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Effect of toothbrushing on fluoride release of polyacid-modified composite resins

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Abstract The aim of the study was to evaluate the fluoride release of polyacid-modified composite resins (Dyract, Compoglass) submitted to brushing abrasion. Twenty samples were taken from each material and stored in a buffer solution (pH 4.0) for 12 days. Each day, the samples were transferred to a fresh solution. Ten samples of each material were brushed in an automatic tooth-brushing machine (250 strokes, 260-g load) every fourth day. The remaining samples were not subjected to brushing. Fluoride content of the solutions was measured with a fluoride sensitive electrode after the addition of TISAB. Statistical analysis demonstrated significant differences between the two materials with regard to cumulative fluoride release within the 12 days of the experiment. However, no difference was observed between the fluoride release of the brushed samples compared to the unbrushed specimens. This was true for both, the cumulative fluoride release and its release on the day following brushing. It is assumed that regular brushing of the tested materials did not influence their release of fluoride and that brushing of polyacid-modified composite resins does not lead to maintaining their initially high level of fluoride release.

Key words Polyacid-modified composite resin · Fluoride · Abrasion · Toothbrushing

Introduction

It has been documented that fluoride is released from polyacid-modified composite resins (compomers). The fluoride release is claimed to hamper demineralization at the margins of a restoration [10]. However, the maximum release of fluoride occurs within the first day after hardening, thereafter falling to a plateau [1,14,27]. It has

been suggested that the low amount of fluoride released after a few days is insufficient to be cariostatically effective [20]. Therefore it would be of great interest to counteract the initial high loss of fluoride and to maintain a continuously increased level of fluoride released from restorative materials. Conventional glass ionomer cements are considered to act as a rechargeable fluoride release device following the application of fluoridation regimes [11,26]. However, there is no commonly held view in the literature on whether compomer materials can also be replenished with fluoride [4,22]. In studies dealing with the fluoride release of restorative materials, it is common practice to use polished specimens. Usually, the surfaces of these specimens were additionally not treated after polishing. However, in the oral cavity the surfaces of restorations are subjected to various influences due to mastication, toothbrushing or the impact of low pH-values after erosive and cariogenic challenges, respectively. Jost-Brinkmann [19] recently showed that treatment of the surface of a polyacid-modified composite resin with an air-polishing device resulted in an increased fluoride release. The air-polishing of the specimens counteracted the decrease of fluoride release, which occurred within the first few days. It is not known, whether this is also true for tooth-brushing which affects the surface of restorative materials as well [13,23]. It would be desirable that tooth-brushing also leads to a maintenance of a constantly high fluoride release from fluoride-containing materials. Cariogenic or erosive challenges are causing a decrease of the pH value at tooth surfaces. It is generally accepted that the cariostatic activity of fluoride depends mainly on the presence of fluoride in the liquid phase around and in the outer surface layer of a tooth at low pH values [24,25]. Hence, it would be of great importance to maintain a high fluoride release from restorative materials particularly at low pH values.

The aim of the present study was, therefore, to assess the effect of brushing polyacid-modified composite resins on the maintenance of a high fluoride release in an acidic environment.

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Table 1Batch-no., fluoridecontent and mean size of fluori-dated particles of the tested ma-terials

Product	Batch-no.	Fluoride content (wt %)	Particle size
Compoglass	708547	10.08 in the Ba-Al-fluorosilicate-glass 2.47 in form of YbF ₃	1.60 μm 0.25 μm
Dyract	S9501188Z	8.70 in the Sr-Al-Na-fluorosilicate-glas 1.50 in form of SrF_2	s 2.50 μm 1.50 μm

Materials and methods

In this study, the polyacid-modified composite resins Dyract (DeTrey Dentsply, Konstanz, Germany) and Compoglass (Vivadent, Schaan, Liechtenstein) were investigated (Table 1).

