A COMPARATIVE MICROLEAKAGE ANALYSIS OF ION-RELEASING SELF-ADHERABLE MATERIALS

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ABSTRACT

INTRODUCTION: Despite the advancements in restorative materials, there are certain drawbacks, including microleakage resulting from insufficient wall adaptation. An effective seal at the tooth/restoration interface is crucial.

OBJECTIVES: The aim of the study was to investigate microleakage of different self-adhesive restorative materials.

MATERIAL AND METHODS: Class-V cavities were prepared and divided into seven experimental groups with regards to applied material and pre-treatment procedures (n = 12). Cavities were restored with self-adhesive materials, such as hybrid glass ionomer (HGI) without pre-treatment (PT), HGI with PT, glass carbomer (GCP) without PT, GCP with PT, alkasite without adhesive resin (AR), alkasite with AR, nanohybrid composite resin with AR, and compared in terms of microleakage. Data were analyzed using Kolmogorov-Smirnov, Kruskal-Wallis, and Mann-Whitney U tests, with SPSS.

RESULTS: Significant difference in microleakage was found among enamel and dentin (p < 0.05). Leakage values from enamel were obtained as follows: alkasite with AR = nanohybrid composite resin with AR < alkasite without AR < HGI with PT < HGI without PT = GCP with PT < GCP without PT, while leakage values obtained from dentin included alkasite with AR = nanohybrid composite resin with AR = alkasite without AR = HGI with PT < HGI without PT = GCP with PT < GCP without PT.

CONCLUSIONS: Microleakage properties are material-dependent and may vary due to ingredients. Alkasite with AR could be an alternative to nanohybrid composite due to better sealing ability of both enamel and dentin, while alkasite without AR and HGI with PT are the preferred materials for dentin. GCP without PT showed the highest microleakage.

KEY WORDS: alkasite, hybrid glass ionomer, glass carbomer, nanohybrid composite resin, microleakage.

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INTRODUCTION

Over the past years, demands have been raising for a restorative material with better physical properties, good esthetics, and long-lasting performance, with acceptable cost. Despite the developments in restorative materials, there are certain drawbacks, including microleakage, marginal staining, secondary caries, post-op sensitivity, and pulpal and periapical pathology progress due to insufficient wall adaptation. Therefore, at this point, an effective seal at the tooth/restoration interface is crucial. Nowadays, various direct restorative material options are available from amalgam to tooth-colored esthetic restoratives, with each having its' own advantages and disadvantages. Despite a great change in directly placed dental restorative materials, simple and basic choices are still in demand [1-3].
Self-adhesive, tooth-colored restorative materials are user-friendly options because of shorter application times, simplicity, and less sensitive technique, especially in the presence of moisture in the cavity [4]. They provide a chemical bond to dental hard tissues by self-etching and/or self-adhesive monomers [5].

For a restorative material, bioactivity it also important, which defines antibacterial effect of a material to inhibit or treat infection [6]. Glass ionomer cements (GICs) have been used since the 70s of the last century as base materials, luting agents, and restorative materials in various areas of dentistry. GICs provide chemical bond with hard dental tissues by chelation of carboxyl group of acid polymeric chains and calcium ions (Ca^{2+}) in the apatite of enamel and dentine [7]. Due to bioactive characteristics of fluoride releasing GICs, hard tissue remineralization and protection against secondary caries can be achieved in contrast to materials, such as composite resin, amalgam, and dental ceramic that are bioinert [8].

GICs are susceptible to moist in the first 10 min following curing. To overcome this major clinical problem, surface coatings, including cocoa butter and petroleum jelly, waterproof varnishes, methyl methacrylates, amides, and light-curing resins are recommended [9]. Many modifications have been performed to conventional GICs to discard disadvantages of brittleness, which may lead to fracture, poor wear resistance, inadequate surface properties, and moisture sensitivity [6].

Lately, in order to prevent moisture sensitivity, and increase hardness and resisting of material under occlusal forces, a powder/liquid ratio, particle size, and distribution were changed, and hybrid glass ionomer (HGI) materials were introduced [10]. EQUIA Forte Fil (GC; Tokyo, Japan) is commercially available HGI, with better physical and mechanical properties incorporating ultrafine, highly reactive glass particles spread in higher molecular weight polyacrylic acid. It is used together with a nano-filled coating material (EQUIA Forte Coat, GC; Tokyo, Japan), which optimize marginal seal and wear resistance, and is indicated as a permanent restorative material if there is no high occlusal stresses [9].

