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Original article

Effect of Mechanical Loading and Simulated Aging on the Microleakage of Metal-Reinforced and Experimental Glass Ionomer Cements

Mohamed Abour

Faculty of Medicine, Al-Joufra University, Libya
Email. *Mohdabour@ju.edu.ly*

Keywords.
Glass-Ionomer Cement; Metal-Reinforced Glass-Ionomer; Microleakage; Dye Penetration; Class II Restoration; Mechanical Loading.

Abstract

Marginal microleakage is commonly used as an in vitro indicator of restorative materials' sealing performance, although it does not directly predict clinical longevity. Glass-ionomer cements (GICs) chemically interact with tooth mineral and are used where adhesion and fluoride release are advantageous. This study compared dye penetration around a metal-reinforced GIC (Hi-Dense) and an experimental high powder-to-liquid ratio GIC (EXPT), before and after mechanical load cycling. We received 80 freshly extracted, non-carious human molars, prepared Class II, and restored with one of the two GICs. For each material, specimens were allocated to an unloaded control or to load cycling intended to represent one day, one week, or one month. Specimens were coated with nail varnish except for a 1-mm perimeter around the restoration, immersed in 0.1% neutral red for 24 h at 37°C, sectioned, and examined at ×15 magnification. Dye penetration was scored separately at cervical and occlusal margins. Group comparisons were analysed using the Mann–Whitney test with a 95% confidence level. Dye penetration was generally limited, and no section received a score greater than 2. At the occlusal margin after one month of cycling, EXPT showed greater leakage than Hi-Dense (reported $p \leq 0.002$). At cervical margins, EXPT showed greater penetration than Hi-Dense at the reported one-day ($p \leq 0.002$) and one-month ($p \leq 0.03$) loading periods. No significant material difference was reported in unloaded specimens. The authors reported no major overall influence of cycling duration on leakage patterns, although occlusal leakage in Hi-Dense was lower after the one-month condition. Under the conditions of this in vitro experiment, the metal-reinforced Hi-Dense material exhibited less dye penetration than EXPT at both margins, particularly under selected loading conditions. These findings support a comparative laboratory advantage for Hi-Dense, but clinical effectiveness cannot be inferred from dye penetration alone.

Introduction

The integrity of the tooth–restoration interface is important to the performance of direct restorations. Marginal gaps and interfacial pathways may permit fluid movement and the passage of small molecules, and have been associated with marginal staining, postoperative sensitivity, marginal degradation, and conditions that can contribute to secondary caries. However, microleakage is a laboratory endpoint rather than a direct measure of clinical failure, and its relationship with long-term outcomes is not straightforward [1–4]. Several laboratory methods have been used to assess leakage, including dye penetration, radioisotope tracers, bacterial or molecular tracers, fluid filtration, microscopy, electrical methods, and ageing protocols involving thermal or mechanical cycling. Dye penetration remains widely used because it is inexpensive and straightforward, but results depend on dye chemistry, exposure time, sectioning plane, scoring criteria, and observer interpretation. Reviews have emphasised the lack of standardisation across microleakage studies and the limited ability of any single in vitro method to predict clinical performance [1–3].

Glass-ionomer cements are acid–base restorative materials formed by the reaction of fluoroaluminosilicate glass with aqueous polyalkenoic acid. Their advantages include adhesion to enamel and dentine, fluoride release, and the development of an ion-exchange zone at the tooth interface [5–8]. Adhesion is generally attributed to interactions between carboxylate groups in the cement and calcium-containing mineral in dental hard tissues. Surface conditioning with polyacrylic acid can remove the smear layer and improve wetting without the aggressive demineralisation associated with some other adhesive approaches [5,6]. Nevertheless, conventional GICs remain sensitive to early moisture imbalance and can have limitations in fracture toughness, wear resistance, and strength under sustained loading [7–10]. Attempts to modify GICs have included changes in powder-to-liquid ratio, glass composition, particle size, and incorporation of metallic particles or metal-containing glass.

