

ORIGINAL ARTICLE

## Effect of different surface treatments on the shear and microtensile bond strength of resin-modified glass ionomer cement to dentin

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### Abstract

**Objective.** The aim of this study was to evaluate the effect of different surface treatments on the microtensile bond strength ( $\mu$ TBS) and shear bond strength (SBS) of resin-modified glass ionomer cement (RMGIC) to dentin. **Materials and methods.** Fifty-two extracted human molars were flattened to obtain dentin surfaces. For SBS assessment 40 teeth were divided into four groups according to their surface treatments (acid etching, Er:YAG laser QSP mode, Er:YAG laser MSP mode and control-SiC) ( $n = 10$ ). A plastic cylinder was placed over the differently treated dentin surfaces and RMGIC was placed into the rings and polymerized. Twelve teeth were used for the  $\mu$ TBS test. The treated dentin surfaces described above were restored with 4 mm high RMGIC and light cured; then, the specimens were sectioned into serial sticks ( $n = 15$ ) and  $\mu$ TBS and SBS were tested for failure in a testing machine with a 1 mm/min crosshead speed. The data were analyzed by one-way ANOVA and Tukey HSD tests ( $\alpha = 0.05$ ). **Results.** Acid etching showed significantly higher SBS than the other groups ( $p < 0.05$ ). Er:YAG QSP and MSP-treated groups showed higher SBS values than the control group, but the difference was not statistically significant ( $p > 0.05$ ). Er:YAG MSP showed the highest  $\mu$ TBS value followed by acid etching, whereas the control group exhibited the lowest value ( $p < 0.05$ ) and the differences between the control group and Er:YAG QSP were not significant ( $p > 0.05$ ). **Conclusions.** The application of Er:YAG MSP mode and acid etching to dentin can be used for improving the bond strength of RMGIC.

**Key Words:** acid etching, Er:YAG lasers, glass ionomer, shear strength, tensile strength

### Introduction

Conventional glass ionomer cements (GICs) were developed in the early 1970s [1]. The favorable characteristics of GICs include continuous fluoride release, inhibiting of bacterial acid metabolism and activity, biocompatibility, chemical bonding to both enamel and dentin and effective bonding in a moist environment without the need for an additional bonding agent layer [2–5]. Nevertheless, GICs have some disadvantages, such as handling properties, esthetics and strength, prolonged setting time, moisture sensitivity during initial setting, dehydration and rough surface texture, which can hamper mechanical resistance [6].

Resin-modified glass ionomer cements (RMGIC) have been developed in an attempt to improve the chemical and physical properties of conventional GICs, combining their properties with those of composite resins [5]. These provide better retention and seal due to their chemical bonding to tooth structure, which results in a reduction of microleakage and keeps the marginal gap in non-enamel margins [7]. RMGICs now have an established and important place in restorative dentistry; they are used as final restorative materials and as luting cement, lining cement and pit and fissure sealant. They are particularly useful where reliable, long-term adhesion is required, such as in non-carious cervical lesions [8], and there is some evidence that they may be

associated with a reduced incidence of secondary caries [9] and also the remineralization of 'affected' (inner carious) dentin [6,10]. However, while they have higher bond strength than conventional formulations, RMGICs still have lower bond strength than composite resins, which have caused the development of different bonding protocols in order to improve their properties [5,7,11,12]. Due to their broad use, it seems necessary to evaluate the behavior of RMGICs bonded to hard dental tissues and also to assess which surface treatments will produce the best outcome.

