

Can Resin Coatings Improve the Flexural Properties of Highly Viscous Glass Ionomer Cements?

(Bolehkah Salutan Resin Meningkatkan Sifat Lentur Simen Ionomer Kaca Sangat Likat?)

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ABSTRACT

This study examined the effect of self-adhesive resin coatings on the flexural properties of three highly viscous glass ionomer cements (HVGICs), specifically Shofu Zirconomer (ZR), GC Equia Forte (EQ), and SDI Riva Self Cure (RV). Custom-made Telfon molds were used to produce 60 beam-shaped specimens ($12 \times 2 \times 2$ mm) for each material. The specimens were finished, measured, and randomly divided into three groups of 20. Ten specimens in each group were left uncoated, while the remaining ten were coated with their respective manufacturers' resin coats. All specimens were immersed in distilled water, artificial saliva or citric acid at 37 °C for seven days and subjected to flexural testing with a 5 KN load at a crosshead speed of 0.5 mm/min till fracture occurred. Flexural data were analysed using one-way ANOVA/Tukey's post-hoc test and independent sample T-test ($\alpha=0.05$). Mean flexural modulus ranged from 0.72 ± 0.13 to 13.19 ± 1.00 GPa while mean flexural strength ranged from 4.32 ± 0.84 to 45.83 ± 4.82 MPa after immersing in the different mediums. The flexural modulus/strength of uncoated ZR and RV were generally comparable or greater than when coated. However, EQ was mostly improved when coated regardless of immersion mediums. RV and EQ generally offered the best flexural performance when uncoated and coated respectively.

Keywords: Flexural properties; highly viscous glass ionomer cement; resin coatings

ABSTRAK

Impak salutan resin terhadap sifat lenturan simen kaca ionomer (HVGIC) telahpun dikaji. Bahan kajian terdiri daripada Shofu Zirconomer (ZR), GC Equia Forte (EQ) dan SDI Riva Self Cure (RV). Untuk setiap bahan, sebanyak 60 spesimen yang berbentuk rasuk ($12 \times 2 \times 2$ mm) telah dihasilkan dengan menggunakan acuan yang dibuat khas. Semua spesimen dilicin, diukur dan dibahagikan secara rawak kepada tiga kumpulan. Daripada 20 spesimen yang terkandung di dalam setiap kumpulan, sepuluh spesimen telah disalut dengan resin khas masing-masing, manakala spesimen lain tidak disalut. Selepas itu, semua spesimen direndam dalam air suling, air liur tiruan dan asid sitrik selama tujuh hari pada suhu 37 °C. Kajian sifat lenturan dijalankan dengan beban sebanyak 5KN dan kelajuan kepala melintang 0.5 mm/min sehingga kepatahan berlaku. Data sifat lenturan dianalisis dengan ujian sehala *post-hoc* ANOVA/Tukey's dan sampel tak bersandar T-test ($\alpha=0.05$). Purata sifat lenturan modulus adalah antara 0.72 ± 0.13 hingga 13.19 ± 1.00 GPa, manakala purata sifat lenturan kekuatan adalah antara 4.32 ± 0.84 hingga 45.83 ± 4.82 MPa, selepas direndam pada pelbagai rendaman. Sifat lenturan ZR dan RV yang tidak disalut didapati setanding atau lebih baik berbanding dengan yang disalut dengan resin. Walau bagaimanapun, EQ didapati bertambah baik apabila disalut dengan resin, tanpa mengambil kira jenis rendaman. RV menawarkan sifat lenturan terbaik apabila tidak disalut, sebaliknya EQ menawarkan sifat lenturan terbaik apabila disalut dengan resin.

