

Microtensile dentin bond strength of fifth with five seventh-generation dentin bonding agents after thermocycling: An *in vitro* study

BRUHVI POPTANI, GOHIL K. S.¹, JAISHREE GANJWALE², MANISHA SHUKLA³

Abstract

Objectives: The objective of this *in vitro* study was to compare the microtensile dentin bond strength (μ TBS) of five seventh-generation dentin bonding agents (DBA) with fifth-generation DBA before and after thermocycling. **Materials and Methods:** Ten extracted teeth were assigned to fifth generation control group (optibond solo) and each of the five experimental groups namely, Group I (G-Bond), Group II (S³ Clearfil), Group III (One Coat 7.0), Group IV (Xeno V), and Group V (Optibond all in one). The crown portions of the teeth were horizontally sectioned below the central groove to expose the dentin. The adhesive resins from all groups were bonded to the teeth with their respective composites. Specimens of sizes 1 × 1 × 6 mm³ were obtained. Fifty specimens that bonded to dentin from each group were selected. Twenty-five of the specimens were tested for debonding without thermocycling and the remaining were subjected to thermocycling followed by μ TBS testing. The data were analyzed with one-way ANOVA and Dunnett's-test for comparison with the reference group (Vth Generation). **Results:** There was no significant difference ($P > 0.05$) between the fifth- and seventh-generation adhesives before and after thermocycling. The results of our study showed significantly higher value ($P < 0.05$) of μ TBS of seventh-generation Group II (Clearfil S³) compared to the fifth-generation before and after thermocycling. **Conclusion:** The study demonstrated that the Clearfil S³ bond had the highest μ TBS values. In addition, of the five tested seventh-generation adhesive resins were comparable to the fifth-generation DBA.

Keywords: Clearfil S3, G-bond, microtensile bond strength, optibond solo, one coat 7.0, optibond all in one, thermocycling, Xeno V

Introduction

The use of bonded composite restorations has revolutionized today's dental practice by being able to replace the lost tooth tissue in an invisible and conservative way with immense success. Given the enormity of the number of commercial products available in the field of adhesion, the evaluation of bond durability is important, as the stability of the bond between the restoration and tooth substrate may be related to long-term clinical success of tooth colored restorations. Since the components of seventh-generation adhesives differ from those of fifth-generation adhesive systems, the thermal stresses created at the bonding interface might also vary.

Department of Conservative Dentistry and Endodontics, Karnavati School of Dentistry, Ahmedabad, ¹Sumandeep Vidyapeeth, Baroda, ²Department of Community Medicine & Central Research Services, Pramukhswami Medical College, Karamsad, Anand, Gujarat, ³Goenka Institute of Dental Sciences, Piplaj, Gandhinagar, India

Correspondence: Dr. Bruhvi Poptani, Karnavati School of Dentistry, Uvarsad, Ahmedabad, Gujarat, India.
E-mail: bruhvip@yahoo.com

Although the most reliable conclusion about the performance of dental adhesive systems in the oral environment is derived from long-term clinical trials, long-term aqueous storage of bonded specimen and/or subjecting it to thermal cycling may give some information about the degradation of material as it induces stress between a tooth substrate and a restorative material and is an indicator of the aging process of the restoration.

Certain experiments such as the microtensile test (μ TBS), introduced in the early 1990s, are designed to test structural integrity that can be recognized as experiments that seek to simulate the clinical situation.^[1] It has the potential ability to calculate the average tensile stress at the adhesive interface up to 100 MPa.

In the light of these developments, this study has been undertaken which compares μ TBS of an etch and rinse adhesive, with five seventh-generation bonding agents with a nontrimming version.

Materials and Methods

Sixty noncarious extracted human permanent molars were selected for the study. Teeth were handled as per the recommendations of OSHA and CDC and stored in 0.5% Chloramine T solution immediately after extraction at room temperature for not more than 1 month. The teeth were randomly divided into two groups, the control group of 10 teeth and the experimental group with five subgroups of 10

Access this article online	
Quick Response Code: 	Website: www.contempclindent.org
	DOI: 10.4103/0976-237X.101079

teeth each according to the bonding agent used. The crown portions of all the teeth were horizontally sectioned below the central groove to expose the dentin with an Isomet saw (Buehler, USA) of blade size 300 μm at a speed of 125 rpm per minute. The exposed dentin was examined under a microscope (Lieca, Germany) at magnification of 6.3 \times . The specimens with affected caries were discarded. The selected specimens were mounted on an acrylic block after which the sectioned teeth were ground on a silicon carbide grit of 600 size on an Ecomet machine (Buehler, USA) at 100 rpm for 30 s each under a constant flow of water to produce a smear layer. After cleaning with distilled water for 1 min to remove any excess debris, the surfaces were washed and dried with oil-free compressed air.

