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BOND STRENGTH OF GLASS-HYBRID AND GLASS-IONOMER MATERIALS TO SOUND AND CARIES-AFFECTED PRIMARY DENTINE

KOHEZIJSKA TRDNOST HIBRIDNIH MATERIALOV NA OSNOVI STEKLENIH IN STEKLO-IONOMERI VEZIV ZA ZDRAVO IN S KARIESOM OGROŽENO PRIMARNO DENTALNO MEDICINO

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Less invasive caries management is recommended for primary dentition, but little is known about how primary dentine demineralisation affects the adhesion of new formulations of glass-ionomer (GI) restorative materials. The study aimed to evaluate the microtensile bond strength (μTBS) of a glass-hybrid (GH), high-viscosity (HV-) and resin-modified (RM-) GI restoratives to sound (SD) and caries-affected (CAD) primary dentine. Occlusal cavities were prepared in 60 primary molars and randomly divided into SD and CAD. Teeth were divided into subgroups (n=6) and restored with a GH (Equia Forte HT), two HV-GI (Equia Fill, Ketac Molar), and two RM-GI (Fuji II LC, Photac Fill) materials. After thermal aging, μTBS and mode of failure were analysed. The overall bond strength was higher for SD than for CAD ($p<0.05$, Mann-Whitney test). For both SD and CAD, Equia Forte and Equia Fill showed higher μTBS compared to other GI restoratives ($p<0.05$, Kruskal-Wallis test). The most frequent mode of failure was adhesive, followed by mixed failure, with no differences between the subgroups ($p>0.05$, chi-square test). Novel restorative formulations are likely to provide better bonding properties to both primary SD and CAD compared to previous generations of GI materials.

Keywords: glass-hybrid cement, glass-ionomer cement, primary dentine, adhesion

V novejšem času je trend uporabe manj invazivnih posegov pri zdravljenju oziroma odstranitvi zobne gnilobe (caries) v primarnem zobozdravstvu (stomatologiji). Toda malo je še znanega, kako na primarno demineralizacijo zobovine vpliva adhezija novih sestav (formulacij) polnil na osnovi steklo-ionomernih (GI; angl.: glass-ionomer) hibridnih materialov za popravilo zob. V tem članku avtorji opisujejo študijo, ki je pomagala ovrednotiti mikro-natezno vezivno (kohezijsko) trdnost (μTBS) hibrida na osnovi stekla (GH), visoko viskoznost (HV-) in modifikacijo vezivne smole (RM-) GI za zdrave (SD) in s kariesom povzročeno (CAD) primarno zobovino. Avtorji so pripravili okluzivne jamice (pripravljene za polnilo) na 60 primarnih kočnikih in jih nato naključno porazdelili v: SD in CAD. Zobe so porazdelili v podskupine (n=6) in jih obnovili s komercialnimi materiali: GH (Equia Forte HT), dve HV-GI (Equia Fill, Ketac Molar) in dve z RM-GI (Fuji II LC, Photac Fill). Po toplotni obdelavi so avtorji raziskave ugotavljali μTBS in način zloma oziroma odpovedi. V splošnem so ugotovili, da je kohezijska trdnost za SD višja kot je pri CAD ($p<0.05$, Mann-Whitney test). Pri obeh; SD in CAD, materiala Equia Forte in Equia Fill imata višjo μTBS v primerjavi z ostalimi GI materiali za popravilo oz. obnovo ($p<0,05$, Kruskal-Wallis test). Najbolj pogost vzrok za okvaro oziroma odpoved je bila adhezija, tej je sledil mešani način odpovedi. Pri tem pa avtorji niso opazili razlik med podskupinami ($p>0,05$, angl.: chi-square test). Nove formule (sestave) materialov za obnovo zobovine imajo očitno boljše kohezijske (vezavne) lastnosti, tako pri primarnem SD kot tudi CAD v primerjavi s prejšnjo generacijo GI materialov.

Keywords: steklo-hibridni cement, steklo-ionomerni cement, primarna dentalna medicina, adhezija

1 INTRODUCTION

Restorative glass-ionomer cements (GIC) are highly suitable for use in paediatric dentistry due to the possibility of minimal cavity preparation, a relatively short and simple application procedure, and a reduced occurrence of secondary caries.[1] Brittleness and relatively low wear resistance are often considered as the main drawbacks of conventional (C) GIC.[2] Different modifications, such as metal-reinforced GIC (cermets), resin-modified (RM) GIC, high-viscosity (HV) GIC, and, recently, glass-hybrid (GH) materials were introduced to overcome the disadvantages of conventional formulations.

