Print ISSN: 2321-6379
Online ISSN: 2321-595X

Original Article

An *In vitro* Study to Evaluate the Shear Bond Strength of an "Amorphous Calcium Phosphate"-Containing Orthodontic Adhesive

Nihar Ranjan Sahoo[1], Rashmita Sahoo[2]

[1]Assistant Professor, Department of Dentistry, MKCG Medical College, Berhampur, Odisha, India, [2]Clinical Tutor, Department of Oral Medicine and Radiology, Institute of Dental Sciences, Bhubaneswar, Odisha, India

Abstract

Introduction: Decalcification of tooth enamel around orthodontic brackets continues to be a perennial problem in orthodontics. The success of fluoride as a cariostatic agent is attributed to two functions: (1) Bactericidal effect at higher concentrations and (2) remineralization of enamel to form fluorohydroxyapatite. Amorphous calcium phosphate (ACP)-filled methacrylate composites have demonstrated the potential to remineralize carious enamel lesions by releasing supersaturated levels of calcium and phosphate ions in proportions favorable for the formation of hydroxy apatite over an extended period of time. Aegis Ortho™*, an ACP-containing adhesive, has been marketed for the use as a light-cured orthodontic adhesive with similar properties to previously used resins. However, to even be considered for clinical use, they must first provide satisfactory shear bond strength.

Purpose: The purpose of the study was to determine whether an ACP-containing adhesive (Aegis Ortho) provides an acceptable level of shear bond strength to function as an orthodontic adhesive.

Materials and Methods: Sixty extracted premolars were randomly divided into three groups for orthodontic bonding. Group 1 used a composite resin adhesive (Transbond XT), Group 2 was bonded with an ACP-containing adhesive (Aegis Ortho), and Group 3 used a resin-modified glass ionomer (Fuji Ortho LC). All bonded teeth were stored in distilled water at 37°C for 24 h before debonding. Shear bond strength and adhesive remnant index (ARI) were recorded for each specimen.

Results: The mean shear bond strengths for the three test groups were as follows: Group 1 (10.83 ± 3.03 MPa), Group 2 (4.04 ± 2.12 MPa), and Group 3 (10.99 ± 2.48 MPa). The statistical analysis showed that there was no statistically significant difference in bond strength between Group I and Group III. Groups II and III exhibited lower ARI scores than Group I, but a majority of specimens had greater than 50% of the cement removed along with the bracket during debonding except Group I.

Conclusion: Orthodontic resin containing ACP has significantly lesser shear bond strength to function as an orthodontic adhesive.

Key words: Amorphous calcium phosphate, Orthodontic adhesive, Shear bond strength

INTRODUCTION

The introduction of the acid etch technique by Buonocore[1] in 1955 and the development of orthodontic resins by Newman *et al.*[2] in 1965 has replaced banding with bonding, marking a new era in orthodontic history. Today, direct bonding has become the most popular method and clinical standard for attaching orthodontic brackets. A common requirement for all resin-based composite adhesive systems is some form of enamel etching due to which there is loss of surface enamel and subsequent caries susceptibility. Because of this glass ionomer has been suggested as an alternative due to their distinct advantage of fluoride release. Despite this advantage, studies have reported poor bracket bond strength in the range of 2.4–5.4 MPa[3] and higher bracket detachment rate than composite resin systems. This was a major shortcoming. Addition of small amount of light-activated resin to glass ionomer cements (GICs) was found to improve the physical and mechanical properties of GICs. These came to be known

Access this article online

www.ijss-sn.com

Month of Submission : 06-2020
Month of Peer Review : 07-2020
Month of Acceptance : 07-2020
Month of Publishing : 08-2020

AQ1

Corresponding Author: D/O. Bijendra Kumar Sahoo, Ranihat, Gopal Sahi Near Radha Krishna Temple (Tungi Market), Po-Buxibazar, Cuttack - 753 001, Odisha, India.

as "resin-modified GICs" and were introduced to the dental profession in 1988. Studies in orthodontic literature reported improvement of bond strength from 5.4 to 18.9 MPa with these resin-modified GICs.[4,5]

Decalcification of tooth enamel around orthodontic brackets continues to be a perennial problem in orthodontics. The success of fluoride as a cariostatic agent is attributed to two functions:[6] (1) A bactericidal effect at higher concentrations and (2) remineralization of enamel to form fluorohydroxyapatite which is less soluble in an acidic medium than hydroxy apatite.

