Effect of acid etching on bond strength of nanoionomer as an orthodontic bonding adhesive

Saba Khan, Sanjeev K. Verma and Sandhya Maheshwari

ABSTRACT

Aims: A new Resin Modified Glass Ionomer Cement known as nanoionomer containing nanofillers of fluoroaluminosilicate glass and nanofiller 'clusters' has been introduced. An in-vitro study aimed at evaluating shear bond strength (SBS) and adhesive remnant index (ARI) of nanoionomer under etching/unetched condition for use as an orthodontic bonding agent.

Material and Methods: A total of 75 extracted premolars were used, which were divided into three equal groups of 25 each: 1-Conventional adhesive (Enlight Light Cure, SDS, Ormco, CA, USA) was used after and etching with 37% phosphoric acid for 30 s, followed by Ortho Solo application 2-nanoionomer (Ketac™ N100, 3M, ESPE, St. Paul, MN, USA) was used after etching with 37% phosphoric acid for 30 s 3-nanoionomer was used without etching. The SBS testing was performed using a digital universal testing machine (UTM-G-410B, Shanta Engineering). Evaluation of ARI was done using scanning electron microscopy. The SBS were compared using ANOVA with *post-hoc* Tukey test for intergroup comparisons and ARI scores were compared with Chi-square test.

Results: ANOVA (SBS, F = 104.75) and Chi-square (ARI, Chi-square = 30.71) tests revealed significant differences between groups ($P < 0.01$). The mean (SD) SBS achieved with conventional light cure adhesive was significantly higher ($P < 0.05$) (10.59 ± 2.03 Mpa, 95% CI, 9.74-11.41) than the nanoionomer groups (unetched 4.13 ± 0.88 Mpa, 95% CI, 3.79-4.47 and etched 9.32 ± 1.87 Mpa, 95% CI, 8.58-10.06). However, nanoionomer with etching, registered SBS in the clinically acceptable range of 5.9–7.8 MPa, as suggested by Reynolds (1975). The nanoionomer groups gave significantly lower ARI values than the conventional adhesive group.

Conclusion: Based on this in-vitro study, nanoionomer with etching can be successfully used as an orthodontic bonding agent leaving less adhesive remnant on enamel surface, making cleaning easier. However, in-vivo studies are needed to confirm the validity of present findings.

Key words: Adhesive remnant index, etching, nanoionomer, orthodontics, shear bond strength

INTRODUCTION

Acid etching being used conventionally has certain disadvantages such as localized enamel decalcification and fracture of enamel.[1,2] Approximately 96% patients undergoing fixed appliance therapy show signs of enamel demineralization.[3] Fluoride releasing property of resin modified glass ionomer cement (RMGIC) has potential for prevention of white spot lesions.[4] RMGIC demonstrates weaker bond as compared to conventional composites.[5,6] Nanotechnology has been used to modify orthodontic bonding adhesives to improve their physical properties. Nanoparticles of Titanium dioxide, silicon dioxide and silver have been added to composites to induct anti-bacterial activities.[7] Nanoionomer is a type of RMGIC composed of fluoroaluminosilicate (FAS) glass, nanofillers,

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and nanofiller "clusters" combined to improve mechanical properties.[8] Nanoionomer is the merger of nanotechnology and FAS technology. It has been suggested that nanoionomer has the advantage of readily flowable consistency which may make it superior to conventionally use composite resin. Nanoionomer easily flows into bracket mesh and coats the enamel better and thus helps in reduction of caries under the bracket.[9] Certain *in-vitro* tests have revealed that nanoionomer has the capability of creating a caries inhibition zone after acid exposure.[10] With the recent upsurge in concern for prevention of white spot lesions, several studies have been performed under different bonding condition, all of which promote etching prior to bonding.[11] Recently, papain gel has also been tested, as an alternative to phosphoric acid, however, comparable bond strength has been reported,[12] deproteinization with sodium hypochlorite has also been proposed.[13] Both dual and light cured RMGICs are being used by the orthodontists, depending on the material used and enamel pretreatment prior to bonding, the bond strength achieved may vary.[11] This is the first study which evaluates the role of acid etching on shear bond strength (SBS) and adhesive remnant index (ARI) scores of orthodontic brackets bonded with nanoionomer. The null hypothesis presumed was that etching has no effect on shear bond strength and bond failure sites when nanoionomer is used as orthodontic bonding adhesive compared to a conventional adhesive.

