International Journal of Dental Sciences and Research, 2018, Vol. 6, No. 4, 90-94
Available online at http://pubs.sciepub.com/ijdsr/6/4/3

DOI:10.12691/ijdsr-6-4-3

Microshear Bond Strength of Bioactive Restorative Materials to Dentin

Ola M. Sakr[1,2,*]

[1]Department of Conservative Dentistry, College of Dentistry, Qassim University, Kingdom of Saudi Arabia
[2]Department of Operative Dentistry, College of Dentistry, Misr University for Science and Technology, Egypt
*Corresponding author: olasakr2004@yahoo.com

Abstract **Purpose**: To evaluate the microshear bond strength of bioactive restorative (BAR) materials to human dentin surface and estimate the clinical durability and serviceability of tested restorative materials **Materials and Methods**: Eighty dentin samples obtained from caries free human premolars and randomly divided into four groups (n = 20) according to application tested restorative materials First group (A) specimens tested direct application of bioactive restoratives BAR material to dentin without etching step. Second group (B) specimens tested for direct application of BAR material to dentin with etching step. Third group (C) specimens tested for direct application of bioactive base (BAB) material to dentin. The fourth group (D) specimens tested for direct application of nanofilled restorative (NFR) material to dentin with etching step, before application of tested restorative materials, a hollow cylinder (2.0 mm height/0.75mm internal diameter) was placed on the treated dentin surfaces. Tested restorative material inserted into the tube and cured. After artificial saliva storage for 5 days, the tube removed and microshear bond strength was determined in a universal testing machine at a crosshead speed of 0.5 mm/min. Data presented as mean and standard deviation (SD) values. Regression model using two-way Analysis of Variance (ANOVA) used in testing significance to evaluate the microshear bond strength of tested restorative materials. **Results:** It was found that group (B) BAR material bonded to etched dentin recorded the highest microshear bond strength mean value (12.18±1.39 MPa) followed by group (D) NFR material mean value (12.06±1.89 MPa) then group, (C) BAB materials mean value (9.96±1.27 MPa) while group (A) BAR material to unetched dentin recorded the lowest microshear bond strength mean value (9.88±2.11 MPa). The difference between all groups was statistically significant as indicated by one-way ANOVA (P=0.003>0.05). Tukey's post-hoc showed non-significant (P>0.05) difference between (groups A and C) or (groups B and D). **Conclusions:** Saliva is a necessary component as calcium and phosphate supplement for proper bond strength of BAR to etched dentin. BAR and NFR have a good and clinically accepted microeshear bond strength. BAB difficult to be used in open sandwich techniques. NFR proper adhesive selection lead to proper collagen encapsulation with qualified bonded interface.

Keywords: *Bioactive Restoratives (BAR), Bioactive Base (BAB), Bioactive Glass Particles (BAG) Nano Filled Restoratives (NFR), Dentin, Microshear Bond Strength*

Cite This Article: Ola M. Sakr, "Microshear Bond Strength of Bioactive Restorative Materials to Dentin." *International Journal of Dental Sciences and Research*, vol. 6, no. 4 (2018): 90-94. doi: 10.12691/ijdsr-6-4-3.

1. Introduction

One of important goals of restorative dentistry is to introduce proper functionally and esthetically accepted restorations. New techniques and materials in combination with new concepts for tooth surface preparation and caries removal have been developed in conservative dentistry [1,2].

Recently, GV Black principle of extension for prevention, has been shifted to the concept of 'minimal intervention dentistry. This approach stressed on the achievement of a more conservative cavity design [3,4]. In most clinical teeth caries removal cases resulted in large areas of exposed dentin. So, bond strength on dentin is a critical, important issue for the restoration retention in place especially for those cavities/preparations with minimal mechanical retention means like cervical restorations [5,6,7].

Large exposed dentin surface showed another problem with opened dentinal tubules with subsequent post-operative dentin hypersensitivity [8].

Most of clinically available resin composites showed polymerization shrinkage during its setting reaction with creation of marginal gap. This tooth restoration interface increased as a result of functional cyclic mechanical loading. These marginal gaps can act as suitable anchorage sites for bacterial colonies with high susceptibility rate of recurrent caries [9].