Metal-reinforced materials have been developed to improve handling and wear-related performance, although reviews have cautioned that reinforcement does not necessarily improve every mechanical or adhesive property [9,11]. Their sealing behaviour therefore needs to be evaluated directly rather than assumed from their composition alone. Class II restorations provide a useful model because the occlusal and cervical margins are located in different substrates and experience different geometries and stress patterns. Cervical margins may lie in dentine or cementum, where the mineral

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content and substrate structure differ from enamel. In addition, loading of the cusps can deform the tooth–restoration complex and may challenge the marginal interface. Previous work on silver-reinforced GICs has investigated the effects of storage, thermal cycling, and mechanical loading, with results dependent on the material and experimental protocol [12–14]. The present study aimed to compare the in vitro microleakage of a metal-reinforced glass-ionomer cement (Hi-Dense) with an experimental GIC with a high powder-to-liquid ratio (EXPT), at both cervical and occlusal margins, after storage in distilled water and selected periods of mechanical load cycling. The null hypothesis was that the two materials would not differ in dye penetration at either margin under unloaded or loaded conditions. Table 1 shows the mechanical load cycling for one day, one week and one month.

Table 1: Mechanical load cycling

Condition	Number of cycles
Unloaded control	0
One-day equivalent	685
One-week equivalent	4,808
One-month equivalent	20,833

Materials and Methods

Study Design and Specimens

This in vitro comparative study evaluated marginal dye penetration in Class II restorations made with two glass-ionomer cements (GICs): a metal-reinforced commercial material (Hi-Dense) and an experimental material with a high powder-to-liquid ratio (EXPT). Eighty freshly extracted, non-carious human molars were cleaned with an ultrasonic scaler and disinfected in 10% formalin saline for 10 minutes. Each tooth was mounted in an acrylic base. The source manuscript does not report the reason for extraction, the tooth-storage interval before preparation, formal randomisation procedures, or ethics approval.

Cavity Preparation and Restoration

A Class II cavity was prepared on either the mesial or distal surface of each tooth. The buccolingual width of the occlusal portion was at least one-third of the intercuspal dimension, and the buccolingual width of the proximal box was at least one-third of the faciolingual dimension. The occlusal depth was approximately 1 mm into dentine beyond the dentino-enamel junction. The proximal box extended approximately 1 mm below the cemento-enamel junction, and the gingivo-axial width was approximately 1 mm from the gingival tooth surface (Figure 1).

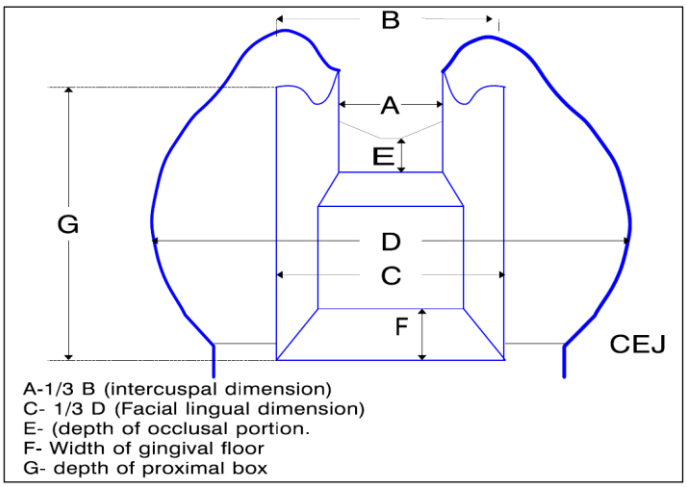


Figure 1. Schematic of the Class II cavity preparation and its principal dimensions.

A, one-third of the intercuspal dimension (B); C, one-third of the faciolingual dimension (D); E, depth of the occlusal portion; F, width of the gingival floor; and G, depth of the proximal box. CEJ, cemento-enamel junction.

The preparations were cleaned with pumice and a prophylaxis brush in a slow-speed handpiece to remove debris, rinsed with deionised distilled water, and dried with oil-free compressed air for 5 seconds. A 25% polyacrylic acid solution was applied for 30 seconds; the cavities were then rinsed with water for 15 seconds and dried for 5 seconds. An automatrix band coated with silicone spray (Releasil) was placed around each tooth as a separating medium. The GIC capsule was activated by applying pressure to the capsule guard for 3 seconds and mixed for 15 seconds in a Silamat Plus amalgamator. The material was removed from the capsule with the supplied spatula and condensed into the cavity using instruments moistened with isopropyl alcohol. Restorations were covered with light-cured protective resin (Hi-Glaze) and

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cured for 20 seconds. Restored teeth were stored in a thermostatically controlled oven at 37°C for 60 minutes before removal of the matrix band. Gross excess material was removed with a 12-fluted carbide bur (Jet FG 7902) using a high-speed handpiece.