Kata kunci: Salutan resin; sifat lenturan; simen kaca ionomer

INTRODUCTION

The introduction of glass ionomer cements (GICs) by Wilson and Kent in the early 1970s was much welcomed as they have several noteworthy features such as chemical adhesion to tooth structure and fluoride ion release/recharge that offers cariostatic ability (Mousavinasab & Meyers 2009; Wilson & Kent 1972). However, conventional GICs showed poor mechanical properties, low wear resistance as well as moisture sensitivity during the early phase of setting (Sidhu & Nicholson 2016). Thus, it cripples their suitability for the restoration of posterior dentitions. To overcome, efforts were made to improve their physico-mechanical properties by optimizing powder/liquid ratios and incorporating different fillers (Friedl et al. 2011), culminating in the development of highly viscous glass ionomer cements (HVGICs). Studies on the clinical performance of HVGICs had indicated comparable success rates between HVGICs and amalgam in posterior restorations (Gurgan et al. 2015; Mickenautsch 2016). Given this promising evidence, HVGICs can potentially replace the unaesthetic silver amalgam restorations.

Water is a critical component of HVGICs. It acts as the solvent for polyacrylic acid and the medium in which the acid-base setting reaction takes place. Eventually, water is an integral part of the set material (Sidhu & Nicholson 2016). However, GICs are vulnerable to both moisture contamination and dehydration during their early setting phase. These phenomena give rise to micro-cracks, volumetric changes, and surface erosions, which can significantly compromise mechanical properties of it (Lohbauer et al. 2011; Nicholson & Czarnecka 2008). Prior means of mitigating early water contamination and loss involved the application of varnish or petroleum jelly but they were susceptible to salivary wash-out and loss during function (Gorseta et al. 2016). With the application of resin coatings on GICs, it can effectively overcome the early loss of a protective coat. Multiple studies have indicated possible enhancements to surface hardness, flexural strength as well as aesthetics by the minimization of water contamination, elimination of voids, the provision of smooth and lustered surfaces (Bonifácio et al. 2012; Diem et al. 2014; Sukumaran & Mensudar 2015).

Since HVGICs with self-adhesive resin coatings had been promoted as materials with enhanced durability for posterior teeth, characterization of their capacity for stress loading is essential to ensure structural integrity and longevity of restorations (Šalinović et al. 2019). However, the mechanical properties of restorative materials are

also influenced by their surrounding oral environments. It is therefore prudent to expose restorative materials to different immersion mediums to better depict their potential clinical performance (Briso et al. 2011; Kooi et al. 2012). Given the scarcity of data on the relation of contemporary HVGICs and their chemical environment, this study aimed to verify the effects of various chemical environments on the flexural properties of resin coated and uncoated HVGICs. Our null hypotheses were: (a) the flexural properties of HVGICs are not affected by resin coating, (b) there is no difference in flexural modulus and strength among different HVGICs, and (c) chemical environments do not influence the flexural properties of HVGICs.

MATERIALS AND METHODS

LIST OF MATERIALS AND SAMPLE SIZE CALCULATION

Table 1 presents the technical profiles of the assessed HVGICs (Zirconomer [ZR], Equia Forte [EQ], and Riva Self Cure [RV]) and their respective manufacturers' resin coats (C).

A minimum total sample size of 108 (i.e. $n=8$) was established with the G*Power Software version 3.1.9.331. Based on a previous study by (Yap et al. 2021), the effect size is hypothesised from the formula $\frac{M_1 - M_2}{s}$, where $M_1 - M_2$ is the difference between group means; s is the standard deviation of either group. The total sample size was generated via analysis of variance (ANOVA) model with an effect size of 0.5, alpha error of 0.05, and power of 95% for 18 groups.

SPECIMEN PREPARATION AND IMMERSION PROTOCOL

Custom Teflon moulds were used to fabricate 60 beam-shaped specimens according to Mini Flexural Test specifications ($12 \times 2 \times 2$ mm) for each material as it has been reported that mini-flexural test may be more clinically relevant (Yap et al. 2018). The materials were manipulated according to manufacturers' instructions, compacted into custom-made moulds, compressed between two glass slides, and allowed to self-cure for 5 min at 27 °C (i.e. ambient temperature). After removal from their moulds, the specimens were finished with fine polishing discs (Sof-Lex, 3M ESPE, St Paul, MN, USA). A digital calliper (ABS Digimatic, Mitutoyo, Kawasaki, Japan) was used to measure the specimens and to ensure uniformity and parallelism of the opposing surfaces. Sixty specimens of each HVGIC were fabricated and randomly distributed into three groups of twenty.