Thus far, our specialty had focused mainly on protocols for fluoride intervention. However another mechanism can also favor remineralization dynamics: Amorphous calcium phosphate (ACP)-filled methacrylate composites have demonstrated the potential to remineralize carious enamel lesions by releasing supersaturated levels of calcium and phosphate ions in proportions favorable for the formation of hydroxy apatite over an extended period of time. This was considered a novel approach and a departure from the many fluoride-containing resin-based materials. The first commercially available ACP-containing orthodontic resin received FDA approval in February 2004.

ACP has the properties of both preventive and restorative materials that justify its use in dental cements, sealants, composites, and more recently, orthodontic adhesives. ACP-filled composite resins have been shown to recover 71% of the lost mineral content of decalcified teeth.[7] One ACP-containing adhesive, Aegis Ortho™* has been marketed for use as a light-cured orthodontic adhesive with similar properties to previously used resins.

Studies have demonstrated there mineralization potential of ACP-containing materials.[7-9] However, to even be considered for clinical use, they must first provide satisfactory shear bond strength. There are hardly any studies in orthodontic literature which investigates the bond strength of ACP-containing materials. Therefore, the aim of this study was to determine whether Aegis Ortho adhesive provides an acceptable level of shear bond strength to function as an orthodontic adhesive. This product was compared with the commonly used orthodontic adhesives/cements, Transbond XT™** and Fuji Ortho LC™*** in India. Similar to the claims made by the ACP adhesive manufacturer, the use of resin-modified GIC (Fuji Ortho LC) has been said to be more effective in preventing white spot lesions than conventional composite resins.[10] Using this cement as another comparative group allows comparison of adhesives marketed for the purpose of white spot prevention. The efficacy of the ACP-containing adhesive in preventing demineralization was not the focus of this study, but if proven to have adequate bond strength, this feature will be explored in future research.

- *-Harry J Bosworth Company, Skokie, Illinois
- **-3M Unitek, 2724, South Peck Road, Monrovia, CA 91016, USA
- ***-GC America Inc., Alsip, Ill.

MATERIALS AND METHODS

Sixty extracted human premolars, free of visible caries, decalcification, fractures, peculiar morphology, fractures, and other defects were collected and stored in distilled water at room temperature and remained in distilled water at all times except when brackets were being bonded and debonded. The distilled water was changed periodically to inhibit bacterial growth.

The teeth were randomly divided into three groups – Group I, Group II, and Group III, all containing 20 specimens.

- Group I – Samples were bonded using Transbond XT (3M/Unitek Corp, Monrovia, Calif.)
- Group II – Samples were bonded using Aegis Ortho (Harry J Bosworth Co., Skokie, Ill) [Figure 1], and
- Group III – Samples were bonded using Fuji Ortho LC (GC America Inc., Alsip, Ill).

Grooves were made on the root surface of each sample and then embedded in a cylindrical colored acrylic block of polymethyl methacrylate so that only the coronal portion of the specimen was exposed [Figure 2]. The crowns were oriented along the long axis of the blocks and were stored in distilled water at room temperature in a closed airtight container. Sixty 022-MBT metal maxillary premolar

Figure 1: Aegis Ortho™ amorphous calcium phosphate-containing adhesive

brackets were used (Gemini Series, 3M Unitek). The base area of each bracket was calculated (mean = 10.12 mm^2) using image analysis equipment.

Before bonding, all teeth were cleaned using coarse, oil free pumice with a rubber prophylaxis cup, rinsed with water, and dried for 10 s using an air-water syringe. The teeth were etched with 35% phosphoric acid gel (Harry J. Bosworth Co.) for 30 s and then thoroughly rinsed with sterile water from an air/water syringe and dried with oil free and moisture free air until enamel had a frosty appearance.

