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Effect of *in vitro* Aging of Resin Microinfiltrant Restoration on Shear Bond Strength of Orthodontic Bracket Adhesive.

Abstract

Objective: To determine the impact of resin infiltration subjected to in vitro conditions on the shear bond strength (SBS) and adhesive remnant index (ARI) scores of orthodontic bracket adhesive.

Materials and Methods: One hundred and twenty bovine incisors were randomly divided into 4 equal groups: control group (Group I), demineralized control group (Group II), ICON control group (Group III), and aged-ICON experimental group (Group IV). Artificial white spot lesions were created in groups II, III, and IV. Micro-invasive resin was utilized in group III, immediately before bracket bonding, and group IV, aging of the resin was conducted prior to bracket bonding. All samples were subjected to thermocycling after bracket bonding and prior to SBS and ARI testing and evaluation.

Results: Significant differences were found between the shear bond strengths of group II and groups I and III ($p<0.001$). Group IV was not significantly different in SBS values than any other group. There are significant differences between the ARI data of group II and groups I, III, and IV; as well as a significant difference between the ARI of groups III and IV.

Conclusions: Aged resin micro-infiltration does not significantly affect the shear bond strength of orthodontic brackets and increases bond strength when applied immediately prior to bonding to demineralized enamel. Resin micro-infiltration of demineralized enamel decreases the likelihood of enamel fracture at debond, whether applied immediately prior to bonding or treated at a prior appointment. However, immediate resin micro-infiltration prior to bracket bonding had significantly lower ARI scores indicating a lower potential risk of enamel fracture than aged resin micro-infiltration.

Key Words: Enamel, Demineralization, Microinfiltrant resins, White spot lesions, Preventative, Self-etch system

INTRODUCTION

In the era of preventative dentistry, the early intervention of initial smooth surface carious lesions is becoming the new standard of care. This early intervention includes the use of micro-invasive infiltrant resins. Not only is this procedure beneficial in the cessation of progression of carious lesions, it also provides an esthetic benefit to the patient in diminishing the appearance of white spot lesions (WSLs).[1,2] This procedure has been widely advocated for use in the post-orthodontic patient who develops initial smooth surface caries (white spot lesions) during treatment. According to Feng, C.H. and X. Y. Chu, as well as Paris, S. et al, resin infiltration is successful in camouflaging the appearance of WSLs post orthodontics.[1,2] Some advocates have even suggested resin infiltrant use prior to orthodontic treatment to prevent smooth surface lesions from developing. Ogodescu et al. suggested patients who present for orthodontic treatment with existing WSLs should be treated with ICON prior to bonding to aid in the cessation of progression of existing lesions and protect intact enamel from the development of lesions.[3] This is a viable recommendation with particular consideration for interdisciplinary patients, orthognathic surgical cases, or patients who are unable to maintain good oral hygiene despite reiteration of oral hygiene education.[3]

One of the challenges associated with resin infiltration is the intra-operability of provider's communication of the completion of this procedure. According to DMG America, resin infiltration with ICON of smooth surface lesions is not detectable clinically or radiographically.[4] A brief history from the patient or requesting operative dentistry notes may lend itself to the confirmation of this procedure; however, these methods can prove unreliable and time consuming. Some patients often have a vague understanding of the procedure being employed to reduce white spot lesions, such as microabrasion, resin microinfiltration or other restorative procedures. Other confounding variables are chair time and monetary compensation. Until recently there was not a specified dental preventative code for reimbursement of resin infiltration. Once implemented the amount reimbursed was not reflective of the chair time and material cost needed to complete the procedure. One smooth surface infiltrated with resin is approximately half the compensation of a one surface anterior resin restoration.[5] With proper isolation technique, completing one dental arch of resin infiltration could take as long as 45 minutes. Since bonding appointments are already a time commitment for the patient, it may be beneficial to accomplish the preventative treatment at a separate appointment prior to the bonding appointment. However, one must investigate how this will affect the bond strength of the bracket.