MATERIALS AND METHODS

The present *in-vitro* study was conducted on 75 healthy premolar teeth extracted for orthodontic purpose. The premolars were screened with hand lens under transillumination for the presence of any enamel cracks. Fluorosed, hypoplastic or carious teeth were discarded. The teeth were stored in normal saline (0.9 N NaCl) at room temperature; storage solution was changed periodically to inhibit the growth of microbial pathogens. Tooth was mounted vertically in 2 cm × 1 cm × 2 cm PVC pipe filled with autopolymerizing acrylic resin. Mounting was done in such a way that the loading blade of the universal testing machine (UTM-G-410B, Shanta Engineering) was parallel to long axis of the tooth. The teeth were randomly divided into three groups with 25 specimen in each group [Table 1]:

- Group 1 – Conventional bonding adhesive group
- Group 2 – Nano-ionomer group without acid etching
- Group 3 – Nanoionomer group with acid etching.

Table 1: Composition of the adhesives used in the study

Adhesive	Composition	Manufacturer
Enlight (Group 1)	Bis-GMA, moisture displacing fluoride releasing sealant/bond enhancer	Ormco
Ketac™ N100 (Group 3)	Two types of surface treated silica/zirconia N100 fillers approximately 5-25 nm and N100 clusters approximately 1.0-1.6 microns, Fluoroaluminosilicatenano of 1 micron	3M, ESPE

Bis-GMA – Bisphenol A-glycidyl methacrylate

All the tooth specimens were gently polished (for 10 s) with an oil free pumice solution to clean the enamel surface.[14,15] Orthodontic brackets Sapphire Series 022' MBT Upper Left Bicuspid bracket with hooks (Modern Orthodontics, Ludhiana) were bonded on all teeth.

Enlight Light Cure Adhesive (SDS, Ormco, CA, USA) was used in the conventional adhesive group. After acid etching with 37% phosphoric acid, Ortho Solo sealant was applied on the etched tooth surface. Bonding agent was applied on the bracket mesh and positioned on the tooth surface. Extra bonding agent was removed with an explorer. The adhesive was cured for 30 s.

For the nanoionomer group Ketac™ N100 (3M, ESPE, St. Paul, MN, USA) was used. For Group 3, acid etching was performed using 37% phosphoric acid for 30 s. Rest of the procedure for Groups 2 and 3 were similar. Ketac N100 primer was applied to semi-dry enamel surface. Brackets were bonded with Ketac N100 paste and cured for 20 s. For standardization of film thickness of the cement, in both the groups brackets were positioned with a seating pressure of 10 ounces for 10 s, which was applied on the bracket using a Dontrix gauge (E.T.M Corporation, Monrovia, California, USA).[16]

SBS and ARI Measurements

Debonding and shear bond testing were performed after 24 h from bonding using a digital universal testing machine (UTM-G-410B, Shanta Engineering) [Figure 1]. The specimen was clamped in the attachment and a tangential load directed at the ligature groove was applied by the loading plunger at a crosshead speed of 1 mm/min. The load obtained was then divided by the bracket base area which was 9.152 mm^2 (measured by Optical Profile Projector); to obtain SBS in Megapascal (MPa). After debonding, all teeth specimen were examined under a Scanning Electron Microscope (JEOL, JSM-6510 Series). ARI scoring (0-3) was recorded as given by Artun and Bergland.[17] ARI demonstrated mode of failure, occurring either at bracket-adhesive interface leaving greater ARI or at enamel-adhesive interface with lesser ARI.