With the invention of HV-GIC, the use of GIC has been extended to the posterior region of permanent dentition, i.e., occlusal and small proximal cavities. A high powder-liquid ratio and an increased molecular

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weight of the polyacid ensure improved physical properties of the HV-GIC.[3] GH technology comprises two types of fillers: fluor-alumino-silicate glass particles of the same structure as in HV-GIC, and ultrafine (<4 μm) glass particles dispersed evenly in the structure of the glass powder. Polyacrylic acid of the GH cements has a higher molecular weight compared to the HV cements. These characteristics reportedly create a restorative material of enhanced microhardness and flexural strength and similar compressive and tensile strength compared to HV-GIC.[4]

The restoration of primary teeth is among the main indications for all types of restorative GIC. There is a general agreement that C-GIC show higher failure rates, while RM-GIC show clinical success comparable to resin composites in regards to cavity restoration in primary teeth.[5–8] Nevertheless, previous studies evaluated older GI formulations, which are rarely used today, and the literature still lacks information about the performance of novel GI formulations as direct restorative materials in primary teeth.

Restorative materials bond to various substrates in clinical conditions. It has been adopted that sound tooth tissues and dental tissues that may be remineralised should be preserved during the carious tissue removal.[9] Dentine caries consists of two layers that have different morphological, biochemical, bacteriological and physiological characteristics. The outer layer is irreversibly changed, characterized by intense demineralisation, denatured collagen fibres, and is infected. The inner carious dentin is reversibly demineralised, shows an unaltered collagen network, it is not infected, and may be remineralisable.[10] Clinically, the golden aim is to remove the infected and necrotic layer to control the progression of the lesion and in order to assure adequate support to the restoration, while leaving the affected layer to preserve the integrity of the tooth structure. Therefore, restorative materials are required to adhere to different surfaces including caries-affected dentine (CAD), characterized by reduced mineral content, increased porosity and altered collagen structure.

The current evidence recommends less-invasive caries management in primary teeth, such as selective caries removal or no caries removal (Hall technique),[6] and the placement of restorative material over caries-affected (CAD) or caries-infected dentine. The bond strengths of composite materials are usually lower to CAD than to sound dentine, and even lower with caries-infected dentine, because the hybrid layer, although thicker, is poorly infiltrated.[11,12] On the contrary, it has been reported that GIC chemical bonding is not affected by partial dentin demineralisation. GIC chemically bonds to the calcium ions in hydroxyapatite (present in both sound and demineralised dental tissues) thus being able to bond undisturbedly to both SD and CAD.[13] GIC seals the lesion and enables pulpo-dentinal complex to heal.[14] Still, the effect of dentine demineralisation on the adhesion of restorative materials has not been extensively studied in primary dentition.

The aim of the study was to evaluate the microtensile bond strength (μTBS) of a GH, HV- and RM-GI restoratives to sound (SD) and CAD primary dentine.

2 EXPERIMENTAL PART

The study was approved by the local ethics committee (Document 36/1 issued on March 7, 2022). Intact human primary molars extracted for exfoliation or orthodontic reasons were used for the study. After extraction, soft debris and periodontal ligaments were removed, and each tooth was inspected for cracks, hypoplasia and white spot lesions. The teeth were stored in physiological saline at +4 °C until used for experiments (up to 1 month after extraction).

The same operator performed all the experimental procedures. Occlusal cavities (4 × 3 × 2) mm were prepared in sixty primary molars. Cavity dimensions were measured with a ruler and marked on the occlusal surface before preparation. Four reference points were placed to guide the creation of a cavity approximately 3 mm in width and 4 mm in length. Preparation was performed using a diamond bur ISO 806 314 173 524 016 (Diaswiss S.A., Nyon, Switzerland) under water cooling, respecting the tooth morphology. A depth of 2 mm was achieved by measuring with an endometer and placing a stopper on the bur to maintain precision.