Preparation of the samples and analysis of fluoride release

Twenty samples were taken from each material. The materials were put into cylindrical cavities (height 1.6 mm; diameter 20.0 mm) of disc-shaped molds of a fluoridefree acrylate resin (Technovit 4071, Kulzer, Wehrheim, Germany). The polyacid-modified composite resins were applied in three to four portions which were lightcured for 60 s each. After hardening, all specimens were stored in a humid atmosphere for 24 h at 37° C. The specimens were ground flat with water-cooled carborundum discs (500–1000 grit; Water Proof Silicon Carbide Paper, Struers). The samples were scrutinized for porosity with the naked eye. The ones that were porous were discarded from the study.

Then each specimen was stored in 3 ml acidic solution (pH 4.0) mixed as described previously [1] for a period of 12 days. Polypropylene flasks (Greiner, Frickenhausen, Germany) with a tapered shape were used for storing the specimens. The tapered shape enabled the samples to be placed inside the flasks in a way that the polished surfaces of the specimens were totally covered with the acidic solution. The samples were transferred daily to a fresh solution. Before their transferal to the new solution. ten specimens of each material were brushed every fourth day with 250 brushing strokes (200 strokes / min) The remaining samples were not brushed. Brushing abrasion of the specimens was performed with an automatic toothbrushing machine (VDD Elektronik, Freiburg, Germany) described in detail previously [2]. Toothbrushes with medium bristle stiffness were used (Clips Wechselkopf Medoral, Diedenhofen, St. Augustin, Germany) and renewed after brushing five specimens from the same experimental group. In order to ensure brushing of the complete surface of the tested materials, it was necessary to alter the toothbrushes. Thus, the heads of the toothbrushes were cut off and fixed in a way, so that the long axes of the head of the toothbrushes were perpendicularly aligned to the direction of the brushing movement. Brushing was carried out at a load of 260 g in 20 ml of an abrasive slurry. The abrasive slurry was prepared by mixing 5 ml artificial fluoridefree saliva [21] with 1 g dentifrice. Nonfluoridated dentifrice based on the formulation of Elmex (Gaba, Therwil, Switzerland) with an REA-value of 4.2 ± 0.3 and a RDAvalue of 77 ± 2 was used for preparing the slurry [5]. The slurry was renewed for each specimen.

The solutions were buffered with an equal amount of TISAB II (Orion Research, Cambridge, Mass., USA). Fluoride in the solutions was analyzed with a specific fluoride electrode (Orion Research). The amount of fluoride eluted from the materials was converted into micrograms of F⁻ released per unit surface area of the specimens (μ g/cm²).

Analysis of variance with repeated measurements was applied to the data for statistical analysis. Significance was set at $P \leq 0.05$.

Determination of material loss due to brushing

In order to determine the effect of the brushing procedure on the surface of the materials, five specimens of each material were prepared as described above and also polished with a diamond spray (3 and 1 μ m; DP-Spray P3, Struers). The surfaces of these samples were covered with tape (Tesa, Beiersdorf, Hamburg, Germany) leaving an exposed area of 1.8 mm×10.0 mm in the center of the restorative materials. Polishing of the samples with diamond spray and covering part of the surfaces was done to ensure reference surfaces were available when measuring the depth of the abrasion grooves. The samples were stored in buffer solution for 4 days and subjected to a single implementation of the brushing procedure as described above.

After brushing, the tapes were removed and the abrasive wear in the brushing grooves was quantitatively assessed with a laser profilometer (Microfocus; UBM Messtechnik, Ettlingen, Germany) as previously described [3]. Additionally surface texture was evaluated recording the average (R_a -value) and maximum (R_{max} value) peaks of the surface profile. Comparisons between the brushed and unbrushed samples were performed by Student's *t*-test.

Results

Data of the cumulative fluoride release from the tested materials during the 12 days of the experiment are presented in Fig. 1. The following mean values (SD) were

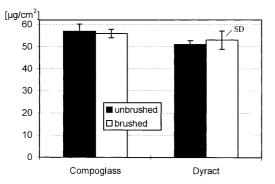


Fig. 1 Mean cumulative fluoride release and standard deviation (SD) of brushed and unbrushed samples after 12 days

recorded for the materials: Compoglass [unbrushed: 57.0 (3.2) μ g/cm², brushed: 55.8 (1.9) μ g/cm²]; Dyract [unbrushed 50.7 (1.8) μ g/cm², brushed: 53.1 (4.1) μ g/cm²]. Differences between the materials were statistically significant (*P*=0.0001). With both the brushed and unbrushed samples, Compoglass revealed a higher fluoride release than Dyract. However, no statistically significant difference was observed between the cumulative fluoride release of the brushed compared to the unbrushed samples for either of the materials (*P*=0.2617).

