days 1, 3, 7, 14, 21, 28, 29, 30, 31, 32, and 33, the specimens were removed from the incubator. Each time, 5.0 mL of the solution was collected from each container and mixed with 5.0 mL of TISAB to provide a constant pH and eliminate aluminum interference. Before each test, the fluoride electrode was recalibrated, carefully washed with deionized water, and dried with a paper towel. After testing, the specimens were immediately returned to the incubator. The sample solutions were changed daily to prevent cumulative measurements.
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By measuring the fluoride in ppm in a known volume of water, the total amount of fluoride ions released from each specimen in μg/cm² could be calculated. This was done by multiplying the ppm (1 ppm = 1 μg/mL) by the water sample volume (10 mL) divided by the surface of the sample disk.

**Fluoride recharge**

On day 28 of the experiment, all specimens were washed with distilled water for 20 seconds, gently dried with paper towels, exposed to a 0.221% sodium fluoride solution (1,000 ppm fluoride) for 5 minutes, and washed again with distilled water for 20 seconds. Subsequently, two specimens of each group were placed in 8 mL of distilled water, and the fluoride release was measured after 24 and 48 hours to observe the release of absorbed fluoride. Fluoride recharges were also performed on days 30, 31, and 32 and reassessed 24 and 48 hours later.

**Statistical analysis**

Statistical analysis was performed with SPSS (IBM). Descriptive statistics included means, standard deviations, medians, minimums, and maximums for each of the six groups. Analysis of variance (ANOVA) with multiple comparisons and the Kruskal-Wallis test were employed at a .05 level of significance to identify differences in fluoride release between the groups.

**RESULTS**

The mean values for the released fluoride are listed in Tables 1 and 2 as well as in Fig 1. Biofix released the highest amount of fluoride during the first 24 hours (P < .05). Fuji Ortho LC also had had a high release, though it was significantly less (P < .05).

**DISCUSSION**

Due to the routine clinical application of orthodontic adhesives, there is a growing interest in using them as fluoride-releasing vehicles. A constant low dose fluoride release is considered most effective because it reduces the solubility of the enamel and enhances its remineralization and its transformation into...
Fluorapatite. This complex mineral has a stable spatial structure and a higher resistance to acid attacks as a result.\textsuperscript{18} Basically, fluoride and calcium ions are released from dissolved fluorapatite to form calcium fluoride, which covers the nondissolved crystals and interferes with the diffusion of the acid, consequently decreasing the release of calcium and fluoride ions from the enamel.

Because of its antienzymatic action, fluoride also has an antibacterial property, thus reducing plaque accumulation on restorations.\textsuperscript{19–22} This factor plays a synergetic role in decreasing the development of secondary caries and promotes longevity of restorations. Overall, fluoride-releasing orthodontic adhesives contribute to an optimal oral pH balance\textsuperscript{23} by reducing the periods of demineralization and increasing the remineralization cycles, mainly in the enamel adjacent to orthodontic attachments. Since there are different fluoride-containing orthodontic adhesives, the objective of this study was to assess their fluoride release over time.