A shift towards nano-technology, which defines using materials of size ranging from 1 to 100 nm, allowed for improved mechanical properties [11]. Therefore, another development related to changes in GICs based on nano-fillers is glass carbomer (GCP) (GCP Dental, Ridderkerk; The Netherlands). There is a common belief that the material transforms into a fluorapatite-like structure in time [12]. Depending upon nano-fluoride hydroxyapatite particles, the contact between liquid and powder grains increases. Thus, mechanical properties of GCP is enhanced [9]. Furthermore, a strong light source is recommended to cure GCP [9].

As a relatively new restorative material, Cention N alk-asite (Ivoclar, Vivadent; Schaan; Liechtenstein) has been introduced into a sub-group of composite resins. Cention N is a self-curing powder/liquid restorative material, with optional additional light-curing, which is capable of releasing acid-neutralizing ions. It is urethane dimethacrylate (UDMA)-based, tooth-colored filling material, with high flexural strength and low elastic modulus. It can be used with or without adhesive application, and self-adhesion to dentin and enamel is anticipated. With regards to the patented ingredient, called ‘isofiller’, polymerization shrinkage decreases, resulting in reduced microleakage [2]. Moreover, it was shown that clinical performance of Cention N is quite satisfying. In a study evaluating failure rate of Cention N restoration in 6 month, the material was observed to have effective marginal integrity as Tetric N Ceram (Ivoclar, Vivadent; Schaan, Liechtenstein) but with lesser surface characteristics [13].

Since long-term clinical studies in the literature are lacking, it is quite important to evaluate Cention N. Even though there are few studies on this subject, self-adhesive restorative materials have not yet been fully evaluated.

OBJECTIVES

The objectives of the current study were to investigate the microleakage of 3 different type, commercially available self-adherable ion-releasing materials with regards to pre-treatment procedures, and to compare them with each other to contribute to the literature.

Null hypothesis assessed was that microleakage of all tested restorative materials do not differ significantly from each other.

MATERIAL AND METHODS

The study started with ethical approval obtained from Pamukkale University Faculty of Medicine Human Ethics committee (number: 60116787-020/47595). All experimental stages of the study were conducted in Pamukkale University Faculty of Dentistry Research Laboratory and Pamukkale University Faculty of Technology Laboratory.

EXPERIMENTAL PROCESS

Eighty-four freshly extracted (within one month before the experiment) human, non-carious permanent molar teeth, which did not differ greatly in terms of morphology, were chosen. Before cavity preparation, all teeth were cleaned using pumice and prophylaxis rubber. Teeth were controlled with an optical microscopy (Discovery V20, Carl Zeiss; Gottingen, Germany) at ×30 magnification for any hypoplastic defects, caries, restorations, or cracks. Specimens with wear, dentin sclerosis, or discoloration were excluded from the study. Soft tissue remnants and dental plaque on the teeth were cleaned with a periodontal curette. The teeth were disinfected with 0.5% chloramine T solution (Fisher Scien-
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TABLE 1. Self-adhesive materials used in each group, their composition and application procedures

