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Effect of Thermocycling on Interfacial Gap-formation in Class V Cavities and Mechanical Properties of Spherical Silica Filler Addition to Resin-modified Glass Ionomer Restorations

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The effects of thermocycling at 20,000 cycles and addition of silanized spherical silica filler (SF) on resin-modified glass ionomer cement (RMGIC) restorations were investigated. A RMGIC added with an untreated spherical silica filler (UF) was used as a comparison. Marginal gaps in Class V tooth cavities, compressive strength, diametral tensile strength, flexural strength, and shear bond strengths to enamel and dentin were examined. All thermocycled samples showed decreased frequency of marginal gap formation as compared to the 24-hour samples, with reduction of 73% to 95%. At the immediate condition, after 24 hours, and after thermocycling, the addition of 10 wt% SF yielded the most favorable results in terms of marginal gap formation in Class V cavities, compressive strength, flexural strength, and shear bond strength to enamel. Diametral tensile strength and flexural strength were also increased significantly by the addition of 5 wt% SF. Further, shear bond strength tests showed that the addition of SF had no effect on bonding capability to enamel and dentin.

Key words: Resin-modified glass ionomer cement, Silanized spherical silica fillers, Thermocycled condition

INTRODUCTION

Resin-modified glass ionomer cements (RMGICs) were developed with the aim of improving the setting characteristics and mechanical properties of conventional glass ionomer cements (GICs)[1]. In a previous study, it was reported that the addition of spherical silica fillers to a RMGIC powder improved the workability of the cement[2]. Silanization of filler − which depends largely upon the siloxane bridge (Si-O-Si) formation between the silica surface and silane molecule − has yielded commendable results[3]. The addition of silanized spherical silica fillers (SF) increased the compressive strength, diametral tensile strength, and flexural strength of RMGIC after 24 hours, as well as reduced its water uptake. Previous studies also showed that SF improved the mechanical properties of the cement more than untreated spherical silica fillers (UF)[2,4]. SF addition significantly reduced immediate marginal gap formation in tooth cavities and immediate interfacial gap formation in Class V restorations by up to 63%, and the setting shrinkage of RMGIC by up to 66%[4,5].

Although RMGICs set immediately upon light curing, it was suggested that restorations should be polished after 24 hours[4,6]. The presence of photopolymerizable monomers like hydroxyethyl methacrylate (HEMA) in RMGIC formulations means that less water is available for the acid-base reaction − which may consequently be retarded[7]. It has been debated that the acid−base reaction in RMGICs does not contribute substantially to the setting process − which is light-initiated. However, a study has demonstrated the evidence of a delayed acid-base reaction for RMGIC, which leveled off after 168 hours of cement mixing[8]. Besides, in a previous study, we showed that the addition of 5 wt% SF increased the shear bond strengths to human enamel and dentin after 24 hours[4].

However, during mastication, restorations in the tooth cavity are subjected not only to shear stress, but also compressive and tensile stresses and temperature fluctuations. Some studies showed that thermocycling between 5℃ and 55℃ may or may not influence the shear bond strength of resin materials[9,10]. However, the ISO TR 11450 standard[11] indicates that a thermocycling procedure comprising 500 cycles in water between 5℃ and 55℃ is an appropriate artificial aging test. A recent literature review[12] further concluded that 10,000 cycles correspond to approximately one year of in vivo functioning, rendering the 500 cycles as proposed by the ISO standard very minimal to mimic long-term bonding effectiveness. For clinical relevance, it is undoubtedly and undisputedly important to study the effect of long-term thermocycling on RMGICs. For a more comprehensive analysis of the results obtained from such an investigation, it is also necessary to make a

comparison with the immediate condition. This is because in dental practice, dentists are accustomed to polishing restorations directly after the curing procedure, which might impart a negative influence on the resistance of restorations[6,13,14].

In the present study, the first hypothesis was that the addition of SF to RMGIC would not decrease the interfacial integrity in Class V restorations after being thermocycled for 20,000 times. The second hypothesis was that the addition of spherical silica filler would not significantly decrease the mechanical properties of RMGIC after 20,000 thermocycles. The third hypothesis was that the mechanical properties of spherical silica filler-added RMGIC after 20,000 thermocycles would be significantly higher than that at the immediate condition and after 24 hours.

MATERIALS AND METHODS

Materials

The RMGIC material used in this study was Fuji II LC EM (Powder Lot No.0507201, Liquid Lot No.0507141, GC Corp., Tokyo, Japan) with a recommended powder/liquid ratio (P/L) of 3.0. The powder was fluoroalumino silicate glass, and the liquid was composed of methacrylic acid ester, polyacrylic acid, and water[2,4,5].