For Group I, a layer of Transbond XT primer was applied to the tooth surface and photopolymerized for 10 s. Then, Transbond XT paste applied to the base of the bracket which was then placed on the tooth with firm pressure. Excess adhesive was removed from around the base of the bracket and the adhesive was then light cured for 40 s, positioning the light source on each interproximal side for 20 s with the help of QHL75, Dentsply halogen curing light.

For Group II, a layer of Aqua Bond was applied on the etched enamel and gently air dried for 5 s. Then, a thin layer of ACP-containing orthodontic adhesive (Aegis Ortho™, Harry J. Bosworth Co.) was applied to the orthodontic bracket and immediately pressed into the adhesive on the tooth surface. Excess adhesive was removed. For Group III instead of frosty appearance, the tooth surface kept moist after acid etching. The powder and liquid were mixed. The mixed cement was applied to the bracket bases and immediately the brackets were pressed on to the prepared tooth surface. The excess cement was removed. The cement was photopolymerized using the same light and following the same protocol as the first group. The manufacturer's instructions were followed throughout the procedure.

After bracket bonding, all the samples were stored in distilled water at 37°C for 24 h.

Method of Shear Bond Strength Evaluation

The shear strength of bonded teeth was tested using an Instron Universal Testing Machine (UTM) Model No: 1195-5500R [Figure 3]. The sample testing was carried out using a sensitive load cell value of 5000 Newton. This technique of testing shear bond strength has been widely reported in the literature.

Each tooth was oriented with the testing device as a guide and held firmly between the lower cross head of the UTM [Figure 4] so that its labial surface would be parallel to the applied force during the shear bond strength tests. A gingivo-occlusal load that produced a shear force at the bracket-tooth interface was applied to the bracket by means of a looped soft stainless steel wire of size 21 gauge attached to the upper cross head of the machine. The results of each test were recorded in Newton (N) on a graphic plotter. Shear bond strengths were measured at a crosshead speed of 0.5 mm/min. The bond strength was calculated in megapascals.

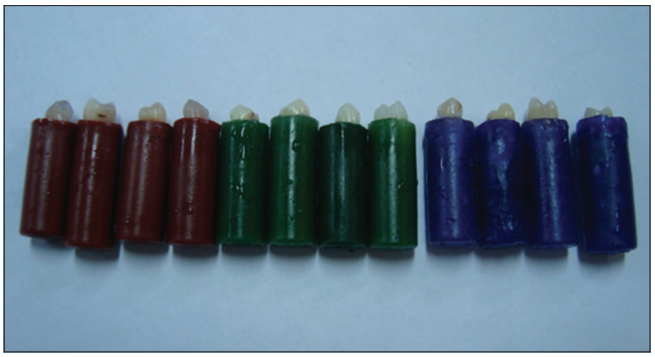

Figure 2: Acrylic blocks with teeth embedded

Figure 3: Instron universal testing machine

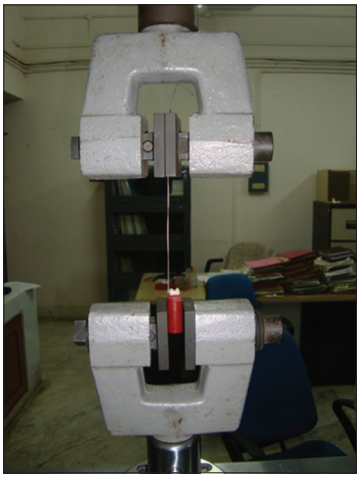

Figure 4: Shear bond strength jig mounted on universal testing machine

After debonding, each tooth was examined under stereomicroscope named Carl Zeiss Jena to determine the amount of adhesive remaining on each tooth according to adhesive remnant index (ARI).[11]

OBSERVATIONS AND RESULTS

The values obtained on testing the shear bond strength of samples in Groups I, II, and III were recorded in Newton and then converted into MPa (megapascal). Routine statistical analysis such as mean, maximum and minimum, range, and standard deviation was calculated for each group.

