

Bonding of contemporary glass ionomer cements to different tooth substrates; microshear bond strength and scanning electron microscope study

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ABSTRACT

Objective: This study was conducted to evaluate the microshear bond strength (μ SBS) and ultramorphological characterization of glass ionomer (GI) cements; conventional GI cement (Fuji IX, CGI), resin modified GI (Fuji II LC, RMGI) and nano-ionomer (Ketac N100, NI) to enamel, dentin and cementum substrates. **Materials and Methods:** Forty-five lower molars were sectioned above the cemento-enamel junction. The occlusal surfaces were ground flat to obtain enamel and dentin substrates, meanwhile the cervical one-third of the root portion were utilized to evaluate the bonding efficacy to cementum substrate. Each substrate received microcylinders from the three tested materials; which were applied according to manufacturer instructions. μ SBS was assessed using a universal testing machine. The data were analyzed using two-way analysis of variance (ANOVA) and Tukey's *post-hoc* test. Modes of failure were examined using stereomicroscope at $\times 25$ magnification. Interfacial analysis of the bonded specimens was carried out using environmental field emission scanning electron microscope. **Results:** Two-way ANOVA revealed that materials, substrates and their interaction had a statistically significant effect on the mean μ SBS values at *P* values; <0.0001 , 0.0108 and 0.0037 respectively. RMGI showed statistically significant the highest μ SBS values to all examined tooth substrates. CGI and RMGI show substrate independent bonding efficiency, meanwhile; NI showed higher μ SBS values to dentin and cementum compared to enamel. **Conclusion:** Despite technological development of GI materials, mainly the nano-particles use, better results have not been achieved for both investigations, when compared to RMGI, independent of tooth substrate.

Key words: Cementum, conventional glass ionomer, dentin, enamel, microshear bond strength, nano-ionomer, resin-modified glass ionomer

INTRODUCTION

Since their introduction in 1972, glass ionomer (GI) cements have been widely used as dental restorative materials, in particular in restoration of cervical lesions.^[1] Despite the advantages associated with the use of conventional GI (CGI) cements as; their chemical bonding efficacy to tooth substrate, long-term fluoride

ion release, low coefficient of thermal expansion and relatively ease of use, yet they suffer from several problems as; moisture sensitivity, low mechanical strength and impaired translucency.^[2]

In an attempt to overcome these problems, several innovations were done. In 1988 resin modified GI (RMGI) cements were introduced by adding polymerizable

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hydrophilic resin to CGI formulations. This material gained wider acceptance in the dental professions as adding light polymerizable (hydroxyethyl methacrylate [HEMA]) resin to the GI enhanced its handling characteristics, increased its working time and improved its esthetic properties.^[3] However, RMGI cements exhibit shrinkage and substantial water sorption owing to the hydrophilic nature of its contents.^[4] Recently a nanofilled RMGI restorative material (nano-ionomer [NI]) was launched into the market. Apart from user-friendliness, the major innovations of these materials involve incorporation of nanotechnology, which allowed a highly packed filler composition (approximately 69%) in the form of nano-sized fillers and nanoclusters.^[5]

GI cements are the material of choice when dealing with root caries or noncarious cervical lesions.^[6] In such areas, the operator is apt to deal with cementum substrate. The chemical composition of cementum is different from enamel and dentin. Cementum in the cervical area is an acellular extrinsic fibre cementum. This substratum is a nonuniform human cementum, which can be represented as a woven fabric-like material that provides tissue porosity and permeability. One of the surface features that distinguish cementum from dentin is the lack of patent tubule orifices, which may alter its bonding capability.^[7,8]

Searching the literature, It was evident that almost all research work was concerned with bonding efficacy to enamel and dentin, however, the bonding efficacy to the cementum was scarcely addressed. Thus, this

study was carried out to determine the bonding efficacy of the nanofilled RMGI (NI) restorative material to enamel, dentin, and cementum and compare it to conventional RMGI restorative material and packable conventional CGI cements. The null hypotheses examined that (1) there is no difference in the bonding performance between the three tested GI cements; CGI, RMGI, and NI when applied to enamel, dentin and cementum. (2) Each tested GI cement bond equally to enamel, dentin and cementum substrates.