Experimental Groups and Mechanical Loading

The 80 teeth were divided into eight groups of 10: four groups for each material. For each material, one group was an unloaded control (n = 10), and three groups underwent mechanical cycling intended to represent one day, one week, or one month of function. Specimens were stored in deionised distilled water before loading. Each tooth was placed under a 12.25-mm-diameter ball bearing attached to the upper crosshead of a universal testing machine (Instron, High Wycombe, UK). The bearing contacted the buccal and lingual cusps without directly contacting the restoration, allowing outward cusp deflection during loading. Teeth remained immersed in water during cycling. The cycle rate was one cycle per second, the crosshead speed was 25 mm/min, and the applied load ranged from 9 to 180 N. The cycle counts were 685 for the one-day condition, 4,808 for the one-week condition, and 20,833 for the one-month condition. These counts were derived from the assumption that 250,000 cycles in an artificial oral environment approximate one year in the human mouth.

Dye Penetration and Sectioning

Following loading, specimens were blotted dry and gently air-dried, taking care to avoid desiccation. Three layers of nail varnish were applied to the external tooth surface, leaving an approximately 1-mm-wide uncoated perimeter around the cavity margin and restoration. Each layer was allowed to dry before the next was applied. The teeth were immersed in 0.1% neutral red dye solution for 24 hours at 37°C. After immersion, they were removed, rinsed, and scrubbed with a brush to remove surface dye, then stored in deionised distilled water at room temperature until sectioning. Each tooth was mounted on a cylindrical holder with sticky wax and sectioned using a 0.2-mm diamond band saw (Exact, Mederex, Germany). One mesiodistal section and four buccolingual cuts were made, producing sections approximately 1 mm thick (Figure 2). The original report states that two section scores were recorded for cervical dye penetration and 16 for occlusal dye penetration. Sections were examined under a Wild photo microscope (Wild Heerbrugg Ltd.) at ×15 magnification.

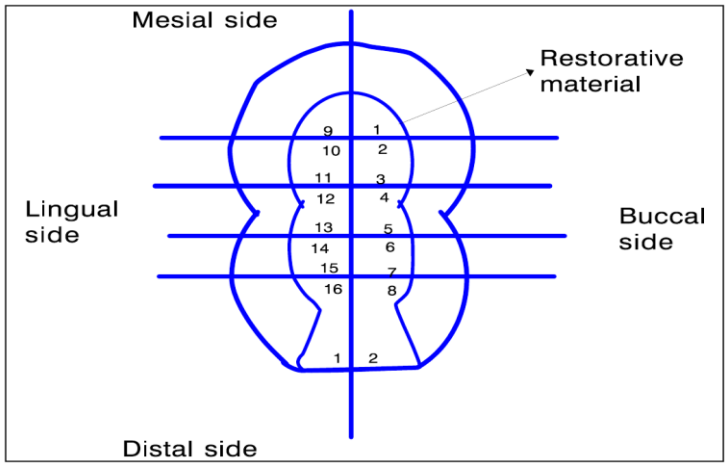


Figure 2. Schematic of the sectioning plan for a restored tooth.

A mesiodistal section and four buccolingual cuts were used to obtain sections for assessment of dye penetration; numbered regions indicate the section locations used in the original protocol.

Microleakage Scoring

Dye penetration along the cervical (C) and occlusal (O) tooth–restoration interfaces was assessed using the ordinal scoring system illustrated in Figure 3. The scale was reported as follows: 0, no dye penetration; 0.5, penetration of less than one-quarter of the cervical floor or occlusal wall; 1, penetration of less than half the cervical floor or occlusal wall; 2, penetration along the full cervical floor or occlusal wall; 3, penetration extending up to half the axial wall at the cervical margin or the occlusal floor; and 4, penetration extending along the whole axial wall at the cervical margin or the occlusal floor.

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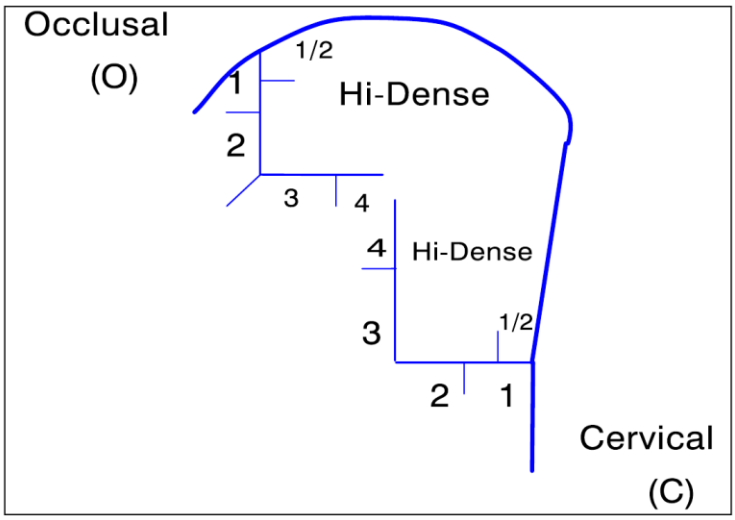