Data were analyzed using computer software, Statistical Package for the Social Sciences (SPSS) version 10. Data are expressed in its mean, median, and standard deviation. Analysis of variance (one-way ANOVA) was performed as parametric test to compare different groups. Duncan's multiple range (DMR) test was used as *post hoc* analysis to elucidate individual comparisons between groups. For all statistical evaluations, a two-tailed $P < 0.05$ was considered statistically significant.

The shear bond strength for brackets bonded with Transbond XT™ in Group I was 10.83 ± 3.03 MPa, for Aegis Ortho™ in Group II was 4.04 ± 2.12 MPa, and for Fuji Ortho LC in Group III was 10.99 ± 2.48 MPa. Fuji Ortho LC was found to have the highest and Aegis Ortho™ the lowest shear bond strength. *Post hoc* DMR test for individual group showed that there was no statistically significant difference in bond strength between Group I and Group III. One-way ANOVA showed that Group II (Aegis Ortho) has significantly lower bond strength than both Group I (Transbond XT) and Group III (Fuji Ortho LC) [Table 1], [Figure 5].

ARI

After debonding, the enamel surface of each tooth was examined with a stereomicroscope under ×10 to determine the amounts of residual adhesive remaining on each tooth. A modified ARI suggested by Artun and Bergland[11] was used to quantify the amount of remaining adhesive using the following scale: 0, no composite left on enamel surface; 1, less than half of composite left on enamel surface; 2, more than half of composite left on enamel surface; and 3, all composite left on enamel surface [Table 2].

The mean ARI score for Transbond XT was 1.4 in Group I, for Aegis Ortho, it was 0.67 in Group II, and for Fuji Ortho LC, it was 0.87 in Group III. Kruskal–Wallis ANOVA was used as non-parametric tool to compare ARI scores between different groups [Table 3], [Figure 6]. It showed that there was statistically significant difference between the mean ARI values of Group I and Groups II and III. For all statistical evaluations, a two-tailed $P < 0.05$ was considered statistically significant.

Table 1: Analysis of variance (one-way ANOVA) of shear bond strength (MPa) comparing different groups

Group	Mean	±SD	F value	*p* value
I	10.83[b]	3.03	71.389	<0.001
II	4.04[a]	2.12		
III	10.99[b]	2.48		

[a,b]Means with same superscript do not differ each other (Duncan's multiple range test)

Table 2: Number of samples and their distribution according to adhesive remnant index

Group	Adhesive remnant index scores			
	0	1	2	3
I	2	5	9	4
II	5	10	3	2
III	5	9	3	3

ARI scores: 0 – no composite left on enamel surface; 1 – less than half of composite left on enamel surface; 2 – more than half of composite left; and 3 – all composite left

Table 3: Kruskal–Wallis ANOVA comparing adhesive remnant index scores between different groups

Group	Mean	Median	±SD	H value	*p* value
I	1.40	2.00	0.74	9.841	< 0.05
II	0.67	1.00	0.49		
III	0.87	1.00	0.52		

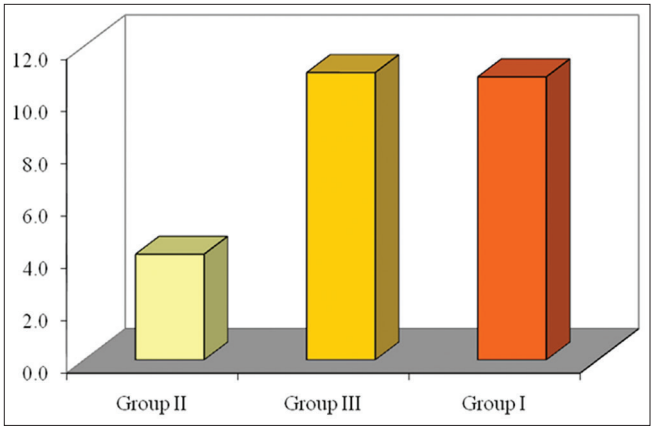

Figure 5: Comparison of mean shear bond strength

DISCUSSION

Until recently, most of the attention has been on fluoride, either placed topically in various concentrations or incorporated into restorative materials with varying