MATERIALS AND METHODS

Experimental design

Forty-five extracted, noncarious lower first molars were collected, cleaned from debris and stored in distilled water with 0.5% thymol. Three GI - based restorative materials were tested: Conventional high viscosity GI cement (Fuji IX, GC Corporation, Tokyo, Japan), RMGI cement (Fuji II LC, GC Corporation, Tokyo, Japan) and NI cement (Ketac N 100, 3M ESPE, NI, St Paul, MN, USA). Material's composition and manufacturer are presented in Table 1. For each material, 10 molars were used to test their bond strength to different tooth substrates; enamel, dentin, and cementum while 5 teeth were utilized for the ultrastructural examination of the interface.

Microshear bond strength testing

Specimen preparation

Each molar was sectioned 1 mm coronal to the cervical line to separate the crown from the roots using a diamond disk (K6974, Komet, Germany) in a low-speed handpiece under copious water cooling.

Table 1: Material's composition and manufacturer

Restorative materials	Composition	Manufacturer
Ketac conditioner	10% polyacrylic acid	3M-ESPE Dental products, St. Paul, MN, USA
Fuji IX (high viscosity GI capsule)	Powder: 95% fluoro-alumino-silicate glass, 5% polyacrylic acid Liquid: 50% distilled water, 40% polyacrylic acid and tartaric acid, 10% polybasic carboxylic acid	GC corporation, Tokyo, Japan
Fuji II LC (light cure RMGI capsule)	Powder: 100% fluoro-alumino-silicate Liquid: 35% HEMA, 25% distilled water, 24% polyacrylic acid, 6% tartaric acid and 0.10% camphorquinone	
Ketac varnish	Bis-GMA and traces of TEGDMA	3M-ESPE Dental products, St. Paul, MN, USA
Nano Primer (self-etching primer) Ketac N100 (nanofilled RMGI cement, NI)	40-50% water, 35-45% HEMA, 10-15% copolymer of acrylic and itaconic acids Paste A: 40-55% silane treated glass, 20-30% silane treated zirconia, 5-15% PEGDMD, 5-15% silane treated silica, 1-15% HEMA, 3-5% glass powder, 2-5% bisphenol A diglycidyl ether dimethacrylate and 0-1% TEGDMA Paste B: 40-60% silane treated ceramic, 20-30% copolymer of acrylic and itaconic acids, 10-20% water, 1-10% HEMA	

HEMA: Hydroxyethyl methacrylate, Bis-GMA: Bisphenol glycidyl methacrylate, TEGDMA: Triethyleneglycol dimethacrylate, PEGDMD: Polyethylene glycol dimethacrylate, RMGI: Resin modified glass ionomer, GI: Glass ionomer, NI: Nano-ionomer

The roots were separated, and each root was embedded in self-cured resin (Rapid Repair, DeguDent GmbH, Hanau, Germany). The flat cementum regions from the mesial and distal surfaces of the mesial and distal roots respectively, at cervical root third, were used as a bonding substrate, after grinding with wet 600 grit silicon carbide paper. The presence of cementum was previously verified under a stereomicroscope, (Carl Zeiss, Germany) at $\times 25$ magnification. The coronal portion of each tooth was embedded vertically in self-cured resin that the occlusal surface was used for testing. The occlusal enamel was then removed, and 1 mm of dentin was ground, thus exposing superficial occlusal dentin surrounded by an enamel rim. The thickness of the enamel should be at least 1.5 mm wide to be utilized for microshear bond strength (μ SBS) testing.^[9] A uniform smear layer was created by abrading the occlusal surface (enamel and dentin) with 600 grit silicon carbide paper.^[10]

Application of the restorative materials

All materials were applied following the manufacturer's instructions. For CGI and RMGI, Ketac dentin conditioner was applied to enamel, dentin or cementum substrates for 20 s using a microbrush (Shofu, Japan). It was then rinsed thoroughly with air-water spray and the excess water was blotted with cotton pellet. For NI group, Ketac N100 Primer was applied for 15 s. The primer was then air-thinned with an air syringe for 10 s and light cured with a light curing unit (DB 686-1, COXO, China) at an intensity of 1200 mW/cm² for 20 s.