TABLE 1. Technical profiles and manufacturers of the materials evaluated

Material (Abbreviation)	Manufacturer	Type and Curing Method	Resin/Liquid	Filler/Powder
Zirconomer (ZR)	Shofu Inc. Koyoto, Japan	Zirconia/ reinforced glass ionomer (chemically cured)	Polyacrylic acid solution, tartaric acid	Fluoroaluminosilicate glass, zirconia oxide, pigments, others
GC Equia Forte (EQ)	GC Industrial Co. Tokyo, Japan	Bulk-fill glass ionomer (chemically cured)	Polyacrylic acid, tartaric acid	Fluoroaluminosilicate glass, polyacrylic acid powder, surface treated glass
Riva self-cure HV (RV)	SDI Limited, Bayswater, Australia	High viscosity glass ionomer (chemically cured)	Polyacrylic acid, tartaric acid	Fluoroaluminosilicate glass
Zirconomer Coat	Shofu Inc. Koyoto, Japan	Resin (light cured)	Methyl methacrylate, Dimethyl aminoethyl methacrylate	Not Applicable
GC Equia Forte Coat	GC Industrial Co. Tokyo, Japan	Resin (light cured)	Methyl methacrylate, phosphoric acid ester monomer, butylated hydroxytoluene	Not Applicable
Riva Coat	SDI Limited, Bayswater, Australia	Nano-filled resin (light cured)	Urethane dimethacrylate, Triethylene glycol dimethacrylate, Polyethylene glycol dimethacrylate	Nano-filler

Ten specimens were left uncoated, while the remaining ten were coated with their respective manufacturers' resin coats by using a micro-brush. The resins were applied with a single stroke motion to ensure homogeneity of resin on all surfaces. They were then light-polymerized in two overlapping irradiations of ten seconds per surface with a LED curing light (Bluephase N, Ivoclar Vivadent, Schaan, Liechtenstein). The device had an output irradiance of 1200 mW/cm², wavelength of 385-515 nm, and an exit window of 8 mm. It was re-charged after every ten specimens and

a radiometer (Bluephase Meter II, Ivoclar Vivadent, Schaan, Liechtenstein) was used to verify the consistency of its performance.

All specimens were then immersed for seven days at 37 °C in distilled water (DW), artificial saliva (AS), or 0.02N citric acid (CA). All specimens were immersed in a sealed container and the pH of all immersion mediums were evaluated daily with a digital pH meter (Eutech pH2700, Singapore) to ensure standardization. Table 2 depicts the composition of artificial saliva used. The pH value of the artificial saliva and citric acid was 6.8 and 2.6, respectively.

TABLE 2. Composition of the artificial saliva

Components	Concentration (mg L ⁻¹)
NaCl	125.6
KCl	963.9
KSCN	189.2
KH ₂ PO ₄	654.5
Urea	200.0
NaSO ₄ •10H ₂ O	763.2
NH ₄ Cl	178.0
CaCl ₂ •2H ₂ O	227.8
NaHCO ₃	630.8

FLEXURAL TESTING

On the 7th day, the specimens were subjected to flexural testing in a 3-point bending configuration using a universal testing machine (Shimadzu Corp. Kyoto, Japan) with a load cell of 5 KN and a crosshead speed of 0.5 mm/min until fracture occurred. The following formulas were used to calculate the flexural modulus and flexural strength of all materials: -

Flexural modulus, E'

$$E' = \left(\frac{F}{D}\right) \left(\frac{L^3}{4BH^3}\right)$$

where F/D is the slope, in newtons per millimeter, measured in the straight-line portion of the load-deflection graph. L is the support span in millimeters (10 mm). B is the width of the specimen in millimeters. H is the height of the specimen in millimeters.

*The initial SI unit for flexural modulus was in Megapascal. It was later converted to Gigapascal (GPa). Flexural strength, σ , in Megapascals (MPa)

$$\sigma = \frac{3PL}{2BH^2}$$

where P is the maximum load exerted on the specimen in Newtons. L , B , H were defined in the flexural modulus equation.