Samples were grouped and adhesives applied according to manufacturer's instructions [Table 1]. They were light cured with a LED APOZA (440–490 nm, Taiwan). A resin composite of the same manufacturer was used to build the core of 3 mm using a Teflon tape. The composite resin with a layer of 1.5 mm was cured initially for 20 s, and a second layer of

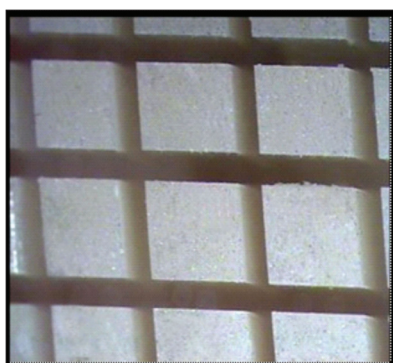


Figure 1: Sectioning done with an isomet saw

approximately the same thickness was placed and cured with LED (APOZA, Taiwan) for 20 s. The Teflon tape was removed, and each side was further cured for 20 s. The teeth were stored in distilled water for 24 h in a humidity chamber.

Microsectioning

Ten teeth from the control Group and each experimental group bonded with the composite resin were sectioned with the Isomet saw (Low speed Buehler, USA) along X, Y, and Z-axis to produce 70–80 specimens of size of 1 \times 1 \times 6 mm³ [Figure 1]. The beams were measured with a vernier caliper to the accuracy of 0.5 μm (Mitutoyo, Japan). Fifty specimens that bonded to dentin were selected from each group. Twenty-five of these specimens were subjected to the μTBS testing using a mini-Instron machine (Chatillon LF plus, UK) without thermocycling. The other twenty-five specimens were subjected to thermocycling and were similarly tested.

After aligning the jaws of the tensile testing machine in a straight line, the specimens were attached to the fixture with antislip property of a mini Instron machine in such a way that the gauge length would not be less than 2 mm [Figure 2]. Tensile load was applied until specimen failure. This load obtained in Newtons was divided by the surface area ($l \times b$) at fracture of specimen, measured with the vernier caliper, to obtain values in Mega Pascals (MPa = Newton/mm²).

Thermocycling was performed for 1000 cycles in a cyclic chamber stored in distilled water maintained at 5° C and 55° C with a dwell time in each bath for 30 s. The transfer time was 5–10 s.^[2]

Results

The results were analyzed by calculating the mean μTBS

Table 1: Fifth- and seventh-generation bonding agents

Bonding agent	Technique	Composition	Composite used	pH
Optibond Solo Plus Control Group Fifth Gen	Etch 15 s on dentin, rinse 15 s, Light brushing motion with applicator gently for 5s Air dry for 15 s, light cure 20 s	BIS-GMA, HEMA, GPDM, ethanol, barium, aluminum borosilicate glass, fumed silica, sodium hexafluorosilicate, camphoroquinone	Premise (nano composite)	1.5
G-bond Group I Seventh Gen	Apply and leave undisturbed for 10 s, dry with air for 5 s, light cure for 10s	4 MET,UDMA, phosphate monomer, DMA component, fumed silica filler, acetone, water, photo initiator	Solare-P (hybrid composite)	1.8
Clearfil S ³ Group II Seventh Gen	Apply for 20 s, air dry, light cure for 10 s	MDP, bis-GMA, HEMA, water, ethanol	APX-Clearfil (nano composite)	2.7
One Coat 7.0 Group III Seven Gen	Apply priming resin for 20 s, dry lightly, light cure for 30 s	UDMA, HEMA, glycerodimethacrylate, polyakenoate methacrylate, and amorphous silica, ethanol	Synergy 6 (nano composite)	1.9
Xeno V Group IV Seventh Gen	Apply two coats with rubbing for 20 s, dry for 5 s and light cure for 20 s	Bifunctional acrylamides, acrylamido alkylsulfonic acid, inverse functional phosphoric acid ester, acrylic acid, butylated benzenediol, water, <i>tert</i> -butanol, camphoroquinone.	Ceram X duo (nano composite)	1.8
Optibond all in one Group V Seventh Gen	Apply two coats with agitation, 20 s each, air dry, light cure for 20 s	Glycerol phosphate dimethacrylate, mono- and di-functional methacrylate esters, water, acetone, ethanol	Premise (nano composite)	2.5

