The Effect of the Pre-Conditioning Step on the Shear Bond Strength of Nano-Filled Resin-Modified Glass-Ionomer to Dentin

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ABSTRACT

Objectives: The purpose of this study was to evaluate the effect of the pre-conditioning step using different dentin conditioners on the shear bond strength (SBS) of the nano-filled resin-modified glass ionomer (RMGI) to dentin.

Methods: Twenty-four human molars were used in this study. Subsequent to teeth sectioning in the mesio-distal direction and creation of the smear layer, the teeth were divided into six groups with and without the pre-conditioning step. Dentin surfaces were restored with RMGI cylinders and the specimens were stored in distilled water at 37ºC for 24 hours. SBS test were performed using a Universal Testing Machine at a crosshead speed of 0.5 mm/minute. Statistical analysis was performed using One-Way ANOVA followed by Duncan's Multiple Range Test. RMGI/dentin interface was evaluated using SEM operated at 12 Kv at 2000X magnification.

Results: 35% phosphoric acid and EDTA yielded significantly higher SBS (12.0±1.8 and 11.9±4.4 MPa, respectively), compared to all tested groups. In addition, 25% polyacrylic acid (8.6±3.1 MPa) reported significantly higher SBS than the nano-filled RMGI when applied according to manufacturer instructions (5.5±2.2 MPa). When nano-filled RMGI was applied without its nano-primer, or directly over 25% polyacrylic acid conditioned dentin exhibited 100% pre-test failure, and their SBS were expressed as 0.0±0.0 MPa. SEM revealed that the nano-primer was unable to decalcify and hybridize the dentin surface.

Conclusions: Based on the results of the current study, the pre-conditioning step effectively improves the SBS of nano-filled RMGI to dentin. The self-adhesiveness of nano-filled RMGI was not proved in this study. (Eur J Dent 2011;5:150-156)

Key words: Dentin conditioners; Bond strength; Nano-filled resin-modified glass-ionomer.

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INTRODUCTION

Since Wilson and Kent's introduction of glass ionomer cement (GIC) during the early 70's, several modifications have been made to improve its properties.[1] Glass ionomer cements are considered the most effective biomaterial that bond to hard tooth substrates.[2,3] Comparing the retention rates of class V adhesive restorations, glass iono-

mers showed the most effective and durable bond to tooth structure.[4]

Resin-modified glass ionomer (RMGI) is a combination of glass ionomer fillers and water-soluble monomer in an aqueous solution of poly-acrylic acid.[5] While conventional GIs bond to tooth substrate by ion-exchange,[6] RMGIs bond to tooth substrates through both ion-exchange and micro-mechanical interlock.[7,8]

A newly introduced nano-filled RMGI restor-ative (Ketac Nano-100, 3M-ESPE) undergoes both glass ionomer and free radical reactions. According to its manufacturer, "Ketac Nano-100 restorative is a new technical development that combines the benefits of a resin modified light cure glass ionomer and bonded nano-filler tech-nology". Nano-filled RMGI is a light-cured paste/paste system that is delivered from a multi-dose "Clicker™ Dispenser". Its chemistry is based on metacrylate modified polyalkenoic acid, which was firstly employed in Vitrebond liner/base ma-terial (Ketac N-100, Technical product profile, 3 M-ESPE).

Nano-primer is a one-component acidic prim-er, which is essential to bond the nano-filled RMGI to dentin (Ketac Nano-100 technical product pro-file) and it does not require the pre-conditioning step of the dentin surface.[3] The manufacturer claims that the bond strength of nano-filled RMGI to dentin using the nano-primer is equivalent to the greatest number of GIs used. This claim was not confirmed in the study by Coutinho et al.[3] Con-ventional RMGI (Fuji II LC) showed a significantly higher μTBS to both enamel and dentin than that recorded with nano-filled RMGI.