One possible approach to increase the restorations resistance to recurrent caries possibility is to add agents that have negative effect to the micro-organisms and/or enhance remineralization of demineralized hard dental tissues. A lot of published original researches and reviews showed the efficient antibacterial properties of different, commercially available bioactive glass (BAG) compositions against many different bacterial species [10,11,12,13].

However, A little data available about clinical performance of bioactive glasses containing dental composites, Ion leaching of bioactive composites is a questionable mechanism in relation with mechanical properties of bioactive materials Another concern should be considered that BAG fillers intimate adherence to the composite matrix is questionable which may result in unsuitably low mechanical properties [14,15,16]. This study goal is evaluating microshear bond strength of some commercially available bioactive restorative and base materials to dentin.

2. Materials and Method

2.1. Preparation of the Specimens

Forty permanent premolar teeth, caries-free, extracted for orthodontic were collected. Each tooth was scaled to remove any remaining soft tissue and stored in distilled water. Separation of crowns from the roots were 2 to 3 mm apical to the cementoenamel junction using a diamond saw (Isomet, Buehler, Lake Bluff, IL, USA) under water cooling at a low speed. Crowns were sectioned into two halves with a diamond disc, under water-cooling. Each specimen was embedded in self-curing acrylic resin (Meliodent, Heraeus Kulzer, Dormagen, Germany) into Teflon molds exposing specimen's labial surfaces. Enamel were removed then gentle polishing of dentin surfaces using a 600-grit silicon carbide paper under running water to prepare a flat dentin surfaces 0.5 mm deep to dentinoenamel junction guided by 2 grooves created using small abrasive round bur. Specimens were examined under a 40-x stereomicroscope (Leica, MZ 12, Leica AG, CH-9435 Heerbrugg, Switzerland), to exclude any cracked or hypoplastic defected specimen.

2.2. Specimens Classification

Eighty dentin specimens were prepared and randomly divided into four groups (n = 20) according to tested restorative materials. First group (A) specimens were tested for direct application of bioactive restoratives material (BAR) to dentin without etching step. Second group (B) specimens were tested for direct application of bioactive restoratives material (BAR) to etched dentin. Third group (C) specimens were tested to direct application of bioactive base (BAB) material to dentin. The fourth group (D) specimens were for direct application of nanofilled restorative material (NFR) to dentin with etching and bonding steps. Tested material data were listed in Table 1.

2.3. Application of Tested Restorative Materials to Dentin

Dentin surfaces were cleaned with water spray for 5 seconds and dried with cotton pellet. For all specimens, hollow cylinders 2.0 mm in height were cut from micro-bore tygon tubing (Norton Performance Plastic; OH, USA) with an internal diameter of 0.75mm and were placed to dentin surfaces.

For group (A): Placing the mix tip on Activia syringe, inserting syringe into Activia Spenser and snap into place using firm pressure. Dispensing Activia BAR material using gentle pressure. To ensure an even mix of base and catalyst, dispense 1-2 mm of material onto a mixing bad and discard this material. Using small applicator, mixed material was applied into the small tube on dentin surface. Light curing for 20 secs using a halogen light source (Visulux curing unit, Vivadent; Schaan, Liechtenstein). The output of the light curing unit was regularly checked (500 mW/mm^2). Final covering layer with Glycerin as oxygen barrier.

For group (B): Etching of prepared dentin surface for 10 secs with 35% phosphoric acid. rinse and dry, removal of excess moisture with cotton pellet. Small plastic tube place on etched surfaces. Placing the mix tip on Activia syringe, inserting syringe into Activia Spenser and snap into place using firm pressure. Dispensing Activia BAR using gentle pressure. To ensure an even mix of base and catalyst, dispense 1-2 mm of material onto a mixing bad and discard this material. Using small applicator, mixed material was applied into the small tube on dentin surface. Light curing as followed in group (A) specimens. Final covering layer with Glycerin as oxygen barrier.

