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In Vitro Weight Loss of Dental Composite Resins and Glass-Ionomer Cements Exposed to a Challenge Simulating the Oral Intake of Acidic Drinks and Foods

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Abstract: Specific conditions of the oral cavity, such as intake of acidic drinks, foods, and drugs, represent a damage both for teeth as well as restorative materials. The aim of this in vitro study is to assess the influence of an acidic challenge on the weight loss of biomimetic restorative dental materials (composite resins and glass-ionomer cements, respectively). Seven products recently available in the marked have been tested in this study for the two kinds of materials, respectively. Resin composites were divided into Groups 1A–7A, whereas glass-ionomer cements into Groups 1B–7B. A total of six samples was considered for each group, among which two were stored into distilled water (control samples) whereas the other four were immersed into soft drink (Coca-Cola, Coca-Cola Company, Milano, Italy) for 7 days. Respectively, after 1, 3 and 7 days, weight was assessed for each sample and the percentage weight loss was calculated. For all the composite resins (Groups 1A–7A), no significant intergroup or intragroup differences occurred for the weight loss values ($p > 0.05$). Conversely, all glass-ionomers (Groups 1B–7B) showed a significant and progressive weight loss after 1, 3, and 7 days of acid challenge ($p < 0.05$) (intragroup differences). This reduction was significantly lower in case of GC Equia Forte + Coat and ChemFil Rock, with respect to the other cements ($p < 0.05$) (intergroup differences). In conclusions, all the biomimetic composite resins showed a reliable behavior when exposed to acidic erosion, whereas glass-ionomers cements generally tended to solubilize. However, the additional use of a protective layer above these latter materials could reduce this event. Despite these results appear to be interesting from a clinical point of view, future morphological evaluations should be conducted to evaluate the superficial changes of the materials after acidic explosion.

Keywords: dentistry; conservative; restorative; resin composites; glass-ionomer cements; acid exposure; acidic drinks

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1. Introduction

Along with periodontal disease, dental caries is one of the most frequent oral pathologies which represents the major cause of tooth loss [1]. This process corresponds to an infection of the hard surface of the tooth which leads to the dissolution of its mineral component, hydroxyapatite [2]. With the aim of restoring both the function as well as the aesthetic of compromised teeth, direct restorations by means of dental biomaterials are one of the main therapeutic options [3]. A wide range of materials by different manufacturers have been proposed for this treatment, in particular composite resins and glass-ionomers [4]. Specifically, the formers allow to carry out permanent dental restorations, with good aesthetic and adequate mechanical/chemical characteristics; conversely, glass-ionomers are

more frequently used for temporary restorations, besides a frequent use in orthodontics and pedodontics [3]. The good clinical performance of restorations is one of the main focuses which clinicians should achieve. However, despite their initial intrinsic properties, several conditions are recognized to compromise materials' stability, including intrinsic (e.g., eating disorders) and extrinsic factors (e.g., acidic diet, intake of acidic drugs, and unproper oral hygiene), besides the normal wear consequent to the exposition in the oral cavity [5–8]. Accordingly, functional stability of restorative materials should be periodically controlled to replace them when necessary.

Based on these considerations, several research studies have been conducted to assess the change of restorative materials exposed to artificial saliva mimicking normal oral conditions. These studies generally show a surface decrease of microhardness as well as an increase of roughness [3]. However, the abovementioned storage medium does not properly mimic more challenging situations, like the case of high intake of acidic drinks or foods. Accordingly, many other Authors have focused on the action of acid storages on restorative dental materials. In fact, the intake of acid substances with the diet is increasing with a high risk of dental wear, biomaterials' degradation, and risk of restoration failure [9,10].

The aim of the present study is to evaluate and compare the action of acidic challenges on the weight loss of biomimetic restorative materials from different manufacturers, including resin composites and glass ionomers. The two null hypotheses of the study are that no intergroup and intragroup significant differences occur considering the weight loss after acidic expositions of the resin composites and glass ionomers tested.

2. Materials and Methods

2.1. Materials Tested

Seven different resin composites and glass ionomers have been, respectively, considered in this study and subdivided into groups. The list of the materials tested, their specific compositions, and the relative manufacturers are shown in Tables 1 and 2.

Table 1. Composite resins tested in this study.