The daily fluoride release of the materials is presented is Table 2. Statistical analysis revealed a significantly continuous decline of the fluoride release within the experimental period for all materials (P=0.0001). This was true for both, the brushed and unbrushed specimens. Moreover, for all materials no significant difference was detectable between the unbrushed and the brushed samples irrespective of which day it was in the experiment (P=0.4987). Similar findings were also found for days 4, 8 and 12, which directly followed the brushing procedure.

For all materials under test, no visual differences in surface texture were observed between the brushed and the unbrushed areas of the samples. The results of the surface texture evaluation and the profilometrical analysis of the wear of the brushed samples are presented in Table 3. Brushing abrasion of the two materials was neg-

Table 3 Mean values of the surface roughness parameters R_a and R_{max} , and the depth of the brushing groove [µm] after a single implementation of the brushing procedure. Standard deviation is given in parentheses

	Compoglass	Dyract
R _a -value	0.05 (0.02)	
Unbrushed Brushed <i>P</i> value ^a	0.25 (0.03) 0.35 (0.03) <i>P</i> =0.0008	0.24 (0.05) 0.43 (0.12) <i>P</i> =0.0114
R _{max} -valu		
Unbrushed Brushed <i>P</i> -value	2.38 (0.69) 4.98 (1.27) <i>P</i> =0.0038	2.37 (0.26) 5.03 (2.28) <i>P</i> =0.001
Depth of brushing groove	0.82 (0.47)	2.76 (0.73)

^a*P*-values of the comparison between the brushed and unbrushed samples.

ligibly small. Surface roughness-values were statistically, significantly different between the brushed and unbrushed samples of both materials.

Discussion

The aim of the present study was to determine the fluoride release of polyacid-modified composite resins after toothbrushing. The samples were stored in an acidic medium for a period of 12 days. Fluoride release of polyacid-modified composite resins and (resin-modified) glass ionomer cements is increased in acidic solutions compared to a neutral medium [4,1,15]. Moreover, it could be demonstrated that brushing abrasion of these materials is significantly enhanced under acidic conditions [3]. In the present study, an acidic storage medium was therefore chosen in order to enhance both, fluoride release and brushing abrasion of the tested materials. Moreover, storage in the acidic buffer solution simulated the oral conditions during an erosive or a cariogenic challenge.

In the present study, the materials were submitted to brushing abrasion every fourth day. We could previously

Table 2 Mean, daily fluoride release $[\mu g/cm^2]$ of the tested materials within the period of 12 days. Standard deviation is given in parentheses

Day	Compoglass		Dyract	
	Unbrushed	Brushed	Unbrushed	Brushed
1 2 3 4 ^a 5 6 7 8 ^a	$\begin{array}{c} 8.8 \ (0.5) \\ 6.1 \ (0.5) \\ 5.4 \ (0.5) \\ 5.5 \ (0.5) \\ 4.6 \ (0.4) \\ 4.7 \ (0.3) \\ 4.5 \ (0.4) \\ 4.5 \ (0.4) \\ 4.5 \ (0.4) \\ 4.0 \ (0.2) \end{array}$	7.9 (0.5) 5.9 (0.5) 5.5 (0.6) 5.3 (0.3) 4.4 (0.2) 4.5 (0.3) 4.3 (0.4) 4.4 (0.1) 4.2 (0.1) 4.2 (0.1) 4.2 (0.1) 4.3 (0.4) 4.4 (0.1) 4.4 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5 (0.1) 4.5	8.2 (1.1) 6.5 (0.5) 4.8 (0.2) 4.4 (0.2) 3.7 (0.2) 3.9 (0.2) 3.7 (0.1) 3.8 (0.1) 2.5 (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1) (0.1)	$\begin{array}{c} 8.5 \ (0.7) \\ 6.9 \ (0.7) \\ 5.1 \ (0.5) \\ 4.6 \ (0.4) \\ 3.7 \ (0.4) \\ 4.0 \ (0.4) \\ 3.9 \ (0.4) \\ 3.9 \ (0.4) \\ 2.7 \ (0.4) \end{array}$
9 10 11 12 ^a	4.0 (0.3) 3.3 (0.2) 3.3 (0.2) 2.3 (0.1)	4.2 (0.1) 3.2 (0.2) 3.2 (0.2) 3.1 (0.1)	3.5 (0.1) 2.9 (0.1) 2.8 (0.1) 2.7 (0.1)	3.7 (0.4) 3.1 (0.3) 3.0 (0.3) 2.8 (0.3)