Silanized spherical silica filler (GC Corp., Tokyo, Japan), which had an average particle diameter of 0.3 μm with γ-methacryloxypropyl trimethoxysilane (γ-MPTS) (KBM 503, Shin-Etsu Chemical Co., Tokyo, Japan), was prepared as previously described[2,4,5].

RMGIC powder was modified by initially mixing it with either SF or UF at different weight percentages (5 wt%, 10 wt%, and 20 wt%) before mixing it with Fuji II LC EM liquid. Prepared cement powders were described as SF5, SF10, SF20, UF5, UF10, and UF20 — hence indicating the type of filler added and filler content in weight percentage. Both the mixing time and preparation time were 30 seconds each. As for the P/L ratio, each one was chosen based on the maximum compressive strength value of cement as given in a previous study[4]. In the present study, the CONTROL and BASE specimens served as controls. For CONTROL, Fuji II LC EM was mixed with P/L=3.6 (the P/L ratio at which maximum compressive strength was achieved); for BASE, it was P/L=3.0 (manufacturer's recommended P/L). SF5, SF10, and SF20 were mixed with P/L ratios of 4.0, 4.4, and 4.0 respectively; while UF5, UF10, and UF20 were mixed with P/L ratios of 4.4, 4.4, and 4.0 respectively.

A visible light curing unit (New Light VL-Ⅱ, GC Corp., Tokyo, Japan; irradiation diameter: 10 mm) was used for activating the specimens, and close contact was ensured between exit window of the lamp and the celluloid strip that covered the specimens. Using a radiometer (Demetron/Kerr, Danbury, CT, USA), light intensity was checked and maintained at 450 mW/cm^2.

Human premolars, extracted for orthodontic reasons, were used for gap measurement in Class V tooth cavities as well as for measurement of shear bond strengths to enamel and dentin. After extraction, the teeth were stored immediately in distilled water at about 4℃ for a maximum period of three months before use. Since occlusal dentin tends to give a lower bond strength than proximal or buccal dentin[15,16], and that dentinal tubule orientation and location significantly influence mechanical strength test results[17,18], buccal surfaces were used for marginal gap measurement in Class V cavities while proximal surfaces were used for shear bond strength measurements in this study.

All procedures, except for cavity preparation and mechanical testing, were performed in a thermohygrostatic room maintained at 23±0.5℃ and 50±2 % relative humidity.

Interfacial gap-formation measurement in Class V cavities

Ten human premolars for each material were prepared. A round Class V cavity on the buccal region of each tooth was prepared with a tungsten carbide bur (200,000 rpm) and a fissure bur (8,000 rpm) under wet conditions to a depth of 1.5 mm with a diameter of 3.5 mm. Cavity preparation was placed 1.0 mm above the cementoenamel junction (CEJ), and cavosurface walls were finished to a butt joint. This design differed from a typical, clinical Class V cavity in that cavity corners were at geometric box angles to prepare a constant-volume model[5,19]. One cavity was prepared in each tooth, and its dimensions were measured using a vernier caliper (U39818, Mitutoyo, Kawasaki, Japan). Each cavity was treated with a Cavity Conditioner (Lot No.0405271, GC Corp., Tokyo, Japan) for 10 seconds according to manufacturer's instructions, rinsed thoroughly with distilled water, air-sprayed, and filled with the material using a syringe tip (Centrix C-R Syringe System, Centrix, Shelton, CT, USA).

Covered with a celluloid strip, the material was light-cured for 20 seconds. Ten surfaces were polished immediately after light activation. Another ten were polished after 24-hour storage in distilled water at 37℃, and the last 10 surfaces were polished after being thermocycled for 20,000 times between 5℃ and 55℃ with a dwell time of one minute each (Percolation Tester, KE-1, Kuraray Engineering Co., Kurashiki, Japan). The outer surfaces of restorations were polished with abrasive points (Silicone Mide, Shofu, Kyoto, Japan) in wet condition to avoid desiccation and breakdown through rinsing with distilled water. Each tooth was sectioned in a

buccolingual direction through the center of the restoration with a low-speed diamond saw (Isomet, Buehler Ltd., Lake Bluff, IL, USA). The presence or absence of marginal gaps was inspected at 14 points (each 0.5 mm apart) under a traveling microscope (1000 ×, Measurescope, MM-11, Nikon, Tokyo, Japan). A point with no gaps had a value of 0, while 1 indicated the presence of gap. The overall sum of 14 points examined was calculated and expressed as the sum of each sample[5,19].