For group (C): BAB was applied to dentin with the same steps of material application used in group (A)

For group (D): Dentin surfaces were etched with 35% phosphoric acid, Scotchbond, for 10 secs, and rinsed with water spray for 10 s. Excess water was removed with cotton pellet. Bonding agent (Adper Single bond 2) were applied with a disposable brush, 2 consecutive coats for 10 s with gentle agitation using a fully saturated applicator. Gently air thin for five seconds in evaporative solvents. light cured for 10 s using a halogen light source (Visulux curing unit, Vivadent; Schaan, Liechtenstein). The output of the light curing unit was regularly checked (500 mW/mm^2). Small plastic tube place on etched surfaces. Using small applicator, nanocomposite Filtek Z350 XT material were applied into the small tube on dentin surface.

All specimens were stored in artificial saliva at 37°C for 5 days. The tygon tubing around tested materials cylinders was removed by gently cutting the tube into two hemi cylinders using a feather-edge blade.

2.4. Microshear Bond Strength Testing

Microshear bond strength testing were performed using NEXYGEN from Lloyd Instruments. Each specimen with its own bonded restorative material micro-cylinders were tightened with tightening screws to the lower fixed compartment of a materials testing machine (Model LRX-plus; Lloyd Instruments Ltd., Fareham, UK) with a loadcell of 5 kN and data were recorded via computerized software (Nexygen-MT Lloyd Instruments).

An orthodontic loop (0.014" in diameter) were wrapped around the tested restorative materials and aligned with the loading axis of the upper movable compartment of the testing machine. A slow shearing load with tensile mode of force was applied via materials testing machine at a crosshead speed of 0.5 mm/min. The debonding loads were recorded in Newton.

Table 1. Composition, lot number, and manufacture of the tested materials

Material	Composition	Lot number	Manufacture
ACTIVIA Bio active restorative (BAR)	56% by weight reactive glass particles that mimic physical and chemical properties of natural teeth., shock absorbing ionic resin component containing acidic monomer with antimicrobial properties .no Bisphenol A, No BisGMA, no BPA derivatives.	150318	PULPDENT Corporation
ACTIVIA Bio active base /liner (BAB)	Bioactive ionic resin with reactive glass	160406	PULPDENT Corporation
Scotchbond etchant gel	35 % phosphoric acid	N 110268	3M ESPE
Adper Single bond 2	(10% colloidal nanofiller) BisGMA, HEMA, dimethacrylates, ethanol, water, photoinitiator system and a methacrylate functional copolymer of polyacrylic and polyitaconic acids	N353034	3M ESPE
Filtek Z350 XT (NFR)	(20 nm silica filler 4-11 nm zirconia filler) as 72.5% by w filler bis-GMA, UDMA, TEGDMA, PEGDMA and bis-EMA resins	N339145	3M ESPE

Microshear bond strength in MPa (τ) calculated with following equation;

$$\tau = P/ \pi r2$$

where; τ =bond strength (in MPa), P =load at failure (in N), π =3.14, r = radius of micro-cylinder (in mm)

2.5. Statistical Analysis

Data analysis was performed in several steps. Initially, descriptive statistics for each group results. One-way ANOVA followed by pair-wise Tukey's post-hoc tests were performed to detect significance between groups. Statistical analysis was performed using Graph-Pad Prism version 4.00 for Windows, Graph-Pad Software, San Diego California USA. P values ≤ 0.05 are considered to be statistically significant in all tests.

3. Results

The mean values and standard deviation (SD) of microshear bond strength test results for all groups are summarized in Table 2 and graphically drawn in Figure 1.

It was found that group B recorded the highest microshear bond strength mean value (12.18±1.39 MPa) followed by group D mean value (12.06±1.89 MPa) then group, C means value (9.96±1.27 MPa) while group A recorded the lowest microshear bond strength mean value (9.88±2.11 MPa). The difference between all groups was statistically significant as indicated by one-way ANOVA (P=0.003>0.05). Tukey's post-hoc showed non-significant (P>0.05) difference between (groups A and C) or (groups B and D)

Numerical data were explored for normality by checking the distribution of data, calculating the mean and median values as well as using the tests of normality (Kolmogorov-Smirnov and Shapiro-Wilk tests). Data showed non-parametric distribution and were presented as mean, median, standard deviation (SD) and range values.

Kruskal-Wallis test was used to compare between the four groups. Mann-Whitney U test was used for pair-wise comparisons when Kruskal-Wallis test is significant. Bonferroni's adjustment was performed for pair-wise comparisons.