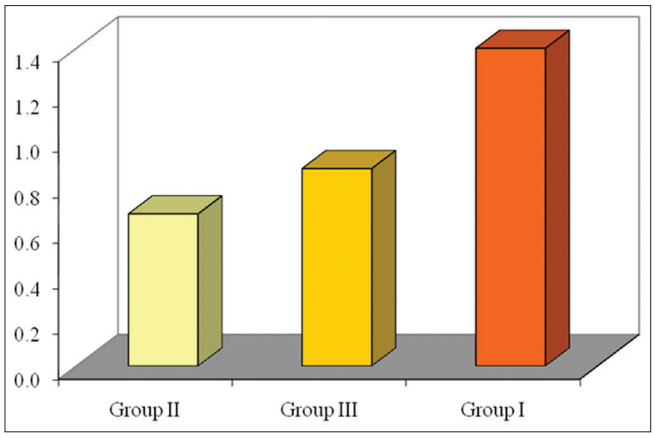

Figure 6: Comparison of adhesive remnant index

degrees of fluoride release, but the development and incorporation of ACP materials in dentistry is a different approach to reverse the effects of demineralization on enamel surfaces. There is no doubt that fluoroapatite is less soluble than hydroxyapatite, but exactly how much fluoride released from a restorative material to inhibit caries is still unknown.[6]

ACP-containing materials are a new class of "smart" materials which get self-activated in low pH oral environments and return to a dormant state when the pH returns to normal. Skrtic *et al.*[9] demonstrated that ACP accelerates the tooth's natural calcium phosphate remineralization process to prevent demineralization that could be due to microleakage or poor oral hygiene.

It is one of the first "smart" materials in dentistry to receive lukewarm reviews. Ariston pHc™ (Vivadent, Schaan, Lichtenstein) was marketed in Europe as a restorative material that released calcium, hydroxyl, and fluoride ions, but little scientific information was generated about its actual ion-release effects.[12] Again, van Dijken[13] reported a clinically unacceptable failure rate using Ariston pHc™, which contained a calcium source but no phosphate. The calcium release was largely ineffective because the resin chemistry did not favor continuous release.

The first commercially available ACP-containing materials were a sugar-free chewing gum containing casein phosphopeptide-ACP (CPP-ACP) known as Recaldent™ (Recaldent Pty. Ltd., Melbourne, Australia) and an ACP-containing toothpaste, Enamelon™ (Enamel, Cranbury, NJ).[14] Studies showed that CPP-ACP chewing gum resulted in a dose-related increase in enamel subsurface remineralization. This increased enamel remineralization was consistent with the previous studies showing the anticariogenic and remineralization potential of CPP-ACP in solution.[15,16] Although there is a growing body of evidence to support ACPs remineralizing potential, there is concern over the mechanical properties of ACP-containing materials.

In the early 1990s, the first bioactive mineral ion-releasing composites were developed, based on a polymer matrix phase and a filler phase of ACP.[8] The ACP had to be stabilized by pyrophosphate ions to retard conversion of ACP back to apatite, and because of ACPs high solubility, a substantial release of calcium and phosphate ions was possible. These levels were found to be sustainable and could promote remineralization in tooth enamel.

Unfortunately, pyrophosphate-stabilized ACP-filled composites are mechanically weak because ACP does not reinforce the composite like silanized fillers do in most resin-based composites. Previous investigators suggested that ACP-containing dental materials should be limited to situations when mechanical demands are less, such as in pit and fissure sealants or bases and liners.[17] Skrtic *et al.*[9] demonstrated that ACP-containing composites can be made stronger by the addition of glass-forming agents and with silica or zirconia-hybridized ACP in Bis-GMA/TEGDMA/HEMA/ZrDMA-based composites.