STATISTICAL ANALYSIS

Flexural modulus/strength data were computed and analysed using the SPSS® statistical software (Version 26.0, IBM Corp. New York, USA). Shapiro-Wilk test was applied to assess the normality of data. As data were found to be normally distributed, parametric analyses were performed at a significance level $p < 0.05$. The independent sample T-test was used to evaluate differences in flexural properties between uncoated and coated specimens after immersing in various mediums. Inter-material and inter-medium differences were determined with one-way ANOVA and Tukey's post-hoc test.

RESULTS

The mean flexural modulus and flexural strength values for the various HVGICs and immersion mediums are shown in Table 3. Figures 1 and 2 illustrate the mean flexural properties of all materials when grouped according to different immersion mediums. Mean flexural

modulus ranged from 0.72 ± 0.13 to 13.19 ± 1.00 GPa while mean flexural strength ranged from 4.32 ± 0.84 to 45.83 ± 4.82 MPa after immersing in different mediums. Table 4 presents the comparison of flexural properties between uncoated (ZR, EQ, RV) and coated (ZRC, EQC, RVC) specimens, while statistical analyses among immersion mediums and materials are presented in Tables 5 and 6 respectively.

COMPARISON BETWEEN COATED AND NON-RESIN COATED

The effect of resin coating on flexural properties was observed to be material and medium dependent. As seen in Table 4, the flexural properties of uncoated ZR and RV were generally comparable or greater than when resin-coated except for exposure to DW. When exposed to DW, the flexural modulus and strength of ZR were significantly greater when coated with resin. Conversely, the flexural properties of EQ were significantly better when resin coated for most mediums. When immersed in DW, the flexural modulus of uncoated EQ specimens was significantly higher than coated ones.

COMPARISON BETWEEN DIFFERENT MATERIALS

Inter-material comparisons (Table 5) indicated that RV and EQ usually offered the best flexural performance when uncoated and coated, respectively. Significant differences in flexural modulus were as follows: Uncoated - $RV > EQ > ZR$ for all mediums; Coated - $EQC > ZRC > RVC$ for DW and $EQC \geq RVC > ZRC$ for AS/CA. With regards to flexural strength, the significant differences were: Uncoated - $RV > EQ > ZR$ for all mediums; Coated - $EQC > ZRC > RVC$ for DW, $EQC > RVC > ZRC$ for AS/CA.

COMPARISON BETWEEN DIFFERENT MEDIUMS

Inter-medium comparisons (Table 6) indicated rather mixed findings for the coated specimens. Significant differences in flexural modulus between mediums were: Uncoated - $DW > AS \geq CA$ for all HVGICs; Coated - $DW > AS > CA$ for ZRC, $DW > AS = CA$ for EQC, and $AS > DW = CA$ for RVC. Concerning flexural strength, the significant differences were: Uncoated - $DW > CA = AS$ for ZR/EQ and $DW = AS > CA$ for RV; Coated - $DW \geq AS > CA$ for ZRC/EQC and $AS > DW = CA$ for RVC.

TABLE 3. Mean flexural modulus (GPa) and flexural strength (MPa) values for various restorative materials with standard deviation in parentheses

Materials/Mediums		Distilled water		Artificial saliva		Citric acid	
		Flexural modulus	Flexural strength	Flexural modulus	Flexural strength	Flexural modulus	Flexural strength
Zirconomer	Uncoated	1.88 (0.44)	21.89 (4.86)	1.21 (0.31)	15.14 (3.79)	1.12 (0.30)	15.13 (3.32)
	Coated	6.04 (0.81)	34.28 (4.56)	1.45 (0.28)	19.68 (2.80)	0.72 (0.13)	4.32 (0.84)
Equia Forte	Uncoated	12.16 (0.84)	39.74 (2.61)	2.21 (0.41)	26.36 (4.95)	2.00 (0.46)	21.06 (4.66)
	Coated	8.87 (0.84)	40.70 (7.01)	7.24 (0.92)	42.11 (6.08)	7.63 (0.62)	33.96 (3.31)
Riva	Uncoated	13.19 (1.00)	45.83 (4.82)	10.10 (1.08)	41.97 (8.42)	2.55 (0.36)	26.38 (4.26)
	Coated	2.70 (0.40)	26.71 (1.57)	7.87 (1.52)	35.05 (4.80)	1.82 (0.50)	23.59 (4.16)