After mixing of CGI and RMGI capsules using an amalgamator (Silamat Plus, Vivadent, Austria) and manual mixing of equal volumes of NI, the materials were injected into polyethylene tubes of 0.9 mm in diameter and 0.7 mm in height. For RMGI and NI group, the restoration was light-cured for 20 s. Light intensity was periodically checked using a radiometer (Kerr; Orange, CA, USA). Then, Ketac glaze was painted on the top of CGI and RMGI cylinders only and light-cured for 10 s. All polyethylene tubes were then removed, and the bonded specimens were stored in distilled water for 24 h. In coronal specimens, enamel and dentin substrates received four microcylinders for each. In each root specimen, each surface received two microcylinders (Each molar received four microcylinders for enamel, dentin, and cementum).

Testing of specimens

Microshear bond strength testing was done using a universal testing machine (Model LRX-plus, Lloyd

Instruments; Fareham, UK). Each specimen with its bonded microcylinders was secured with tightening screws to the lower fixed compartment of the universal testing machine. A loop of orthodontic stainless steel wire (0.014 inch in diameter) was wrapped around the bonded microcylinder as close to its base as possible and aligned with the loading axis of the upper movable compartment of the testing machine. The specimens were stressed in shear using a load cell of 5 KN at a crosshead speed of 0.5 mm/min. The shear force at failure was recorded and converted to shear stress in MPa units using computer software (Nexygen-MT Lloyd Instruments, Fareham, UK). For each molar, the four readings obtained from enamel, dentin and cementum substrates were averaged to obtain one reading for each substrate from each molar ($n = 10$).

Data were analyzed using two-way analysis of variance (ANOVA) to determine the effect of restorative material, tooth substrate and their interaction on mean μ SBS values. Comparing between the three materials for each tooth substrate and between the three substrates for each restorative material was carried out using One way ANOVA followed by Tukey's *post-hoc* test. The significance level was set at $P \leq 0.05$. Statistical analysis was performed with SPSS 16.0, Inc., (SPSS, Inc., Chicago, IL, USA) for Windows.

Fractographic analysis

Following microshear testing, specimens were examined using a stereomicroscope (Carl Zeiss, Germany) at $\times 25$ magnification to evaluate the fracture mode. The mode of failure was identified as: Type I (adhesive failure at the interface), Type II (mixed adhesive failure at the interface + cohesive in the restorative material), Type III (cohesive in the restorative material).

Interfacial analysis of the bonded interface

Fifteen molar were utilized for ultrastructural analysis of the interface (five for each restorative material). The specimens were prepared in a similar manner for μ SBS. The restorative materials (Fuji IX, Fuji II LC and Ketac N100) were applied to the different substrates as described before but in one layer of 2 mm in thickness. A transparent mylar strip (Moyco, USA) was placed on top of the restorative material until setting.

Teeth were then sectioned bucco-lingually using a diamond disc at a slow speed under copious water cooling. The sectioned specimens were finished with Soflex discs (3M-ESPE Dental products, St. Paul, MN, USA) of descending grits. The specimens were then

placed into an ultrasonic cleaner (Elma-Transsonic 460Hz, Germany) in distilled water for 30 min to remove the smear layer. Specimens were examined under low vacuum without gold sputtering using environmental field emission scanning electron microscopy (Quanta Field Emission Gun 250, Holland 115/230 V, Inspect SFEI, Phillips, Holland), and representative environmental scanning photomicrographs of enamel-, dentin- and cementum-restoration interface were obtained at $\times 500$ magnification.

RESULTS

Microshear bond strength

Two-way ANOVA revealed that restorative materials, substrates examined, and their interaction had a statistically significant effect on mean μ SBS values at P values; <0.0001 , 0.0108 and 0.0037 respectively [Table 2].

Mean and standard deviation of the tested groups are presented in Table 3. It was evident that RMGI showed the highest statistical significant μ SBS to all tested substrates compared to CGI and NI. CGI and RMGI showed equal bonding performance to enamel, dentin, and cementum. Meanwhile, NI showed statistically significant lower μ SBS values to enamel compared to dentin and cementum.

Fracture mode analysis results are presented in percentages in Table 4. It was evident that the highest percent of cohesive failure (Type III) was present in the CGI group.

Environmental field emission scanning electron microscope

All tested groups showed a gap-free junction with the existence of a filler-free zone of variable thickness, except at the NI-enamel interface at which a gap was evident [Figure 1]. The greatest thickness of the filler-free zone was observed for the NI-enamel interface. Several cracks were seen propagating within the CGI cement.