Compressive strength measurement

For compressive strength measurement, 10 specimens were prepared for each material and condition. Mixed in 30 seconds using a plastic spatula on a mixing pad, the RMGIC specimens were syringe-loaded into a cylindrical Teflon split mold (with a depth of 6.0 mm and diameter of 3.0 mm), covered with a glass plate, and clamped. The specimens were light-cured for 60 seconds on each side, with due consideration to the thickness of the glass plate and the specimens[5]. Then, they were removed from the mold and tested immediately after 24-hour storage in distilled water at 37℃ or after being thermocycled for 20,000 times, as mentioned above. Compressive strength was measured using a universal testing machine (Autograph DCS-2000, Shimadzu, Kyoto, Japan) with a crosshead speed of 0.5 mm/min as outlined in ISO 7489-1986, with a maximum external force of 200 kgf.

Diametral tensile strength (DTS) measurement

Light curing and testing procedures for DTS were the same as those described for compressive strength measurement. The samples for DTS test were prepared in a cylindrical Teflon split mold (h=3.0 mm, d=6.0 mm), where 10 samples were prepared for each material and condition[5].

Flexural strength measurement

A Teflon split mold with internal dimensions of 25 mm×2 mm×2 mm was used to prepare the rectangular samples for flexural strength test. After mixing within 30 seconds using a plastic spatula on a mixing pad and syringe-loaded, the mold was covered with a glass plate and clamped. The samples, 10 for each material and condition, were light-cured for 60 seconds at three overlapping sites. The three-point bending method was used to measure flexural strength with a 20-mm span. Crosshead speed of the universal testing machine (5565, Instron, Canton, MA, USA) was 0.5 mm/min, and a maximum external force of 10 kgf was applied to the midpoint of the test beam. Flexural strength was then calculated thereby[5,19].

Shear bond strengths to enamel and dentin

Shear bond strengths to enamel and dentin — which comprised the cavity wall — was measured to evaluate the bonding durability between the filling material and the cavity. Bond strengths to flat enamel and dentin surfaces were determined immediately after light activation, after distilled water storage for 24 hours at 37℃, and after thermocycling. Specimens (N=10 for each material and condition) were obtained from human premolars embedded in slow-setting epoxy resin. Proximal flat enamel and dentin surfaces were obtained by grinding with a wet silicon carbide paper (#1000) followed by treatment with the Cavity Conditioner for 10 seconds. To prepare the samples for shear bond strength measurement, a cylindrical Teflon split mold (3.6 mm in diameter, 2.0 mm in height) was used to minimize the stress exerted on the specimens during their retrieval. Each material was placed into the Teflon mold set on the enamel or dentinal surface using a syringe tip and hardened by light curing[5]. At the immediate condition, after 24-hour water storage, and after being thermocycled for 20,000 times, the specimens obtained were mounted on a universal testing machine (Autograph DCS-2000, Shimadzu, Kyoto, Japan). Shear stress was then applied at 0.5 mm/min with a maximum external force of 50 kgf.

Statistical analysis

Results of mechanical properties measurement and bonding to tooth structure were analyzed statistically using ANOVA and Tukey's test with level of significance set at 0.05. Differences among the marginal gaps in Class V cavities were compared statistically using non-parametrical t-test, and the linear correlations among the properties were analyzed using Pearson product-moment correlation[5,19–21].

RESULTS

Summed interfacial gap-formation in Class V tooth cavities

Table 1 lists the effects of spherical silica fillers on marginal gap formation in Class V tooth cavities. After thermocycling for 20,000 times, the frequency of marginal gaps in the tooth cavities reduced significantly. It should also be pointed out that the reduction effect was also shown after water storage for 24 hours. Nonetheless, when compared to the 24-hour samples, the thermocycled samples had similar or less marginal gaps.

At the immediate condition, the highest frequency of marginal gaps was shown by the Base specimen (RMGIC with P/L=3.0). However, after 24-hour storage and after thermocycling, the highest frequency was shown by UF20. For example, after 24 hours, the marginal gap frequency of SF10 was only 55% that of UF20. In terms of comparison between the immediate condition and after 24-hour storage, marginal gaps in Class V cavities in the lat-

ter condition were only 16 to 25%: SF5 showed the biggest reduction with a value of 16%, while the 24-hour control and base materials were 18% and 17% respectively. Then, like the 24-hour samples, the thermocycled materials also showed less marginal gaps with values of reduction between 73% and 95% — except for SF10, but the difference was not significant.

