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ASSESSMENT OF MICROTENSILE BOND STRENGTH OF DIFFERENT RESIN CEMENTS USED FOR LUTING OF INDIRECT MOD RESIN COMPOSITE INLAYS TO DENTIN AFTER AGING IN DIFFERENT MEDIA

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ABSTRACT

Purpose: To assess the microtensile bond strength (μTBS) of self-adhesive and self-etch resin cements used for luting indirect MOD resin composite inlays to dentin after aging in distilled water and lactic acid.

Materials and Methods: Forty-eight sound human molars were selected, disinfected and a standardized Class II MOD cavity was prepared in each molar to be restored with indirect resin composite inlay restoration (SR Nexo, Ivoclar Vivadent AGSchaan, Liechtenstein). The molars were divided into three groups (n=16) according to the type of resin cement used for luting the restorations: total etch resin cement (All-Bond 2/Choice), self-etch resin cement (Panavia F2.0) and self-adhesive resin cement (Maxcem). According to the aging medium, every group was subdivided into two subgroups (n=8). Half of the specimens from each subgroup (n=4) were immersed in the aging medium for 24h while the other half for 168h. Specimens were sectioned to produce beams with a cross-sectional area of 1 mm2. Microtensile bond strength was evaluated using a universal testing machine and modes of bond failure were assessed using a stereomicroscope. Data was analyzed using a Three-Way analysis of variance (ANOVA) Test and Tukey's post hoc multiple comparison test.

Results: Panavia F2.0 specimens stored in distilled water for 24h revealed the highest μTBS values. While, All-Bond 2/Choice specimens stored for 168h in lactic acid showed the weakest values. Adhesive failure (at resin cement/dentin) was the predominant failure pattern.

Conclusions: Resin cement type and aging media/time had a substantial effect on the microtensile bond strength at cement/dentin interface.

KEYWORDS: Indirect resin composite inlays, resin cement, microtensile bond strength

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INTRODUCTION

The increasing demands for esthetic restorations and minimally invasive treatment procedures resulted in an increased popularity of resin composite restorative materials. In addition to being directly applied to restore posterior teeth, resin composite can be used for indirect restorations, particularly in medium-to large sized cavities, provided that adequate tooth structure is available for adhesive cementation.[1] Laboratory-processed resin composite inlays have many advantages when compared to direct restorations such as increased mechanical properties, low polymerization shrinkage, enhanced wear resistance, superior esthetics, good proximal contact and occlusal morphology.[2,3]

Cementation stage is a significant factor in warranting the durability of resin composite inlays. Selecting the appropriate resin cement, in order to achieve a strong bond at dental substrate/resin cement interface, could be perplexing due to the availability of a variety of dental adhesives.[4–7] According to the type of tooth substrate pretreatment, resin cements could be categorized into: (1) total etch adhesive cements, (2) self-etch (SE) cements, (3) self-adhesive (SA) cements.[8–15] The use of the conventional etch-and rinse luting agents entails tooth substrate pre-conditioning, priming, application of the bonding agent and finally application of the resin cement. However, these materials have been shown to be sensible to handling with time consuming application techniques.[16,17] On the other hand, SE adhesives merge the etching and priming phases into one step, by including acidic monomers followed by the application of adhesive resin.[18–21] Many advancements have been introduced in order to simplify the luting procedure and establish a strong, long-lasting bond to the tooth structure such as the development of SA luting agents. These materials do not require any pretreatment of the dentin surface, and thus have attracted the attention of clinicians and manufactures as well.[22,23]

Cements should form strong bonds and possess high strength under tension.[24,25] Also, they should have appropriate impedance to dissolution in oral fluids or acids formed in dental plaque so as to decrease the risk of cement degradation and bond degeneration which can result in restoration failure.[26,27] The influence of acids produced by dental plaque was assessed and it was shown that lactic and other acids could adversely affect the resin materials and cause them to soften and their surface to degenerate.[26,27] In addition, it was noticed that, greater micro-morphological damage occurs in resin-based materials after a regimen of acid challenge, when compared to storage in artificial saliva or distilled water.[28]

Microtensile bond strength to dentin is thought to be one of the most important factors influencing the clinical efficacy of resin cements and the durability of inlay restorations.[28] Therefore, laboratory investigation is crucial for the evaluation of microtensile bond strength (μTBS) of self-adhesive and self-etching resin cements used for luting indirect MOD resin composite inlays to dentin after specimens' storage in lactic acid and distilled water.