Several new alternative preparation techniques for hard dental tissues have been suggested to improve the bond strength of restorative materials. Erbium lasers provide several advantages, such as a lack of vibration, pressure and noise, all of which make patients feel good under high- or low-speed preparations and the reduced need for local anesthesia [13,14]. Durable adhesion is required for successful restoration. The bond strength of dental resins to the tooth structure is affected by several factors. These include the types of adhesive systems, restorative materials and the method of cavity preparation [15–17]. Several characteristics of lased dentinal tissue have previously been considered as advantageous for resin bonding. They include the formation of a microscopically rough substrate surface without demineralization, open dentinal tubules without smear layer production and dentin surface sterilization [18–20]. Moreover, RMGICs can bond to unground enamel and dentin according to the manufacturer's recommendations; therefore, the following hypothesis was tested: different surface treatments to dentin by Er:YAG QSP and MSP modes and acid etching do not affect the micro tensile bond strength ( $\mu$ TBS) and shear bond strength (SBS) of RMGIC. There are few studies concerning the surface characteristics and shear bond strength (SBS) of the RMGIC to the hard tissues of teeth and composite resin following surface treatments with laser irradiation [7,11]; however, thus far, no studies evaluating microtensile bond strength ( $\mu$ TBS) with laser preparation are available in the literature and ongoing studies are being performed to assess the performance of RMGICs as a restorative material. Therefore, the aim of the present study was to assess the effects of different surface treatments on  $\mu$ TBS and SBS of RMGIC to dentin.

## Materials and methods

Prior to the study, a power analysis using G\*Power (version 3.0.10; Franz Faul, Christian-Albrechts-University, Kiel, Germany) was performed to estimate the sample size. It showed that a total sample size of 60 for  $\mu$ TBS assessment ( $n = 15$ ) would give 90% power (actual power = 0.9193; critical  $F = 2.7694$ ; non-centrality parameter = 16.224) to detect significant

differences with a 0.52 effect size at a  $p < 0.05$  significance level. For SBS assessment, a total sample size of 40 ( $n = 10$ ) would give 85% power (actual power = 0.8681; critical  $F = 2.8662$ ; non-centrality parameter = 14.400) to detect significant differences with a 0.60 effect size at a  $p < 0.05$  significance level.

A total of 52 non-carious human third molars were selected and stored in 0.1% thymol solution at 4°C for a month after extraction. The teeth were cleaned with a scaler and water/pumice slurry were embedded in the polyester resin. Then, the occlusal enamel was completely removed to expose a mid-coronal flat dentin surface. All prepared surfaces were polished with 600-grit paper for 60 s to create a standard and clinically relevant smear layer. Afterwards, all samples were examined under a stereomicroscope at 40 $\times$  magnification to ensure that no enamel was left on the surface of the dentin.

### $\mu$ TBS assessment

To determine the  $\mu$ TBS, 12 teeth were used. The teeth were divided into four groups, as defined below, according to the surface treatments ( $n = 15$ ):

- Group C: The RMGIC (Fuji II LC; GC Corporation, Tokyo, Japan) was applied on a flat dentin surface without surface treatment. These specimens were used as the control.
- Group E: The specimens were etched with 37.5% phosphoric acid for 15 s, rinsed with distilled water for 15 s and gently dried for 5 s. Then, the RMGIC was applied to the dentin surface according to the manufacturer's instructions.
- Group QSP: Er:YAG quantum-square pulse mode (QSP) laser was applied to the dentin, then the RMGIC was applied to the dentin.
- Group MSP: Er:YAG medium-short pulse mode (MSP) laser was applied to the dentin, then the RMGIC was applied to the dentin.

For laser treatment of the dentin surface, a contact handpiece (H14 C, Fotona d.d, Ljubljana, Slovenia) with a sapphire tip (8 mm long, 1.3 mm diameter) was used for irradiation. The Er:YAG laser with QSP and MSP mode (100  $\mu$ s) treatment were carried out with settings of 120 mJ, 10 Hz 1.20 Watt for 4 s. Water spray level was 6 and the distance of the sapphire tip was 2 mm from the target surface during irradiation. The irradiation distance was standardized using a custom-made apparatus consisting of a holder that positioned the handpiece in such a way that the laser beam was delivered perpendicular to the sample surface at a constant working distance from the target site.