With a multitude of benefits to the prevention of white spot lesions, one must inquire about the effect a micro-invasive composite has on the bond strength of orthodontic brackets. Jia et al. concluded caries infiltrants do not significantly affect the shear bond strength of traditional etch and rinse systems and may enhance the shear bond strength of self-etching adhesives.[6] According to Baka et al. resin infiltrants decreased the likelihood of enamel fracture upon bracket removal from demineralized tooth structure.[7] These studies deploy table top execution without *in vivo* aging simulation. As the aforementioned studies have demonstrated, bonding directly over resin infiltration maintains the bond strength of etch and rinse systems and increases the bond strength of self-adhesive systems. The considerations of this study include the effects of previously restored demineralized lesions on the bond strength after subjected to the oral environment. This protocol replicates several clinical scenarios to include multi-phase

(Interceptive early care, phase I, phase II, etc), preventative care (struggling oral hygiene, special needs, or difficult behavior), and orthognathic surgical patients. The doctor must consider if there should be special considerations for the bonding protocol with the pre-existing resin infiltration. In an effort to decrease prolonged chair time, consideration could be made for separating the prophylactic or prescribed ICON infiltration at an appointment prior to bonding brackets. The null hypothesis of this study states there is no difference between the shear bond strength or ARI of brackets bonded to bovine enamel based on type of surface treatment.

MATERIALS AND METHODS

Bovine Enamel Selection and Preparation

One hundred and fifty bovine incisors were procured and stored in artificial saliva composed of Na_3PO_4, 3.90 mM; NaCl, 4.29 mM; KCl, 17.98 mM; $CaCl_2$, 1.10 mM; $MgCl_2$, 0.08 mM; $NaHCO_3$, 3.27mM and distilled water; titrated with H_2SO_4 to pH 7.2 at room temperature prior to and throughout the protocol.[8] Teeth were chosen based on a set of inclusion criteria used to ensure suitability with the protocol design and adequate bonding environment. Teeth were excluded if there was evidence of coronal caries, enamel fractures or hypomineralization in the bonding area, gross staining extending into the bonding area, or less than eighty percent of the root remaining. One hundred and twenty bovine incisors were randomly divided into 4 equal groups. Thirty samples were allocated to each group: control group (Group 1), demineralized control group (Group 2), ICON control group (Group 3), and aged-ICON experimental group (Group 4). See Table 1 for a schematic explanation of all groups. Three groups were subjected to a demineralization process consisting of samples placed into a demineralizing solution (pH 4.8) consisting of: 40mL of 0.1 mol/L Lactic Acid, 500 mg/L Hydroxyapetite, 20 g/L Cabopol.[7] The demineralizing solution was changed every two days for three weeks and maintained at a temperature of 37ºC.

Table I: Protocol Summary

	Demineralization	ICON	
Control (Group 1)	No	No	Bond Bracket → Thermocycle → SBS Test
Demin Control (Group 2)	Yes	No	Bond Bracket → Thermocycle → SBS Test
ICON Control (Group 3)	Yes	Yes	Bond Bracket → Thermocycle → SBS Test
ICON Experimental (Group 4)	Yes	Yes	Age ICON (Thermocycle) → Bond Bracket → Thermocycle → SBS Test

Bonding Procedure and Application of Brackets

Precoated 3M Unitek Victory Series APC II maxillary right central incisor brackets with .022 MBT Rx were bonded to the specimens using the following protocol: Pumice each tooth with toothbrush for 5sec. Rinse completely and dry. The saturated tip of the Transbond SEP applicator was rubbed onto tooth surface for 5 seconds (Transbond Plus Self Etching Primer, 3M Unitek, Puchheim, Germany). An oil and moisture-free air source delivered a gentle air burst for 1-2 seconds to dry primer into a thin film. Brackets were placed on the specimens with a fixed 500 grams of pressure. Excess material was carefully removed with microbrushes at 2.7X magnification. Light-curing of the precoated brackets were performed a total of twelve seconds each, three seconds per side (incisal, gingival, mesial, distal) with a VALO curing light (VALO Corded LED Curing Light, Ultradent Products, Inc, South Jordan, Utah, USA).