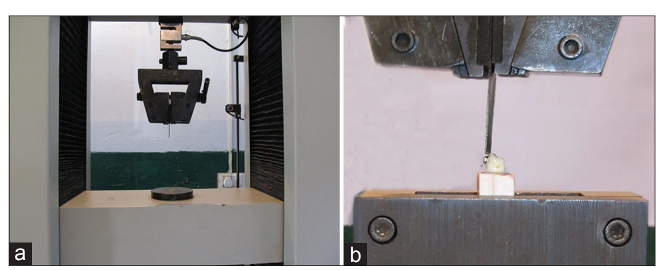

Figure 1: Universal testing machine (a) showing the orientation for alignment loading blade of UTM (b)

Statistical Analysis

Mean, standard deviation and 95% confidence intenrvals were calculated for the SBS values. The SBS values were compared using ANOVA with *post-hoc* Tukey test for intergroup comparisons. ARI scores were compared using the Chi-square test. *P* >0.05 was considered statistically significant.

RESULTS

The Shear bond strength in the conventional bonding adhesive group (Group 1) was 10.59 ± 2.03 Mpa with 95% CI of 9.74-11.41.The SBS in nanoionomer group without etching (Group 2) was 4.13 ± 0.88 Mpa with 95% CI of 3.79-4.47 Mpa and in the etched nanoionomer group (Group 3) it was 9.32 ± 1.87 Mpa with 95% CI of 8.58-10.06 Mpa. as shown in Tables 2 and 3. SBS was significantly higher in Group 1 as compared to the other groups. Between nanoionomer groups, etched-nanoionomer group was associated with higher SBS. The ANOVA test showed these differences to be significant with a F Value of 104.75 with *P* < 0.001. *Post Hoc* tuckey test showed significant differences in all the groups of adhesive with highest mean difference in SBS between conventional and nanoinomer without etching group (6.47 Mpa; 95% CI 5.34-7.60) as shown in Table 3. Etching had improved the SBS which is demonstrated by reduced difference in the SBS of conventional and etched Nanoinomer group (1.28 mpa; 95% CI 0.14-2.41).

The ARI scores were higher for the conventional group 20/25 (80%) tooth having ARI of 2 or 3 [Table 4], as compared to nanoinomer groups in which the unetched group had only 2/25 (8%) tooth with ARI of 2 or 3, whereas etched group had 11/25 (44%) tooth with ARI of 2 or 3. These differences were found to be statistically significant (*P* < 0.001). The difference in ARI Scores of etched and unetched nanoinomer groups was also significant with *P* Value of 0.017 as shown in second part of Table 4. Therefore the nul hypothesis for this study was fully rejected.

Table 2: The SBS (Mean (SD); 95% CI) in different study groups*

Adhesive	SBS (Mpa)	95% CI (Mpa)
Conventional	10.59±2.03	9.74-11.41
Nanoionomer etched	9.32±1.87	8.58-10.06
Nanoionomer	4.13±0.88	3.79-4.47
P	<0.001	

*Values expressed as mean±SD. SD – Standard deviation; SBS – Shear bond strength; Mpa – Megapascal; CI - Confidence interval

Table 3: Inter-group comparison of mean SBS values using the *post-hoc* Tukey test

Dependent variable	Adhesive groups	Mean difference	*P*	95% CI
SBS (Mpa)	Conventional			
	Nanoionomer etched	1.28	0.023	0.14-2.41
	Conventional			
	Nanoionomer	6.47	<0.001	5.34-7.60
	Nanoionomer etched			
	Nanoionomer	5.19	<0.001	4.06-6.33

SBS – Shear bond strength; Mpa – Megapascal; CI – Confidence interval

Table 4: The adhesive remnant index scores on debonded bracket surfaces (ARIs) with different Adhesive agents

Adhesive group	*N*	ARI			
		0	1	2	3
Conventional	25	0	5	10	10
NanoIonomer etched	25	3	11	7	4
Nanoionomer	25	9	14	2	0
Total	75	12	30	19	14
Chi-square=30.71, df=6, *P*<0.001					