Group	Material	Type	Composition	Filler Content % (*w*/*w*)	Manufacturer	Lot #
1A	ENAMEL Plus HRi Bio Function	Microfilled hybrid composite	Matrix: urethane dimethacrylate (UDMA), tricyclodecane dimethanol dimethacrylate(TCDDA), no comonomers and no Bis-GMA Filler: glass filler, high dispersion silicon dioxide, fluorine	74 (*w*/*w*)	Micerium S.p.A., Avegno, Italy	2018006379
2A	Essentia	Microfilled hybrid composite	Matrix: urethane dimethacrylate (UDMA), Bis-MEPP, Bis-EMA, Bis-GMA, TEGDMA Filler: prepolymerised fillers, barium glass, fumed silica	81 (*w*/*w*)	GC Corporation, Tokyo, Japan	151109C
3A	Filtek Supreme XTE	Nanofilled composite	Matrix: Bis-phenol A diglycidylmethacrylate (Bis-GMA), triehtylene glycol dimethacrylate (TEGDMA), urethane dimethacrylate (UDMA), bis-phenol A polyethylene glycol diether dimethacylate Filler: silica nanofillers (5–75 nm), zirconia/silica nanoclusters (0.6–1.4 μm)	78.5 (*w*/*w*)	3M ESPE, St. Paul, MN, USA	N748173
4A	ENAMEL Plus HRi Flow	Microfilled hybrid composite	Matrix: urethane dimethacrylate (UDMA), Butanedioldimethacrylate Diurethandimethacrylate, Filler: glass filler, high dispersion silicon dioxide	53 (*w*/*w*)	Micerium S.p.A., Avegno, Italy	2017008768

Table 1. *Cont.*

Group	Material	Type	Composition	Filler Content % (*w*/*w*)	Manufacturer	Lot #
5A	SDR Flow	Microfilled hybrid composite	Matrix: modified UDMA, EBPADMA, TEGDMA Filler: barium and strontium alumino-fluoro-borosilicate glasses	47.3 (*w*/*w*)	Dentsply Sirona, Ballantyne Corporate Pl, Charlotte, USA	2003000392
6A	Ceram.X Universal	Nanoceramic composite	Matrix: methacrylate modified ploysiloxane, dimethacylate resin, ethyl-4(dimethylamino) benzoate, iron oxide pigments, titanium oxide pigments, aluminum sulfo silicate pigments Filler: barium-aluminum-borosilicate glass (1.1–1.5 μm), methacrylate functionalized silicon dioxide nano filler (10 nm)	76 (*w*/*w*)	Dentsply Sirona, Ballantyne Corporate Pl, Charlotte, USA	1507000661
7A	Gradia direct Flow	Microfilled hybrid composite	Matrix: urethanedimethacrylate (UDMA), dimethacrylate camphorquinone Filler: fluoro-alumino-silicate glass silica powder	67 (*w*/*w*)	GC Corporation, Tokyo, Japan	140606A

Table 2. Glass-ionomers tested in this study.

Group	Material	Composition	Manufacturer	Lot #
1B	Voco IonoStar Plus	Powder: fluoro-alumino-silicate glass, polyacrylic acid, tartaric acid Liquid: polyacrylic acid solution	Voco GmbH, Cuxhaven, Germany	1620354
2B	Voco IonoStar Plus + Easy Glaze	Powder: fluoro-alumino-silicate glass, polyacrylic acid, tartaric acid Liquid: polyacrylic acid solution Light Curing Protective Coating	Voco GmbH, Cuxhaven, Germany	1620354 Easy Glaze 1411097
3B	GC Equia Forte	Powder: fluoro-alumino-silicate glass, polyacrylic acid powder, pigment Liquid: polyacrylic acid, distilled water, polybasic carboxylic acid	GC Corporation, Tokyo, Japan	161020A
4B	GC Equia Forte + Coat	Powder: fluoro-alumino-silicate glass, polyacrylic acid powder, pigment Liquid: polyacrylic acid, distilled water, polybasic carboxylic acid Light Curing Protective Coating	GC Corporation, Tokyo, Japan	161020A Coat 1605131
5B	3M ESPE Ketac Universal Aplicap	Powder: Al-Ca-La fluorosilicate glass, copolymer acid (acrylic and maleic acid) Liquid: polyalkenoic acid, tartaric acid, water	3M ESPE, St Paul, MN, USA	634330
6B	GC Fuji TRIAGE CAPSULE	Powder: fluoro-alumino-silicate glass Liquid: polyacrylic acid, distilled water	GC Corporation, Tokyo, Japan	1611011
7B	ChemFil Rock	Powder: zinc-modified fluoro alumino silicate glass Liquid: polyacrylic and itaconic acid	Dentsply Sirona, Ballantyne Corporate Pl, Charlotte, USA	1607000503