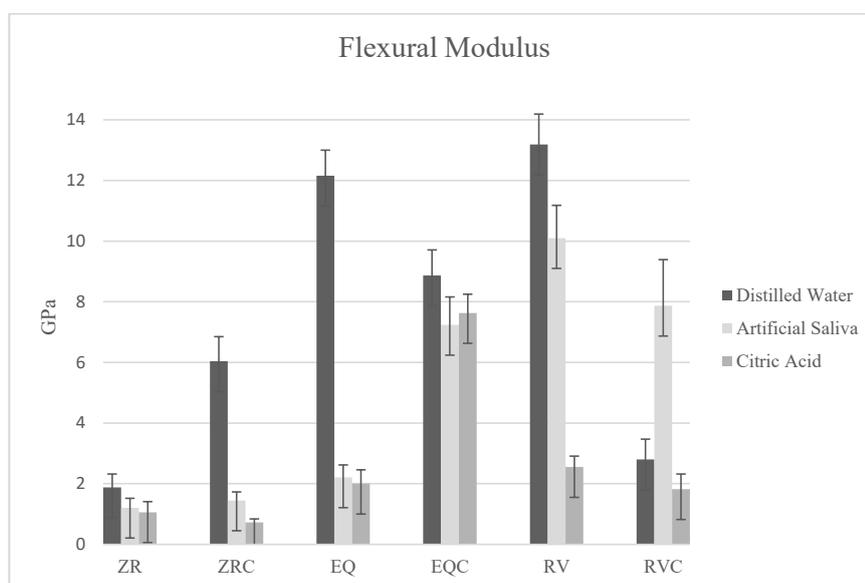


FIGURE 1. Mean flexural modulus (GPa) with standard deviation error bars of all materials when immersed in all three mediums

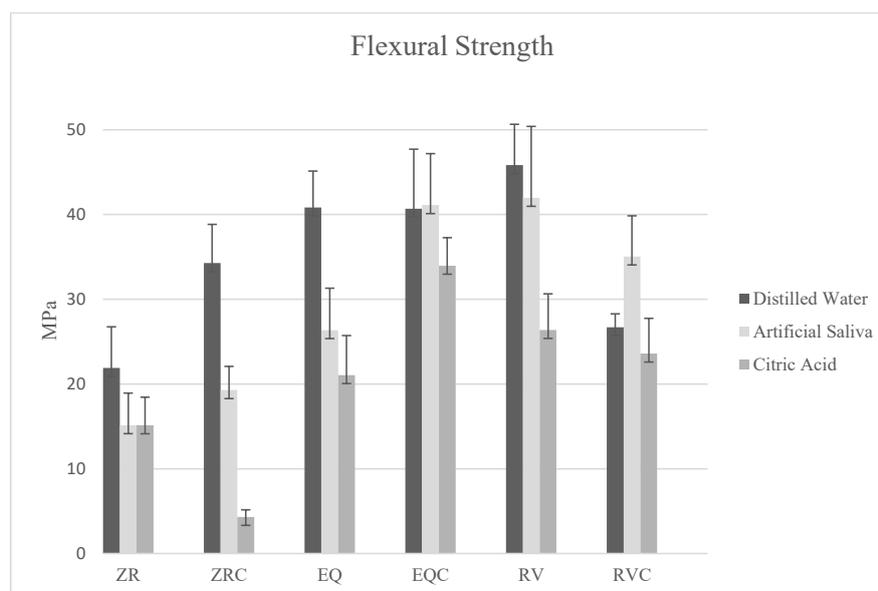


FIGURE 2. Mean flexural strength (MPa) with standard deviation error bars of all materials when immersed in all three mediums

