Effect of ageing media on shear bond strength of metal orthodontic brackets bonded with different adhesive systems
(A comparative in-vitro study)
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ABSTRACT

Background: The aim of this study was to evaluate the shear bond strength (SBS) and adhesive remnant index (ARI) of different orthodontic adhesive systems after exposure to aging media (water storage and acid challenge).

Materials and methods: Eighty human upper premolar teeth were extracted for orthodontic purposes and randomly divided into two groups (40 teeth each): the first group in which the bonded teeth were stored in distilled water for 30 days at 37°C, and the second group in which the bonded teeth were subjected to acid challenge. Each group was further subdivided into four subgroups (10 teeth each) according to the type of adhesive system that would be bonded to metal brackets: either non-fluoride releasing adhesive (NFRA), fluoride releasing adhesive (FRA), Fluoride releasing bond with self-etching primer (FRBSP), or powder and liquid orthodontic fluoride releasing adhesive (PLFRA). After 30 days of water storage and acid challenge ageing procedures, the SBS was determined using Instron testing machine with a crosshead speed of 1 mm/min. The ARI was assessed using a stereomicroscope with 10 X magnification.

Result: The SBS testing revealed significant differences (p < 0.05) among the four tested adhesive systems in water storage and acid challenge groups using ANOVA F-test. In both groups, the NFRA subgroup exhibited the highest mean SBS value, followed by FRASP, then FRA subgroups, while the PLFRA subgroup had the lowest value of mean SBS. The independent t-test showed non-significant differences in mean SBS values between water storage and acid challenge groups. In respect to the ARI analysis, the Chi-square test showed significant differences among the tested adhesive systems.

Conclusion: The shear bond strength of the fluoride releasing adhesive system was less than that of the non-fluoride releasing adhesive system, but still above the clinically acceptable range.

Keywords: Fluoride releasing adhesive, Acid challenge, Water storage, Shear bond strength.(Received: 20/8/2020; Accepted: 1/11/2020)

INTRODUCTION

Fixed orthodontic appliances are still associated with a high risk of white spot lesions (WSLs) formation, even with the improvements in materials and treatment mechanics. The prevalence of the WSLs during orthodontic therapy expressed to a range from 13% to 75%.(1) The maintenance of oral hygiene is impeded by components of fixed orthodontic appliances, which encourage the plaque accumulation around the bracket base. These lesions can occur through a short duration of about 4 weeks, which is usually within the intervals of orthodontic treatment appointments.(2) The prevention of the WSLs must be the first goal of an orthodontist. Accordingly, the most essential way for averting WSL development is the patient education and motivation. The other means have been utilized for reducing the extent of WSLs are dentifrice, mouthwash, gels, and varnishes; all are formulated with fluoride.(3)

In restorative dentistry and orthodontics, the fluoride-releasing bonding system, Clearfil Liner Bond F (Kuraray Medical Inc, Okayama, Japan) has been advanced. It contained a specially treated sodium fluoride (NaF), which was effective in reducing the demineralization while maintaining the bonding strength.(4) The Light Bond paste and sealant have been developed as polyacid modified composite resins, with a patented monomer of fluoride-releasing property and it has been shown that the bond strength was maintained while fluoride would be regularly released into the mouth.(5) Resin modified glass ionomer cements (RMGIC) have been combined with the preferable properties of composite resin including the shear bond strength (SBS) and fluoride releasing feature of glass ionomer cement. Several RMGICs have been evaluated for SBS, one of them was Fuji Ortho LC (GC Company, Tokyo, Japan) which had a bond strength of a comparable value to composite resins.(6) Oncag et al.(7) evaluated the effect of acidic soft drinks on the SBS of orthodontic brackets and found that the bracket retention was adversely affected. Under the SEM, they observed the formation of erosive defects on the enamel surface around the adhesive. So far to our knowledge, the fluoride-releasing adhesives have not been tested for the SBS under the effect of acidic attack. Accordingly, the objective of the current study was to evaluate the shear bond strength and the
adhesive remnant index of the different orthodontic adhesive systems after exposure to aging media (water storage and acid challenge) for 30 days.