MATERIALS AND METHODS

Three different types of resin composite cements were chosen in this study for luting laboratory processed resin composite inlays, SR Nexo (Ivoclar Vivadent AGSchaan, Liechtenstein) including: total etch dual-cured All-Bond 2/Choice (Bisco, Inc., Schaumburg, IL, USA), self-etch dual-cured Panavia F2.0 (Kurary medical, Okayama, Japan) and self-adhesive dual-cured Maxicem (Kerr, Orange, CA, USA). The materials' full description is provided in **Table 1 and 2**. All materials were used following the manufacturers' instructions. A light curing unit (LED Bluephase C5, Ivoclar, Vivadent, Amherst, NY, USA) with an output density of 655 mW/cm^2 was used for curing. The irradiance was checked before each procedure using Demetron LED light meters (Demetron Research Corp., Danbury, CT, USA).

TABLE (1). Resin composite cements used in this study

Resin cement	Components	Composition	Manufacturer
All-Bond 2/ Choice	Uni-Etch	32% Phosphoric acid gel, xanthum gum thickener	Bisco, Inc., Schaumburg, IL, USA
	Primer A	NTG-GMA, acetone, ethanol, water	
	Primer B	BPDM, photoinitiator, acetone	
	Pre-Bond resin	Bis-GMA, TEGDMA, benzoyl peroxide, BHT	
	D/E bonding resin	Bis-GMA, UDMA, HEMA	
Panavia F 2.0	ED primer:		Kurary medical, Okayama, Japan
	Primer A	HEMA,10- MDP, chemical initiator, water, 5-NMSA	
	Primer B	5-NMSA, chemical initiator, water panavia F2.0	
	A paste	Quartz, glass,10- MDP, methacrylate, photoinitiator	
	B paste	Silanated barium glass, NaF, methacrylate, chemical initiator	
Maxcem		GPDM, functional metacrilates, initiators, stabilizers, barium glass and aluminium–fluoride–silicate glass	Kerr, Orange, CA, USA

TABLE (2). Indirect resin composite restorative system used in the study

Materials	Composition	Manufacturer	Batch no.
SR Nexo liner	Dimethacrylates (48wt.%), barium glass filler, silicone dioxide (51wt.%), additional contents are catalysts, stabilizers and pigments (<1wt.%).	Ivoclar Vivadent AG Schaan, Liechtenstein	S14322
SR Nexo paste Layering materials (incisal & dentin)	Dimethacrylates (17-19wt.%), copolymer and silicone dioxide (82-83wt.%), inorganic filler (64-65wt.%), inorganic filler (64-65wt.%) (<1wt.%).	Ivoclar Vivadent AG Schaan, Liechtenstein	SR Nexo Dentin S37979 SR Nexo Incisal R29525
SR Nexo stain	Dimethacrylates (47-48wt.%), copolymer and silicone dioxide (49-50wt.%), additional contents are catalysts, stabilizers and pigments (2-3wt.%).	Ivoclar Vivadent AG Schaan, Liechtenstein	R60689
SR Gel	Glycerine, silicone dioxide and aluminium oxide	Ivoclar Vivadent AG Schaan, Liechtenstein	R81357
Universal polishing paste	Emulsion of aluminium oxide, ammonium oleate, petroleum distillate and water	Ivoclar Vivadent AG Schaan, Liechtenstein	P83174

Forty-eight freshly extracted human permanent molars were collected for the current study. The ethics committee at Mansoura University granted ethical approval before commencement of the study. All molars were caries, restorations and crack-free. They were hand-scaled (Zeffiro, Lascod, Florence, Italy), disinfected in aqueous solution of 0.5% chloramine for 48 hours, and stored in distilled water in a refrigerator until use. The distilled water was changed periodically every 5 days. For teeth fixation, cylindrical polyvinyl chloride (PVC) rings were filled with chemical cured acrylic resin (Acrostone, Cairo, Egypt), in which the roots of selected teeth were inserted to a level just 2 mm beneath the cementoenamel junction. Before cavity preparation, a primary impression was taken for each tooth. Then, a kit for inlay preparation (Komet, Brasseler GmbH & Co. KG, Lemgo, Germany) together with a high-speed handpiece with a water cooling system were used for MOD cavity preparation.