Compressive strength

Table 2 lists the compressive strength measurement results. On the overall, all samples at the immediate condition showed lower compressive strength values than those after 24-hour storage and thermocycling. Except for UF20, the post-thermocycling compressive strength of all RMGICs was 11 to 20% higher than the compressive strength values after 24 hours. Moreover, for all the three conditions, SF10 consistently showed the highest compressive strength value, whereby the highest value of 221.5±8.0 MPa was yielded after thermocycling. In contrast, UF20 showed the lowest compressive strength value after 24-hour storage and thermocycling.

Diametral tensile strength (DTS)

Table 3 shows the DTS measurement results. After thermocycling, there were no significant differences among the SF specimens. Further, SF10 and SF20 were not significantly different from UF5. It should be highlighted that following 24-hour storage and thermocycling, all SF-added RMGICs and UF5 showed significant differences ($p<0.05$) from UF10, UF20, control and base specimens. As for comparison between the 24-hour storage and thermocycled conditions of each specimen, there were no significant differences for all RMGICs. In terms of comparison

Table 1 Interfacial gap-formation in Class V cavities (points)

Material	P/L	Immediately	After 24 hours	After 20,000 thermocycles
SF5	4.0	82 [a]	13 [e*]	12 [e*]
SF10	4.4	63 [d]	11 [e*]	12 [e*]
SF20	4.0	80 [a,b]	16 [e*]	14 [e*]
UF5	4.4	66 [c,d]	14 [e*]	12 [e*]
UF10	4.4	70 [b,c,d]	17 [e*]	15 [e*]
UF20	4.0	79 [a,b,c]	20 [e*]	19 [e*]
Control	3.6	84 [a]	15 [e*]	11 [e*]
Base	3.0	109 [e]	19 [e*]	15 [e*]

SF: Silanized spherical silica filler-added Fuji II LC EM.
UF: Untreated spherical silica filler-added Fuji II LC EM.
Control: Fuji II LC EM mixed with P/L 3.6.
Base: Fuji II LC EM mixed with P/L 3.0.
Same alphabets indicate no significant differences among the mean values within same condition analyzed using the t-test ($p>0.05$).
Same symbol (*) indicates no significant differences among the conditions of same material analyzed using the t-test ($p>0.05$).
N=10.

Table 2 Compressive strengths of RMGICs (MPa)

Material	P/L	Immediately	After 24 hours	After 20,000 thermocycles
SF5	4.0	158.3 ± 10.9 [a,b]	187.0 ± 4.0 [a,b]	220.2 ± 8.9 [a]
SF10	4.4	173.1 ± 9.7 [a]	196.0 ± 9.0 [a]	221.5 ± 8.0 [a]
SF20	4.0	157.1 ± 9.2 [b]	191.0 ± 9.0 [a]	218.6 ± 9.6 [a,b]
UF5	4.4	167.0 ± 7.9 [a,b]	185.0 ± 6.0 [a,b]	205.9 ± 8.0 [c]
UF10	4.4	158.6 ± 7.6 [a,b]	174.0 ± 10.0 [b,c]	208.5 ± 10.9 [b,c]
UF20	4.0	129.2 ± 5.6 [c]	150.0 ± 8.0 [d*]	146.3 ± 7.8 [d*]
Control	3.6	128.8 ± 5.8 [c]	170.0 ± 7.0 [c]	180.4 ± 7.2 [e]
Base	3.0	111.0 ± 7.9 [d]	153.0 ± 4.0 [d]	178.0 ± 7.3 [e]

SF: Silanized spherical silica filler-added Fuji II LC EM.
UF: Untreated spherical silica filler-added Fuji II LC EM.
Control: Fuji II LC EM mixed with P/L 3.6.
Base: Fuji II LC EM mixed with P/L 3.0.
Same alphabets indicate no significant differences among the mean values within same condition analyzed using the t-test ($p>0.05$).
Same symbol (*) indicates no significant differences among the conditions of same material analyzed using the t-test ($p>0.05$).
N=10.

between the immediate and thermocycled conditions, DTS values increased by 18 to 56% after thermocycling. In particular, SF5 exhibited the largest increase in DTS value while SF10 exhibited the lowest increase.

Flexural strength

Table 4 shows the flexural strength measurement results. All materials showed higher flexural strength values after thermocycling. Upon comparison between the 24-hour and thermocycled specimens, SF10 showed the highest increase at 31% while UF5 showed the lowest increase at 11%. In terms of comparison between the immediate and thermocycled specimens, SF10 also showed the highest increase at 122% while the flexural strength of UF20 increased by only 63%. With the same comparison scenario, the control specimen increased in flexural strength by 77%, while the base specimen increased in flexural strength by 97%.