[Figure 3]. The statistical analysis was done using SPSS version 14 (Chicago, IL, USA) to compare the influence of thermal cycling on μ TBS of fifth- and seventh-generation bonding agents. For the initial comparison of the μ TBS of various seventh-generation bonding agents as compared to the fifth-generation bonding agent before and after thermocycling, one-way ANOVA was applied and when there was a significant difference ($P < 0.05$) found among the μ TBS of the bonding agents in ANOVA, then to find where this difference lied, the *post hoc* test was performed using Dunnett's test (for value more than the control group), considering the fifth-generation bonding agent as the control [Table 2].

There was no statistical difference in the μ TBS of the fifth- and seventh-generation adhesives before and after thermocycling, except for the seventh generation group II which showed significantly higher values ($P = 0.016$) of μ TBS after thermocycling (Box Plot 1).

Discussion

The fundamental principle of adhesion to tooth substrate involves removal of calcium phosphates, by which microporosities are created in both enamel and dentin surfaces followed by infiltration and subsequent *in situ* polymerization of resin within the created surface microporosities. Along with micromechanical bonding, the self-etch adhesives show chemical interaction between functional monomers and tooth substrate components which have recently gained attention. Of the three categories of self-etch adhesives, mild ($\text{pH} > 2$), intermediate ($\text{pH} 1.5$), and strong self-etch adhesive ($\text{pH} < 1$), we have selected the mild and intermediate types of self-etch adhesives since they demineralize dentin very shallowly, leaving hydroxyapatite crystals around the collagen fibrils, enabling intimate chemical interaction.

Table 2: Comparison of seventh generation bonding agents with the fifth generation bonding agent before and after thermocycling

Generation	Sample type, n	Mean	SD	P value
Control Group Fifth Gen	btc	40.3580	2.10624	Reference ^a
	atc	36.7244	7.01933	Reference ^b
Group I	btc	34.0132	5.27223	1.000 ^a
	atc	37.4184	8.14161	0.721 ^b
Group II	btc	36.8920	8.84063	1.000 ^a
	atc	42.5896	13.87237	0.016 ^{b,c}
Group III	btc	33.5796	2.65564	1.000 ^a
	atc	33.4384	4.82898	0.997 ^b
Group IV	btc	31.5956	2.43593	1.000 ^a
	atc	31.1476	2.25503	1.000 ^b
Group V	btc	35.2084	3.52148	1.000 ^a
	atc	32.6544	3.99229	0.999 ^b

^aBefore thermocycling (btc): seventh generation as compared to fifth, ^bAfter thermocycling (atc): seventh generation as compared to fifth, ^cSignificant difference observed (P value < 0.05 considered significant), n was 25 in each the categories.

The dentin bond strength was chosen as due to the highly variable morphological and compositional nature of dentin plus the presence of smear layer that makes resin–dentin bonding more difficult to attain compared to enamel.

Optibond solo plus was selected as a control material since the bonding ability of this adhesive to be satisfactory on both enamel and dentin.^[3-5] It has the highest filler loading (26%) among all fifth-generation adhesives for enhanced strength, which favors a stronger bond by acting as a relatively flexible layer that absorbs stresses due to the contraction of the first layer of the composite and eliminates the problem of inhibition by oxygen. It has ethanol as carrier that makes the adhesive less sensitive to the wetness of the acid-etched dentin surface and therefore less technique sensitive.