Figure 3. Schematic representation of the original ordinal dye-penetration scoring system at the occlusal (O) and cervical (C) margins.
The diagram illustrates the extent of penetration along the tooth–restoration interface; score definitions are reproduced in Section 2.5.

Statistical Analysis
The original report states that results were summarised using means and medians and that comparisons were performed with the Mann–Whitney test at a 95% confidence level in Minitab, release 6. Exact p-values, effect sizes, confidence intervals, and a clear definition of the statistical unit were not fully reported in the source manuscript. Because multiple sections were obtained from each tooth, section-level observations may be clustered within teeth; tooth-level data or an analysis accounting for this clustering would be preferable.

Materials and Equipment
Hi-Dense and experimental GIC: Shofu, supplied by Advance Healthcare Ltd, Kent, UK. Universal testing machine: Instron, High Wycombe, UK. Acrylic: De Trey/Dentsply. Mechanical mixer: Silamat Plus, Vivadent. Silicone spray: Releasil, Dow Corning Ltd, Barry, UK. Light-curing unit: EuroMax, De Trey/Dentsply. Diamond band saw: Exact, Mederex, Germany. Photomicroscope: Wild Heerbrugg Ltd.

Results
Across the two materials and all experimental conditions, dye penetration was generally limited. No sections were assigned scores greater than 2. The original manuscript reports that four of the 640 sections examined for Hi-Dense and 12 of the 640 sections examined for EXPT received a score of 2. The number of sections examined was 20 per group at the cervical margin and 160 per group at the occlusal margin for one day, one week and one month cycling. . For unloaded specimens, no statistically significant difference between Hi-Dense and EXPT was reported at either margin. At the occlusal margin after the one-month-equivalent loading condition, EXPT showed significantly greater dye penetration than Hi-Dense (reported $p \leq 0.002$). At the cervical margin, EXPT showed greater penetration than Hi-Dense after one day (reported $p \leq 0.002$) and one month (reported $p \leq 0.03$) of cycling. The original manuscript did not report a significant cervical difference at the one-week condition. The original authors reported that mechanical cycling did not produce a major overall change in cervical leakage for either material or in occlusal leakage for EXPT. Occlusal dye penetration in Hi-Dense was lower in the one-month-equivalent group than in the earlier loading groups.

Table 2. Distribution of section-level microleakage scores
Hi-Dense glass-ionomer cement

Cycling condition	C: S0	C: S0.5	C: S1	C: S2	O: S0	O: S0.5	O: S1	O: S2
Unloaded	14	6	0	0	129	25	5	0
One day	13	1	6	0	119	8	30	3
One week	8	6	6	0	123	8	28	1
One month	12	5	3	0	147	8	5	0

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EXPT experimental glass-ionomer cement

Cycling condition	C: S0	C: S0.5	C: S1	C: S2	O: S0	O: S0.5	O: S1	O: S2
Unloaded	7	7	6	0	120	28	12	0
One day	5	10	5	0	115	30	14	1
One week	6	5	9	0	112	18	16	4
One month	4	7	9	0	112	27	14	7

Note. C = cervical; O = occlusal; S = score. The original tables also contained columns for scores 3 and 4; all entries for these scores were zero and are omitted here for readability. Counts are section counts, not independent tooth counts. Cervical and occlusal counts must not be pooled as though they represented equivalent observations.

Discussion

This in vitro study found less dye penetration around the metal-reinforced Hi-Dense GIC than around the experimental high powder-to-liquid ratio material, with statistically significant differences reported at selected cervical and occlusal loading conditions. The findings are consistent with the possibility that formulation and handling characteristics influence the adaptation of GICs to cavity walls. They do not, however, establish that metal reinforcement itself caused the difference, because the two materials may differ in more than the presence of an alloy phase, including powder composition, particle characteristics, viscosity, and working behaviour. GICs adhere to tooth tissues through a combination of ionic interaction and development of an interfacial ion-exchange zone. This interaction is influenced by substrate mineral content, surface preparation, cement maturation, and the quality of contact at placement [5–8]. The enamel margin generally offers a more mineral-rich substrate than dentine or cementum, whereas the cervical margin in this Class II design extended below the cemento-enamel junction.