Shear bond strengths to enamel and dentin

The effects of spherical silica fillers on the shear bond strength of RMGIC to enamel and dentin are listed in Tables 5 and 6 respectively. After thermocycling, all materials — except UF10 and UF20 — showed no significant differences in shear bond strength. The largest shear bond strength value to enamel after thermocycling was shown by SF10 (21.1 ± 3.1 MPa). However, the largest shear bond strength value to dentin after thermocycling was shown by SF5 (15.2 ± 1.9 MPa). For shear bond strength to enamel, both 24-hour and thermocycled specimens yielded higher values than the immediate specimens — although thermocycled specimens showed lower values than the 24-hour specimens. For shear

Table 3 Diametral tensile strengths of RMGICs (MPa)

Material	P/L	Immediately	After 24 hours	After 20,000 thermocycles
SF5	4.0	24.74 ± 2.6 ^{a,b}	38.48 ± 2.8 ^{a*}	38.59 ± 4.1 ^{a*}
SF10	4.4	26.08 ± 2.4 ^{b}	37.06 ± 6.2 ^{a*}	36.02 ± 4.2 ^{a,b*}
SF20	4.0	24.14 ± 3.2 ^{a,b}	36.16 ± 4.2 ^{a*}	36.66 ± 2.4 ^{a,b*}
UF5	4.4	23.76 ± 3.0 ^{a,b,c}	39.82 ± 5.0 ^{a*}	35.61 ± 2.8 ^{b*}
UF10	4.4	24.62 ± 2.4 ^{a,c}	30.56 ± 4.6 ^{b*}	31.09 ± 2.4 ^{c*}
UF20	4.0	22.20 ± 2.6 ^{a,c}	27.52 ± 3.0 ^{b*}	26.09 ± 1.8 ^{d*}
Control	3.6	23.08 ± 1.2 ^{a,b,c}	31.28 ± 6.2 ^{b*}	31.36 ± 2.0 ^{c*}
Base	3.0	20.42 ± 1.2 ^{c}	29.78 ± 3.2 ^{b*}	30.50 ± 1.3 ^{c*}

SF: Silanized spherical silica filler-added Fuji II LC EM.
UF: Untreated spherical silica filler-added Fuji II LC EM.
Control: Fuji II LC EM mixed with P/L 3.6.
Base: Fuji II LC EM mixed with P/L 3.0.
Same alphabets indicate no significant differences among the mean values within same condition analyzed using the t-test ($p>0.05$).
Same symbol (*) indicates no significant differences among the conditions of same material analyzed using the t-test ($p>0.05$).
N=10.

Table 4 Flexural strengths of RMGICs (MPa)

Material	P/L	Immediately	After 24 hours	After 20,000 thermocycles
SF5	4.0	39.9 ± 2.9 ^{a}	62.9 ± 8.3 ^{a,b}	74.8 ± 5.7 ^{b}
SF10	4.4	38.9 ± 2.3 ^{a,b}	65.7 ± 4.8 ^{a}	86.2 ± 6.6 ^{a}
SF20	4.0	32.2 ± 2.5 ^{d,e}	58.6 ± 8.8 ^{a,b,c}	70.5 ± 5.8 ^{b}
UF5	4.4	34.7 ± 2.2 ^{c,d}	55.0 ± 5.6 ^{b,c}	61.1 ± 5.7 ^{c}
UF10	4.4	30.9 ± 2.4 ^{e}	50.9 ± 5.4 ^{c}	63.9 ± 3.6 ^{c}
UF20	4.0	24.9 ± 2.3 ^{f}	34.1 ± 3.0 ^{d}	40.5 ± 3.6 ^{d}
Control	3.6	35.9 ± 2.5 ^{b,c}	56.2 ± 3.7 ^{b,c}	63.6 ± 4.3 ^{c}
Base	3.0	30.8 ± 1.2 ^{e}	51.7 ± 6.1 ^{c}	60.7 ± 5.2 ^{c}

SF: Silanized spherical silica filler-added Fuji II LC EM.
UF: Untreated spherical silica filler-added Fuji II LC EM.
Control: Fuji II LC EM mixed with P/L 3.6.
Base: Fuji II LC EM mixed with P/L 3.0.
Same alphabets indicate no significant differences among the mean values within same condition analyzed using the t-test ($p>0.05$).
Same symbol (*) indicates no significant differences among the conditions of same material analyzed using the t-test ($p>0.05$).
N=10.