DISCUSSION

This study was conducted to elaborate the bonding efficacy of GI cements to different tooth substrates (enamel, dentin, and cementum) via the use of μ SBS test and environmental field emission scanning electron microscope (EFSEM). Three GI cements were evaluated; high viscosity chemical-cure CGI cement, RMGI, and NI. NI, Ketac N 100, is light

Table 2: Two-way ANOVA

Source of variation	df	Sum of squares	Mean square	F	P
Material factor	2	863.1	431.5	83.57	<0.0001
Substrate factor	2	49.52	24.76	4.795	0.0108
Interaction	4	87.32	21.83	4.227	0.0037
Residual	81	418.3	5.164		

ANOVA: Analysis of variance

Table 3: Means \pm SD of μ SBS values in MPa of all tested groups

Substrate	Materials						P
	GI cement		RMGI		NI		
	Mean	SD	Mean	SD	Mean	SD	
Enamel	5.19 ^b	1.38	11.08 ^a	2.45	2.8 ^{c,B}	1.06	$<0.001^*$
Dentin	4.82 ^b	1.68	11.37 ^a	2.64	7.47 ^{b,A}	2.5	$<0.001^*$
Cementum	4.78 ^b	1.82	12.86 ^a	3.06	6.27 ^{b,A}	2.6	$<0.001^*$
P	0.825		0.309		0.004		

*Significant at $P \leq 0.05$. Superscripts with different letters are statistically significantly. ^{A,B}Superscripts with uppercase letters are used for comparison between substrates in the same column. ^{a,b,c}Superscripts with lower case letters are used for comparison between materials within the same row. μ SBS: Microshear bond strength, RMGI: Resin modified glass ionomer, GI: Glass ionomer, SD: Standard deviation, NI: Nano-ionomer

Table 4: The percentages of fracture modes of all tested groups

Substrate	Type	Material (%)		
		GI	RMGI	NI
Enamel	Type I	25	15	42.5
	Type II	52.5	77.5	52.5
	Type III	22.5	7.5	5
Dentin	Type I	40	15	40
	Type II	50	82.5	60
	Type III	10	2.5	0
Cementum	Type I	12.5	7.5	15
	Type II	57.5	82.5	55
	Type III	30	10	30

RMGI: Resin modified glass ionomer, GI: Glass ionomer, NI: Nano-ionomer

curing RMGI modified using bonded nanofillers, and nanocluster fillers, along with fluoroaluminosilicate glass.^[11] A RMGI (Fuji II LC) was chosen to elucidate the value of the latter modification. A CGI cement (Fuji IX) was compared as control.

In this study enamel, dentin and cementum specimens were obtained from the same tooth for more standardization of the chemical composition and the degree of mineralization when comparing the three substrates with each other. In the present study, μ SBS test was used to evaluate the bonding efficacy. This test is considered a relatively simple test that permits efficient screening of adhesive systems, regional and depth profiling of a variety of substrates,