ICON

All samples in groups 3 and 4 were subjected to the following ICON (ICON, DMG America, Ridgefield Park, NJ, USA) manufacturer's protocol: Applied an ample amount of Icon-Etch onto the surface. Periodically massaged etch over the 2 minute period. Removed excess material with a cotton wad. Suctioned off Icon-Etch and rinsed with water for at least 30 seconds. Dry with oil-free and water-free air. Applied an ample amount of Icon-Dry onto the lesion, and allowed to set for 30 seconds. Dried the lesion thoroughly with oil-free and water-free air. Applied an ample amount of Icon-Infiltrant massaged periodically for 3 minutes and added material to keep the surface wet. Removed excess material with a cotton wad and dental floss. Light-cured Icon-Infiltrant for 40 seconds.

THERMOCYCLING

Thermocycling was selected to age the ICON and bracket adhesive as this process replicates the stress of the intraoral environment by thermally expanding and contracting the adhesive-enamel interface. Per El-Mowafy, et al, 3,000 thermocycles is greater than the average orthodontic treatment time.[9] 10,000 thermocycles were selected to age the ICON in Group 4 prior to bracket bonding. All groups were subjected to 5,000 rounds of thermocycling after bracket bonding. The thermocycling consisted of distilled water between 5º - 55ºC, with a dwell time in each bath of 30secs and a transfer time of 15secs.

SHEAR BOND STRENGTH AND ADHESIVE REMNANT INDEX

After thermocycling was completed, the bovine specimens were embedded into cold cure acrylic resin.[10] Immediately, samples were subjected to the shear bond strength (SBS) test and adhesive remnant index (ARI) recorded. The acrylic-tooth blocks were mounted in the Instron Universal Testing Machine holder and positioned such that the crosshead contacted the brackets between the bovine enamel and bonding pad. The crosshead speed was set to 1 mm/min. The crosshead was lowered until contact was made with the bracket. The load increased until the bracket debonded from the tooth. The maximum load achieved for each test was recorded on computer software in units of newtons (N). Each measurement was converted into megapascals (MPa) using the following equation: $1\ N/mm^2 = 1\ MPa$. The maximum load in newtons was divided by the surface area of the bracket's bonding pad (surface area = 10.52 mm^2) to yield a measurement in megapascals. The maximum shear bond strength of each of the samples was recorded. A univariate ANOVA was utilized to evaluate the SBS data with a Tukey Homogenous subset post-hoc test utilized to locate the significant differences ($\alpha = 0.05$). Brackets were recovered and adhesive remnant index (ARI) was examined by two calibrated investigators at 2.5X magnification. ARI Scores were assessed for each sample according to Table 2. According to Veh et al., a low ARI score is indicative of less risk of enamel fracture at debond.[11] ARI data was analyzed with a Kruskal Wallis test and differences were determined via a Mann-Whitney analysis. All analyses completed using IBM SPSS v24.

Table II: Adhesive Remnant Index Scoring System

Adhesive Remnant Index (ARI) Score	Observation based on percentage (%) of adhesive found on enamel after bracket failure
0	100
1	$50<X<100$
2	$0\leq X<50$
3	0

RESULTS

Shear Bond Strength

Descriptive statistics and statistical comparison data represented in Table 3 for all shear bond strength data. SBS graphically represented in Figure 1. Significant differences were found between the shear bond strengths of the demineralized control group and the control group and ICON control group ($p<0.001$). The experimental ICON group was not significantly different in SBS values than any other group.