Table 5 Shear bond strengths to enamel surface (MPa)

Material	P/L	Immediately	After 24 hours	After 20,000 thermocycles
SF5	4.0	7.9 ± 1.6 [a]	27.5 ± 5.1 [a]	20.7 ± 4.2 [a]
SF10	4.4	7.3 ± 2.0 [a,b,c]	22.5 ± 5.3 [b,c*]	21.1 ± 3.1 [a*]
SF20	4.0	6.9 ± 1.4 [a,b,c]	19.7 ± 4.9 [c,d*]	17.1 ± 4.7 [a*]
UF5	4.4	7.0 ± 1.4 [a,b,c]	21.2 ± 4.7 [b,c,d]	15.6 ± 4.2 [a,b]
UF10	4.4	6.4 ± 2.2 [a,b,c]	18.0 ± 4.9 [d]	13.1 ± 3.3 [b,c]
UF20	4.0	6.0 ± 1.0 [c]	12.1 ± 3.9 [e]	8.7 ± 1.9 [c]
Control	3.6	7.6 ± 1.7 [a,b]	24.8 ± 3.8 [a,b]	19.3 ± 3.5 [a]
Base	3.0	6.4 ± 1.7 [b,c]	23.9 ± 3.4 [a,b*]	20.4 ± 2.6 [a*]

SF: Silanized spherical silica filler-added Fuji II LC EM.
UF: Untreated spherical silica filler-added Fuji II LC EM.
Control: Fuji II LC EM mixed with P/L 3.6.
Base: Fuji II LC EM mixed with P/L 3.0.
Same alphabets indicate no significant differences among the mean values within same condition analyzed using the t-test ($p>0.05$).
Same symbol (*) indicates no significant differences among the conditions of same material analyzed using the t-test ($p>0.05$).
N=10.

Table 6 Shear bond strengths to dentin surface (MPa)

Material	P/L	Immediately	After 24 hours	After 20,000 thermocycles
SF5	4.0	7.6 ± 2.2 [a]	13.5 ± 3.6 [a*]	15.2 ± 1.9 [a*]
SF10	4.4	7.4 ± 1.5 [a]	10.3 ± 3.6 [b,c]	13.6 ± 3.7 [a,b]
SF20	4.0	7.9 ± 3.7 [a]	10.3 ± 3.6 [b,c*]	11.0 ± 0.9 [a,b,c*]
UF5	4.4	7.7 ± 1.6 [a]	11.5 ± 3.3 [a,b*]	10.8 ± 0.8 [b,c*]
UF10	4.4	7.5 ± 2.6 [a]	10.0 ± 2.5 [b,c*]	9.1 ± 1.3 [c,d*]
UF20	4.0	7.3 ± 2.5 [a*]	8.2 ± 2.4 [c*]	6.0 ± 3.0 [d*]
Control	3.6	6.5 ± 2.4 [a]	13.5 ± 3.8 [a*]	13.2 ± 3.5 [a,b,c*]
Base	3.0	6.4 ± 2.3 [a]	10.7 ± 3.3 [a,b,c*]	9.3 ± 1.9 [b,c,d*]

SF: Silanized spherical silica filler-added Fuji II LC EM.
UF: Untreated spherical silica filler-added Fuji II LC EM.
Control: Fuji II LC EM mixed with P/L 3.6.
Base: Fuji II LC EM mixed with P/L 3.0.
Same alphabets indicate no significant differences among the mean values within same condition analyzed using the t-test ($p>0.05$).
Same symbol (*) indicates no significant differences among the conditions of same material analyzed using the t-test ($p>0.05$).
N=10.

bond strength to dentin, both 24-hour and thermocycled specimens also showed higher values than the immediate specimens. In particular, with SF-added RMGICs, the thermocycled specimens showed higher values than the 24-hour specimens, while other materials showed the inverse tendency.

Table 7 presents the correlations among the properties. Significant correlations were observed between shear bond strength to tooth enamel and flexural strength for the immediate, after-24-hour, and thermocycled conditions (r噂0.79, $p<0.05$, $n=8$). Significant correlations were also observed between marginal gaps in Class V cavities and compressive strength or DTS, especially in the immediate and after-24-hour conditions (r噂0.83, $p<0.05$, $n=8$). In addition, there was a significant correlation between compressive strength and DTS (r 噂 0.88, $p<0.01$, $n=8$). However, significant correlations between flexural strength and compressive strength or DTS were observed only after 24 hours or after thermocycling (r噂0.76, $p<0.05$, $n=8$). Likewise, significant correlations between shear bond strength to enamel and shear bond strength to dentin were observed only after 24 hours and after thermocycling (r 噂 0.83, $p<0.05$, $n=8$).