Teeth were randomly divided into two groups: SD and CAD. Teeth were embedded in the condensation silicone (Zetaplus, Zhermack SpA, Badia Polesine, Italy), so the cavity floors were parallel to the floor. CAD was formed by administering drops of demineralising solution (1.5 mM $CaCl_2$, 0.9 mM KH_2PO_4, 50 mM acetic acid; pH=4.5 (adjusted with NaOH) into the cavity (up to 1 mm) for 7 days at the room temperature.[15] Cavities were regularly examined for evaporation of the demineralising solution (every 12 hours), and the solution was replenished accordingly. Teeth were randomly assigned to the groups (n=6), and restored with a GH (Equia Forte HT, GC Int, Tokyo, Japan- EF), two HV-GI (Equia Fill, GC Int, Tokyo, Japan- E; Ketac Molar, 3M ESPE, St. Paul, MN, USA- KM), and two RM-GI (Fuji II LC, GC Int, Tokyo, Japan- F-II, Photac Fill, 3M ESPE, St. Paul, MN, USA- PF) materials according to manufacturers' instructions (**Table 1**).

Samples were immersed in artificial saliva (1.2 mmol/L $CaCl_2$, 0.72 mmol/L KH_2PO_4, 30 mmol/L KCl; pH = 7.0 (adjusted with KOH))[16] at 37 °C for 7 days (CIBAC45 Cooling circulating bath, COLO, Novo Mesto, Slovenia), and subsequently exposed to the thermal aging (10.000 cycles in aqueous media[17] 5 °C–55 °C–5 °C with a corresponding dwell sequence 30 s–1 s–30 s–1 s–30 s (ThermocycleR v1.0, Belgrade, Serbia)).

Table 1: Restorative materials

Group	Material type	Clinical procedure
A) Equia Forte HT	glass-hybrid restorative	1. Preparation of the tooth Cavity was conditioned with 10 % polyacrylic acid (GC Dentine Conditioner, GC Int.) for 20 s using a cotton pellet, rinsed thoroughly with water and gently air-dried with an air syringe 2. Application of the material Capsule was activated, set into the amalgamator, mixed for 10 s at high speed, and the mixed capsule was loaded into the capsule applier; the material was syringed directly onto the tooth 3. Setting of the material Contours of the restoration were formed and the material was allowed to set for about 2 min. 4. Protection of the material External surfaces of the material were protected with coat (Equia Forte HT Coat, GC Int.), which was light cured for 20 s
B) Equia Fill	high-viscosity conventional restorative GIC	1. Same as A.1. 2. Same as A.2. 3. Same as A.3. 4. External surfaces of the material were protected with coat (Equia Coat, GC Int.), which was light cured for 20 s
C) Fuji II LC	light-cured resin-modified restorative GIC	1. Same as A.1. 2. Same as A.2. 3. Contours of the restoration were formed and the material was light-cured for 20 s 4. External surfaces of the material were coated with varnish (Fuji Coat LC, GC Int.), which was light cured for 10 s
D) Ketac Molar	high-viscosity conventional restorative GIC	1. Preparation of the tooth Cavity was conditioned with 20% polyacrylic acid (Ketac Conditioner, 3M ESPE) for 10 s using a cotton pellet, rinsed thoroughly with water and gently air-dried with an air syringe 2. Same as A.2. 3. Same as A.3.
E) Photac Fill	light-cured resin-modified restorative GIC	1. Same as A.2. 2. Same as C.3.

Samples were sectioned in mesial/distal and buccal/lingual direction across the adhesive interface using a water-cooled diamond-impregnated low-speed saw (Isomet Low Speed Saw; Buehler, Lake Bluff, IL, USA) to form twelve 0.9×0.9 mm stick microspecimens per group. The thickness of sticks was examined with a digital nonius (Digital caliper 0-150 mm, ZKG, Hong Kong, China). Microspecimens were allowed to age in artificial saliva[16] at 37 °C for one week (CIBAC45 Cooling circulating bath, COLO).

Microspecimens that de-bonded during sectioning were recorded as "pre-testing failure".

Sticks were fixed to the testing machine (BISCO Dillon Quantrol, Fairmont, MN, USA) with an acrylate glue (Loctite Super Bond Liquid, Henkel, Düsseldorf, Germany), and tested at a loading rate of 0.5 mm/min until failure. μTBS was expressed in MPa as imposed force at the time of failure (N) by the bonding area (mm^2). Mode of failure (adhesive, cohesive, mixed) was analysed using optical digital microscope (5M 300x USB Digital Microscope, Mustech Electronics, Shenzhen, China).