### Table 2 Means and standard deviations (SDs) of the fluoride release from the test adhesives from 28 days to 33 days

<table>
<thead>
<tr>
<th>Material</th>
<th>Mean ± SD fluoride release</th>
<th>28 d*</th>
<th>29 d</th>
<th>30 d*</th>
<th>31 d*</th>
<th>32 d*</th>
<th>33 d</th>
</tr>
</thead>
<tbody>
<tr>
<td>Biofix</td>
<td>10.58 (0.33)\textsuperscript{A}</td>
<td>12.81 (1.07)\textsuperscript{A}</td>
<td>9.95 (0.45)\textsuperscript{A}</td>
<td>9.23 (0.41)\textsuperscript{A}</td>
<td>9.55 (0.00)\textsuperscript{A}</td>
<td>7.96 (0.00)\textsuperscript{A}</td>
<td></td>
</tr>
<tr>
<td>Fill Magic</td>
<td>10.50 (1.28)\textsuperscript{A}</td>
<td>10.98 (0.82)\textsuperscript{B}</td>
<td>9.15 (0.53)\textsuperscript{A,C}</td>
<td>8.75 (0.53)\textsuperscript{A}</td>
<td>8.43 (0.41)\textsuperscript{A,E}</td>
<td>9.95 (1.87)\textsuperscript{B,C,D}</td>
<td></td>
</tr>
<tr>
<td>EagleBond</td>
<td>8.83 (0.17)\textsuperscript{B}</td>
<td>9.71 (0.33)\textsuperscript{C}</td>
<td>8.35 (1.02)\textsuperscript{B,C}</td>
<td>9.79 (0.20)\textsuperscript{A,D}</td>
<td>7.88 (0.16)\textsuperscript{B,E}</td>
<td>6.68 (0.41)\textsuperscript{A}</td>
<td></td>
</tr>
<tr>
<td>Orthobond</td>
<td>8.67 (0.16)\textsuperscript{B}</td>
<td>9.87 (0.41)\textsuperscript{C}</td>
<td>7.00 (0.68)\textsuperscript{D}</td>
<td>11.06 (0.41)\textsuperscript{B}</td>
<td>7.96 (0.00)\textsuperscript{B,E}</td>
<td>6.92 (0.20)\textsuperscript{A}</td>
<td></td>
</tr>
<tr>
<td>Transbond XT</td>
<td>8.59 (0.62)\textsuperscript{B}</td>
<td>10.58 (1.20)\textsuperscript{B,C}</td>
<td>7.80 (1.08)\textsuperscript{D}</td>
<td>6.76 (0.00)\textsuperscript{C}</td>
<td>11.78 (2.56)\textsuperscript{C}</td>
<td>8.59 (2.49)\textsuperscript{A,C}</td>
<td></td>
</tr>
<tr>
<td>Fuji Ortho LC</td>
<td>10.19 (0.62)\textsuperscript{A}</td>
<td>14.25 (0.67)\textsuperscript{D}</td>
<td>10.66 (0.67)\textsuperscript{A}</td>
<td>10.98 (0.97)\textsuperscript{B}</td>
<td>14.33 (0.79)\textsuperscript{D}</td>
<td>11.30 (0.62)\textsuperscript{B,D}</td>
<td></td>
</tr>
</tbody>
</table>

Identical letters indicate the absence of a statistically significant difference (P > .05). *, fluoride recharge.

Fig 1 Fluoride release from the tested adhesives between hour 1 and day 33. The blue arrows indicate repeated fluoride recharges.
The study was performed by using deionized water so the amount of total fluoride ions released into the medium was quantified without the interference of saliva.\textsuperscript{3,24} The approximately 4-week evaluation period was based on the fact that after this time, the first control appointment would be scheduled. Although the evaluation was extended to 33 days, further studies with a longer time frame seem desirable since orthodontic adhesives usually remain in the oral cavity during the entire treatment (mean, 30 months).

Fuji Ortho LC served as a control because its fluoride release was proven by other studies.\textsuperscript{3,13,25} In the present investigation, the highest fluoride release was found for Biofix and Fuji Ortho LC during the first day. This is described in the literature as the burst effect.\textsuperscript{13,26} It is important because it allows enamel remineralization by the formation of a calcium fluoride reservoir.\textsuperscript{13} The immediate release pattern was also observed elsewhere.\textsuperscript{19}

On day 3, the fluoride release from Biofix and Fuji Ortho LC had decreased by 40%. The other adhesives, however, kept their level of fluoride release over the study period despite small fluctuations. From day 14 until the end of the experiment, Fuji Ortho LC released more fluoride than Biofix, although this difference was not always significant.

In general, Fuji Ortho LC had the highest fluoride release throughout the entire experiment, followed by Biofix, Fill Magic Orthodontic, and Transbond XT. EagleBond had the lowest release of fluoride.

To assess a material’s recharge and subsequent release capacity (i.e., to simulate a topical fluoride application), all specimens were exposed to a 0.221% sodium fluoride solution on day 28. After the first recharge, all materials had an increased fluoride release that decreased again after 48 hours. Further recharges did not achieve the same result as the first recharge, indicating that the boost capacity is somewhat limited. Clinically, this result is important since the orthodontist can help maintain an increased level of fluoride by applying a sodium fluoride solution at every maintenance appointment.

It remains questionable, though, whether the amount of fluoride released by orthodontic adhesives is sufficient to prevent demineralization. It was demonstrated in rats kept on a cariogenic diet that a fluoride release of as little as 0.5 to 1.0 μg/cm² per day reduced white spot formation by 38% over 38 days.\textsuperscript{27} Thus, the frequency of fluoride application appears to be more important than the concentration of the preparation.\textsuperscript{12} Still, initial high concentrations of fluoride ions are also effective, as a combination of both application methods.\textsuperscript{26}

Despite the differences, every adhesive material demonstrated a fluoride release of > 1.0 mg/cm² per day, which suffices to prevent (at most) or reduce (at least) the formation of white spot lesions during orthodontic treatment.

**CONCLUSION**

The results of this study for are:

- Biofix released initially the highest amount of fluoride.
- EagleBond released the least amount of fluoride during the evaluation period.
- All orthodontic adhesives studied released significant amounts of fluoride to prevent or reduce white spots.
REFERENCES