MATERIALS AND METHODS

After inspection of 138 human upper first premolars extracted for orthodontic purposes, only 80 teeth were involved that had an intact buccal surface and free from caries, restorations, cracks, fluorosis and not subjected to any chemical treatment. They were stored in 1% Chloramine-T solution for one week and subsequently kept in deionized water until conducting the bonding procedures.\(^{(8)}\) The teeth were divided into two groups (40 teeth each): the first group in which the bonded teeth would be saved in distilled water for 30 days at 37°C and the second group in which the bonded teeth would be subjected to acid challenge. Each group was subdivided into four subgroups equivalent to the bonding procedures (10 teeth each) and mounted in auto-polymerized acrylic blocks before bonding. The acrylic blocks were coded to facilitate the randomization procedure.

Brackets

Eighty upper first premolar stainless-steel brackets of Discovery® Smart type (Dentaurum company, Ispringen, Germany) were used in this study. The prescription of upper premolar bracket was MBT system with slot size 0.022×0.030 of an inch and the bracket’s bonding surface area is 10.56 mm\(^2\).

Bonding procedures

At room temperature, the bonding procedure was performed by one of the four adhesive systems according to the manufacture instructions as followed:

1. Non-fluoride releasing adhesive: The enamel surfaces were etched with 37% phosphoric acid etching gel (PerfectEitch-E, Perfection Plus, UK) for 30 seconds, then washed for 10 seconds and air-dried gently. A thin film of Transbond XT primer was applied to the etched enamel surfaces, then polymerized by a LED light curing unit (O-light, Woodpecker, China) for 10 seconds.

2. Fluoride releasing adhesive: The teeth were bonded with Light Bond paste and sealant (Reliance Orthodontic Products, Itasca, Illinois, USA). The liquid etchant (37% phosphoric acid) was applied to the buccal tooth surfaces for 30 seconds, then washed for 30 seconds and air-dried gently. The fluoride releasing sealant resin was painted with a disposable brush in a thin uniform coating, followed by mild air-drying, and then cured for 30 seconds.\(^{(5)}\)

3. Fluoride releasing bond with self-etching primer: The teeth were bonded with Clearfil Liner Bond F (Kuraray Noritake Dental Inc., Okayama, Japan) and Transbond XT paste. The self-etching primer was applied for 20 seconds, then dried with a mild air flow. The Clearfil Liner Bond F which had fluoride releasing property, was applied, gently air flowed to create a uniform bond film, and light cured for 10 seconds.\(^{(9)}\)

4. Powder and liquid orthodontic fluoride releasing adhesive: the teeth were bonded with Fuji Ortho LC (GC Company, Tokyo, Japan). The etching gel (37% phosphoric acid) was applied for 30 seconds, then washed for 10 seconds. The bonding area was not completely desiccated through the bonding procedure. The cement was prepared by one scoop of powder and two drops of liquid on a mixing pad using a plastic spatula to achieve a glossy consistency.\(^{(10)}\)

In the four bonding procedures, the bracket base was coated with an adhesive paste or cement, and placed at the center of the buccal tooth surface. A load (200 gm) was placed on each bracket using a surveyor for 10 seconds to achieve uniform adhesive thickness.\(^{(11)}\) Any excess of adhesive was removed by dental explorer before the curing. The LED light curing unit with curing intensity 1200 mw/cm\(^2\) was applied for 40 seconds (10 seconds from each side of bracket).\(^{(10)}\) Once the bonding procedures were completed, the bonded teeth of first group were stored in the incubator in distilled water inside sealed containers at 37°C for 30 days with daily refreshment, in order to avoid the cumulative effects.\(^{(12,13)}\) While the bonded teeth in the second group stored in deionized water for 24 hours at 37°C prior to the acidic challenge experiment. The acidic solution (pH=2.5) of 500 ml was prepared by gradual addition of 1.5 ml of HCl [1M] in distilled water. The acidic challenge was performed by immersing the samples in the acidic solution through a protocol of three session per day, 5 min each, with equal intervening periods (2 hour) for 30 days. The samples were stored in distilled water (pH=6) at 37°C for the remaining time in order to mimic the wet oral environment. After each session, each storage medium was periodically renewed, and before and after each session, the samples were rinsed with water and air dried.\(^{(13)}\)