The results of our study demonstrated higher μ TBS of seventh-generation Group II (*Clearfil S³*) DBA, when compared with fifth-generation DBA, which is in agreement with other studies.^[6,7] In addition, the other experimental seventh-generation DBAs used in this study was comparable to the fifth-generation control group.^[8-11] However, some authors

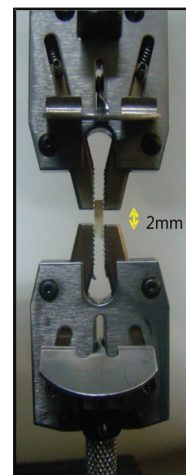


Figure 2: Specimen mounted for microtensile bond strength

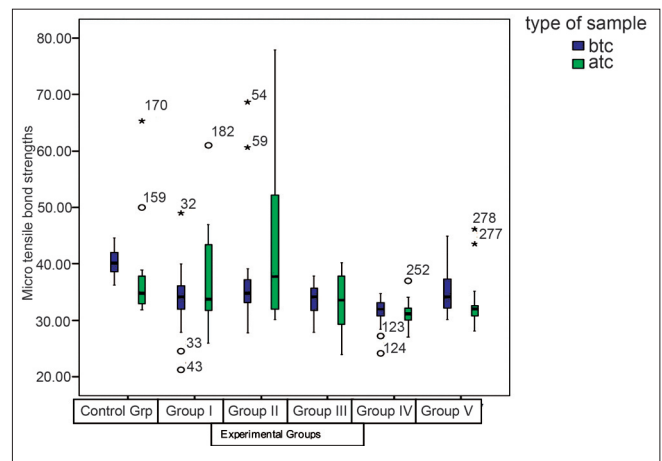


Figure 3: Comparative graph of fifth vs. seventh-generation bonding agents

have stated in their studies that the self-etch adhesives have lower bond strength as compared to total-etch adhesives.^[4,12-14] The reason behind such variations in the results of μ TBS testing may be due to the variations in bonding agents tested and the methodology used.

The higher μ TBS of Group I (*G-bond*) is due to the interface formed by *G-bond*, which is expected to be stronger and more durable as the surface of the dentin is decalcified only slightly and there is almost no exposure of the collagen fibers. The carboxylic group of 4-MET renders *G-bond* monomers hydrophilic, but less reactive than UDMA in hydrogen bonding with water and that functions as proton donors that bond ionically with calcium in hydroxyapatite. Thus, an extremely thin interface nano-interaction zone (300 nm) is formed as opposed to the traditional hybrid layer appellation that provides resistance to acute debonding stresses and better bond durability and survival of adhesion, minimizing voids. *G-Bond's* 5% filler further seals the tubules and decreases pulpal sensitivity. Strong air-blowing of the primed surface as suggested in *G-bond* accelerates the evaporation of solvent – acetone and the resultant water droplets formed due to phase separation. The excess of nonpolymerizable hydrophilic components (water, acetone, and glutaraldehyde) may give rise to hydration forces that repel water at film boundaries, hence less water sorption. Aromatic rings present in *G-bond* are more stable.^[15] A decrease in bond strength of *G-bond* compared to Group II (*Clearfil S³*) might be attributed to blisters in *G-bond* that are associated with the distribution of non-hydrogen-bonded water (5193 cm^{-1}), as *G-Bond* is HEMA-free adhesive. In the absence of this monomer with the high water-dispersion capacity, water in contact with hydrophobic groups creates an entropically unfavorable condition that leads to water separation and arrangement in small droplets contributing to phase separation.

Significantly higher μ TBS of Group II (*Clearfil S³*) is due to the 10-methacryloyloxydecyl dihydrogen phosphate (MDP) adhesive monomer molecular structure that allows for chemical interaction, and the calcium salt of MDP is highly insoluble that provides more intense and stable molecular adhesion to a hydroxyapatite-based substrate. The formulation includes a proprietary “Molecular Dispersion Technology”, enabling a two-phase liquid, (hydrophilic/hydrophobic component) in a homogenous state at the molecular level, reportedly resulting in reduction and/or loss of water droplets at the adhesive interface and therein a superior bond.

Clearfil S³ showed a significant increase in μ TBS after TC which could be explained by the more stable adhesive potential of 10-MDP. In addition, higher μ TBS for Group I (*G-bond*) and Group II (*Clearfil S³*) after TC^[9,16] was due to a river pattern or cleavage appears on the fractured surfaces of the adhesive systems. Microcracks present in these specimens increase the bond strength of the adhesive resins through

crack pinning, crack branching, and plastic deformation of the resin composite called microcrack-induced toughness effect.^[17] However, a decrease in bond strength after TC has also been reported.^[18]