For cavity preparation standardization, a custom made apparatus was designed and constructed at the Production Engineering and Mechanical Design Department, Faculty of Engineering, Mansoura University to secure the handpiece in place. The cavities were prepared with the following dimensions; 4 mm buccolingually, 3 mm deep at the isthmus, 4 mm deep at the mesial and distal surfaces and the boxes were 1.5 mm at the base towards the pulp. Then, a secondary impression was recorded for each cavity and cast into die stone.

For resin composite inlays construction, model sealer was applied to harden and protect the surface of the die stone, followed by the placement of two thin coats (3 minute/coat) of SR model Separator (Ivoclar Vivadent AG Schaan, Liechtenstein). A thick coat of SR Nexco Liner (Ivoclar Vivadent AG Schaan, Liechtenstein) was applied to the cavity walls and floor then light-cured. The restoration was built up by incremental application and curing of SR Nexo Dentin followed by the placement and curing of a final layer of SR Nexo Incisal. Then, the outer surfaces of the inlays were coated with SR Nexo gel and placed in a furnace (Targis Power TP3 Upgrade, Ivoclar Vivadent AG Schaan, Liechtenstein) to complete the polymerization process. Flexible discs (Sof-Lex XT Pop On, 3M ESPE) were used for finishing the inlays following the recommended grit sequence. Then, polishing of the inlays was achieved using leather buff wheels and Universal Polishing Paste (Ivoclar Vivadent AG Schaan, Liechtenstein). Lastly, in order to achieve a robust bond to the luting resin cement, sandblasting of the internal surfaces of the inlays was done using 80-100 μm AL_2O_3 at 1 bar pressure.

According to the type of resin cement used to bond the inlay restoration to the tooth structure, the 48 specimens were divided into three groups (n=16). The cementation process of each group was done following the manufacturer's instructions. After the bonding procedure, each group was further subdivided into two subgroups (n=8) according to the type of storage media either distilled water with a pH of 7 or 0.01M buffered lactic acid with a pH of 4. Four specimens in each subgroup were stored for 24h while the remaining ones were stored for 168h.

Specimens of all subgroups were sectioned using a low speed wafering blade in an automated diamond saw (Isomet 4000, Buehler Ltd., Lake Bluff, IL, USA) under abundant water cooling. Each specimen was longitudinally and repeatedly sectioned in the buccolingual and mesiodistal directions to produce beams with a cross-sectional area of approximately 1 mm^2. To separate these beams, a final cut was made horizontally at the level of the cemento-enamel junction. Beams from the central area of the tooth only were selected where 4 beams were obtained from each tooth to get 16 beams/subgroup. Each beam consisted of dentin, resin cement and the indirect resin composite. The thickness of each beam was checked using a digital caliper (Mitutoyo, Tokyo, Japan).

To measure microtensile bond strength, each beam was fixed in a universal testing machine (Instron, MA, USA) with a load cell of 500 N and

subjected to tensile forces at a cross-head speed of 0.5 mm/min till bonding failure. Finally, the bond strength between dentin and inlay-resin cement was calculated (Bluehill Lite software, Instron, MA, USA).

A stereomicroscope was used to examine the fragments of specimens and assess the modes of bond failure which can be categorized into cohesive failure (in dentin or resin cement or in composite), adhesive failure (resin cement/dentin or resin cement/composite inlay) and mixed failure.[29]

Data was collected and statistically analyzed using a SPSS software package (SPSS Inc., Chicago, Illinois, USA). Data was checked for normal distribution using Shapiro-Wilk Test. The influence of the three variables (type of resin cement, storage media and storage time) and the interaction of these factors on the micro-tensile bond strength were analyzed using a Three-Way ANOVA Test. The μTBS means between the tested groups were compared using Tukey's post hoc multiple comparison test with statistical significance set at α=0.05.

RESULTS

Microtensile bond strength data showed a normal distribution pattern ($p>0.05$) when analyzed with Shapiro-Wilk Test. The 'type of resin cement', 'storage media' and 'storage time' significantly affected the μTBS results as indicated by Three-Way ANOVA Test. According to the results of Tukey's post hoc multiple comparison Test as mentioned in **Table 3**, specimens bonded using SE Panavia F2.0 and stored in distilled water for 24 h showed the highest μTBS values when compared to other groups. On the contrary, the lowest μTBS was exhibited by specimens bonded using the All-Bond 2/Choice (etch-and rinse resin cement) and stored in lactic acid for 168h. Specimens stored for 168h in either distilled water or lactic acid showed significantly lower μTBS when compared to the 24h storage time ($p<0.05$). In all groups, adhesive failure (at resin cement/dentin) was the prevalent type of failure, as described in **Table 4**, compared to cohesive failure (within resin cement) and mixed failure (dentin, resin cement and resin composite).