The interaction of acids to tooth structure has been evaluated extensively; nevertheless, it has never been elucidated.[9] The use of different den-tin conditioners did not only improve the bond strength of self-etching primers to dentin,[10,11] but also improved the shear bond strength (SBS) of the glass ionomer adhesive.[12] Accordingly, the purpose of this study was to evaluate the effect of the pre-conditioning step on the SBS of nano-filled RMGI to dentin. The null hypothesis tested is that the pre-conditioning step will not affect the SBS of nano-filled RMGI to dentin.

MATERIALS AND METHODS

Twenty-four caries-free non-restored hu-man molars were used in this study. The teeth were collected and stored in a 0.1% thymol solu-tion for not more than one month. Each tooth was sectioned in a mesio-distal direction down to the root into two halves, using a low-speed diamond disc under copious water irrigation; then, the two halves were completely separated using a surgi-cal chisel,[12] yielding 48 halves. Each half was fixed from its dentin side to the base of a metallic rect-angular mould with double-faced adhesive tape. Auto-cured acrylic resin material (Acroston, un-der exclusive license of Acroston Dental Compa-ny-Cairo, Egypt) was poured to completely cover each half. After hardening of the acrylic resin, each dentin surface was wet ground using 600 grit SiC papers for 60 seconds to create a standardized smear layer.

Experimental procedures

The 48-halves were equally divided into 6 groups (n=8) according to the dentin surface treat-ment procedures. Materials, compositions and manufacturers are summarized in Table 1. Before the application of each dentin treatment, double-faced adhesive tape, with a hole of 3 mm in diam-eter was fixed over the dentin surface to limit the bonded surface area.

Group 1: The nano-filled RMGI was applied according to manufacturer instructions. Nano-primer was applied for 15 seconds, air-dried using gentle, compressed-air for 10 seconds, and light cured for 10 seconds with a halogen light-curing unit (Hilux, Dental Curing Light Unit, Benlioglu; Ankara, Turkey) with an output of 600 mW/cm^2. Before the application of the nano-filled RMGI, a split Teflon mould, 2 mm in thickness with a cen-tral hole of 3 mm in diameter, was fixed over the dentin surface. The material was dispensed over a pad with the aid of its Clicker™ Dispenser, mixed using a metallic spatula for 20 seconds, and ap-plied over the cured primer, then, the material was light cured for 20 seconds.

Group 2: Nano-filled RMGI was directly applied over the dentin surface, without the application of the nano-primer and light cured as in Group 1.

Group 3: The dentin surface was conditioned using 25% polyacrylic acid for 10 seconds, rinsed with a copious air/water spray for 10 seconds and, blot dried using sterile cotton, leaving the dentin surface visibly moist (wet bonding). Nano-filled

RMGI was applied directly over the dentin surface, without the application of the nano-primer and light cured, as in Group 1.

Group 4: The dentin surface was conditioned with 25% polyacrylic acid as in Group 3. Nano-primer and nano-filled RMGI were applied as described in Group 1.

Group 5: The dentin surface was etched using 35% phosphoric acid for 15 seconds, rinsed for 20 seconds and blot dried as in Group 3. Nano-primer and nano-filled RMGI were applied as described for Group 1.

Group 6: The dentin surface was conditioned with EDTA solution for 60 seconds, rinsed for 20 seconds and blot dried as in Group 3. Nano-primer and nano-filled RMGI were applied as described for Group 1.

Subsequent to light curing of the nano-filled RMGI, the Teflon mould was dissembled, and the dentin surfaces- with their attached RMGI cylinders- were stored in distilled water for 24 hours at 37°C.

SBS testing

After 24 hours, each specimen was mounted to the universal testing machine (LR5K series, Lloyd Instrument; Fareham, UK). A specially fabricated, metallic, chisel-bladed instrument was positioned as accurately as possible on the nano-filled RMGI/dentin interface. The test was run at a crosshead speed of 0.5 mm/minute until failure. The load recorded in Newton was divided over the calculated surface area and the SBS was retrieved in MPa. Groups 2 and 3 were not tested, as all specimens of these two groups failed before testing during the 24-hour storage period, and their SBS was expressed as 0.0 MPa for statistical analysis.