2.2. Sample Size Calculation

Sample size calculation (alpha = 0.05; power = 80%) was performed considering a continuous variable. Concerning the primary outcome (% of weight loss), an expected mean of 1.75 was hypothesized, with a standard deviation of 0.85 [11]. The expected difference between the means was supposed to be 1.35, and therefore 6 specimens were requested for each group.

2.3. Samples' Preparation

2.3.1. Composite Resins

Each sample of the materials tested was inserted into silicon rings (height 2 mm, internal diameter 6 mm, and external diameter 8 mm) to obtain equal specimens. Molds were positioned above a dark opaque paper background with a polyester matrix strip interposed, to obtain a smooth surface under the material, as well as to avoid light reflection from the bottom thus reducing artificial hardening of this area. For each product, the A2 Vita shade has been chosen to avoid the effects of colorants on light-curing [12].

Each mold has been slightly overfilled and a second polyester matrix strip (Mylar strip, Henry Schein, Melville, NY, USA) was positioned on the top to avoid oxygen interfering with the polymerization of the most superficial layer of the composite [13]; to extrude the excess composite resin and obtain a flat surface, a glass slide was pressed against the upper polyester film and removed before curing [14].

Each sample was light-cured for 40 s with the LED unit Celalux 2 (Voco, Cuxhaven, Germany), and then removed from the mold without conducting polishing. Before every use, the cordless curing unit was maintained at full charge, and irradiance was checked with a radiometer (SDS Kerr, Orange, CA, USA). The distal end of the light guide was placed perpendicular to the surface of the matrix strip, and positioned concentrically with the mold, before starting the light-curing of the samples, which only took place on their external (top) side. Exclusively one light polymerization mode was used, with an output irradiance of 1000 mW/cm^2 [12].

All the samples were subsequently weighed with a Mettler-Toledo precision balance (AE1633, Mettler-Toledo SPA, Novate Milanese, Milan, Italy) with metering accuracy of 0.01 mg. Subsequently, for each composite resin, specimens were immersed in 50 mL of a soft drink (Coca-Cola Classic, Coca-Cola Company, Milano, Italy) (acidifying agent: phosphoric acid; measured pH value: 2.4) at room temperature (18 $\pm$ 1 °C)

After 24 h, each specimen was removed from the liquid using tweezers, then dried with blotting paper, left undisturbed for 60 min to completely dry, weighed with the precision balance as previously described, and then immersed again in the storage medium. This procedure was subsequently repeated after 3 and 7 days since the first immersion.

2.3.2. Glass-Ionomer Cements

Following the same procedure previously described for resin composites, each sample of the materials tested was inserted into silicon rings (height 2 mm, internal diameter 6 mm, and external diameter 8 mm) to obtain equal specimens. Before this step, glass-ionomer cements have been vibrated according to the indications of every single manufacturer. All the samples were then weighed with the same precision balance used for the resin composites.

Subsequently, for each glass-ionomer cement, specimens were immersed in 50 mL of a soft drink (Coca-Cola Classic, Coca-Cola Company, Milano, Italy) (acidifying agent: phosphoric acid; measured pH value: 2.4) at room temperature (18 $\pm$ 1 °C). The subsequent procedures were the same as described for composite resins in Section 2.3.1.

2.4. Statistical Analysis

Data have been subjected to analysis of variance (One-way ANOVA) followed by Bonferroni's post hoc tests. Analyses were performed using Prism 4.0 (GraphPad Software, San Diego, CA, USA). Two-tailed p values of 0.05 were considered statistically significant.

3. Results

3.1. Composite Resins

As shown in Table 3 and in Figure 1, the exposure to the acidic drink of all composite resins tested has caused no significant weight loss after 1, 3, and 7 days. No significant intergroup or intragroup differences have been assessed ($p > 0.05$).