A 4 mm-high resin layer was created in two increments on the dentin samples, after each increment was light cured with a LED light curing unit (Elipar S10, 3M ESPE, St. Paul, MN) according to the

manufacturer's recommendations. The specimens were stored in distilled water at 37°C for 24 h and, after storage, the specimens were sectioned with a diamond saw (Buehler, Lake Bluff, IL) under water cooling, perpendicularly to the bonding surface to create multiple beam-shaped sticks with a cross-sectional surface area of ~1.6 mm<sup>2</sup>. Each stick was measured with a digital caliper (Mitutoyo, Tokyo, Japan). Three sticks were randomly selected from each of the teeth ( $n = 15$ ). The specimens were then attached to an Instron testing machine (Dillon, Tronix Inc., Fairmont, MN) and were stressed at a crosshead speed of 1 mm/min until failure occurred. The load at failure was recorded in Newtons (N) and the bond strength was calculated in Mega Pascals (MPa) by dividing the load at failure by the adhesive surface area (mm<sup>2</sup>). Failure modes were evaluated by a single operator under a stereomicroscope (Nikon type102, Tokyo, Japan) at 40× magnification. Failure modes were categorized into three types: adhesive = 100% adhesive failure between the tooth substrate and adhesive resin; cohesive = 100% cohesive failure in the tooth substrate; mix = mixed failure with adhesive failure and cohesive failure in the tooth substrate. The data were analyzed using one-way ANOVA and Tukey HSD tests ( $\alpha = 0.05$ ).

#### SBS assessment

Forty teeth were used. Subsequently, the specimens were randomly assigned to four groups with 10 teeth in each group and the surface treatment applications described previously for  $\mu$ TBS assessment were performed for these four groups. A plastic cylinder with a 2 mm height and a 2.38 mm diameter surface area was placed over the previously treated dentin surfaces and the RMGIC was placed into the rings and light cured with a LED light curing unit according to the manufacturer's recommendations from the side of the cylinder. The samples were stored in distilled water for 24 h at 37°C and SBS was tested for failure using a knife-edge blade in a universal testing machine (Testometric, Lancashire, UK) with a 1 mm/min crosshead speed. The load at failure was recorded in Newtons (N) and the bond strength was calculated in MPa by dividing the load at failure by the adhesive surface area (mm<sup>2</sup>). The failure modes described previously were categorized for  $\mu$ TBS assessment. The data were analyzed using one-way ANOVA and Tukey HSD tests ( $\alpha = 0.05$ ).

#### Scanning electron microscopy (SEM) analysis of the dentin surface

A treated dentin surface from each group was used for the SEM evaluations. For dentin observation, the specimens were cleaned in an ultrasonic bath for 10 min and fixed in a Karnovsky solution for 1 h.

Table I. Mean shear bond strength values (MPa) and standard deviations ( $\pm$ SD) of each group.

Groups	$n$	Mean $\pm$ SD
Acid etching	10	6.61 $\pm$ 1.99 <sup>a</sup>
Er:YAG MSP	10	3.24 $\pm$ 1.03 <sup>b</sup>
Er:YAG QSP	10	2.77 $\pm$ 1.07 <sup>b</sup>
Control	10	2.53 $\pm$ 1.02 <sup>b</sup>

Different letters label statistically significant differences according to the post-hoc test ( $p < 0.05$ ).

Afterwards, they were rinsed with distilled water and then dehydrated in a graded series of alcohol solutions (70, 90 and (3×) 100%) for 10 min at each concentration. Specimens were sputter-coated with gold and examined using a SEM (Evo LS10, Carl Zeiss, Oberkochen, Germany). The entire surface was scanned and the most representative areas were photographed at 1500× magnification.

#### Results

The mean SBS values and standard deviations (SD) for the groups are presented in Table I. A comparison between the groups revealed that group etching showed a significantly higher bond strength than the other groups ( $p < 0.05$ ). Er:YAG QSP and MSP-treated groups showed higher SBS values than control group, but the difference was not statistically significant ( $p > 0.05$ ). The modes of failure for the specimens after the SBS test are presented in Table II.

The mean  $\mu$ TBS and standard deviations of the groups are presented in Table III. Group MSP showed the highest  $\mu$ TBS value followed by group etching, whereas control group exhibited the lowest value ( $p < 0.05$ ). The differences between control group and QSP were not significant ( $p > 0.05$ ). The modes of failure for the specimens after the  $\mu$ TBS test are presented in Table IV.

#### SEM observation of dentin surfaces

The scanning electron microscope micrographs (Figures 1A–C) show the morphology of the dentin surface after acid etching and laser irradiation. The surface is regular, without a smear layer and with opened

Table II. Distribution of failure modes within groups for SBS ( $n = 10$ ).

