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Evaluation of the Influence of Different Bonding Systems at Different Time Intervals on the Shear Bond Strength of Composite Bonded To Biodentine -An In Vitro Study

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Introduction

Overtime there has been a continuous search for dental materials that present an ideal combination of good mechanical, physicochemical and biologic properties. In the early 1990s the development of mineral trioxide aggregate (MTA) introduced a new class of dental calcium silicate cements (CSCs) based on Portland cement, which was originally developed as a perforation repair material.[1] Beside a long setting time the major drawbacks of MTA are its relatively low compression and flexural strength, which are lower than those of dentine .These factors have limited its application to low stress-bearing areas. Hence, MTA cannot be used as a base, core build-up material or as temporary restoration.[2]

This triggered the development of new formulas of calcium silicate-based cements to overcome these drawbacks and keep the advantages. Biodentine (Septodont,St.-Maur-des-Fossés, France) can be considered as an outcome of this process. It is a high-purity dental material composed of tricalcium silicate, calcium carbonate, zirconium oxide, and a water-based liquid containing calcium chloride as the setting accelerator and water as reducing agent. The initial setting reaction takes approximately 12 minutes, however, through impedance spectroscopy tests it has been shown

that at least two weeks are necessary for the material to finally set.[3]

Biodentine is indicated for use as a dentin substitute under different restorations and as a repair material due to its good physical and chemical properties, good sealing ability, high compressive strength, short setting time, biocompatibility, bioactivity and biomineraization properties.To complete the final restoration in a single visit, an adhesive restorative material can be applied over partially set Biodentine layer. However, the potential of restorative materials to attach to Biodentine is not well know.[4] Therefore, it is important to identify materials that are compatible in relation to the interface between the two different materials. Understanding such behavior will be of great importance to complete final restoration.

Patient demand for esthetic restorations has generated an interest in the advancement of adhesive dentistry. Bonding to enamel has become a predictable technique; however, bonding to dentin has evolved significantly with the development of various bonding systems.[5]

The strength with which restorative materials bond to Biodentine is unclear. Also the proper timing for composite resin restoration over Biodentine placement has not been studied extensively because etching, rinsing, and priming all affect the bond strength of Biodentine. To date, a limited number of studies have been carried out on the bond strength between Biodentine and composite using various bonding systems at different time intervals.

Therefore, the aim of this in vitro study was to evaluate and compare the shear bond strength of three different bonding systems- Prime & bond NT, Clearfil SE bond and Single Bond Universal at different time intervals -12 min, 24 hr and 2 weeks and to evaluate the mode of failure under stereomicroscope for composite bonded to Biodentine.

Materials and Method

Three commercial adhesive systems, Prime & Bond NT (etch-and-rinse adhesive system), Clearfil SE Bond (two-step self-etch adhesive system) and Single Bond Universal Adhesive (one-step self-etch adhesive systems) were tested in this study and applied as recommended by the manufacturers. The materials used are listed in Table 1.

Specimen Fabrication-A total of 120 acrylic blocks containing a central hole with a 6mm diameter and a 3mm height were prepared. Biodentine was mixed according to the manufacturer's instructions. The acrylic blocks were fully filled with Biodentine. Then, the specimens were stored at 37˚C with 100% humidity for 12 minutes, 24 hours and 2 weeks to encourage setting.

The specimens were randomly divided into four groups (n =30) according to the dental adhesive systems- Control (No Adhesive) - (A); (etch-and-rinse adhesive system) Prime and bond NT (Dentsply, USA) - (B) ;(2-step selfetch adhesive system) Clearfil SE Bond, (Kuraray, Japan) – (C); (1-step self-etch adhesive systems) Single Bond Universal Adhesive(3M ESPE,USA) - (D) .Each group was further subdivided into three subgroups (n=10) according to different time interval i.e.12 min, 24 hr and 2 weeks. After 12 minutes in group 1, no adhesive system was applied (n=10), whereas in groups 2, 3, and 4, the corresponding adhesive system was applied over Biodentine according to the manufacturer's instructions. A composite material (Filtek Z-350, 3M ESPE, U.S.A) was applied into a cylindrical shaped plastic matrix with an internal diameter of 3mm and a height of 3 mm. Light curing was administered with a light emitting diode light-curing Unit (QTH75**TM** curing light , Dentsply, Germany) with an intensity of 500 mV/cm^2 for 20 seconds. This procedure was repeated at 24 hours and 2 weeks respectively.