The comparable value for μ TBS of Group III (*One Coat 7.0*) has been attributed to its contents – HEMA, water, and ethanol. HEMA is known to prevent phase separation in addition to solubilizing insoluble monomers in water, improves hydrogen-bonded interaction with collagen fibers exposed by acid-etching thus, enhancing the bonding of resin to the collagen fiber. *One Coat 7.0* and *Clearfil S³* utilize ethanol as the primer component solvent. Ethanol increases the miscibility of the monomer and water. In polar chemistry, solutions with similar solubility parameters (δ) are miscible and can permeate those polymers causing them to swell. The δ_t for ethanol is $26.1 \text{ (J/cm}^3)^{1/2}$ which is near to that of water wet-collagen δ_w $30.6 \text{ (J/cm}^3)^{1/2}$ hence acts as a better wetting agent and a diluent for bis-GMA.^[19] The HEMA–alcohol mixture has been shown to produce high bond strength values to dentin. *One Coat 7.0* and *Clearfil S³* contain nanofillers, fumed silica similar to that used in *G-Bond* which deliver a homogenous bond layer, improve mechanical strength, abrasion resistance, and marginal integrity. Like *G-Bond*, *One Coat 7.0* uses UDMA as a resin monomer; however, the balance of water-acidic monomers and resin monomers in self-etch adhesives is paramount in optimizing bond efficacy to dentin. *One Coat 7.0* has phosphoric acid mono-methacrylate (comparable to MDP) and a methacrylated polyacrylic acid that results in chemical bonding with the tooth surface.^[20]

High μ TBS of Group IV (*Xeno V*) before thermocycling may be due to the chemical composition. *tert*-Butanol as a solvent provides the formulation with a well-balanced polarity. It uses acrylic amide resins instead of acrylic ester resins which are more stable in an aqueous form thus, less prone to hydrolysis by water. *Xeno V* uses inverse functionalized phosphoric acid esters instead of ester-functionalized phosphoric acid esters that have a hydrolysis stable ether bond. The acrylamido alkylsulfonic acid that might account for a very good interprismatic penetration into enamel; however, its penetration is shallow as is with other self-etch adhesives. The bifunctional acrylic amides used as crosslinkers ensure the formation of a dense resin network upon light curing and promote penetration of the bigger crosslinking monomers into the tooth substrate. *Xeno V* after TC showed lower values of μ TBS compared to the control group, but the difference was insignificant. The reason may be a complex process of phase separation that has been shown to occur in one-component, HEMA-free SEAs.^[21]

Group V (*Optibond all in one*) showed comparable values of μ TBS with the control group before thermocycling as it incorporates acetone and ethanol both as solvents and water. Higher μ TBS in *Optibond all in one* may be due to the phosphate (PO_4^{2-}) of a general-purpose DMA (GPDMA)

molecule which promotes chemical bonding to HA in dentin. There was a decrease in μ TBS of *Optibond all in one* observed post-thermocycling but not significantly different from the control group. Acetone-based adhesives because of its low vapor pressure and difference in its solubility parameter from wet-collagen do not infiltrate the demineralized collagen mesh fully and remove all residual solvents, especially water. Moreover, the effect of ethanol and acetone together as a solvent has not been tested as yet. GPDMA increases water sorption. When HEMA is cured in the presence of water, polymerization is incomplete and a porous hydrogel is formed that allow water to permeate through the adhesive layer, compromising long-term bonding effectiveness.^[8,10]

Although there was a decrease in the μ TBS of the seventh-generation DBA after TC (except Groups II and I), the comparative decrease in the μ TBS of the fifth-generation DBA after TC was more. The stronger etching process followed by thermal stress may destabilize the collagen leading to a decrease in bond strength. A low rate of polymerization of bonding resin within the hybrid layer has been shown for one-bottle adhesive, which may also lead to rapid degradation of resin–dentin bonds.

Attaining very high μ TBS is not necessarily an indicator of clinical success. Other parameters, such as chemical interaction with the tooth surface and bond stability over the long-term, may be important for the clinical success of bonded restorations. The results of the bond strength study should thus be complemented with microleakage studies and validated by the findings of *in vivo* trials.