<table>
<thead>
<tr>
<th>Groups</th>
<th>Material/ manufacturer/ Batch number</th>
<th>Material composition</th>
<th>Application procedure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hybrid glass ionomer</td>
<td>EQUIA Forte/GC Tokyo, Japan/ 1804061</td>
<td>Liquid: Polycrylic acid, distilled water, polybasic carboxylic acid. Powder: Fluoro-aluminum-silicate glass, polyacrylic acid powder, pigment, methyl methacrylate, colloidal silica, camphorquione, urethane methacrylate, phosphoric ester monomer.</td>
<td>Activate the capsule and mix in a high-frequency mixer. Apply EQUIA Forte directly into the cavity preparation after mixing for 10 s using a mixing device. Remove excess material.</td>
</tr>
<tr>
<td>Coating</td>
<td>EQUIA Forte Coat/ GC Europe, Leuven, Belgium/0086</td>
<td>Methylmethacrylate, colloidal silica, camphorquione.</td>
<td>Apply EQUIA Forte Coat and additional light for 20 s using a LED lamp at a distance of 1 mm (1,200 mW/cm², standard power curing mode of VALO™ Cordless, Ultradent, South Jordan, UT 84095, USA) perpendicularly to the material surfaces.</td>
</tr>
<tr>
<td>Alkasite</td>
<td>Cention N/ Ivoclar Vivadent AG, Bendererstrasse, Schaan, Liechtenstein/ W93722</td>
<td>Filler: Barium aluminum silicate glass, ytterbium trifluoride, isofiller (Tetric N-Ceram technology), calcium barium aluminum fluorosilicate glass, calcium fluoro silicate glass. Liquid: UDMA, DCP, Tetramethyl-xylol-diurethane dimethacrylate, PEG-400 DMA.</td>
<td>Dispense powder and liquid next to each other on a mixing pad. Mix the first portion of powder with the entire liquid dispensed. Once the components have been thoroughly mixed, add the remaining powder and mix again, until a homogeneous consistency is achieved (45-60 s). The working time is 3 min from the start of mixing (when no light curing). Apply additional polymerized light for 20 s, using a LED lamp at a distance of 1 mm (1,200 mW/cm², standard power curing mode of VALO™ Cordless, Ultradent, South Jordan, UT 84095, USA).</td>
</tr>
<tr>
<td>Coating</td>
<td>GCP Gloss/ GCP Dental, Vianen, The Netherlands/ 7180616</td>
<td>Modified polysiloxanes.</td>
<td>Coat the surfaces with GCP Gloss and light-cure for 60 s, with a high output light device (GCP CarboLED, GCP Dental).</td>
</tr>
<tr>
<td>Nanohybrid composite resin</td>
<td>Grandio So/VOCO GmbH, Cuxhaven, Germany/1806497</td>
<td>Glass ceramic filler (particle size 1 μm), silicon dioxide nano-particles (range, 20-40 nm), Bis-GMA, Bis-EMA, TEGDMA, initiators, inorganic pigments, BHT.</td>
<td>Apply the composite resin material in one increment and light cure for 40 s, using a LED lamp at a distance of 1 mm (1200 mW/cm², standard power curing mode of VALO™ Cordless, Ultradent, South Jordan, UT 84095, USA).</td>
</tr>
</tbody>
</table>


*All of the above materials are from shade A2.

Cavity Preparation

Eighty-four box-shaped class V cavities (5 mm width and 2.5 mm depth) were prepared. Gingival cavity edge was finished 1 mm below the enamel-cement border. Cavity edges were not beveled. Cavity preparations were completed by using a flat-end diamond fissure bur No. 836/010 (Maxima Diamond, Henry Schein; Port Washington, NY, USA) in a high-speed aerator handpiece under water cooling. The bur was replaced after every five preparations. For dimension and depth standardization, a periodontal probe (PCP-UNC 15 Hu-Friedy; Leimen, Germany) was applied. All preparations were completed by one clinician for standardization.

Afterwards, prepared cavities were randomly assigned into 7 experimental groups with regards to applied self-adherable ion-releasing material and pre-treatment procedures (n = 12). Group 1: HGI (EQUIA Forte Fil, GC Corp., Tokyo, Japan) without pre-treatment; Group 2: HGI (EQUIA Forte Fil, GC Corp., Tokyo, Japan) with pre-treatment (Dentin Conditioner, GC Corp.; Tokyo, Japan); Group 3: GCP (GCP Fil, GCP Dental; Vianen, The Netherlands) without pre-treatment; Group 4: GCP (GCP Fil, GCP Dental; Vianen, The Netherlands) with pre-treatment (Dentin Conditioner, GC Corp.; Tokyo, Japan); Group 5: Alkasite (Cention N, Ivoclar Vivadent; India) without adhesive resin; Group 6: Alkasite (Cention N, Ivoclar Vivadent; India) with adhesive resin (Clearfil

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The teeth were immersed in 1% methylene blue dye (pH = 7) solution for 24 h, except for the roots. Then, they were washed for 1 min under running tap water, and dried.

A precise diamond saw was used to cut the teeth longitudinally through the center of restorations under continuous water irrigation (Micracut, Metkon, Bursa; Turkey; rotational speed range, 0-300 rpm). Three sections were obtained from each tooth. Sections’ surface was examined under stereomicroscope (Discovery V20, Carl Zeiss; Gottingen, Germany) at ×40 magnification.