SEM evaluation

An additional eight human molars were included to evaluate the nano-filled RMGI/dentin interface for Groups 1, 4, 5 and 6, two teeth per group. The occlusal enamel of each tooth was wet ground using the laboratory trimmer to expose a flat dentin surface surrounded by enamel. Subsequent to enamel removal, the dentin surfaces were wet ground using 600 grit SiC paper for 60 seconds, in order to create a standardized smear layer. The teeth of the above mentioned groups (Groups 1, 4, 5 and 6) were treated as described for SBS

testing. After each treatment, an approximately 2 mm thick nano-filled RMGI block was applied and light cured for 20 seconds. After 24-hour storage period in distilled water at 37°C, each tooth was sectioned mesio-distally using the low speed diamond disc under copious water irrigation to the level of the cervical line into two halves. Then, the two halves were separated by an additional horizontal cut. Two specimens were obtained from each tooth (four specimens per group). The evaluated surfaces were wet ground with 600, 800, 1000 grits and finally with 1200 grit SiC papers.

The evaluated surfaces were acid etched with 35% phosphoric acid for 15 seconds, rinsed for 20 seconds and, then, gently air dried. The specimens were immersed in 5% sodium hypochlorite for 120 seconds, and washed under running water for 5 minutes. The specimens were dehydrated in ascending concentration of ethanol, 50%, 70%, 90% for 20 minutes each and, then immersed in 100% for 1 hour. The specimens were left to dry on absorbent paper in closed container over night.

The specimens were fixed on specimen's holder using double-faced stickers and sputter coated (BAL-TEC, SCD 005 sputter coater, Germany). Nano-filled RMGI/dentin interface was evaluated at 2000X using SEM (Philips XL series, XL30, Holland) operated at 12 Kv.

All data of SBS in MPa was expressed as mean±standard deviation (SD). Statistical analysis was carried out using SAS program (SAS, 1988). One Way Analysis of Variance (Procedure ANOVA of SAS) followed by Duncan's Multiple Range Test were used to test the effect of dentin conditioners on SBS.

RESULTS

SBS results for the effect of different dentin conditioners are shown in Table 2. One way ANOVA followed by Duncan's Multiple Range Test revealed that the pre-conditioning step using 35% phosphoric acid and EDTA solution (Groups 5 and 6, respectively) yielded statistically significant higher SBS compared to all tested groups (12.0±1.8 MPa and 11.9±4.4 MPa, respectively). No statistical significant was recorded between these two above groups. When the dentin was pre-conditioned with 25% polyacrylic acid (Group 4, 8.6±3.1 MPa), it reported a statistically significant higher SBS than that when the nano-filled RMGI

was applied according to manufacturer instructions (Group 1, 5.5±2.2 MPa). SBS of nano-filled RMGI when directly applied either over dentin surface (Group 2) or over conditioned dentin with 25% polyacrylic acid (Group 3) showed significant differences between all groups (0.0±0.0 MPa and 0.0±0.0 MPa, respectively), which exhibited 100% pre-test failure in all their specimens. No significant differences were observed between the two above groups.

SEM evaluation

SEM evaluation of nano-filled RMGI/dentin interface, when the material was applied according to manufacturer instructions (Group 1) revealed no evidence of hybrid layer formation or resin tag extensions. A gap was detected between the restoration and underlying dentin, and the smear layer was observed over the dentin surface (Figure 1). When the dentin surface was pre-conditioned with 25% polyacrylic acid prior to the use of the nano-primer (Group 4), few discrete and very shallow resin tags extensions were noticed, although a thin hybrid layer was observed (Figure 2).

Numerous long, funnel-shaped, resin tag extensions were detected when the dentin surface was pre-conditioned with 35% phosphoric acid prior to the application of the nano-primer (Group 5). A thick hybrid layer was evident with filler distribution at the bottom and within the hybrid layer, as well as around the orifices of the dentinal tubules (Figure 3). Long and thin resin tag extensions were noticed when the dentin surfaces were pre-conditioned with EDTA solution for 60 seconds prior to the application of the nano-primer (Group 6). The hybrid layer was thinner than that detected with Group 5, with a few discrete filler distributions (Figure 4).