DISCUSSION

A thermocycling process of 20,000 times in water between 5℃ and 55℃ is equivalent to water storage with repetitive dynamic loading for 28 days or about one month. Thermocycling test was carried out in this study because it aptly represents the condition in the oral cavity. Indeed, apart from the characteristics of restorative materials, the success of RMGIC restorations is inextricably dependent on the condi-

Table 7 Correlations among the properties analyzed using Pearson product−moment correlation

Correlation between			Immediate		After 24 hours		After 20,000 thermocycles	
			r	p	r	p	r	p
Class V marginal gap	vs	Shear bond strength to enamel	0.18	>0.05	0.60	>0.05	0.78	<0.05
		Shear bond strength to dentin	0.69	>0.05	0.54	>0.05	0.90	<0.01
		Compressive strength	0.87	<0.01	0.88	<0.01	0.66	>0.05
		DTS	0.83	<0.05	0.83	<0.05	0.74	<0.05
		Flexural strength	0.26	>0.05	0.87	<0.05	0.74	<0.05
Shear bond strength to enamel	vs	Shear bond strength to dentin	0.07	>0.05	0.88	<0.01	0.83	<0.05
		Compressive strength	0.39	>0.05	0.41	>0.05	0.55	>0.05
		DTS	0.52	>0.05	0.51	>0.05	0.65	>0.05
		Flexural strength	0.93	<0.001	0.80	<0.05	0.79	<0.05
Shear bond strength to dentin	vs	Compressive strength	0.82	<0.05	0.34	>0.05	0.70	>0.05
		DTS	0.67	>0.05	0.46	>0.05	0.81	<0.05
		Flexural strength	0.11	>0.05	0.63	>0.05	0.83	<0.05
Compressive strength	vs	DTS	0.92	<0.01	0.88	<0.01	0.91	<0.01
		Flexural strength	0.53	>0.05	0.84	<0.01	0.88	<0.01
DTS	vs	Flexural strength	0.62	>0.05	0.76	<0.05	0.82	<0.05

tion of the environment. In the context of the oral cavity, water − as the major component of saliva − plays a major role in filler-matrix bond failures in resin-based matrices. It causes filler elements to leach out; it induces filler failures and filler-matrix debonding, thereby reducing the strength of matrix material because debonded fillers may act as stress concentrators and significantly multiply the number of potential crack growth sites; and it has a plasticizing effect on the matrix[22,23]. However, in this study, the strength of RMGIC restorations increased after water storage for 24 hours or after thermocycling − a stark contrast to the supposed detrimental effects of water. Significant correlations between shear bond strength to enamel and shear bond strength to dentin after 24 hours and after thermocycling for 20,000 times could lead to the assumption that RMGICs obtained their stability after 24 hours with minimal adverse changes after 28 days. Besides, a previous review[12] concluded that 10,000 thermocycles correspond to one year of in vivo degradation. Therefore, the interfacial integrity, mechanical strength, and bond strength produced by these RMGICs should be durable for at least two years of clinical service.

In this study, it was shown through the three tested conditions that the addition of spherical silica filler caused significant improvements in two aspects: reduced sum of interfacial gaps in RMGIC as well as increased mechanical strength. As for the role of spherical silica fillers, it was reported that the addition of spherical silica filler to a RMGIC powder improved the flowability or workability of the cement − based on the latter's rolling performance. Further, SF − whereby silanization of filler depends on the siloxane bridge (Si-O-Si) formation between the silica surface and silane molecule − has been shown to improve the mechanical properties of RMGICs[2,4,5]. These improvements occurred due to one or the combination of the following reasons. First, the SF- or UF-added RMGIC was mixed with a higher P/L in this study. It has been shown in previous studies[4,5] that a higher P/L resulted in a smaller sum of interfacial gaps in the tooth cavity, and at the same time imparted a greater mechanical strength to RMGIC. The second reason is that silica fillers do not shrink; hence the higher the amount of fillers, the smaller would be the shrinkage[5]. Therefore, in this study, the reduced sum of interfacial gaps (as shown through the three tested conditions) and the significantly improved mechanical strength of RMGICs could be attributed to the abovementioned reasons.