The SPSS program ver. 22 was used for the data analysis. Numerical data were described as mean, median, standard deviation, and minimum and maximum. The One-sample Kolmogorov Smirnov test was used to examine the normal distribution. The Kruskal-Wallis test was utilized for multiple comparisons between groups, while the Mann-Whitney U test was used for comparisons between two groups. Attributive data were compared using the chi-square test. Statistical significance was set at 0.05. The sample size was calculated based on a pilot study of 50 samples for the bond strength outcome (ANOVA fixed effects, one way) where the difference between 5 mean values (5 materials) was compared for a significance level of 0.05 and a study power of 95 %. The sample size was determined to be 20 specimens for each material (10 specimens per subgroup) (G*Power program 3.1.9.4. Germany).

3 RESULTS

Results of the μTBS testing are shown in **Table 2**. The overall bond strength was higher for SD than for CAD ($p<0.05$, Mann-Whitney test). When μTBS to SD and CAD was compared for individual materials, difference was significant for E and PF ($p<0.05$, Mann-Whitney test), while EF, F-II and KM showed no differences. For both SD and CAD, EF and E showed higher μTBS

Table 2: Microtensile bond strength [MPa]

	Sound dentine		Caries-affected dentine	
	mean (±SD)	median (range)	mean (±SD)	median (range)
Equia Forte HT	4.05 (3.47)[b]	4.88 (0–7.56)	3.73±2.61[d]	4.28 (0–7.56)
Equia Fill[a]	5.08 (3.20)[c]	6.74 (0–8.06)	2.41±2.18[e]	3.42 (0–4.80)
Ketac Molar	1.40 (1.47)[b,c]	1.23 (0–3.10)	0.79±0.99[d,e]	0 (0–2.18)
Fuji II LC	3.27 (2.96)[b,c]	4.44 (0–6.62)	1.93±2.08[d,e]	1.58 (0–5.25)
Photac Fill[a]	3.12 (2.57)[b,c]	3.30 (0–6.42)	0.93±1.16[d,e]	0 (0–2.53)

[a] significant difference for individual material between SD and CAD (*p<0.05*, Mann-Whitney)
[b,c] significant difference between the materials marked with the same letter in the sound dentine group (*p<0.05*, Mann-Whitney test)
[d,e] significant difference between the materials marked with the same letter in the caries-affected group (*p<0.05*, Mann-Whitney test)

compared to other GI restoratives (p<0.05, Kruskal-Wallis test).

No significant differences with regard to pre-test failure were noticed either between SD and CAD or among the materials tested (p>0.05, chi-square test).

The most frequent mode of failure was adhesive, followed by mixed failure, with no differences between the subgroups (p>0.05, chi-square test; **Figure 1**).

4 DISCUSSION

A strong bond between the tooth structure and the restorative material is crucial for the longevity of a dental restoration. It ensures not only good mechanical properties, but also biological and esthetical success of the restoration.[18] Although the correlation of bond strength and clinical retention of the restorations is uncertain, laboratory studies may give a valuable insight into the physical and mechanical properties of dental materials.[19] A number of techniques is available to test the bond strength. The μTBS test is currently considered as the most appropriate *in-vitro* measure of a material's retention.[20] It enables better stress distribution at the tooth-material interface, and better control of potential varieties between diverse dentine regions.[21] However, preparation of micro-specimens might be sensitive, especially in brittle materials with lower bond strength such some of the GI materials are. Therefore, a higher proportion of pre-testing failures might be expected during the μTBS testing of GIC.

Several procedures were performed to simulate clinical conditions in the present study. The first one was formation of CAD which is a common substrate for restorative material adhesion in paediatric dentistry. Secondly, the tested materials were applied into the prepared cavities instead of placing them in a bulk on a flat dentin surface. The advantage of this step might also be the smaller number of pretesting failures that could have been expected due to delicacy of the primary dentine-GIC interface. In the end, considering that subjecting the specimens to a durability challenge is recommended to simulate clinical conditions,[20,21] specimens were subjected to thermal aging.