DISCUSSION

Self-adhesiveness properties to tooth structure[3], anticariogenicity[13] and minimal tooth/restoration interface microleakage[14] are among the advantages presented by glass ionomer cements. The introduction of RMGIs was aimed to overcome the drawbacks encountered with conventional GIs.[15] A new approach to improve some properties of conventional RMGIs is the introduction of

Table 1. Materials used in this study, their compositions and their manufacturers.

Materials	Compositions	Manufacturers
Ketac Nano-100 (nano-filled RMGI)	Silane treated glass 40-50%, silane treated zirconia oxide silica 20-30%, polyethylene glycol dimethacrylate 5-15 %, silane treated silica 5-15%, HEMA 1-10%, glass powder <5%, BISGMA <5%, TEGDMA <1%.	3M ESPE, Dental products, Seefeld-Germany
Ketac Nano-primer	HEMA 35-45%, water 40-50%, Vetremer copolymer 10-15%, photoinciator. pH≈3	3M ESPE, Dental products, Seefeld-Germany
Ketac Conditioner	25% polyacrylic acid, pH=1.5-2	3M ESPE, Dental products, Seefeld-Germany
Scotchbond etchant	35% phosphoric acid gel	3M ESPE, Dental products, Seefeld-Germany
Ethylenediaminetetraacitic acid (EDTA)	Disodium EDTA 18.87%, Potassium Hydroxide 2.62%, Deionized water 78.53%. pH=7.4	Dentsply, Petropolis, R.J, Brazil

HEMA= 2-hydroxyethyl metacrylate, BISGMA= Bisphenol A Diglycidyl Ether Dimethacrylate, TEGDMA= Triethylene Glycol Dimethacrylate.

Table 2. Means±standard deviations, minimum and maximum in MPa for the effect of different conditioners on SBS of nano-filled RMGI to dentin.

Conditioners	Means±SD	Min	Max	dt
Group 1	5.5±2.2	3.8	9.6	c
Group 2	0 .0±0.0	0	0	d
Group 3	0.0±0.0	0	0	d
Group 4	8.6±3.1	4.9	13.6	b
Group 5	12.0±1.8	8.6	14.1	a
Group 6	11.9±4.4	6.5	19.4	a

SD= Standard deviation. dt= Duncan's Multiple Range Test for the effect of treatment. Min= Minimum. Max= Maximum.

Means with the same letter within each column are not significantly different at P=0.05.

a nano-filled RMGI (Ketac Nano-100, technical material profile). This new material requires the application of nano-primer without the need for a pre-conditioning step. This reduces the number clinical application steps and consequently reduces clinical procedure time.

The use of both mild and aggressive conditioners in this study was aimed to evaluate whether the pre-conditioning step can improve the bond strength and facilitate the hybridization of the nano-primer to dentin. Based on our results, the null hypothesis was rejected. The pre-conditioning step not only significantly improved the SBS of nano-filled RMGI, but also allowed for hybridization to dentin.

In this study, occlusal dentin was used to evaluate the nano-filled RMGI/dentin interface. It was reported that when the dentinal tubules were cut obliquely, RMGI adhesive failed to form resin

tags.[13] In the occlusal dentin, the dentinal tubules were cut with an "open end," which might facilitate the penetration of the resin in the acidic primer to form resin tag extensions.

Although, the shear test, does not actually assess the adhesive bond,[16] it is thus far the most popular technique to measure bond strength. The shear test is the easiest and fastest method and does not require further specimen preparations after the completion of bonding procedures.[17]