As for the influence of curing shrinkage, it was reported that it affects bond strength − especially in Class V cavities − by creating marginal gaps and thereby leading to restoration failures[19,22,24,25]. Since silica fillers do not shrink, previous studies have shown that the addition of spherical silica fillers served to reduce the sum of interfacial gaps in the immediate condition as well as significantly improved the mechanical strength of RMGICs[4,5]. Apart from the contributory factor of silica filler, the factor of water uptake into the RMGIC matrix to form a poly-HEMA complex would contribute to eliminating the marginal gaps too[3,26]. In this study, thermocycling reduced the frequency of marginal gaps more than just mere immersion of specimens in the water for 24 hours. Although there was undoubtedly a shrinkage effect arising from the chemically initiated polymerization reaction[22], the former

effect ensured that the delayed acid-base reaction in RMGIC[8] did not create more shrinkage with fluctuation of temperature between 5℃ and 55℃. Thus, resin-modified glass ionomer luting cements seemed capable of providing sealings that are biologically compatible and which prevent bacterial penetration[27].

As shown in this study, the sum of interfacial gaps formed had significant correlations with many mechanical properties (Table 7). In other words, improved mechanical properties of RMGIC were achieved by using power/liquid ratios higher than that recommended by the manufacturer[5].

Many factors, apart from the mechanical properties of the restorative material, challenge the interfacial integrity of RMGIC restorations. Van Meerbeek et al.[26] reported that resin concentration was highest at the top of the hybrid layer, and lowest near the base of decalcified superficial dentin. If resin did not penetrate through the full depth of the decalcified zone, the non-infiltrated weak collagen layer at the bottom of the zone might perhaps be susceptible to long-term hydrolytic degradation[28]. Therefore, the effect of thermocycling and water storage for 24 hours in 37℃ showed that temperature did not make a remarkable difference in reducing interfacial gaps in Class V cavities.

While a previous study showed that there was no significant correlation between mechanical strength of cement and its bond strength to bovine teeth[29], this study yielded different results. In particular, significant correlations were observed between shear bond strength to tooth enamel and flexural strength at the immediate condition, after 24-hour storage, and after thermocycling. Significant correlations were also observed between interfacial gap formation in Class V cavities and compressive strength or DTS, especially in the immediate condition and after 24 hours. Thus, it could be generally concluded that a RMGIC with higher flexural strength would show higher shear bond strength to enamel, and that the fewer the sum of interfacial gas in a Class V cavity, the stronger the compressive strength and DTS of the restorative material. This was because in the current study, it was shown that there was a significant correlation between compressive strength and DTS.

It has been shown that the addition of SF up to 20 wt% increased the mechanical strength[2,4,5]. At the thermocycled condition, the addition of 10 wt% SF was the most effective in reducing interfacial gaps in Class V cavities as well as improving compressive strength, flexural strength, and shear bond strength to enamel, whereas 5 wt% SF improved DTS and shear bond strength to dentin. Although silanization is important for fillers, it seemed that 10 wt% UF — just like 5 wt% UF — also gave promising results in terms of strength values after thermocycling[5]. However, in terms of shear bond strength, 10 wt% and 20 wt% UF addition resulted in significantly reduced strengthening effect after thermocycling — and shear bond strength is an important indicator of the success or failure of a restoration. It was assumed that this condition occurred because many cracks originated from bubbles or the filler-resin interface[30]. As such, the results of this study further showed evidence that silane, as a filler-matrix coupling agent, enhances the physical properties of RMGICs and allows for adequate wetting and dispersion of the fillers within the considerably more hydrophobic resin matrices, although the latter depends on the hydrophilicity of the silane agent[31].

A previous study has shown that the addition of spherical silica fillers reduces fluoride release from RMGICs[4]. However, there are possible means to fluoride-recharge GICs to ensure continued release of fluoride[32—34]. On this note, the fluoride recharge ability of spherical silica filler-added RMGICs is another important subject worthy of further investigation in future studies.

CONCLUSIONS

A thermocycling process of 20,000 times did not adversely affect the interfacial integrity of Class V cavities restored with SF-added RMGICs. Except for shear bond strengths to enamel and dentin with UF specimens, thermocycling otherwise increased the mechanical properties of spherical silica filler-added RMGICs. Indeed, on the overall, the mechanical properties of spherical silica filler-added RMGICs after thermocycling were significantly higher than at the immediate condition and after 24-hour storage. Further, comparison between the 24-hour and thermocycled conditions showed that flexural strength yielded the most prominent increase after thermocycling. Despite the general improvement exhibited by SF-added RMGICs, it was found that the addition of 5 or 10 wt% of silanized spherical silica fillers to RMGIC powder yielded the best results in improving the mechanical properties of RMGICs.

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