TABLE (3). The mean μTBS results of tested groups and results of Tukey's post hoc multiple comparison Test

Groups	N	Mean±SD
TE-W-24	16	14.244 ± 5.376^{e}
TE-W-168	16	12.384 ± 4.368^{e}
TE-L-24	16	13.728 ± 4.152^{e}
TE-L-168	16	10.248 ± 2.556^{f}
SE-W-24	16	28.02 ± 7.788^{a}
SE-W-168	16	20.784 ± 4.068^{c}
SE-L-24	16	24.876 ± 5.124^{b}
SE-L-168	16	13. 992 ±5.472^{e}
SA-W-24	16	21.348 ± 6.444^{c}
SA-W-168	16	17.34 ± 7.224^{d}
SA-L-24	16	16.992 ± 5.94^{d}
SA-L-168	16	12.504 ± 3.984^{e}

Abbreviations: TE=Total-etch (All-Bond 2/Choice), W=Distilled water, 24=24h, SE=Self-etch (Panavia F2.0), L=Lactic acid, 168=168h, SA=Self-adhesive (Maxcem)

TABLE (4). Modes of failure observed in tested groups

Resin cement		Fracture pattern			Total
		Adhesive	Cohesive	Mixed	
TE	Number	57	7	0	64
	Percentage	89.1%	10.9%	0.0%	100%
SE	Number	44	13	7	64
	Percentage	68.8%	20.3%	10.9%	100%
SA	Number	50	9	5	64
	Percentage	78.1%	14.1%	7.8%	100%

Abbreviations: TE=Total-etch (All-Bond 2/Choice), SE=Self-etch (Panavia F2.0), , SA=Self-adhesive (Maxcem)

DISCUSSION

Generally, residual unreacted monomers are the main constituents that could be released from resin cements within the first seven days after curing. However, any component could be leached out from the cured cement when exposed to several solvents during the first 1-3 days. Hence, in this study, a seven day (168 h) period was chosen as the maximum storage period for the resin cements under investigation.[36] The microtensile test was developed to utilize specimens with approximately 1 mm^2 cross-sectional areas. The reduction in the cross-sectional area leads to a more uniform distribution of stresses within the specimens with the merit of producing of a larger number of specimens from smaller volume of the material.[37,38]

The results of the current study demonstrated lower microtensile bond strength values for specimens that were stored in lactic acid than for the specimens stored in distilled water, especially for 168 h storage time when compared to 24h. Furthermore, specimens in which Panavia F2.0 was used as the luting cement, showed the best results followed by Maxcem and finally All Bond 2/Choice. Moreover, adhesive failure (at resin cement/dentin) was the predominant failure pattern in all groups. This can be explained by the differences in rates of sorption due to the difference in composition of resin matrices in the luting resins used. In All Bond 2/Choice resin cement, extensive hydrogen bonds are formed between polar sites of dimethacrylate monomers found in the organic matrix (-OH- in Bis-GMA, -O- in TEGDMA and Bis-EMA and -NH- in UDMA) and the functional groups found in molecules of lactic acid (-OH hydroxyl group and -COOH carboxyl group). These materials can form hydrogen bonds with acids and water increasing the resin matrix liquid uptake resulting in resin degradation and ultimately a reduction in its strength and mechanical properties.[39] On the other hand, Maxcem resin cement phosphoric acid ester monomers ($PO\text{-}O_3R_3$) form ester groups in the resin matrix that could be hydrolyzed by lactic acid forming alcohol and carboxylic molecules. These reaction products may increase the cement liability to moisture absorption increasing the chances for matrix degradation.

Moreover, chemical erosion of the surface of filler particle could be caused by the acidic pH, allowing for easier bonding and the release of weak leachable ions.[40] The acidic functional monomer 10-MDP (10-methacryloxy decyl-dihydrogen phosphate), that is found in the constituents of Panavia F2.0, has a lengthy carbonyl chain that makes it less susceptible to hydrolysis. In this study, Panavia F2.0 presented the strongest bond strength results when compared to other used cements.[41]

CONCLUSION

Resin cement type and the aging media/time have a substantial effect on the microtensile bond strength of cement/dentin interface. Self-etch resin cement exhibited acceptable μTBS to dentin.

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