The most clinically relevant property of an adhesive system is its bond strength as it determines the clinical longevity of the bonded attachments. In the current study, the mean shear bond strength of Group I (Transbond XT), Group II (Aegis Ortho), and Group III (Fuji Ortho LC) was 10.83 ± 3.03, 4.04 ± 2.12, and 10.99 ± 2.48 MPa, respectively. One-way ANOVA was done to evaluate for significant difference at $P < 0.05$ in the mean shear bond strengths of three groups and *post hoc* DMR test for individual group comparison.

One-way ANOVA analysis showed that there is a statistically significant difference in shear bond strength between Group II and the other two groups (Groups I and III) ($P < 0.001$). However, a *post hoc* Duncan's analysis revealed no statistically significant difference between Groups I and III. Group III showed highest shear bond strength among three groups and Group II showed lowest shear bond strength.

It has been reported that clinically adequate shear bond strengths for metal orthodontic brackets to enamel should range from 5.9 to 7.8 MPa in terms of clinical and 4.9 MPa in terms of laboratory performances as suggested by Reynolds.[18]

The bond strength (10.83 MPa) at which failure was observed for the brackets in the Transbond XT control

group falls within acceptable limits reported in the orthodontic literature.[3-5] The corresponding value for shear bond strength of the Aegis Ortho group was only 4.04 MPa, which is just below the range in terms of laboratory performances as suggested by Reynolds.[18] Fuji Ortho LC exhibited sufficient bond strength to be used as an orthodontic adhesive.

Only a few articles have investigated the incorporation of calcium phosphates into orthodontic adhesives. Dunn[19] examined the bond strength of the same ACP adhesive used in this study and observed that brackets failed at a significantly lower force level (14.2 N) when compared to a traditional composite resin adhesive, as was found in the current study. Kawabata *et al.*[20] investigated the bond strength of a resin cement containing varying amounts of α-tricalcium phosphate and concluded the formulations possessed a clinically acceptable bond strength.

On examining the enamel surfaces of the debonded samples, it was found 45% of Group II brackets and 50% of Group III brackets gave an ARI score of 1 (<50% of adhesive remained on the enamel surface) while 45% of Group I brackets gave ARI score of 2 (more than 50% of adhesive remained on the enamel). This implies in both Groups II and III the bond failure occurred at enamel-adhesive interface and for Group I at adhesive-bracket interface.

Less resin on the enamel surface after debonding corresponds to less work and time spent by the orthodontist to remove the resin. Hence, a lower ARI score is favorable in this situation. Group II did have a significantly ($P < 0.05$) lower ARI score compared to the other two groups. Therefore, the clinician would have to spend very little time removing the ACP-containing adhesive from the tooth. In addition, this difference in ARI scores suggests the ACP-containing adhesive exhibited more of a cohesive failure compared to the composite resin and resin-modified glass ionomer adhesives.

No clinical studies have examined the survival rate of brackets bonded with ACP-containing adhesives. It should be stressed. This study suggests that, if this adhesive is found to have remineralization capabilities, it may be acceptable to some clinicians and patients who have higher caries susceptibility if it prevents white spots. However, as per this study, from the viewpoint of bond strength, Aegis Ortho™ cannot be recommended, as the mean shear bond strength obtained for this material was only 4.04 MPa (±2.12) which is well below the accepted range suggested by Reynolds.[18]

Being an *in vitro* bond strength study, caution is advised in extrapolating the results of the present study to the clinical situation.

CONCLUSION

The result of the present study showed that orthodontic resin containing ACP has significantly lesser shear bond strength as compared to conventional resin-based composite and resin-modified GICs ($P < 0.001$). This would suggest greater tendency for earlier debonding which would be clinically unacceptable.

Fuji Ortho LC displayed acceptable bond strength similar to Transbond XT. Both these groups showed significantly higher bond strength than Aegis Ortho.

Aegis Ortho and Fuji Ortho LC displayed lower value of ARI score compared to Transbond XT.

The complete characterization of this adhesive system will be incomplete without an *in vivo* evaluation.