The significance level was set at $P \leq 0.05$. Statistical analysis was performed with IBM® SPSS® Statistics Version 20 for Windows.

Table 2. Comparison of microshear bond strength test results (Mean±SD) between all groups

Variables		Descriptive statistics			
		Mean±SD	**95 % confidence intervals**		**ANOVA**
			Low	**High**	**P value**
Experimental groups	Group A	$9.88^{B}\pm2.11$	8.37	11.93	0.003*
	Group B	$12.18^{A}\pm1.39$	11.18	13.17	
	Group C	$9.96^{B}\pm1.27$	9.06	10.87	
	Group D	$12.06^{A}\pm1.89$	10.71	13.42	

Different superscript large letter in same column indicating significant between groups (p<0.05), *; significant (p<0.05).

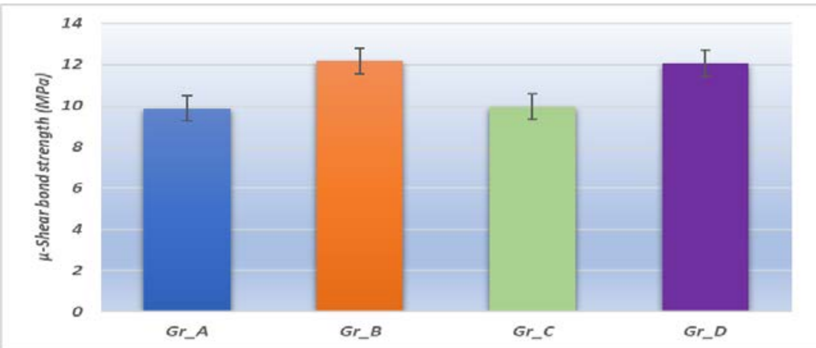

Figure 1. Column chart of µ-shear bond strength mean values for all groups

4. Discussion

The dentin adhesion to restorative materials has been investigated for effective bond and stable restoration. There are a lot of challenges as dentin complex structure, composition and its characteristic moisture [17].

Our results of tested BAR showed significantly highest microshear bond strength to etched dentin. This strong attachment between etched dentin surface involving exposed collagen fibrils with BAR may be attributed to strong affinity and integration between collagen network and modified BAG particles. These findings are coincide to interfaces studies between BAG and dental tissues that showed interdigitation of collagen within modified surface of BAG after 5 days incubation in saliva [17,18,19]. So the intimate bonding based mainly on dentin exposed collagen fibrils as a result of partial demineralization of dentin surfaces due to acid etching procedures. This principle may explain our findings of non-statistically significant difference of low microeshear bond strength in group A (BAR) and group C (BAB) as no etching procedures prior of application of restorative materials [20].

Many studies explained and illustrated that apatite precipitation is formed while BAG bonding to hard and soft tissues in a process where silicic acid is released first then chemisorbed into demineralized collagen via electrostatic bonding to form a polycondensation silica-gel-layer for the precipitation of Ca and PO4 [21-28]. Also Suge T et al (1995) stated that calcium-phosphate compounds formed on the dentin surfaces interface with BAG explained calcium and phosphate ion uptake from the artificial saliva over the storage period, by the resistance of the interaction layer to dissolution [29].

Also, our microshear measures found that group D (NFR) showed high records of microshear bond strength to dentin. As three-step system usually acts as the gold-standard etch-and-rinse adhesives with good restoration retention results. The lower microshear bond strength of (NFR) than (BAR) to etched dentin may be attributed to incomplete infiltration of the adhesive monomer to demineralized dentin layer. these findings in acceptance with many studies who stated that effective collagen encapsulation was happen as a result of simplified adhesives that put together hydrophilic and hydrophobic component in the same bottle impairing proper encapsulation and collagen fibrils protection [30].

Many studies stated that suboptimal polymerization and phase separation reported as important factors of the simplified adhesive interface degradation over time. Particularly as a result of suboptimal polymerization, the interface created by simplified adhesives may result in a semipermeable adhesive layer [31-35].

5. Conclusions

Under the limitation of this study, following points can be concluded:

-Saliva is a necessary component as calcium and phosphate supplement for proper bond strength of BAR to etched dentin.