References

- Sano H, Shono T, Sonoda H, Takatsu T, Ciucch B, Carvalho R, *et al.* Relationship between surface area for adhesion and tensile bond strength—evaluation of a micro-tensile bond test. *Dent Mater* 1994;10:236-40.
- Burrow MF, Taniguchi Y, Nikaido T, Satoh M, Inai N, Tagami J, *et al.* Influence of temperature and relative humidity on early bond strengths to dentin. *J Dent* 1995;23:41-5.
- Toledano M, Osorio R, Ceballos L, Fuentes MV, Fernandes CA, Tay FR, *et al.* Microtensile bond strength of several adhesive systems to different depths. *Am J Dent* 2003;16:292-8.
- Kerby RE, Knobloch LA, Clelland N, Lilley H, Seghi R. Microtensile bond strengths of one-step and self-etching adhesive systems. *Oper Dent* 2005;30:195-200.
- Braga RR, Cesar PF, Gonzaga CC. Tensile bond strength of filled and unfilled adhesives to dentin. *Am J Dent* 2000;20:73-6.
- Kiremitci A, Yalcin F, Gokalp S. Effectiveness of three dentin adhesive systems on adhesion of resin composite to both dentin and ground enamel. *Quint Int* 2004;35:367-70.
- Van Meerbeek B, Vargas S, Inoue S, Yoshida Y, Peumans M, Lambrechts P, *et al.* Adhesives and cements to promote preservation dentistry. *Oper Dent* 2001;26:S119-44.
- Brackett WW, Tay FR, Looney SW, Ito S, Haisch LD, Pashley DH. Microtensile dentin and enamel bond strengths of recent self-etching resins. *Oper Dent* 2008;33:89-95.
- Asaka Y, Amano S, Rikuta A, Kurokawa H, Miyazaki M, Platt JA, *et al.* Influence of thermal cycling on dentin bond strengths of single-step self-etch adhesive systems. *Oper Dent* 2007;32:73-8.
- Van Landuyt KL, Mine A, De Munck J, Jaecques S, Peumans M, Lambrechts P, *et al.* Are one-step adhesives easier to use and better performing? Multifactorial assessment of contemporary one-step self-etching adhesives. *J Adhes Dent* 2009;11:175-90.
- Sensi LG, Lopes GC, Monteiro S, Baratieri Jr LN, Vieira LC. Dentin bond strength of self etching primers/adhesives. *Oper Dent* 2005;30:63.
- McLean JW. Some physical properties of a new cross—linked plastic filling material. *Br Dent J* 1961;110:375-8.
- Inoue S, Vargas MA, Abe Y, Yoshida Y, Lambrechts P, Vanherle G, *et al.* Microtensile bond strength of 11 contemporary adhesives to enamel. *Am J Dent* 2003;16:329-34.
- Chaung S, Chang L, Chang C, Yemen P, Liu J. Influence of enamel wetness on composite restorations using various dentin bonding agents: Part II effects on shear bond strengths. *J Dent* 2006;34:352-61.
- Boullaguet S, Gyst P, Wataha JC, Ciucch B, Cattani M, Godin C, *et al.* Bond strength of composite to dentin using conventional, one-step, and self-etching adhesive systems. *J Dent* 2001;29:55-61.
- Van Meerbeek B, De Munck J, Yoshida Y, Inoue S, Vargas M, Vijay P, *et al.* Adhesion to enamel and dentin: Current status and future challenges. *Oper Dent* 2003;28:215-35.
- Burrow MF, Nopnakeepong U, Phrukkanon S. A comparison of microtensile bond strength of several dentin bonding systems to primary or permanent dentin. *Dent Mater* 2002;18:239-45.
- Loughran GM, Versluis A, Doulas WH. Evaluation of subcritical fatigue crack propagation in a restoration with composite. *Dent Mater* 2005;21:252-61.
- Ikeda M, Tsubota K, Takamizawa T, Yoshida T, Miyazaki M, Platt JA. Bonding durability of single step adhesives to previously etched dentin. *Oper Dent* 2008;33:702-9.
- Nishiyama N, Suzuki K, Komatsu K, Yasuda S, Nemoto K. A 13C NMR study on the adsorption characteristics of HEMA to dentinal collagen. *J Dent Res* 2002;81:469-71.
- Latta (unpublished data) 2007 Scientific compendium-Xeno V, Dentsply, De tray, Germany.

How to cite this article: Poptani B, Gohil K, Ganjiwale J, Shukla M. Microtensile dentin bond strength of fifth with five seventh-generation dentin bonding agents after thermocycling: An *in vitro* study. *Contemp Clin Dent* 2012;3:S167-71.

Source of Support: The Indian Space Research Organization, Ahmedabad – providing technical assistance and necessary support. Central Institute of Plastic Engineering and Technology, Ahmedabad where the thermocycling was performed.. **Conflict of Interest:** None declared.