SEM in this study showed the presence of a smear layer over the dentin surface and the lack of hybridization formation (Figure 1), when the nano-filled RMGI was applied according to manufacturer instructions. This could be attributed to the inability of nano-primer to decalcify the underlying dentin, which might be due to the high pH of the nano-primer (≈3). Our result confirmed the finding reported by Coutinho et al,[3] despite the differ-
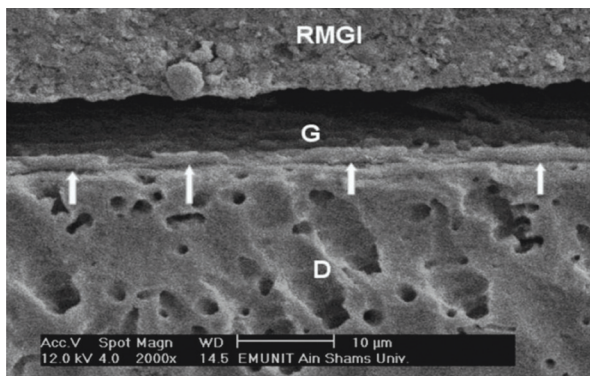

Figure 1. SEMicrograph of nano-filled RMGI/dentin interface for Group 1. No evidence of hybrid layer or resin tag extensions, with a Gap (G) between the restoration and underlying dentin. Smear layer remnants (arrows) are noticed over the dentin surface. RMGI= Nano-filled resin-modified glass-ionomer and D= Dentin.
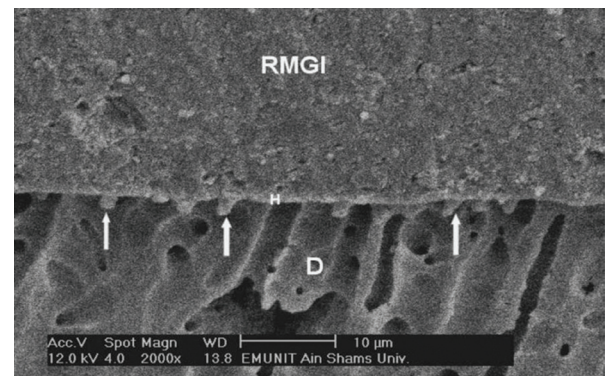

Figure 2. SEMicrograph of nano-filled RMGI/dentin interface for Group 4. Few discrete and very shallow resin tags extensions (arrows) with a thin hybrid layer (H). RMGI= Nano-filled resin-modified glass-ionomer and D= Dentin.
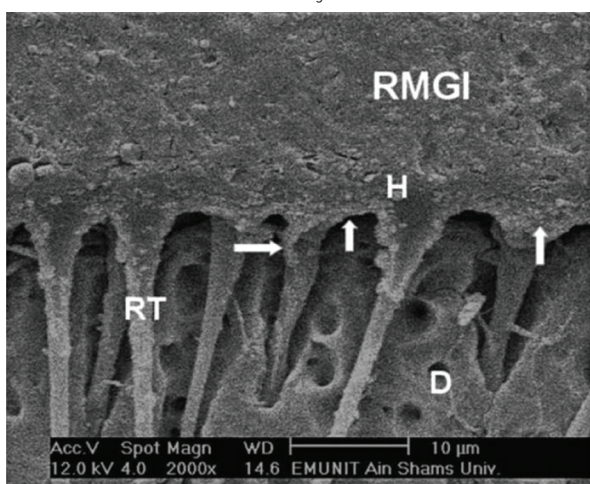

Figure 3. SEMicrograph of nano-filled RMGI/dentin interface for Group 5. Numerous long, funnel-shaped resin tag extensions (RT) with a thick hybrid layer (H). Fillers distributed at the bottom of and within the hybrid layer, as well as around the orifices of the dentinal tubules (arrows). RMGI= Nano-filled resin-modified glass-ionomer and D= Dentin.
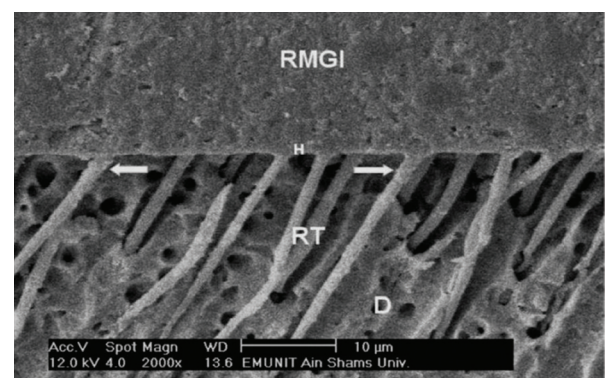