Shear bond strength test

The Tinius-Olsen universal testing machine was used to carry out the shear bond strength test after water storage and acid challenge ageing procedures for 30 days using a 5 KN load cell with a crosshead speed of 1 mm/min.\(^{(14)}\) At the enamel-bracket interface, the load was applied vertically in the occluso-gingival direction from knife-edge rod (which was fixed inside the upper
arm of the universal testing machine) until adhesive failure occurred. The debonding force was recorded in units of Newton and then divided by the surface area of the bracket base (10.56 mm²) to get the readings in megapascal (MPa).\(^{(15)}\)

**Estimation of adhesive remnant index**

The stereomicroscope (Hamilton, Italy) with 10 X magnification was utilized to examine the enamel surface of each tooth and the debonded bracket, in order to assess the predominant site of bond failure. The site of bond failure was scored according to Artun and Bergland\(^{(16)}\) as followed:

- **0 =** No adhesive remained on the tooth surface.
- **I =** Less than 50% adhesive remained on the tooth surface.
- **II =** More than 50% adhesive remained on the tooth surface.
- **III =** All the adhesive is remained on the tooth surface.

**Statistical analysis**

The collected data were analyzed using SPSS (version 25.0, SPSS Inc. Illinois, USA). The statistical analyses involved One-way analysis of variance (ANOVA), Post-hoc Tukey’s HSD test, Chi-square test, and Independent sample t-test. The level of significance \(p<0.05\) was considered for statistical evaluations.

**RESULTS**

Table 1 shows the mean, standard deviation (S.D.), standard error (S.E.) minimum (Min.), and maximum (Max.) values of shear bond strength (SBS) in both ageing groups. In water storage group, the highest mean value of SBS was in NFRA group (26.524 ± 3.767), followed by that of FRBSP group (24.244 ± 4.553), then FRA group (21.408 ± 3.424), and lastly the PLFRA group, which had the lowest mean of SBS (18.346 ± 4.109), while in acid challenge group, the highest mean value of SBS was in NFRA group (25.880 ± 3.938), followed by that of FRBSP group (23.856 ± 4.030), then FRA group (20.900 ± 3.403), and lastly the PLFRA group, which had the lowest mean of SBS (16.779 ± 3.653). Table 2 shows the comparison of mean difference of SBS values among all tested adhesive systems in both ageing groups. The One-way analysis of variance (ANOVA) revealed that there were significant differences between all adhesive systems in both ageing groups. In both groups, the Post-hoc Tukey’s HSD test revealed similar results where there was significant differences between NFRA and PLFRA groups, between NFRA and FRA groups, while non-significant differences between NFRA and FRBSP, FRA and FRBSP, and FRA and PLFRA groups; excepting that the difference was significant between FRBSP and PLFRA in water storage and highly significant in acid challenge group.

Table 3 presents the frequencies and percentages of ARI scores for all tested adhesive systems. In water storage group, the highest frequency of ARI score 0 was found in FRA group, while the highest frequency of score I was found in FRA and FRBSP groups, the highest frequency of score II was found in NFRA group, and the highest frequency of score III was found in PLFRA group. In acid challenge group, the highest frequency of ARI score 0 and I were found in FRA group, while the highest frequency of score II was found in NFRA and FRBSP groups, and the highest frequency of score III was found in PLFRA group. Table 4 shows the comparison of the ARI for all adhesive systems. The chi-square test displayed significant differences among all tested adhesive systems. In both ageing groups, the results demonstrated significant differences between FRA and PLFRA groups, FRA and FRBSP groups. The non-significant differences were found between NFRA and FRBSP, NFRA and PLFRA, and FRBSP and PLFRA groups. The differences between NFRA and FRA groups were significant in water storage and acid challenge groups. The effect of ageing media on the SBS and ARI of the four test adhesive systems was determined by the independent t-test and chi-square test respectively. The results revealed non-significant differences between the water storage and acid challenge groups, as shown in table 5 and table 6.