Shear Bond Strength Test. The polymerized specimens were stored in 100% relative humidity at 37˚C. For shear bond strength testing, the specimens were secured in a holder placed on the platen of the testing machine and then sheared with a knife-edge blade on an instron universal testing machine (±100kN Spectro Private Limited Okhla, New Delhi) at a crosshead speed of 1.0 mm/min. Shear bond strength was calculated in MPa by dividing the peak load at failure with the specimen surface area.

Fracture Analysis. Fractured test specimens were examined under a stereomicroscope at a magnification of ×25 (Spectro Private Limited Okhla, New Delhi). Specimen fractures were classified as follows: cohesive failure exclusively within Biodentine, cohesive failure exclusively within restorative material, adhesive failure that occurred at the Biodentine restorative material interface, or mixed failure when 2 modes of failure happened simultaneously. Fracture analysis was performed by a single observer who was completely uninformed about the experimental groups.

Table 1: Composition

Bonding Agents

Prime & Bond NT (Caulk/Dentsply International Inc., Milford, DE, USA)	Di- and trimethacrylate resin, PENTA, Functionalized amorphous silica, Photoinitiators, stabilizers, Cetylamine, Hydrofluoride and Acetone
Clearfil SE Bond (Kuraray Noritake Dental Inc, Okayama, Japan)	*Primer* MDP, HEMA, Hydrophilic aliphatic dimethacrylate, Dicamphoroquinone, N-diethyl-p-toluidine and Water *Bond* MDP, BisphenolA-glycidyl methacrylate (bis-GMA), HEMA, HydrophobicAliphaticdimethacrylate, Dicamphoroquinone, N-diethyl-p-toluidine and Colloidal silica
Single Bond Universal Adhesive(3M ESPE, USA)	MDP, Dimethacrylate resins, HEMA, Vitrebond™, Copolymer, Filler, Ethanol, Water, Initiators, Silane

Etchant

Scotchbond™ Etchant(3M ESPE, USA)	35% phosphoric acid

Composite

Filtek Z-350 (3M ESPE, USA)	TEGDMA, Bis-GMA, Bis-EMA, Silica, Zirconia

Biodentine (Septodont,Saint-Maur-Des-Foss´Escedex, France)

Powder	Tricalcium silicate, dicalcium silicate, calcium carbonate and oxide, iron oxide, and zirconium oxide
Liquid	Calcium chloride and hydrosoluble polymer

Statistical Analysis- One-way analysis of variance was used to detect differences in bond strength among the experimental groups. Post hoc comparisons were performed using the Scheff´e test.

Results

The mean values and standard deviations of shear bond strengths are given in Table 2. When shear bond strengths of adhesive systems were compared, Significant differences were found between all of the adhesive groups at different time intervals (12 minutes, 24 hours and 2

weeks) (P < .05). Among the three time intervals, the bond strength of group 3 (two step self-etch adhesive) at 2 week period presented significantly higher bond strength values (4.4590±.13034 MPa) than group 1 (control), group 2 (etch-and-rinse adhesive) and group 4 (1-step self etch adhesive systems) at 12- minute period (P < .05).