REFERENCES

1. Buonocore MG. A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces. J Dent Res 1955;34:849-53.
2. Newman GV, Snyder WH, Wilson CE Jr. Acrylic adhesives for bonding attachments to tooth surfaces. Angle Orthod 1968;38:12-8.
3. Wiltshire WA. Shear bond strengths of a glass ionomer for direct bonding in orthodontics. Am J Orthod Dentofacial Orthop 1994;106:127-30.
4. Lippitz SJ, Staley RN, Jakobsen JR. *In vitro* study of 24-hour and 30-day shear bond strengths of three resin-glass ionomer cements used to bond orthodontic brackets. Am J Orthod Dentofacial Orthop 1998;113:402-7.
5. Fricker JP. A new self-curing resin-modified glass ionomer cement for the direct bonding of orthodontic brackets *in vivo*. Am J Orthod Dentofacial Orthop 1998;113:384-6.
6. Cate JM, Featherstone JD. Mechanistic aspects of the interactions between fluoride and dental enamel. Crit Rev Oral Biol Med 1991;2:283-96.
7. Skrtic D, Hailer AW, Antonucci JM, Takagi S, Eanes ED. Quantitative assessment of the efficacy of amorphous calcium phosphate/methacrylate composites in remineralizing caries-like lesions artificially produced in bovine enamel. J Dent Res 1996;75:1679-86.
8. Antonucci JM, Skrtic D, Eanes ED. Bioactive polymeric dental materials based on amorphous calcium phosphate. Polym Prepr 1994;35:460-1.
9. Skrtic D, Antonucci JM, Eanes ED, Eidelman N. Dental composites based on hybrid and surface-modified amorphous calcium phosphates. Biomaterials 2004;25:1141-50.
10. Miller JR, Mancl L, Arbuckle G, Baldwin J, Phillips RW. A three-year clinical trial using a glass ionomer cement for the bonding of orthodontic brackets. Angle Orthod 1996;66:309-12.
11. Artun J, Bergland S. Clinical trials with crystal growth conditioning as an alternative to acid-etch enamel pretreatment. Am J Orthod 1984;85:333-40.
12. Kielbassa AM, Shulte-Monting J, Garcia-Godoy F, Meyer-Lueckel H. Initial *in situ* secondary caries formation: Effect of various fluoride-containing restorative materials. Oper Dent 2003;28:765-72.
13. Van Dijken JW. Three-year performance of a calcium-, fluoride-, and hydroxyl-ions-releasing resin composite. Acta Odontol Scand 2002;60:155-9.
14. Shen P, Cai F, Nowicki A, Vincent J, Reynolds EC. Remineralization of enamel subsurface lesions by sugar-free chewing gum containing

casein phosphopeptide-amorphous calcium phosphate. J Dent Res 2001;80:2066-70.
15. Reynolds EC, Cain CJ, Webber FL, Black CL, Riley PF, Johnson IH, *et al*. Anticariogenicity of calcium phosphate complexes of tryptic casein phosphopeptides in the rat. J Dent Res 1995;74:1272-9.
16. Reynolds EC. Remineralization of enamel subsurface lesions by casein phosphopeptide-stabilized calcium phosphate solutions. J Dent Res 1997;76:1587-95.
17. Park MS, Eanes ED, Antonucci JM, Skrtic D. Mechanical properties of bioactive amorphous calcium phosphate/methacrylate composites. Dent Mater 1998;14:137-41.
18. Reynolds IR. A review of direct orthodontic bonding. Br J Orthod 1975;2:171-8.
19. Dunn WJ. Shear bond strength of an amorphous calcium-phosphate-containing orthodontic resin cement. Am J Orthod Dentofacial Orthop 2007;131:243-7.
20. Kawabata R, Hayakawa T, Kasai K. Modification of 4-META/MMA-TBB resin for safe debonding of orthodontic brackets-influence of the addition of degradable additives or fluoride compound. Dent Mater J 2006;25:524-32.

How to cite this article: Sahoo NR, Sahoo R. An *In vitro* Study to Evaluate the Shear Bond Strength of an "Amorphous Calcium Phosphate"-Containing Orthodontic Adhesive. Int J Sci Stud 2020;8(5):23-29.

Source of Support: Nil, **Conflicts of Interest:** None declared.