Table 1: Descriptive statistics of the shear bond strength test of different groups.

| Group                        | Adhesive system | \(X^2\) | Likelihood Ratio | d.f. | p-value |
|------------------------------|-----------------|--------|------------------|------|---------|
| **Water storage**            |                 |        |                  |      |         |
| Among all groups             | 17.421          | 19.612 | 9                | 0.020|
| NFRA-FRA                     | 8.978           | 11.461 | 3                | 0.009|
| NFRA-FRBSP                   | 9.900           | 9.908  | 3                | 0.823|
| NFRA-PLFRA                   | 2.633           | 2.773  | 3                | 0.428|
| FRA-FRBSP                    | 6.921           | 8.630  | 3                | 0.035|
| FRA-PLFRA                    | 11.700          | 14.967 | 3                | 0.002|
| FRBSP-PLFRA                  | 2.800           | 2.947  | 3                | 0.400|
| **Acid challenge**           |                 |        |                  |      |         |
| Among all groups             | 17.171          | 19.466 | 9                | 0.040|
| NFRA-FRA                     | 8.662           | 11.090 | 3                | 0.011|
| NFRA-FRBSP                   | 0.533           | 0.541  | 3                | 0.910|
| NFRA-PLFRA                   | 1.143           | 1.163  | 3                | 0.762|
| FRA-FRBSP                    | 6.667           | 8.630  | 3                | 0.035|
| FRA-PLFRA                    | 10.800          | 13.725 | 2                | 0.003|
| FRBSP-PLFRA                  | 2.476           | 2.612  | 3                | 0.455|
Table 2: Comparison of the mean shear bond strength test in different groups by ANOVA and Post-hoc Tukey’s HSD test.

| Group          | Adhesive system | Comparison | ANOVA test | Tukey’s HSD test |
|----------------|-----------------|------------|------------|------------------|
|                |                 |            | F-test     | p-value          | Between subgroups | p-value |
| Water storage  | NFRA            | 7.893      | 0.000      | 0.033            | NFRA-FRA          | 0.033 |
|                | FRA             |            |            | 0.582            | NFRA-FRBSP       |        |
|                | FRBSP           |            |            | 0.000            | NFRA-PLFRA       |        |
|                | PLFRA           |            |            | 0.396            | FRA-FRBSP        |        |
|                |                 |            |            | 0.330            | FRA-PLFRA        |        |
|                |                 |            |            | 0.011            | FRBSP-PLFRA      |        |
| Acid challenge | NFRA            | 11.02      | 0.000      | 0.629            | NFRA-FRA         | 0.027 |
|                | FRA             |            |            |                    | NFRA-FRBSP       |        |
|                | FRBSP           |            |            |                    | NFRA-PLFRA       | 0.000 |
|                | PLFRA           |            |            |                    | FRA-FRBSP        | 0.311 |
|                |                 |            |            |                    | FRA-PLFRA        | 0.086 |
|                |                 |            |            |                    | FRBSP-PLFRA      | 0.001 |

Table 3: Frequency distribution and percentages of adhesive remnant index among different adhesive systems in both ageing groups.

| Group          | Adhesive system | ARI scores | 0 | I | II | III | Total |
|----------------|-----------------|------------|---|---|----|-----|-------|
| Water storage  | NFRA            | N          | 2 | 2 | 5  | 1   | 10    |
|                |                 | %          | 20.0 % | 20.0 % | 50.0 % | 10.0 % | 100.0 % |
|                | FRA             | N          | 7 | 3 | 0  | 0   | 10    |
|                |                 | %          | 70.0 % | 30.0 % | 0.0 % | 0.0 % | 100.0 % |
|                | FRBSP           | N          | 3 | 3 | 3  | 1   | 10    |
|                |                 | %          | 30.0 % | 30.0 % | 30.0 % | 10.0 % | 100.0 % |
|                | PLFRA           | N          | 1 | 2 | 3  | 4   | 10    |
|                |                 | %          | 10.0 % | 20.0 % | 30.0 % | 40.0 % | 100.0 % |
| Total          | N               | 13         | 10 | 11 | 6  | 40   |
|                |                 | %          | 32.5 % | 25.0 % | 27.5 % | 15.0 % | 100.0 % |
| Acid challenge | NFRA            | N          | 2 | 2 | 4  | 2   | 10    |
|                |                 | %          | 20.0 % | 20.0 % | 40.0 % | 20.0 % | 100.0 % |
|                | FRA             | N          | 6 | 4 | 0  | 0   | 10    |
|                |                 | %          | 60.0 % | 40.0 % | 0.0 % | 0.0 % | 100.0 % |
|                | FRBSP           | N          | 3 | 2 | 4  | 1   | 10    |
|                |                 | %          | 30.0 % | 2.0 %  | 40.0 % | 10.0 % | 100.0 % |
|                | PLFRA           | N          | 2 | 1 | 3  | 4   | 10    |
|                |                 | %          | 20.0 % | 10.0 % | 30.0 % | 40.0 % | 100.0 % |
| Total          | N               | 13         | 9  | 11 | 7  | 40   |
|                |                 | %          | 32.5 % | 22.50 % | 27.50 % | 17.5 % | 100.0 % |