Bonding of GIC to primary CAD has not received much attention so far. Calvo et al.[22] reported no differences in immediate and long-term (24 months) μTBS of C-GIC, RM-GIC, and nano-ionomer bonded to either SD or CAD. Similarly, Marquezan et al.[23] found that bonding of a RM-GIC was not influenced by the substrate. By contrast, Çehreli et al.[18] showed significantly lower long-term (18 months) μTBS of RM-GIC bonded to primary CAD. The overall results of the present study show higher bond strengths to SD compared to CAD, with significant differences between the two substrates in E and PF groups. In addition, the variability of absolute values of μTBS has been reported in the above-mentioned studies. As a result, drawing reliable conclusions from the available research on bond strength tests between primary dentin and GI materials is challenging due to the limited research available. It is advisable to interpret the

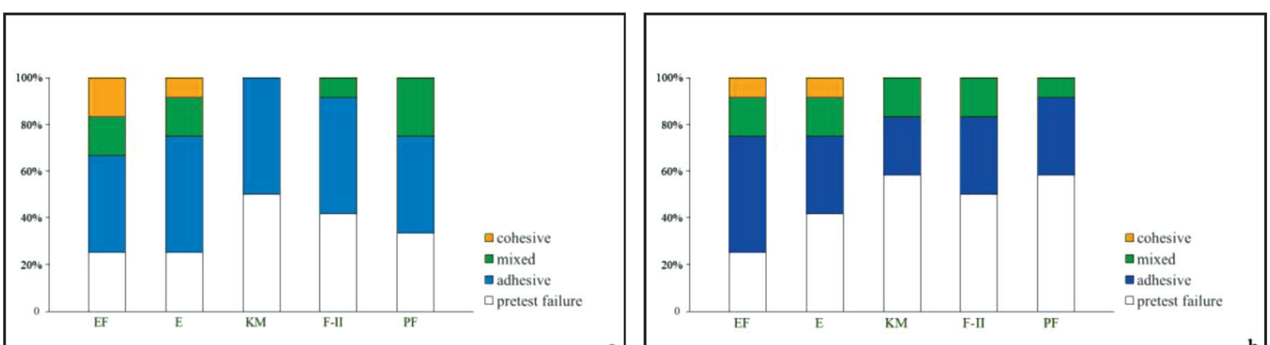

Figure 1: Mode of failure [%] of restorative materials bonded to a) sound dentine, and b) caries-affected dentine EF- Equia Forte HT, E- Equia Fill, KM- Ketac Molar, F-II- Fuji II LC, PF- Photac Fill

results within the context of each study rather than as absolute benchmarks.

Bonding properties of RM-GIC to primary enamel and dentin have been reported to be superior to those of C-GIC.[24] Those materials bond through two mechanisms: chemically and micromechanically, which may favour the durability of the adhesive interface.[22] Therefore, RM-GICs are favoured for the restoration of primary teeth.[5,7,8] In the present study, the novel formulations EF and E, demonstrated increased bond strength to both SD and CAD in comparison to RM-GIC. In addition, the fracture resistance of GH has been reported to be higher than C-GIC when used to restore primary teeth, with no differences between complete and selective caries removal to firm dentin.[25]

Similar to the present study, Calvo et al.[22] showed the predominance of adhesive/mixed failure mode of C-GIC and RM-GIC to both primary SD and CAD. Puwanawiroj et al.[26] also reported the prevalence of mixed/adhesive failures of a HV-GIC to CAD. On the contrary, Burrow et al.[27] described the predominance of cohesive failures into the cement. Although any conclusion could not be drawn from such a small number of the studies that used primary teeth as a substrate, this difference might be related to the different specimen preparation for the μTBS testing. It could be speculated that hourglass shaping (used by Burrow et al.) created additional stress areas within the material that led to crack formation and propagation during the testing, thus favouring cohesive failures.

Tests on permanent teeth reported that GIC tended to fail cohesively within the material, implying that the GIC-tooth bond strengths were higher than the strengths within the GIC bulk.[28] The weaker bond between the GIC and the primary teeth compared to the permanent teeth might explain predominance of adhesive failures in the present study. The occurrence of cohesive fracture in EF and E group could imply higher bonding of these materials to the primary dentine.

The authors acknowledge that not testing the immediate bond strength is a limitation of the present study. The reason for this is that our intention was to reproduce clinical conditions as much as possible and to examine "aged" bond strength. This is believed to predict better the long-term clinical performance of the materials tested.[21,29]

5 CONCLUSIONS

Within the limitations, this *in vitro* study suggests better GIC bonding to SD than to CAD. Compared to GIC, GHC demonstrates less variations in bonding between SD and CAD. Novel restorative GI and GH formulations appear to have superior adhesion properties to both SD and CAD.

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