Groups	Adhesive	Cohesive	Mix
Etching	5	2	3
Er:YAG QSP	9	1	0
Er:YAG MSP	9	1	0
Control	10	0	0

Table III. Mean microtensile bond strength values (MPa) and standard deviations ( $\pm$ SD) of each group.

Groups	<i>n</i>	Mean $\pm$ SD
Er:YAG MSP	15	9.2 $\pm$ 1.6 <sup>a</sup>
Acid etching	15	6.9 $\pm$ 1.5 <sup>b</sup>
Er:YAG QSP	15	5.2 $\pm$ 1.0 <sup>c</sup>
Control	15	5.1 $\pm$ 1.1 <sup>c</sup>

Different letters label statistically significant differences according to the post-hoc test ( $p < 0.05$ ).

Table IV. Distribution of failure modes within groups for  $\mu$ TBS ( $n = 15$ ).

Groups	Adhesive	Cohesive	Mix
Etching	6	4	5
Er:YAG QSP	10	1	4
Er:YAG MSP	8	2	5
Control	12	0	3

dentinal tubules in the acid-etched dentin. In the Er:YAG MSP mode-treated dentin, the surface is irregular, without a smear layer and with opened dentinal tubules. The irregularity of the surface can be attributed to the effects of laser beam scattering. The dentin surfaces treated with QSP mode were perfectly clean and flat, showing wide open dentinal tubules.

## Discussion

RMGICs are widely used in dentistry and pedodontics. They have higher adhesive properties compared with conventional GICs, they can absorb or release fluoride [21] and they can bond to moist environments, eliminating the need to keep the teeth dry during bonding [22]. These properties are advantageous for dental fillings in children, which is a technique-sensitive procedure that requires isolation, especially in the mandibular posterior segment. As dental fillings are exposed to the oral cavity and are intended to serve in the mouth for a long period of time, fluoride release and uptake is thought to reduce the risk of decalcification.

The microtensile measurement is a very sensitive technique and, when materials or substrates with relatively low bond strength values are tested, the specimens tend to fail prematurely during preparation [15]. In the present study a high proportion of the RMGIC specimens failed prior to testing; therefore we made sticks with a 1.6 mm<sup>2</sup> surface area to minimize this problem. If the specimens failed prior to testing, they were excluded from the  $\mu$ TBS calculation.

Cardoso et al. [23] reported that RMGIC bonds to dentin by micro-mechanical and chemical bonding mechanisms; however, micro-mechanical adhesion does not play an important role in the bonding mechanism of RMGIC and chemical interaction remains the main mechanism their bonding relies on. To improve the adhesion of RMGIC, some authors have suggested that the dentin surface should be treated with agents to promote smear layer removal or modification. This procedure tends to improve resin monomer (HEMA) penetration into the dentinal tubules, thus creating micromechanical retention and improving adhesion [5,12,24,25]. In the present study, acid etching of the dentin significantly improved the SBS and  $\mu$ TBS of the RMGIC when compared to the control groups. It may be that phosphoric acid significantly enhances the surface energy of the dentin, thus providing more micro-retention. Phosphoric acid is strong enough to dissolve and, upon rinsing, remove the smear layer. Based on SEM examinations, it was reported that phosphoric acid application clearly exposes the collagen matrix and results in wide open and regular dentinal tubules [26].

The adhesive mechanism of RMGIC is based on the chemical interaction between the carboxylic groups from material and calcium ions from dental substrates associated with the chemical diffusion of polymers on to the surface [11,27]. This means that, for optimal performance, ionomeric cement requires close contact with an homogenous tooth surface [28,29]. As laser irradiation produces the disorganized, indiscriminate destruction of organic and inorganic components [11,30], it also influences the availability of calcium ions on dental structure. Therefore, mechanical and chemical adhesion of RMGIC is inherently affected [29,31]. In the present study, the laser applications to dentin did not increase the SBS