Table 3. Weight loss of composite resins after 1, 3, and 7 day of acid challenge: data are expressed as medium percentage of weight loss.

GROUPS	WEIGHT LOSS (%)		
	1 Day	3 Days	7 Days
1A. ENAMEL Plus HRi Bio Function	0.61	0.62	0.67
2A. Essentia	0.76	0.78	0.81
3A. FILTEK Supreme XTE	0.86	1.20	1.30
4A. ENAMEL Plus HRi Flow	0.35	0.36	0.39
5A. SDR Flow	0.02	0.02	0.5
6A. Ceram.X Universal	1.08	1.12	1.17
7A. Gradia direct Flow	0.02	0.04	0.07

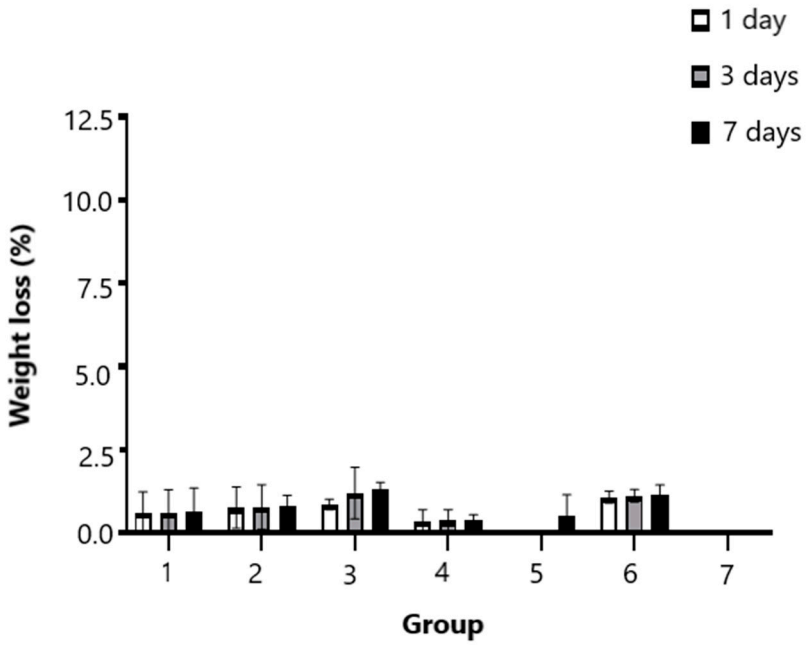

Figure 1. Composite resins: representation of weight loss after acid challenges.

3.2. Glass-Ionomer Cements

As shown in Table 4 and in Figure 2, the exposure to the acidic drink of all glass ionomer cements tested has caused significant and progressive weight losses after 1, 3, and 7 days (intergroup differences) ($p < 0.05$). As regards intragroup differences, group 4B and 7B reported the lowest weight loss with respect to the other groups ($p < 0.05$).

Table 4. Weight loss of glass-ionomer cements after 1, 3, and 7 day of acid challenge: data are expressed as medium percentage of weight loss.

GROUPS	WEIGHT LOSS (%)		
	1 Day	3 Days	7 Days
1B. Voco IonoStar Plus	0.43	4.45	9.39
2B. Voco IonoStar Plus + Easy Glaze	0.16	5.28	8.10
3B. GC Equia Forte	0.37	5.03	9.60
4B. GC Equia Forte + Coat	0.09	0.43	1.11
5B. 3M ESPE Ketac Universal Aplicap	1.40	4.99	8.33
6B. GC Fuji TRIAGE CAPSULE	2.71	5.33	7.71
7B. ChemFil Rock	1.69	2.15	2.97