- BAR and NFR have good and clinically accepted microeshear bond strength.

- BAB difficult to be used in open sandwich techniques

- NFR proper adhesive selection lead to proper collagen encapsulation with qualified bonded interface,

List of Abbreviations

BAR**:** bioactive restoratives
BAB: Bioactive base
BAG: Bioactive glass
NFR: Nano filled restoratives

Acknowledgments

The author gratefully acknowledge Qassim University, represented by the Deanship of Scientific Research ,on the material support for this research under the number (dent-2018-1-14-S-3557) during the academic year 1437 AH /2016 AD"

References

[1] Antunes LA, Pedro RL, Vieira AS , Maia LC. :Effectiveness of high speed instrument and air abrasion on different dental substrates. Braz Oral Res 2008; 22: 235-41.
[2] Neuhaus KW, Ciucchi P, Donnet M , Lussi A. Removal of enamel caries with an air abrasion powder. Oper Dent 2010; 35: 538-46.
[3] Black GV: A work in operative dentistry in two volumes. Chicago: Medico-Dental Publishing. 1917.
[4] Degrange M, Roulet JF: Minimally invasive restorations with bonding. ,Chicago: Quintessence Publishing. 1997.
[5] Finnema KJ, Ozcan M, Post WJ, Ren Y, Dijkstra PU.: In-vitro orthodontic bond strength testing: a systematic review andmeta-analysis. Am J Orthod Dentofac Orthop 2010; 137:615-22. e3.
[6] De Munck J, Mine A, Poitevin A, Van Ende A, Cardoso MV, Van Landuyt KL, et al.: Meta-analytical review of parameters involved in dentin bonding. J Dent Res 2012; 91: 351-7.
[7] Braga RR, Meira JB, Boaro LC, Xavier TA. :Adhesion to tooth structure: a critical review of macro test methods. Dent Mater 2010; 26: e38-49.
[8] Fu Y, Li X, Que K, Wang M, Hu D, Mateo LR, DeVizio W, Zhang YP: Instant dentinal hypersensitivity relief of a new desensitizing dentifrice containing 8.0% arginine, a high cleaning calcium carbonate system and 1450 ppm fluoride: a 3-day clinical study in Chengdu, China. Am Dent J .2010, 23:20A-27A.
[9] Choi KK, Condon JR, Ferracane JL.: The effects of adhesive thickness on polymerization contraction stress of composite. J Dent Res 2000 ;79 :812-7.
[10] Martins CHG, Carvalho TC, Souza MGM, Ravagnani C, Peitl O,Zanotto ED, et al. Assessment of antimicrobial effect of Biosilicate (R) against anaerobic, microaerophilic and facultative anaerobic microorganisms. Journal of Materials Science: Materials in Medicine 2011; 22:1439-46.
[11] Yarborough L, Engle J, Wang M, Ferracane JL, Mitchell JC.: Inhibition of bacteria on a bioactive glass-containing, anti-microbial sealant material. In: AADR/CADR annual meeting and exhibition. 2012.
[12] Mitchell JC, Musanje L, Ferracane JL. Biomimetic dentin desensitizer based on nano-structured bioactive glass. DentMater 2011; 27: 386-93.
[13] Ornaghi BP, Meier MM, Rosa V, Cesar PF, Lohbauer U, Braga RR.. Subcritical crack growth and in vitro lifetime prediction of resin composites with different filler distributions. Dent Mater 2012; 28:985-95.
[14] D. Khvostenkoa, J.C. Mitchellb, T.J. Hiltonc, J.L. Ferracanec, J.J. Kruzica. Mechanical performance of novel bioactive glass containing dental restorative composites. dental materials 29. 2013, 1139-1148.
[15] Elbishari H, Silikas N, Satterthwaite J.: Filler size ofresin-composites, percentage of voids and fracture toughness: is there a correlation? Dent Mater J 2012; 31:523-7.

[16] Drummond JL, Lin LH, Al-Turki LA, Hurley RK.: Fatigue behavior of dental composite materials. J Dent 2009; 37: 321-30.

[17] Efflandt SE, Magne P, Douglas LF, Francis LF : Interaction between bioactive glasses and human dentin. J Mater Sci 2002, 13, :557-565.