First group (*n*=25)	Second group (*n*=25)	df	χ^2	*P* value
Conventional	NanoInomer Etched	3	8.35	0.039
Conventional	NanoInomer	3	28.59	< 0.001
NanoInomer etched	NanoInomer	3	10.14	0.017

The second part shows the result of chi-square test between Adhesive groups

DISCUSSION

Achieving adequate bond strength is imperative for successful orthodontic treatment. Bond failure may occur due to moisture contamination, poor or expired adhesive, careless technique, and/or excessive masticatory forces. SBS values ranging from 5.9–7.8 MPa are sufficient for clinically effective bonding.[18] In our study, SBS for conventional adhesive was significantly higher than the other two groups. However, conventional light cure composite and nanoionomer with acid etching registered SBS in clinically acceptable range, 10.59 ± 2.03 MPa and 9.32 ± 1.87 MPa, respectively. All the brackets failed safely with no enamel damage. SBS achieved with nanoionomer can be said to be more preferable as lesser but clinically acceptable bond strength confers protection to enamel surface from damage during debonding. Nanoionomer used without enamel pretreatment demonstrated significantly low SBS (4.13 ± 0.88 Mpa).

For the nanoionomer group, no effort was made to dry the enamel surface, and bonding was performed on the moist surface. This moisture did not adversely affect the SBS achieved with nanoionomer (9.32 ± 1.87), thus demonstrating that nanoionomer can be successfully used in areas where moisture contamination cannot be controlled.

Choo *et al*. found that bond strength achieved with RMGIC on acid etching and polyacrylic pretreatment were significantly higher than when no etching was performed.[19] Jobalia *et al*. and Chung *et al*. have also reported that, to achieve clinically acceptable bond strength with RMGIC acid pretreatment is required.[20,21] Results of the present study validate Bishara's remark that when the enamel is unetched, the SBS of RMGIC is reduced by half.[22] However, the literature on RMGIC reveals various studies, which have verified that RMGIC achieves clinically acceptable bond strength with no enamel-pretreatment.[23-25] A 96.8% success rate was reported by Silverman *et al*. for RMGIC in a saliva-moistened environment with no acid etching. This is contrary to the findings of our study where we achieved low SBS with nanoionomer without acid etching.

The presentstudy used the ARI as an additional method of investigating the bonding properties of the new orthodontic adhesive which have been previously used.[17,26-28] The average ARI was highest for conventional composite group (2.20 ± 0.76) followed by etched-nanoionomer group (1.48 ± 0.92) and least for nonetched nanoionomer group (0.72 ± 0.61). This can be explained on the basis that etching aids in formation of hybrid layer, which is a part of micro-mechanical bond between adhesive and enamel surface. In concordance with our results, many studies have indicated that bond failure with conventional light-cure adhesives usually occurs at bracket resin interface, leaving most of the residual adhesive on enamel surface[29-31] while studies with GIC have shown that most of the failure occurs at enamel-adhesive interface that is, most of the adhesive adheres to bracket mesh.[32] Bishara *et al*. concluded that etching is a critical variable affecting bond failure location when RMGIC is used, without enamel acid pretreatment bond failure largely occurs at enamel-adhesive interface.[33] Disadvantages of higher ARI is; increased chair-side time taken to mechanically remove remnant adhesive after removing the bracket. Furthermore, there is a risk of enamel damage during mechanical adhesive removal and polishing.[33] Hence, in terms of ARI, nanoionomer was found to be a better bonding agent than conventional composites.

Nanoionomer has certain good qualities like increased flowability, which helps in coating of the enamel during the bonding procedure which might reduce the possibility of caries under brackets during treatment. Fluoride release and recharge might also reduce the possibility of caries/white spot lesions.

CONCLUSION

Nanoionomer can potentially be used as an orthodontic adhesive as:

- Nanoionomer with acid etching demonstrated clinically acceptable SBS
- Lesser remnant adhesive was found with nanoionomer, so lesser clean-up time is required, and lesser chance of enamel damage.

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Conflicts of Interest
There are no conflict of interest.

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