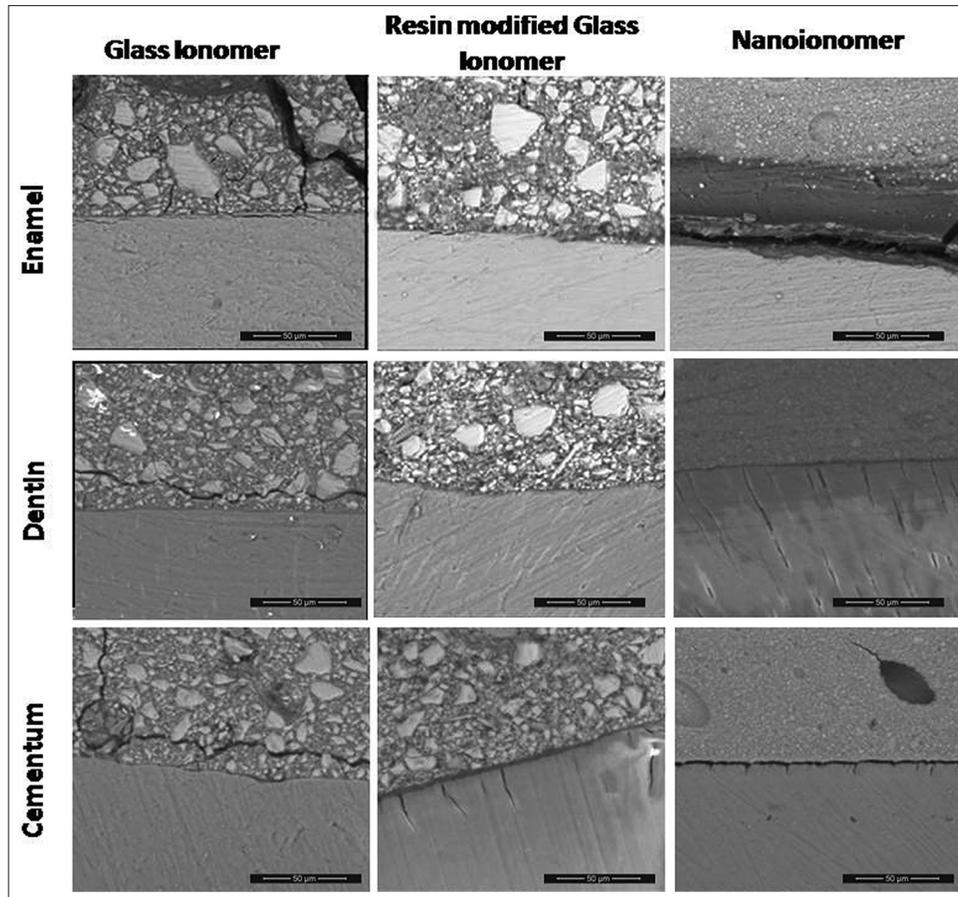


Figure 1: Environmental field emission scanning electron microscope photomicrograph representing tooth-restoration interface of all tested groups at $\times 500$ magnification. It revealed the existence of a filler free zone at the interface of all tested groups. A gap is present at the nanoionomer-enamel interface

with conservation of teeth.^[12] Furthermore, it could be a viable test when evaluating brittle materials, having low modulus of elasticity as GI cements as it lowers the probability of having a crack opening relative to the load applied.^[13] Fractographic analysis of the debonded specimens gives clues about some of the reasons of failure, the usage and limitations of each material.^[14] In addition, evaluation of the interface was carried out using EFSEEM. EFSEEM photomicrographs may give clues about the bond quality at the interface, which can be a complementary tool for bond strength evaluation.^[15] It is a more suitable tool for evaluation of GI cements restoratives which are sensitive to the vacuuming, processing and gold sputtering step which are mandatory in the conventional scanning electron microscope.^[15,16]

Results of the present study revealed that the RMGI showed the highest statistically significant μ SBS values compared to CGI and NI irrespective to substrate examined, thus the first null hypothesis is rejected. This result is in agreement with several authors whom found that the bond strength of RMGI was

significantly higher than CGI cements to enamel,^[17,18] and to dentin.^[8,14,19] In addition, in agreement with Coutinho *et al.*,^[20] whom found that RMGI showed significantly higher bond strength values compared to NI to both enamel and dentin substrates. However, the bonding of GI restoratives to cementum was not previously addressed in literature.

The better bonding performance of the RMGI compared to CGI could be attributed to several theories acting simultaneously; first; to the difference in the rates of adsorption on the calcified tissue surface and crosslinking in the bulk regions of the material.^[21] Light-curing of RMGI causes an initial increase in the ionization rate due to the production of acid (via the photoinitiator), and this results in a very strong adsorbed layer. At the same time, internal cross-linking reactions result from free-radical polymerization processes. Thus, the unsaturated methacrylate groups will polymerize and co-polymerize with the modified polyacrylic acid, forming a polyacrylic acid network entangled within the collagen web.^[22] Second; to the resinous component in RMGI cements

that can form a covalently bonded matrix, allowing the material to have greater fracture strengths than CGI cements.^[17] Fractographic analysis showed that the highest percentage of cohesive failure was present in CGI. Furthermore, FESEM photos revealed existence of multiple cracks within the CGI matrix. Third, this might be attributed to the carboxylic acid copolymers contained in the cement, which provide a more reactive and acidic liquid with increased number of carboxyl groups per chain unit thus increasing its chemical bonding potential.^[8] Forth; to the high HEMA concentration in RMGI cement, which improves its wetting ability and results in better bonding.^[19] The HEMA concentration in the RMGI cement used is 35%; meanwhile, it is completely absent in CGI cement.