Table 4: Comparison of ARI among different adhesive systems in both ageing groups.

| Group          | Adhesive system | N | Mean (MPa) | S.D. | S. E. | Min. | Max. |
|----------------|-----------------|---|------------|------|-------|------|------|
| Water storage  | NFRA            | 10| 26.524     | 3.767| 1.192 | 20.190| 33.190|
|                | FRA             | 10| 21.408     | 3.424| 1.083 | 17.320| 28.380|
|                | FRBSP           | 10| 24.244     | 4.533| 1.440 | 18.860| 31.520|
|                | PLFRA           | 10| 18.346     | 4.109| 1.299 | 13.330| 26.100|

Table 5: Comparison of the effect of ageing media on the mean SBS of the four test adhesive systems.

| Adhesive system | Group          | Mean differences | t-value | p-value |
|-----------------|----------------|------------------|---------|---------|
| NFRA            | Water storage  | 0.644            | 0.374   | 0.713   |
|                 | Acid challenge | 0.508            | 0.333   | 0.743   |
| FRA             | Water storage  | 0.388            | 0.842   | 0.713   |
|                 | Acid challenge | 0.000*           | 1       | 1.000   |
| FRBSP           | Water storage  | 0.000*           | 1       | 1.000   |
|                 | Acid challenge | 0.000*           | 1       | 1.000   |
| PLFRA           | Water storage  | 1.567            | 0.835   | 0.415   |
|                 | Acid challenge | 1.567            | 0.835   | 0.415   |

Table 6: Comparison of the effect of ageing media on the ARI scores distribution of the four test adhesive systems. *Continuity correction test

| Adhesive system | Groups | X² | Likelihood Ratio | d.f. | p-value |
|-----------------|--------|----|------------------|------|---------|
| NFRA            | Water storage | 0.444 | 0.451 | 3 | 0.929   |
|                 | Acid challenge | 0.444 | 0.451 | 3 | 0.929   |
| FRA             | Water storage  | 0.220 | 0.000* | 1 | 1.000   |
|                 | Acid challenge | 0.220 | 0.000* | 1 | 1.000   |
| FRBSP           | Water storage  | 0.343 | 0.345 | 3 | 0.951   |
|                 | Acid challenge | 0.343 | 0.345 | 3 | 0.951   |
| PLFRA           | Water storage  | 0.676 | 0.680 | 3 | 0.878   |
|                 | Acid challenge | 0.676 | 0.680 | 3 | 0.878   |