To prevent bias, the samples were numbered and mixed for blending. Two clinicians scored depth of dye penetration separately at enamel and dentin margins, according to microleakage scores: 0 – no dye penetration; 1 – dye penetration up to half of the wall; 2 – dye penetration for more than half of the wall; 3 – dye penetration of the entire wall [14].

**THERMAL CYCLING AND MICROLEAKAGE EVALUATION**

All of the specimens were subjected to thermal cycling in a thermocycling device (MTE 101 thermocycling machine, Esetron; Ankara, Turkey) alternating between 5 ± 2°C to 55 ± 2°C for 5,000 cycles, with an exposure time of 30 s and a dwelling time of 15 s. Afterwards, root apices were sealed with wax (Modelling Wax, Dentsply; Turkey), and the teeth were covered with two coats of nail varnish to within 1 mm beyond margins of the restorations.

**STATHISTICAL ANALYSIS**

The sample size of each group was determined using a power analysis (G Power 3.1.9.2 software), with statistical difference of \( p = 0.05 \), and effect size of 0.3 at 95% power.

Data were analyzed using SPSS for Windows software (version 22.0; SPSS Inc.; Chicago, IL, USA). Shapiro-Wilk test was applied to determine normality of distribution. Kruskal-Wallis test and Mann-Whitney \( U \)-test

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**FIGURE 1.** Scores of dye penetration on enamel and dentin margins for each group
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A comparative microleakage analysis of ion-releasing self-adherable materials with Bonferroni correction was used to examine statistical differences among the groups.

RESULTS

The mean and standard deviation values of microleakage scores obtained from groups at enamel and dentin margins (Figure 1) and multiple comparisons are presented in Table 2 (p < 0.05). Photographs taken from different groups representing each microleakage score (0, 1, 2, and 3) are shown in Figure 2. There were statistically significant differences among the groups in microleakage value both at enamel and dentin margins (Kruskal-Wallis test, p < 0.05). When leakage values obtained from enamel margins were analyzed, microleakage values increased in Cention N with adhesive = nano-hybrid composite resin with adhesive < Cention N without adhesive < HGI with pre-treatment < HGI without pre-treatment = GCP with pre-treatment < GCP without pre-treatment (Table 2).

When leakage values obtained from dentin margins were analyzed, microleakage values increased in Cen-
tion N with adhesive = Cention N without adhesive = HGI with pre-treatment < HGI without pre-treatment = GCP with pre-treatment < GCP without pre-treatment (Table 2).

**DISCUSSION**

In the present study, a comparative investigation of various ion-releasing self-adhesive restorative materials with regards to microleakage were examined. The null hypothesis that microleakage of all tested restorative materials do not differ significantly from each other was rejected, since the results showed that microleakage properties of restorative materials were material-dependent and may change due to pre-treatment methods.

Microleakage can be defined as a transition of bacteria, fluids, or molecules in microcaps (range, 6-10 μm) between the cavity wall and a restorative material that leads to post-operative sensitivity, pulpal inflammation, and secondary caries [15-17].

In this present study, class V cavities were tested because of weak bonding in this area, which is more prone to microleakage [18]. To discard bias, identically similar teeth were selected, and cavity standardization was performed by one researcher.

Thermal and mechanical stresses affect the lifetime of restorations by creating marginal gaps. In the present study, to simulate the variation of oral conditions, thermocycling was performed. Although radioactive isotopes, scanning electron microscope, neutron activation analysis, and confocal laser scanning microscope can all be used as test methods to evaluate microleakage, dye penetration is still the most popular technique [3, 18]. Hence, a dye material was used for detecting microleakage scores since the diameter of dye molecule (0.80 nm) is smaller than dentinal tubule (range, 1-4 μm) [16-19].

The selection of composite resin is crucial for clinical success, when restoring in posterior loadbearing areas. Composite resins placed in conjunction with certain dental adhesives are believed to lose their sealing ability over time, thus permitting microleakage. The Academy of Operative Dentistry – European section recommends the use of micro-hybrid or nano-hybrid composite resins, with a minimum of 60% filler load by volume. Therefore, in this study, 89% of filled nano-hybrid composite resin was selected as a control group [20].

Dentin interface has challenging properties for bonding, with higher organic content, tubular structure, fluid pressure, and low surface energy, when compared to enamel. Moreover, dentin contains parallel tubules, which run towards the pulp [18]. In line with structural properties, statistically significantly higher microleakage results were obtained in dentin.