Nano-ionomer primer contains the same HEMA concentration as RMGI, but it showed lower bonding performance with all tooth substrates, despite the existence of a thick filler-free zone with gap-free junction with dentin and cementum substrates. This observation is in line with Coutinho *et al.*,^[20] whom attributed this to the difference in the technique of application and the conditioning step. In RMGI cement polyacrylic acid conditioning is applied and rinsed prior to the material application which allows for partial demineralization and removal of the smear layer, giving the opportunity for the HEMA component to enhance the wetting of the surface and the production of microporosity in the different tooth substrates. This could contribute to either increased surface area for chemical bonding with residual hydroxyl apatite or micromechanical bonding through micromechanical interlocking. Meanwhile, with NI, the polyacrylic acid is incorporated in the primer which is applied onto the smear layer and is not rinsed away. In addition, NI primer has a relatively higher pH (3) than polyacrylic acid pH (1.5). This difference could be reflected in the ability of polyacrylic acid to remove the smear layer resulting in distinct performance.^[11,19]

Coutinho *et al.*,^[20] reported that the the filler-free zone at the NI interface most likely represented remnants of the primer that did not polymerize well due to the presence of oxygen, and therefore only showed a nonhomogeneous layer which might have not contributed to the bonding effectiveness. Although the existence of filler free zone is important to act as a stress breaking layer, the increase in its thickness above a certain level appears to be a negating factor similar to the scenario of the undesired increase in the hybrid/adhesive layer thickness.^[23]

However, this FESEM observation is in contrary to El-Askary and Nassif,^[5] whom reported the existence of a gap between the NI and the dentin substrate, when the material was applied according to the manufacturer instructions. This contradiction could be attributed to the differences in the technique adopted for specimen preparations for FESEM examination.

The results also revealed that there was no statistically significant difference between enamel, dentin and cementum substrates with both CGI and RMGI cements. Meanwhile, NI showed significantly lower bond strength values to enamel compared to dentin and cementum. Thus, the second null hypothesis is partially accepted. This result is in partial agreement with Fritz *et al.*,^[24] and Coutinho *et al.*,^[20] whom found no statistically significant difference between enamel and dentin substrates. On the other hand, μ SBS values revealed that NI showed significantly lower bond strength values to enamel compared to dentin and cementum. This result is in line with FESEM photomicrograph, which revealed the existence a gap at NI-enamel interface. This result is in agreement with Uysal *et al.*,^[25] who found a significant decrease in bond strength NI was bonded to the enamel. Compared to the low organic content in the enamel (1% by weight),^[26] the organic content in dentin comprises 20% by weight while cementum comprises 33% by weight.^[7] This might explain the low bonding capacity of NI to enamel. As the high pH of Ketac primer, which acts as a mild self-etch adhesive, make it sensitive to degree of tissue mineralization.^[22] These results are in contrary to Korkmaz *et al.*,^[27] whom found that there was no significant difference observed between enamel and dentin at the NI group.

In the present investigation, no direct relationship between the shear bond strength and the mode of failure observed. This substantiates other results in which high bond strength values were not necessarily related to a cohesive type of fracture. This is in agreement with Sidhu *et al.*,^[14] air voids, cracks, defects or geometric features act as stress raisers when an interface is loaded, which lead to rupture of the "joint" at stresses very much lower than that which should theoretically occur. This was confirmed by the FESEM micrograph which showed many cracks in the CGI extending to the interface which was also observed with several authors.^[14,28-30] The presence of cracks in GI cement might reflect the high percentage of Type III failure present in this group. This would suggest that the bonding configuration surpassed the inherent strength of the GI strength, denoting

that the weak point in this interface is the mechanical properties of the material.

Further studies should be carried out to test the effect of the complex oral environmental conditions on the mechanical and chemical adhesion of different GI cement categories to the different tooth substrates.

CONCLUSION

Under the conditions of the present study, the following conclusions could be derived:

- CGI and RMGI cement showed equal performance to the different tooth substrates, meanwhile NI showed lower bonding performance to enamel compared to dentin and cementum
- RMGI cement showed prompt bonding to enamel, dentin and cementum compared to CGI and NI.

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