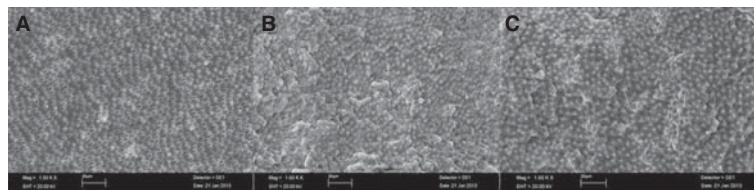


Figure 1. (A) SEM pictures for acid etching: The dentin surface is regular and clean of smear layer, showing wide open dentinal tubules. (B) SEM pictures for Er:YAG MSP: Surface is irregular and clean of smear layer and the dentinal tubules are opened. (C) SEM pictures for Er:YAG QSP: The dentin surface is perfectly clean and flat, showing wide open dentinal tubules (Magnification  $\times 1500$ ).

of RMGIC statistically. These findings are in agreement with the results of previous studies [11,32]. The Er:YAG laser may have caused excessive heat that occurs during cavity preparation, which may cause denaturation of the collagen network and a decrease in dentin permeability and it produced a modified superficial layer in which collagen fibers are poorly attached to the underlying substrate [33]. This may explain why low SBS was observed at laser-irradiated dentin. In addition, Cardoso et al. [23] reported that an Er:YAG laser caused many horizontal cracks in the subsurface of dentin. If these fissures are not infiltrated by RMGIC, they act as a stress raiser and, thus, reduce the bond strength of RMGIC to dentin [34,35]. Ceballos et al. [33] reported that Er:YAG lasers cause zones of sub-surface cracks, so these areas become the weakest part of the adhesive complex. In this study, a higher incidence of adhesive failures was observed in laser groups, indicating poor interaction between the RMGIC and the substrate.

The use of the laser has been recommended to increase the adhesion of the resin to the tooth structure. It has also been recommended for etching or modifying the surface of teeth as a substitute for acid etching [36]; however, the effectiveness of this technique is controversial [37–41]. The Er:YAG laser initially vaporizes water and other hydrated organic components of the tissue; upon vaporization, the internal pressure increases inside the tissue until the explosive destruction of inorganic substances occurs [42]. Since inter-tubular dentin contains more water and has a lower mineral content than peritubular dentin, it is selectively ablated at a higher rate than peritubular dentin, leaving protruding dentinal tubules with a cuff-like appearance (Figure 1). Patent tubules and the absence of a smear layer are additional factors that may enhance bonding to laser-treated dentin [18,33]. Jordehi et al. [43] and Cardoso et al. [23] reported that laser irradiation to dentin does not improve the  $\mu$ TBS of the RMGICs, which is in conflict with the findings of Ekwaropoj et al. [44], probably because of the different power and irradiation settings of the laser devices used. In the present study, the Er:YAG MSP mode increased the  $\mu$ TBS of the RMGIC; however, the Er:YAG QSP mode application increased the bonding effectiveness, but was not as expected. The Er:YAG MSP mode had deeper grooves on tooth surfaces than did the Er:YAG QSP mode according to the AFM evaluation [45]. It is likely that, in the current study, Er:YAG MSP mode showed higher  $\mu$ TBS by providing more micro-retention on the dentin surface than Er:YAG QSP mode. The Er:YAG MSP and QSP modes increased the SBS when compared to the control group, but not significantly. Acid etching increased both  $\mu$ TBS and SBS values and Er:YAG MSP mode application increased the  $\mu$ TBS of RMGIC, so the hypothesis is rejected.

A study by Hibst [46] reported that a laser beam can cause the formation of a layer of tiny flakes after tooth preparation and suggested that this layer should be removed mechanically or chemically before application of the filling material. We think that these flakes could have covered the dentin surface and reduced the bond strength. To overcome this problem, Gutknecht et al. [47] and De Carvalho et al. [48] suggested acid etching of the laser-prepared cavity. Within the limitations of this study, RMGIC showed higher SBS values to acid-etched dentin when compared to laser-treated dentin; however, the Er:YAG MSP mode-treated dentin demonstrated the highest  $\mu$ TBS value. Acid etching and laser applications to dentin can, therefore, be applied to increase the bonding effectiveness of RMGICs.

**Declaration of interest:** The authors report no conflicts of interest. This study was an oral presentation at the 18th BASS Congress, 25–28 April 2013, Skopje, Macedonia.

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