Table 3 shows the fracture modes of the experimental groups. Most of the observed modes of failure were cohesive in Biodentine and adhesive failure. No specimens failed cohesively with in composite resin **(Fig.1).**

A B C

Table 2: Mean shear bond strength values of adhesives (MPa) to Biodentine

Group	N	12 Min.	24 Hr.	2 Weeks
Group A: Control	10	1.3270±.24400	1.5920±.09271	1.9700±.12238
Group B: Prime and Bond NT	10	1.7720±.27458	1.9550±.08960	3.2510±.40498
Group C: Clear fill SE Bond	10	1.1860±.24500	1.3060±.16788	4.4590±.13034
Group D: Single Bond Universal Adhesive	10	1.9030±.11898	2.7420±.16158	3.0130±.13022

Table 3: Fracture modes of the specimens after shear bond test

MODE	GROUP A			GROUP B			GROUP C			GROUP D		
	12 min	24 Hr	2 weeks	12 min	24 Hr	2 weeks	12 min	24 Hr	2 weeks	12 min	24 Hr	2 weeks
Adhesive	10	10	10	3	4	3	3	2	3	3	4	3
Mixed	0	0	0	2	1	1	2	2	1	3	1	2
Cohesive in Biodentine	0	0	0	5	5	6	5	6	6	4	5	5
Cohesive in composite resin	0	0	0	0	0	0	0	0	0	0	0	0

Discussion

Biodentine is a calcium silicate mixture based material that has polycarboxylate-based hydrosoluble polymer system described as “water reducing agent” to reduce the overall water content of the mix, along with calcium chloride as a setting accelerator. The combined effect reduces the setting time to 12 min and increases the compressive strength.[6]

To complete the final restoration, we should use a material that is compatible with Biodentine and can be applied over partially set material. Therefore, it is important to identify materials that can be applied over biodentine and can allow for immediate final restoration placement.[7] In addition, the longevity of the restorative materials depends partly on the bond strength of these restorations with the substrate.[8] Different methods are available to analyze the bond strength in-vitro. The most common method to evaluate adhesive properties of restorative materials is a shear bond strength assessment. This method has the advantage of being more easy to perform.[9] The superior performance of two-step self- etch adhesive systems (Clearfil SE bond) could be due to the following reasons: The solvent, water which is present in Clearfil SE bonding agent is in low concentration, the hydrophylicity of functional monomers is low[10], polymerization is to a greater degree[10], the underlined dentin undergoes limited etching and demineralization over a longer period of time[11], the presence of ethanol in one-step self-etch adhesive (Single Bond Universal) and acetone in etch and rinse adhesive (Prime and Bond NT).[12]

The functional monomers are delivered into the hybrid layer by the organic solvents, such as ethanol and acetone which act as carriers and water chasers. Acetone is more volatile than ethanol because acetone has vapor pressure 200 mm Hg at25°C where else Ethanol has 54.1 mm Hg.[13]Acetone is preferred as a solvent medium, due to its better hydrolytical stability of the functional monomers when compared to ethanol. Therefore it might be one of the reasons for superior bond strength of prime and bond adhesive in comparison to single bond. Therefore, the reasons for the low bond strength of one-step self-etching primers may be due to the following reasons: The

combination of acidic hydrophilic and hydrophobic monomers into a single step may compromise polymerization of the adhesive[14], the inherent low strength of the adhesive polymer[14], the lower degree of polymerization of the resin monomer because of a major solvent/oxygen inhibition effect during light activation of these materials[14] and the incompatibility between the adhesive and the restorative material[14].

The initial setting reaction takes approximately 12 minutes, however,through impedance spectroscopy tests it has been shown that at least two weeks are necessary for the material to finally set.[3]As mentioned earlier, it is during this period that the calcium carbonate crystals are still being formed. This setting time represents an improvement compared to other calcium silicate-based dental materials such as MTA, which takes more than two hours to reach an initial set.[3] In the present study the shear bond strength of Biodentine increased significantly from 12 min to two week. This may be explained by the fact that that the setting reaction of CSCs might continue for more than a month.[15]

As Biodentine has a smaller particle size and uniform components that may contribute to better adhesion and interlocking with the dentine, which consequently results in cohesive failures within the filling material.[16] In addition, the ability of Biodentine to form tag-like structures into dentinal tubules increased the micromechanical attachment.[17]

Conclusion

This in vitro study found statistically significant differences between all the three adhesive systems at each of the 3 time intervals. However, Biodentine has shorter setting time than MTA (12 min); the highest bond strength value was obtained for two-step self-etch adhesive at a 2 week period.

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