TABLE 4. Comparison of flexural modulus (GPa) and flexural strength (MPa) between uncoated (UC) and coated (C) materials after immersing in the various mediums

Materials	Differences					
	Flexural modulus (GPa)			Flexural strength (MPa)		
	Distilled Water	Artificial Saliva	Citric Acid	Distilled Water	Artificial Saliva	Citric Acid
Zirconomer	C > UC	UC = C	UC > C	C > UC	C > UC	UC > C
Equia Forte	UC > C	C > UC	C > UC	C = UC	C > UC	C > UC
Riva	UC > C	UC > C	UC > C	UC > C	UC > C	UC = C

UC=uncoated, C=resin coated. > indicates statistically significant differences, while = denotes no statistically significant differences

TABLE 5. Comparison of flexural modulus (GPa) and flexural strength (MPa) values between materials after immersing in various mediums

Mediums	Differences			
	Flexural modulus (GPa)		Flexural strength (MPa)	
	Uncoated	Coated	Uncoated	Coated
Distilled Water	RV > EQ > ZR	EQC > ZRC > RVC	RV > EQ > ZR	EQC > ZRC > RVC
Artificial Saliva	RV > EQ > ZR	EQC = RVC > ZRC	RV > EQ > ZR	EQC > RVC > ZRC
Citric Acid	RV > EQ > ZR	EQC > RVC > ZRC	RV > EQ > ZR	EQC > RVC > ZRC

*ZR=Zirconomer, EQ=Equia, RV=Riva. C denotes resin coated. > indicates statistically significant differences, while = denotes no statistically significant difference

TABLE 6. Comparison of flexural modulus (GPa) and strength (MPa) values between different immersion mediums for various materials

Materials	Differences			
	Flexural modulus (GPa)		Flexural strength (MPa)	
	Uncoated	Coated	Uncoated	Coated
Zirconomer	DW > AS = CA	DW > AS > CA	DW > CA = AS	DW > AS > CA
Equia Forte	DW > AS = CA	DW > AS = CA	DW > CA = AS	DW = AS > CA
Riva	DW > AS > CA	AS > DW = CA	DW = AS > CA	AS > DW = CA

*DW=distilled water, AS=artificial saliva, CA=citric acid. > indicates statistically significant differences, while = denotes no statistically significant difference

DISCUSSION

The effect of resin coating of HVGICs on the flexural properties was investigated with the following immersion mediums, namely DW, AS, and CA. As resin coating influenced the flexural modulus and flexural strength, and significant differences were observed among materials and mediums, all three null hypotheses were duly rejected. The selection of evaluated materials represented the spectrum of contemporary HVGICs

available including zirconia (ZR), ultrafine glass particle (EQ), and 'ionglass' filler (RV) reinforced materials. DW served as the control medium, while AS and CA simulated neutral and acidic oral environments. The configuration of the three-point-bending mode had been theorized to represent the *in-vivo* scenario of delivery of masticatory forces from the opposing cusp (Ramashanker et al. 2011). Flexural modulus is the capability of a material to bend. In mechanical terms, it is the ratio of stress to strain during

deformation. The fundamentals are similar to elastic modulus, which denotes a material's rigidity. Flexural strength is an indication of the amount of stress and force that a material can withstand before fracturing. These two flexural properties are key indicators in determining the suitability of restorations, especially in high stress-bearing posterior dentitions. Hence, materials with high flexural modulus and strength are greatly desirable.

COATED AND NON-RESIN COATED

The impact of resin coating on flexural properties were both material and medium dependent. For ZR and RV, uncoated specimens generally performed better than coated ones. On the other hand, the flexural performance of EQ was mostly better when resin coated. Findings may be explained in part by the relative early moisture sensitivity of the HVGICs, their dynamic ionic interactions with the surrounding environments, and the constituents of coating resins. The superior performance of uncoated ZR and RV may be contributed by the increase of 'bound' water during early water exposure and consequently leading to hydration (Leirskar et al. 2003). Moreover, HVGICs have been reported to interact with environmental calcium and phosphate leading to improved mechanical properties especially under acidic conditions (Wang & Yap 2009; Wang et al. 2007). While ZR and RV Coat are unfilled resins, EQ coat contained nanofillers that may enhance its flexural properties. Reportedly, the application of nano-filled resin coatings may enhance the flexural strength of light and chemical-cured GICs (Bagheri et al. 2017). However, further research is still required to validate the latter including the evaluation of flexural properties after the application of EQ Coat to ZR and RV.