Greater leakage at cervical margins is therefore biologically and technically plausible, and has also been described in other Class II microleakage investigations [13]. Nonetheless, the present study's cervical and occlusal observations cannot be directly compared quantitatively because different numbers of sections were scored at the two margins and the scoring geometry differed. Metal-reinforced GICs have been studied as an approach to improving the clinical handling and wear performance of conventional materials. A review by Wasson noted that improvements in wear resistance were reported, but that improvement in strength or adhesion was not consistently demonstrated and that methodological variation limited comparisons [11]. The present results add a material-specific observation about dye penetration, rather than proving a general benefit of alloy addition. The more condensable handling of Hi-Dense, described by the original investigators, may have helped the cement wet and adapt to cavity surfaces. This is a plausible explanation, but adaptation was not measured directly and should be treated as a hypothesis. The absence of a consistent increase in leakage with increasing load duration may reflect the limited magnitude and geometry of the applied loading, the conditioning period before cycling, or the characteristics of the materials. The machine applied axial force through the buccal and lingual cusps, whereas clinical mastication involves multidirectional contacts, changing contact points, thermal fluctuations, saliva, pH changes, and repeated chemical and mechanical challenges. The experimental setup therefore represents a controlled mechanical challenge rather than a full simulation of the oral environment [15–19].

Prior work on Hi-Dense has also examined storage, thermal cycling, and mechanical loading, reinforcing the importance of considering the specific ageing protocol when comparing microleakage outcomes [12–14]. The lower occlusal leakage observed for Hi-Dense after the one-month-equivalent condition might be related to continued maturation of the cement or random variation in a small sample. GIC properties evolve after initial setting as the acid–base reaction progresses, and early water balance can affect surface and mechanical properties [7,8,21]. However, the present data do not allow maturation to be isolated from other factors, and the apparent reduction should not be interpreted as evidence that mechanical loading improves the seal.

Dye penetration is a useful screening method but has recognised limitations. Dye molecules are smaller than bacteria; dye uptake can depend on chemical interactions with the material, and sectioning may miss or exaggerate local pathways. The literature documents substantial variation in tracers, exposure conditions, sectioning, scoring, and statistical methods, making cross-study comparisons difficult [1–4]. A score of zero means no dye was detected in the examined section under the chosen protocol; it does not prove an impermeable interface or predict long-term clinical success. Similarly, a higher score indicates greater dye penetration under these conditions, not necessarily a directly proportional increase in secondary caries risk. Several methodological limitations should be acknowledged. First, the study used extracted teeth and an in vitro endpoint, so clinical outcomes cannot be inferred. Second, the sample size was 10 teeth per material-condition group, and no a priori sample-size calculation was reported. Third, the report does not describe randomisation, allocation concealment, blinded scoring, intra- or inter-examiner reliability, or handling of missing specimens. Fourth, multiple sections were scored per tooth; treating each section as an independent observation would underestimate uncertainty if clustering was not accounted for. Fifth, exact p-values, effect sizes, confidence intervals, and full specimen-level data were not supplied. Sixth, the control condition and loaded conditions differed by cycling exposure, but the original report does not fully describe all storage durations and whether they were identical

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across groups. Finally, the experimental GIC is not fully characterised, limiting reproducibility and preventing attribution of differences specifically to the alloy component. Future studies should report full material composition and mixing details, use randomised allocation and blinded scoring, prespecify a sample-size calculation, and report tooth-level data alongside section-level observations. More standardised ageing protocols, thermocycling, multidirectional mechanical loading, quantitative image analysis, and complementary methods such as fluid filtration or microscopy could provide a broader assessment. Clinical studies would ultimately be needed to establish whether laboratory differences correspond to restoration survival or patient-relevant outcomes [2–4,22–24].

Conclusion

Within the limitations of this in vitro study, the metal-reinforced Hi-Dense glass-ionomer cement showed less dye penetration than the experimental high powder-to-liquid ratio GIC at both cervical and occlusal margins, with statistically significant differences at selected loading conditions. Mechanical cycling did not produce a consistent increase in leakage across all groups. The findings suggest better marginal sealing by Hi-Dense under the specific experimental conditions used; they do not establish clinical superiority or prove that alloy addition alone caused the difference. Further studies with complete raw-data reporting and clinically relevant ageing protocols are warranted.

Conflict of interest. Nil

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