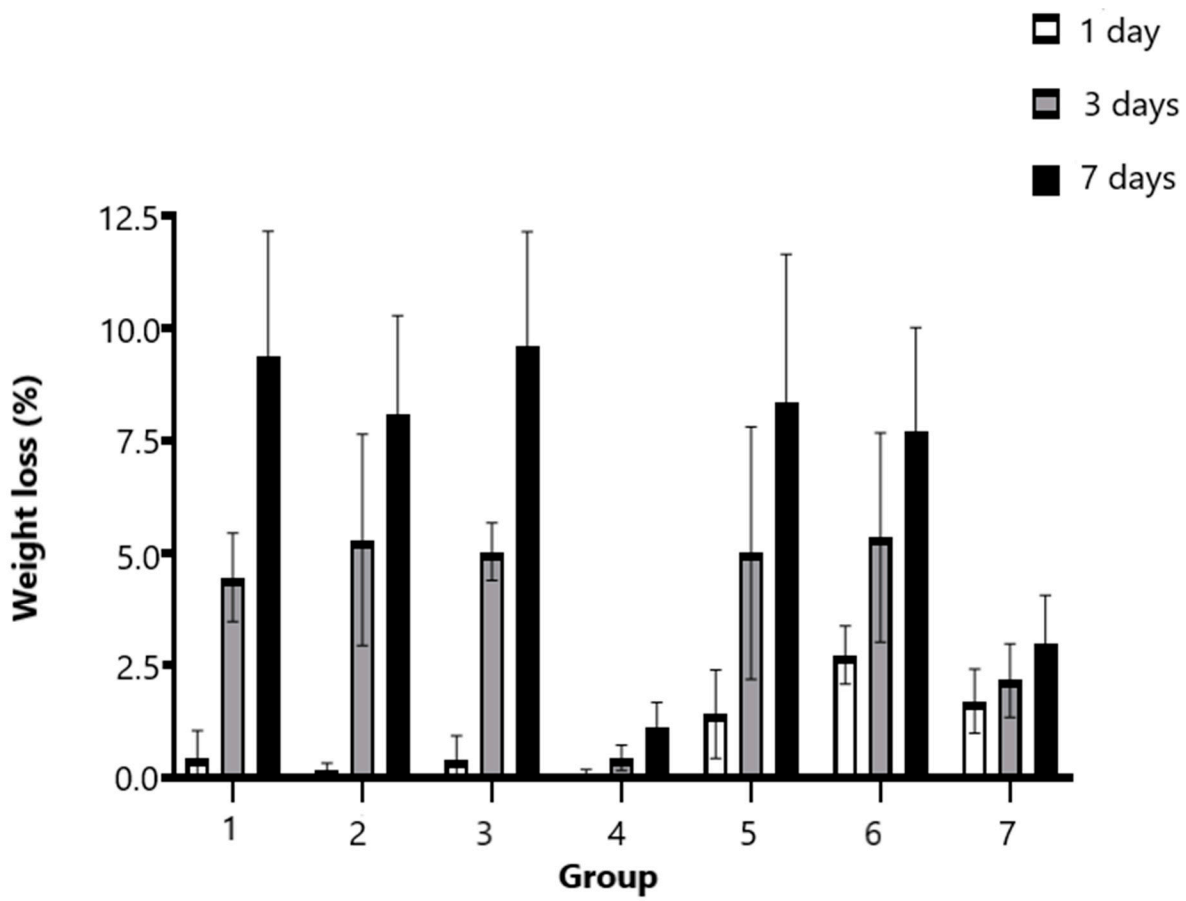

Figure 2. Glass ionomer cements: representation of weight loss after acid challenges.

4. Discussion

In the worldwide population, there is nowadays a large consume of artificially sweetened beverages, sport drinks, energy drinks, and other substances that cause problems to restorative dental materials [15]. Multiple studies have shown that acid substances are capable to jeopardize the microhardness and microroughness of composite resins and glass ionomers [16,17]. As in previous studies [18–20], Coca-Cola has been chosen as medium because it is the most popular acid drink and, additionally, compared to the other most popular ones available in the market, it has the lowest pH value, thus reporting the most significant erosive action [15]. In fact, phosphoric acid is recognized to have a more erosive action with respect to citric acid, this latter generally contained in the other soft drinks. The room temperature was chosen to reproduce the condition with which the soft drink is generally drunk.

In the present report, the behavior of seven different composite resins and seven glass-ionomer cements immersed in the abovementioned soft drink has been evaluated to assess the solubility level of these two different types of materials in acidic medium.

For the composite resins tested, the two null hypotheses considered have been accepted. No significant intragroup and intergroup differences have been assessed. In fact, the exposure of these materials to acidic challenge for 1, 3, and 7 days did not cause a significant weight loss. It is interesting to note that, among the materials tested, no significant differences resulted between flow and no flow composite resins. They both demonstrated a resistance to the weight loss caused by acidic erosion, thus confirming the evolution obtained in the last few years with these restorative materials. It is, thus, feasible to believe that acid erosion on composite resins just interfere with the superficial layer with a reduction of the surface microhardness, without interfering with the total weight of the material.