Figure 4. SEMicrograph of nano-filled RMGI/dentin interface for Group 6. Long and thin resin tag extensions (RT), the hybrid layer (H) is thinner than that detected in Group 5 with a few discrete filler distributions (arrows). RMGI= Nano-filled resin-modified glass-ionomer and D= Dentin.

ences in evaluation methodology between the two studies. They reported that nano-primer could not decalcify the underlying dentin surface, resulting in no evidence of hybridization. The gap noticed in SEMicrographs (Figure 1) could be attributed to the lack of hybridization and resin tag extensions in the underlying dentin, where contraction of dentin could be expected during specimen processing for SEM evaluation, which in turn allows for the separation of such material at its weaker point.

In this study, the significant improvement in the SBS of nano-filled RMGI with the pre-conditioning step could be explained by the SEM results (Figures 2, 3 and 4). It was reported that polyacrylic acid removed the smear layer without widely opening the dentinal tubules.[18] Discrete and short resin tags, with the formation of the thin hybrid layer (Figure 2), might cause the significant improvement in SBS when 25% polyacrylic acid was used.

Regardless of the difference in the aggressiveness of phosphoric acid[19-21] and EDTA solution,[11,22] both reported comparable SBS results. SEM showed that (Figures 3 and 4), both phosphoric acid and EDTA (Groups 5 and 6, respectively) decalcified the underlying dentin, facilitating the diffusivity of the nano-prime through the decalcified dentin surface. Phosphoric acid widely opened the dentinal tubules, resulting in the formation of numerous long and funnel-shaped, resin tag extensions (Figure 3). Furthermore, a thick hybrid layer with filler distributions at the bottom of and within the hybrid layer as well as, around the orifices of the dentinal tubules was evident (Figure 3). On the other hand, EDTA did not aggressively decalcify the dentin surface or widely open the dentinal tubules, resulting in the formation of long and thin resin tags, thinner hybrid layer, and much less filler distributions (Figure 4). It was reported that the formation of the hybrid layer and resin tag extensions is crucial to resist the acute de-bonding stresses[23] that could occur during bond strength testing.[24] As different hybrid layer thicknesses were evident in SEM, given three different dentin conditioners used in this study, our SEM results suggest that the length of the resin tags played a major role in the enhancement of the SBS of nano-filled RMGI for Groups 5 and 6, when compared to all tested groups.

In the experimental design of this study, two further groups were included. One of these two groups, the nano-filled RMGI was bonded directly to the dentin surface with neither nano-primer nor dentin conditioners (Group 2) and the other was the nano-filled RMGI bonded directly on conditioned dentin surface with 25% polyacrylic acid for 10 seconds without the use of the nano-primer (Group 3). These two groups were included to assess whether nano-filled RMGI has the self-adhesiveness property, which is one of the main advantages of the conventional RMGI. Unfortunately, based on the SBS results of this study, nano-filled RMGI lacked the self-adhesiveness property. This result, specifically for Group 2 in this study, was consistent with the results of Coutinho et al,[3] in spite of these studies' differences in bond strength testing methodology. They reported that when nano-filled RMGI was directly bonded to dentin, its bond was limited to the roughness that resulted from dentin surface preparation. Therefore, Groups 2 and 3 were omitted from the SEM section of the study.

Advances in material-manufacturing technology might improve some properties, but on the other hand, it could ruin other important and unique properties of the materials. Based on this fact, additional efforts should be made to carefully assess these newly introduced materials.

CONCLUSIONS

Based on the results of the current study, several conclusions can be drawn:

• The pre-conditioning step effectively improves bonding of the nano-filled RMGI to dentin.

• The self-adhesiveness of nano-filled RMGI was not proved in this study, even with the use of 25% polyacrylic acid conditioner.

• The enhancement in bonding of the nano-filled RMGI depended on the presence of long resin tag extensions in the underlying dentin.

Nevertheless, further studies should be conducted to assess the durability of nano-filled RMGI bond when a pre-conditioning step is used.

ACKNOWLEDGMENTS

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