In the current study, comparatively lower microleakage scores of Cention N over HGI or GCP was observed. This lower scores in Cention N could be related to Ivo-
revealed that Cention N without adhesive showed lesser microleakage than Cention N with adhesive. Different results can be related to changes in dentin structure, size, or location of dentinal tubules within the tooth, which may have an effect on properties of materials.

GICs bond by micromechanical interlocking and chemical bonding to hydroxyapatite in tooth structure. GICs mechanism of adhering tooth can be considered of as a ‘mild self-etch approach’. However, GICs adhere better when combined with etch-and-rinse step [11]. Polyacrylic acid is commonly used as a conditioner both in enamel and dentine pre-treatments, to remove the smear layer [28]. It results in partial demineralization and increased surface area. Therefore, microporosities allow for micromechanical retention [29]. Omitting this polyacrylic acid step saves time and reduce technical sensitivity [11]. However, in this study, in both enamel and dentin, the microleakage scores in HGI without pre-treatment were considered statistically significantly higher than HGI with pre-treatment, and comparable results were obtained in GCP with pre-treatment. According to the literature, higher microleakage results might be responsible, and are possibly related to the lack of additional conditioning steps [11]. Also, HGI with pre-treatment showed comparable results with the control group in dentin (p > 0.05), while higher microleakage scores were noted in enamel (p < 0.05). Thus, clinicians should consider using polyacrylic acid conditioning, especially when adhesion forces are more crucial, like in this study, in cervical lesions. Another reason for microleakage pattern in HGI used in the present study was most likely due to high viscosity, which may be responsible for inadequacy wetting of the tooth surface.

GCP material sets through an acid-base reaction, being a kind of GIC. Water plays a crucial role when bonding GICs to dental substrates, and acts as a medium to provide ion exchange [29]. Moreover, the manufacturer recommends using heat to speed up the setting time of GCP restorative material [30]. Here, the authors found comparatively lesser microleakage scores of Cention N and HGI over GCP. The reason for the highest microleakage scores of GCP may be due to high intensity light curing that may resulted in water evaporation. Ülker et al. [32] observed lesser microleakage scores in high viscosity GIC (EQUA Fil, C Corp.; Tokyo, Japan) when compared to GCP. Moreover, Munerveroglu et al. [32] evaluated microleakage of resin-modified GIC, self-adhesive composite resin GCP, and found more microleakage scores in GCP. This might be explained by a weak adhesion between tooth and GCP. Shebl et al. [31] showed lower bond strength results of GCP when compared to conventional GIC. Also, Munerveroglu et al. [32] observed lesser microleakage scores in high viscosity GIC (EQUA Fil, C Corp.; Tokyo, Japan) when compared to GCP. Moreover, Cehreli et al. [33] conducted a study evaluating primary molars, and found higher microleakage scores in GCP, which presented internal and surface's cracks when compared to GIC.

It has been suggested that polyacrylic acid increase adhesion between GCP and tooth substrate, just like GICs [29]. Therefore, in the present study, to enhance bond strength and lessen microleakage scores of GCP, polyacrylic acid conditioning on enamel and dentin surfaces was performed. GCP with pre-treatment in both enamel and dentin demonstrated statistically significantly lesser microleakage scores than GCP without pre-treatment. In line with this study, Ayar et al. [29] reported statistically significantly lesser bond strengths to enamel and dentin, when using GCP without any surface pre-treatment.

Limitations of the present study included intraoral conditions, which could affect the microleakage. Moreover, in this study, extracted teeth were evaluated, and moisture arising from the pulp chamber was reduced. Indeed, clinical evaluations provide better knowledge about the performance of materials for preliminary studies performed in vitro [31]. Therefore, both in vitro and in vivo future studies are recommended, investigating self-adhesive materials and their sealing ability.

CONCLUSIONS

None of the tested materials were free from microleakage. In this present study, the authors revealed that Cention N is a promising alternative restorative material with lesser microleakage over HIC and GCP, and comparable properties with nano-hybrid composite resin. Particularly in enamel, Cention N combined with an adhesive, is preferred. Clinicians also should keep in mind that conditioning with polyacrylic acid improves sealing ability and decreases microleakage scores of HGI and GCP restorative materials. Therefore, additional pre-treatment is recommended especially in stress bearing areas.

CONFLICT OF INTEREST

The authors declare no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

References