^a Day following brushing

demonstrate that the tested materials revealed a continuously low fluoride release within 28 days after an initially high fluoride release on the first 2–4 days [1]. It seems to be probable that this initial high fluoride release within the first four days is generated mainly by diffusion of fluoride ions from the outermost layer of the materials. Therefore, brushing abrasion in the present study was performed after four days in order to attack and remove the outermost layer exactly after the first few days of pronounced fluoride release. Brushing abrasion (instead of grinding of the surfaces) was performed in order to simulate clinical conditions.

In the present investigation, the specimens were brushed with 250 strokes at a load of 260 g which amounted to 2.6 N. Previous studies had demonstrated that the individual force applied during brushing amounted to 1.6-13.1 N [6,7,12]. It is therefore suggested that the load chosen in the present study simulates a low brushing force. Furthermore, it has been documented that patients brush their teeth at a frequency of 4.5 strokes/s [17]. Taking into account that during meticulous toothbrushing each tooth is brushed approximately for 5-10 s, it may be assumed that during a single period of toothbrushing about 20-40 brushing strokes are applied per tooth. This amounts to 40–80 brushing strokes per day provided that toothbrushing is performed twice a day. Hence, the number of brushing strokes applied every fourth day summarizes and reflects the suggested number of strokes performed within 4 days.

Fluoride release of the two polyacid-modified composite resins decreased continuously within the experimental period. This finding corresponds with several previous investigations [1,13,16,27]. Toothbrushing of the specimens did not influence fluoride release of the tested, fluoride-containing materials. This was true for the cumulative fluoride release and for the fluoride release on the day following brushing. This observation does not correspond with the findings of the previously published study by Jost-Brinkmann [19]. In this particular study, polyacid-modified composite resin specimens were submitted to air-polishing resulting in an increased fluoride release compared with samples that were not air-polished. The outcome of this study suggests that the removal of the outermost layer of the specimens by air polishing may lead to exposure of a layer that is not depleted of fluoride. It is described that air-polishing results in roughening of the surface of polished resin-based restorative materials. This effect can be observed by profilometrical analysis, but sometimes even clinically with the naked eye [8,9,18]. Furthermore it is reported that air-polishing of glass ionomer cements and composite resins leads to surface abrasion of up to 575 µm and 120 µm, respectively. In the present study, the profilometric analysis merely detects minimal wear of the materials due to brushing compared with the values reported after air polishing. Moreover, the increase in surface roughness of the surfaces was small, although statistically significant. Also, changes in surface texture were not macroscopically discernible. It is therefore suggested that the applied brushing procedure does not lead to exposure of a fluoride rich layer in the materials, which had been presumed to occur after air-polishing.

In a previous study, it was shown that the tested materials could not be replenished with fluoride by application of a fluoride containing dentifrice [4]. As a result of the present study, it is concluded that toothbrushing is not able to counteract fluoride loss occurring from the materials during the first few days of the experiment nor maintain a constant fluoride release thereafter at a level as high as within those first few days.

Generally, abrasion of restorative materials is an undesirable effect that had an influence on the longevity of a restoration. Moreover, it is important to notice that the influence of fluoride released from restorations on secondary caries development is still debatable and needs further investigation. Thus it is not reasonable to develop filling materials with lower abrasion resistance, even if this would lead to an elevated and continuous fluoride release.

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