Conversely, for the glass-ionomer cements, the two null hypotheses have been partially refused. Significant intergroup differences have been assessed for each product tested between 1, 3, and 7 days. As to intragroup differences, all glass ionomers have shown a similar weight loss from 1 to 7 days of acid challenge, with exception of GC Equia Forte + Coat (Group 4B) and ChemFil Rock (Group 7B) which showed the lowest reductions with respect to the other groups. According to these results, the loss of weight of glass ionomers immersed in acidic medium has been progressive and it has affected all the products belonging to this typology of restorative materials. The first two glass-ionomer cements tested in this study (Voco IonoStar Plus and Voco IonoStar Easy Glaze) are based on the same material, but the second product contains Easy Glaze as a protective layer which guaranteed a slight reduction of weight loss values if compared to the former product. There was instead a clear difference between Group 3B (GC Equia Forte) and 4B (GC Equia Forte + Coat): even in this case, the two groups consist of the same material, but the second product is also composed of a protective coat which has demonstrated an effective resistance and protection not only after the first day of acidic challenge but also during the subsequent time points. It is relevant to note that, in this study, GC Equia Forte showed the highest percentage of weight loss at the last evaluation (9.60%); considering that the additional coating of this material with a protective layer reported the lowest weight loss after 7 days, this strategy could be very useful to protect glass-ionomer cements. Even Groups 5B, 6B, and 7B demonstrated a significant weight loss during the whole acid challenge, with a higher effect for 3M ESPE Ketac Universal Aplicap and GC Fuji TRIAGE CAPSULE, whereas a quite reduced one for ChemFil Rock. Comparing all the glass-ionomer cements tested in this study, GC Equia Forte + Coat (Group 4B) and ChemFil Rock (Group 7B) appeared to be the most reliable, reporting the least weight variation.

In conclusion, our study shows that all the biomimetic composite resins tested have proved to be well resistant to the acidic medium. On the other hand, some glass-ionomer cements can be subjected to an elevate and progressive loss of weight after exposure to acid beverages due to their different composition with respect to composite resins, the former based on calcium-alumino-fluoro-silicate glass and polyacrylic acid. However, if these materials are coated with a protective material this loss could decrease. Above these considerations, glass-ionomers remain among the most used materials in pediatric dentistry and orthodontics because of their action as fluoride reservoir which increases the concentration of this ion in saliva, plaque, and hard tissues of teeth, thus reducing the incidence of secondary caries [21]. Our results, thus, confirm what has been previously demonstrated in literature.

The main limitation of this report is that it has been conducted in vitro, therefore the buffering capacity of saliva, which contrasts the erosive action of acids [22], has not been considered. In addition to that, weight loss should be evaluated over a longer period (e.g., 30 days). Moreover, the specific composition of each restorative material has been regarded as the only factor influencing the severity of the changes caused by acidic solutions, conversely other in vivo factors, such as alimentary habits and oral hygiene procedures must be considered [3]. Finally, in our study, as in previous ones [23–25], no morphological evaluations were conducted to assess the variations of the materials' surfaces after acidic exposition. Accordingly, despite our results appear to be interesting from a clinical point of view, more detailed analyses should be conducted, encompassing for instance the use of SEM and AFM analysis.

5. Conclusions

Under the limitations of this in vitro study, we can conclude that acidic erosion does not significantly affect the weight of different biomimetic resin composites available in the market, neither considering intra- or inter-group comparisons after an acid challenge of three timepoints ($p > 0.05$). Conversely, all glass-ionomers (Groups 1B–7B) showed a significant and progressive weight loss after 1, 3, and 7 days of acid exposure ($p < 0.05$) (intragroup differences). This reduction was significantly lower for GC Equia Forte + Coat

and for ChemFil Rock, with respect to the other cements ($p < 0.05$) (intergroup differences). However, the use of glass-ionomer cements remains fundamental in orthodontics and pedodontics. In addition to that, specific recent glass-ionomers avoid this limitation by requiring the combination with a protective coat which preserves the characteristics of the bulk material.

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