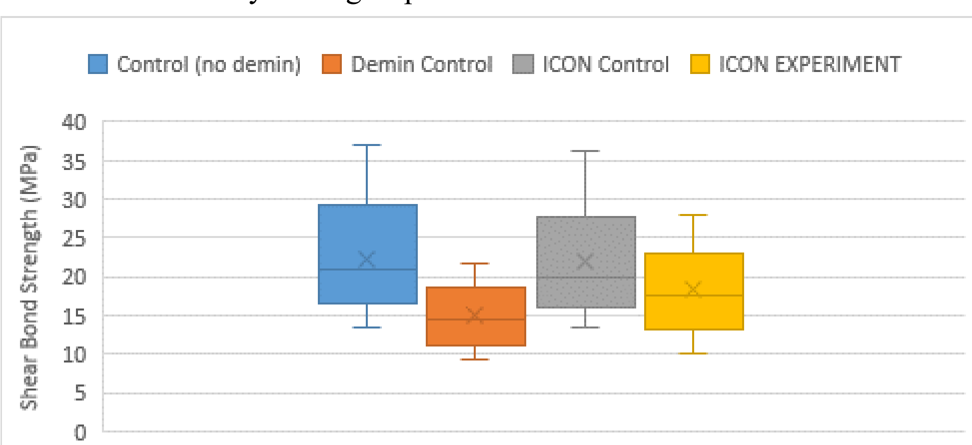

FIGURE 1: SBS Data Distribution

Table III: SBS Descriptive Statistics and Statistical Comparisons

	Mean	Std. Deviation	Min-Max	ANOVA	N	TUKEY
Control (Group 1)	22.38	6.84	13.38-36.98		24	a
Demin Ctrl (Group 2)	14.94	4.23	9.35-21.84	P= 0.001	22	b
ICON Ctrl (Group 3)	22.00	7.27	13.34-36.21		24	a
ICON Experiment (Group 4)	18.29	5.65	10.10-27.99		24	ab

*SBS values which were not significantly different are marked with the same lower case letter

Adhesive Remnant Index

ARI data is ordinal, non-normally distributed data. Descriptive statistics and statistical comparison data represented in Table 4 for all ARI data. A Kruskal Wallis test shows a significant difference among the four groups ($p<0.0001$). A Mann-Whitney analysis was utilized to evaluate the ARI data to locate the significant differences between groups. All analyses completed using IBM SPSS v24. There are significant differences between the ARI data of the demineralized control and all of the other groups (I, III, IV); as well as a significant difference between the ARI of groups III and IV.

Table IV: ARI Descriptive Statistics and Statistical Comparisons

	ARI Scores				Median	Interquartile Range	Kruskal Wallis	N	Mann-Whitney
	0	1	2	3					
Control (Group I)	10	9	5	0	1	1	p < 0.0001	24	bc
Demin Ctrl (Group II)	0	0	8	14	3	1		22	a
ICON Ctrl (Group III)	11	13	0	0	1	1		24	c
ICON Experiment (Group IV)	6	10	8	0	1	1		24	b

*ARI values which were not significantly different are marked with the same lower case letter

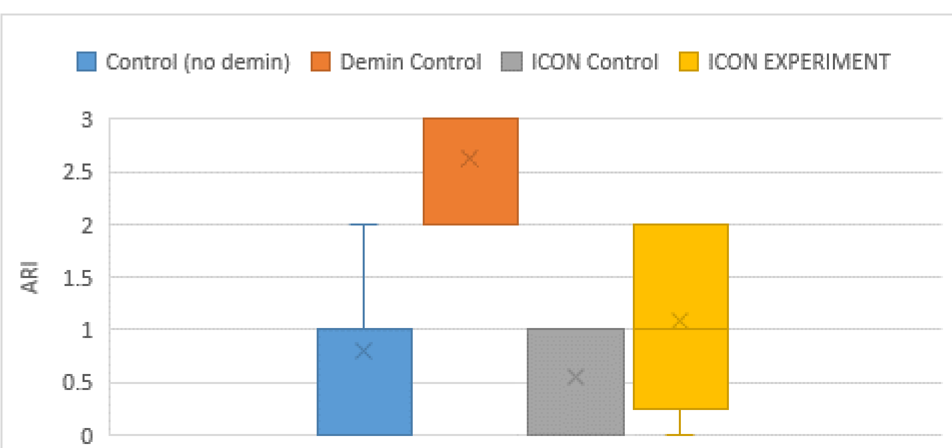

Figure 2: ARI Data Distribution

DISCUSSION

According to Gorelick, up to 24% of patients will present for orthodontic bonding with WSLs present and approximately 50% of orthodontic patients may develop WSLs during orthodontic treatment.[12] Utilization of a resin infiltrant prior to bonding in the treatment of WSLs will not significantly affect the SBS and may decrease the likelihood of enamel fracture at debond.[7,10,11] Significant differences were found in shear bond strength and ARI based on surface treatment; therefore, rejecting the null hypothesis. The control group (Grp I) and ICON control (Grp III) had significantly higher shear bond strengths than the demineralized enamel. There is no difference between the shear bond strength of brackets bonded to aged caries infiltrant treated demineralized enamel and untreated demineralized enamel (Grp II).