INTER-MATERIALS

When the three HVGICs were compared, RV and EQ generally demonstrated the highest flexural modulus and strength when uncoated and coated, respectively. The superior performance of RV found in our study corroborated the findings of Shiozawa et al. (2014). Despite the inclusion of zirconia fillers, ZR often showed the lowest flexural modulus and strength regardless of coating or immersion mediums. In effect, the flexural properties of ZR were about half (or less) that of RV and EQ when immersed in AS. This may be due to the lack of a proper chemical coupling between the zirconia fillers and the polysalt matrix, thus leading to crack propagation around the fillers and decreased mechanical properties. Furthermore, zirconia fillers are prone to

aqueous attack and consequent dissociation (Erdemir et al. 2013). Results corroborated that of a short-term study that indicated that ZR had poorer clinical performance than conventional GICs (Prabhakar et al. 2015). The application of ZR as posterior restorations should thus be done judiciously.

INTER-MEDIUMS

When mediums were compared, immersed in DW generally resulted in significantly higher flexural properties than AS and CA when the HVGIC specimens were uncoated. The finding was consistent with that of earlier studies specifying an increase in strength with early water exposure of HVGICs (Wang et al. 2006). No significant difference in flexural properties was observed between AS and CA for uncoated ZR and EQ specimens. However, uncoated RV specimens seemed to be more susceptible to acid degradation. When immersed in CA, hydrogen ions may diffuse into RV and replace cations in the polysalt matrix. As matrix cations are diminished, more is extracted from the adjacent glass particles leading to leaching (Fukazawa et al. 1987). Although the application of resin coating may provide a certain degree of resistance against chemical degradation induced by environmental acids, they may also mitigate the beneficial effects of environmental water and calcium/phosphate on HVGICs (Wang et al. 2006). Still, the immersion of coated ZR and EQ specimens in DW resulted in the highest flexural properties and exposure to CA usually the lowest. For coated RV specimens, immersion in AS presented significantly higher flexural modulus and strength than in DW and CA. While the exact mechanism for this is unknown, it may be related to the interaction of its reactive 'ionglass' fillers and environmental calcium/phosphate. Fourier-transform infrared (FTIR) analysis of HVGICs immersed in AS had shown a key chemical change in the phosphorous-amide bonding that could contribute to improved flexural properties (Yip & To 2005).

LIMITATIONS OF STUDY

The study had several limitations. First, the selected materials and their proprietary resin coats do not represent all HVGICs. As the effects of resin coating on flexural properties were found to be material and perhaps even resin coat dependent, further research on other HVGICs and a combination of products are necessary. Second, the HVGICs were exposed to the various mediums for only seven days. The immersion period could be prolonged to determine the longer-term impact of the three mediums

on HVGIC maturity. Lengthening the immersion period will also permit the evaluation of the durability of the resin coat in optimum situations. Third, only flexural properties were assessed. The appraisal of other physico-mechanical and biochemical properties such as colour stability, wear resistance, surface hardness, fluoride release, and setting are needed to fully characterize the impact of resin coatings on HVGICs. Additionally, the findings of these *in-vitro* testing should also be correlated to the outcomes of clinical trials.

CONCLUSIONS

Within the limitation of the study, the following conclusion could be made. The effect of resin coating on the flexural properties of HVGICs was material and immersion medium dependent. While the application of a resin coat is not mandatory (or beneficial) for ZR and RV, it is recommended for EQ as flexural properties are significantly improved. Uncoated RV and resin-coated EQ offered the best flexural modulus and strength. Immersion of HVGICs in DW generally presented the highest flexural properties suggesting probable interactions between HVGICs and their environment including saliva.

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