DISCUSSION

The most common adverse effect associated with fixed orthodontic therapy is the white spot lesions around the bonded attachments and its prevalence ranges between 2% and 96%.(17) Many studies evaluated the efficacy of fluoride releasing adhesives as non-patient dependent approach and topical fluoride exposure in reducing the enamel demineralization adjacent to the brackets.(6,18,19)
In the present study, the mean SBS values in all adhesive systems were higher than the clinically acceptable SBS (5.9 to 7.8 MPa) as considered by Reynolds \(^{(20)}\), which means that all the tested adhesive systems can withstand the shear stress to an acceptable level. According to the results of the shear bond strength test, there were statistically significant differences among the tested adhesive system in water storage and acid challenge groups. In both ageing groups, the NFRA had the highest value of mean SBS, while the PLFRA had the least value of mean SBS among the tested adhesives, but above clinically acceptable SBS. This outcome might be based on that the enamel surfaces in this group were conditioned with 37% phosphoric acid instead of the polyacrylic acid conditioner, this would produce rougher enamel surface, and consequently enhanced the bond strength; this explanation was reported by Cacciafesta et al. \(^{(21)}\) and Tanbakuchi et al. \(^{(22)}\) Another possible explanation is that the enamel surfaces were adequately wet during the bonding procedure, otherwise the SBS would be adversely affected if the enamel surface was desiccated. \(^{(23)}\) The current study demonstrated that there was highly significant difference between the NFRA and PLFRA groups, these observations were congruent with Yassaei et al. \(^{(24)}\), who concluded that the Fuji Ortho LC had a significantly reduced SBS values compared to Transbond XT for metal and ceramic brackets. The FRA group had less SBS value than NFRA group, and the difference between these two groups was significant. These results are supported by Benkli et al. \(^{(25)}\), who examined the SBS of metal and ceramic brackets bonded with different bonding agents and observed that the SBS values were less in the Light Bond than Transbond XT for metal and ceramic brackets. The FRBSP group had mildly deceased mean SBS values than NFRA group. This may be attributed to the use of a self-etching primer instead of a conventional acid etchant, which reduces the bonding strength as approved by Cehreli et al. \(^{(26)}\) and Scougall-Vilchis et al. \(^{(27)}\). The result of present study demonstrated that the difference between these two groups was non-significant. This outcome is supported by Raji et al. \(^{(28)}\), who evaluated the SBS of fluoride releasing self-etching primers in comparison with conventional adhesive after thermocycling (500 cycles) and concluded that there were no significant differences in the SBS values between them.

The present study observed that the difference between the FRBSP and PLFRA was significant in water storage and acid challenge. Also, there were no significant differences between the FRA and FRBSP, FRA and PLFRA in both groups. These results supported by Reicheneder et al. \(^{(29)}\), who found that the SBS was higher in Light Bond than Fuji Ortho LC but the difference was non-significant, this may be due to the fact that both adhesive systems had fluoride-releasing ability.

The analysis of ARI in present study showed that the difference between the NFRA and FRA groups was significant in water storage and acid challenge groups, where the ARI score was mainly II for NFRA in first group and 0 for FRA in latter group. These results agreed with Vicente et al. \(^{(30)}\), who found that the Transbond XT left significantly more adhesive remnant on enamel surface than Light bond. In both ageing groups, the distribution of ARI scores in FRBSP group ranged between 0 and II, and the difference in comparison with NFRA group was non-significant, this may be attributed to the usage of the same adhesive paste (Transbond XT) in both groups. These outcomes were consistent with Krobmacher et al. \(^{(31)}\), and Raji et al. \(^{(28)}\) as they found that the difference in ARI scores distribution between these groups was non-significant and the bond failure mostly occurred with some remnants on the enamel surface. The PLFRA group had a high frequency of bond failure (score II and III), indicating more adhesive remained on enamel surfaces. The current study findings demonstrated that the difference was non-significant between the NFRA and PLFRA groups. These findings are supported by Owen et al. \(^{(31)}\) who found the RMGICs adhere strongly to the enamel surface and weakly to metal in contrast to the composite resins, which bond well to both the enamel and metal surfaces. Moreover, the study demonstrated significant differences between FRA and PLFRA, and between FRA and FRBSP groups. These outcomes are supported by Summers et al. \(^{(32)}\), who assessed the SBS and ARI for orthodontic brackets bonded with the light Bond and Fuji Ortho LC, and found that there was a significant difference in distribution of ARI scores between these two groups.

With respect to the effect of ageing media, the current study showed that there were no significant differences in the SBS and ARI scores distribution among the tested adhesive systems in both ageing groups. These results are supported by Navarro et al. \(^{(33)}\), who evaluated the SBS values and ARI of bonded teeth stored in acidic soft drinks and artificial saliva, and concluded that there were no significant differences between the experimental and control groups, this
agreement may be attributed to the use of nearly the same acid challenge protocol.

CONCLUSION

1-The shear bond strength of the fluoride releasing adhesive systems (PLFRA, FRA, FRASP) was less than that of the non-fluoride releasing adhesive system, but above the clinically acceptable range.

2-The ageing media did not affect significantly the SBS and ARI of the four tested adhesive systems.

Conflict of interest: None.

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