Since ICON can increase the bond strength to demineralized enamel, the demineralized enamel could be at an increased fracture risk at debond. However, according to the ARI scores, treatment of demineralized enamel with resin microinfiltration maintains 50% to 100% of the bracket adhesive remaining on the tooth after bracket removal. As other studies have demonstrated, cohesive failures, or failures at the adhesive-enamel interface, increase the likelihood of enamel fracture and surface roughening.[6,7] Therefore, low ARI scores can be attributed to a decreased risk of enamel fracture upon debonding. Group III (ICON Control) had the most favorable ARI scores with 46% receiving the best score (zero, See Table 2) versus Group II (Demin Control) which had the least favorable ARI scores with 64% receiving the worst score (four, See Table 2). There was a significant difference between the ARI scores of ICON Control (Grp III) and aged ICON (Grp IV), Group III being significantly lower. Group IV had 33% of samples receive an ARI score of three, while Group IV did not have any samples score above an ARI of one. Therefore, from an enamel health perspective, it is better to bond to uncontaminated infiltrant resins.

The findings of this study are consistent with others that show increased bond strength to ICON versus demineralized enamel, as well as an increased risk of demineralized enamel fracture at debond when left untreated.[6,13] The increased bond strength to ICON treated demineralized enamel has been linked to the utilization of deep resin tags formed by the infiltration process, increasing micromechanical interdigitation strengthening.[7] The SBS values in this study are significantly higher than some previous studies[7] and consistent with several other studies.[6,10,11,13] Reynolds reported 5.9-7.8 MPa recommendation for adequate bond strength in most clinical orthodontic applications.[14] However, bovine incisors were utilized in this study, which have been found to have a slightly larger enamel crystal pattern. Although this has been implicated in up to 40% bond strength decreases,[15] Ruetterman found that subjecting bovine incisor enamel to thermocycling increased the bond strength one and a half times the SBS obtained from human third molar samples.[16] Therefore, although bovine enamel is an adequate substitution for SBS studies, subjectivity to thermocycling, among other varying protocols, may lead to varying relationships when applied to clinical practice.

The findings of this study have potential clinical implications for the timing and sequence of orthodontic treatment of patients with WSLs. For example, during interceptive orthodontic treatment, if white spot lesions are encountered at debond, after the appropriate remineralization protocol has been executed, infiltration of these lesions may be completed. There will be no special considerations for bonding to the microinfiltrated lesions years later for comprehensive treatment. If a patient presents at initial consultation with white spot lesions, the remineralization or infiltration of these lesions should occur prior to bracket bonding to decrease the likelihood of enamel fracture at debond. If microinfiltration is done in conjunction with bonding, it may also increase the shear bond strength of the brackets to the demineralized enamel. This study provides evidence for flexible practice management strategies. One could either sequence the preventative and bonding appointments to occur simultaneously or over multiple shorter appointments. The flexibility of offering shorter appointments can be advantageous for a patient due to medical conditions, behavior considerations, or other confounding factors.

CONCLUSIONS

- There is no significant difference between the SBS of aged microinfiltrantion (ICON) resin and untreated enamel, demineralized enamel, and ICON treated demineralized enamel. Immediate infiltration of demineralized enamel and untreated enamel have significantly greater SBS than demineralized enamel.
- The risk of enamel fracture from debonding brackets on untreated demineralized enamel is significantly higher than all other groups. Immediate ICON application with bracket bonding has significantly lower ARI scores than the aged ICON group.
- The application of ICON to demineralized lesions without aging significantly increases bond strength relative to untreated demineralized enamel and has significantly lower ARI scores, reducing the risk of enamel fracture at debond.
- The SBS of bonding over aged ICON does not significantly differ from any group (Grp I, II, III) and decreases risk of enamel fracture at debond.

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