[18] Davies, G.C., Bruce, D.M. :A stress analysis model for composite coaxial cylinders ., Journal of Mate Scie ,1997, 32(20), pp. 5425-54.

[19] Lee J-H, Seo S-J, Kim H-W. Bioactive glass-based nanocomposites for personalized dental tissue regeneration. Dent Mater J 2016; 35:710-20.

[20] Lorenzo B∗, Tatjana M, Sandra Rc, Allegra C, Milena C, Leo T, David H., Franklin R. and Annalisa M: Dentin bonding systems: From dentin collagen structure to bond preservation and clinical applications. Dent Mater J. 2 0 1 8, 3 4: 78-96.

[21] Marshall GW Jr, Marshall SJ, Kinney JH, Balooch M. The dentin substrate: structure and properties related to bonding. J Dent. 1997; 25: 441-58.

[22] Ebisawa, Y., Kokubo, T., Ohura, K., Yamamuro, T.. Bioactivity of CaO·SiO2-based glasses: in vitro evaluation. Journal of Materials Science: Materials in Medicine. 1990,1(4), pp. 233-238.

[23] Kokubo, T., Kushitani, H., Sakka, S., Kitsugi, T., Yamamuro, T.. Solutions able to reproduce in vivo surface-structure changes in bioactive glass-ceramic A-W. ,Journal of Biomedical Materials Research, 1990, 24(6), pp. 721-734.

[24] Engqvist H, Schultz-Walz JE, Loof J, Botton GA, Mayer D, Phaneuf MW, et al. :Chemical and biological integration of a mouldable bioactive ceramic material capable of forming apatite in vivo in teeth. J of Biomaterials. 2004; 25: 2781-7.

[25] Hench LL. Bioceramics and the origin of life. J Biomed Mater Res. 1989; 23: 685-93.

[26] Zhong JP, LaTorre GP, Hench LL.. The kinetics of bioactive ceramics part VII: Binding of collagen to hydroxyapatite and bioactive glass. In Bioceramics 7, eds. OH Andersson, A Yli-Urpo, 1994, pp. 61-66.

[27] Sauro S, Thompson I, Watson TF.: Effects of common dental materials used in preventive or operative dentistry on dentin permeability and remineralization. Oper Dent. 2011; 36: 222-30.

[28] Sepulveda P, Jones JR, Hench LL.: Bioactive sol-gel foams for tissue repair. J Biomed Mater Res. 2002; 59:340-8.

[29] Suge T, Ishikawa K, Kawasaki A, Yoshiyama M, Asaoka K, Ebisu S.: Duration of dentinal tubule occlusion formed by calcium phosphate precipitation method: in vitro evaluation using synthetic saliva. Journal of Dental Research1995; 74: 1709-14.

[30] Fabianni M, Lidiane C, Francisco C, Mônica Y b, Lorenzo B and Vicentede Paulo A.. Evaluation of resin/dentin bonded interfaces formed by different adhesive strategies and exposed to NaOCl challenge. Int J Adhesion &Adhesives, 2015, 59: 21-26.

[31] Breschi L, Mazzoni A, Ruggeri A, Cadenaro M, Di Lenarda R, De Stefano Dorigo E.. Dental adhesion review: aging and stability of the bonded interface. Dent Mater 2008; 24(1):90-101.

[32] Argolo S, Mathias P, Aguiar T, Lima A, Santos S, Foxton R, etal.: Effect of agitation and storage temperature on water sorption and solubility of adhesive systems. Dent MaterJ 2014; 34:1-6.

[33] Cadenaro M, AntoniolliF, SauroS, TayFR, DiLenardaR, PratiC, etal. :Degree of conversion and permeability of dental adhesives. Eur J Oral Sci 2005; 113: 525-30.

[34] CardosoMV, Almeida NevesA, MineA, CoutinhoE, VanLanduyt K, DeMunck J, et al.: Current aspects on bonding effectiveness and stability in adhesive dentistry. Aust Dent J, 2011; 56: 31-44.

[35] Tay FR, Frankenberger R, KrejciI, Bouillaget S, Pashley DH, Carvalho RM, etal.. Single-bottle adhesives behave as permeable membranes after polymerization I. In vivo evidence. J Dent 2004; 32: 611-21.