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Evaluation of microtensile bond strength of nanoclay modified adhesive systems using bulk fill resin composite

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***Abstract*---**Background: Composite polymerization creates internal stresses that cause bond loss at the tooth composite interface, cuspal deflection, and enamel crack formation; that are potential restoration failure. Aims: The aim of the present study was to evaluate micro-tensile bond strength of bulk-fill resin composite to human dentine of extracted teeth using nanoclay modified adhesive system (with and without organic modifier). Materials and Methods: A total of 120 caries-free permanent human posterior teeth were used and classified three groups according to adhesive system (each with 40 teeth);

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including Prime&Bond universal™, Dellite®43B and Dellite®LVF modified adhesive systems. Each group was subdivided into two equal subgroups whether thermocycling was conducted or not. Results: Result revealed that there was a significant difference in mean microtensile bond strength of the three tested materials The best results achieved by Dellite®43B followed by Dellite®LVF modified adhesive system finally conventional Prime&Bond universal adhesive system. Difference in Microtensile bond strength between thermocycled and Nonthermocycled was highly significant. Conclusion: Addition of Nanoclay (montmorillonite) with organic modifier to universal adhesive material is valuable to augment the bond strength of bulk fill resin composite to human dentition. (2) Thermocycling adversely affect the bond strength of nanoclay /adhesive system complex to tooth tissue.

***Keywords*---**Montmorillonite, Bulk fill, Dellite® 43B, Dellite® LVF modified Adhesive system, clay.

Introduction

Composite polymerization creates internal stresses that cause bond loss at the tooth composite interface, cuspal deflection, and enamel crack formation; all are principal factors in the potential restoration failure [1] (1). Limitation in the depth of cure causes incomplete polymerization with the possibility of insufficient monomer conversion resulting in inferior physical and biological properties of resin composites [2] (2). As a result, numerous studies have been conducted to reduce the amount of polymerization stress created and increase the depth of cure.

Bulk-fill composites were developed to address some of the flaws in light cure resin composites. Because of their low polymerization stress and strong responsiveness to light curing, they are appropriate for insertion in a 4mm bulk placement or larger. Various photo-initiators that are more transparent and enable light to penetrate through considerably deeper layers account for the increased depth of cure of these materials [3]**.**Low polymerization stresses in bulk-fill composites are related to changes in the organic matrix's filler content and the occurrence of stress inhibitors [4]**.** Compared to traditional resin composites filled in an oblique incremental layering manner [5], they showed less cuspal deflection, and when marginal integrity was tested, bulk-fill composites fared well [6].

When a tiny quantity of montmorillonite (nanoclay) is added to the polymer matrix, the resultant composite's mechanical, physical, and chemical characteristics are significantly improved over traditional materials [7]. The mechanical performance (stiffness and strength) was improved significantly by adding a minor quantity of nanoclay (0.5–5.0wt %) to the polymers [8–10].

Materials and Methods

The following materials were used in this study:

a) Bulk fill resin composite (SDR the unmatched posterior bulk fill material with flowable consistency).
b) 36% phosphoric acid etch conditioner.
c) Prime & Bond universal™ dental adhesive.
d) Modifying materials (two types of montmorillonites):
   - Dellite® 43B (with organic modifier)
   - Dellite® LVF (without organic modifier)

Specimen selection:

After approval of the research ethics committee faculty of dentistry Suez Canal University, this study was carried on a total of 120 caries-free permanent human molar teeth as follow:

a) Teeth were carefully examined for the presence of any carious lesions, cracks or hypoplastic defects; if any were found, they were discarded.
b) The selected teeth were thoroughly washed under running water to remove the blood and were scaled using an ultrasonic scaler to remove any plaque, calculus and soft tissue remnants.
c) The teeth were stored in distilled water at 25ºC, and the water was changed every week.

Specimens grouping

According to the type of adhesive system used, the selected teeth were classified into three main groups (40 teeth each). In the first group, the Prime&Bond universal™ dental adhesive system was used (control group A1).In the second group, Dellite® 43B modified adhesive system was utilized (A2).While in the third group Dellite® LVF modified adhesive system was utilized (A3). Each group was subdivided into two equal subgroups B0, B1 according to whether thermocycling was conducted or not (20 teeth each). [11].

Table (1)
Variables of study

Variable	Symbol	Denote
Adhesive System (A)	A_1	Prime &Bond universal™ dental adhesive system (Control Group)
	A_2	Dellite®43B modified adhesive system
	A_3	Dellite®LVF modified adhesive system
Thermocycling (B)	B_0	Thermocycling was not done
	B_1	Thermocycling was done

Table (2):
Interaction of variables: n=20

		(A) Group-A (Adhesive system)			
		A_1	A_2	A_3	Total
Group-B (Thermo cycling)	B_0	A_1B_0	A_2B_0	A_3B_0	60
	B_1	A_1B_1	A_2B_1	A_3B_1	60
	Total	40	40	40	120

Embedding of teeth in acrylic resin blocks:

The selected teeth were embedded in a rectangular auto-polymerized acrylic resin block (Acrostone, Egypt). The dimensions of the block were (2.5 cm 2.0 cm 1.5 cm). The blocks were prepared using a split mold assembly that allows for the pouring of six blocks simultaneously**.**

Each tooth was embedded in the acrylic while it was in the soft dough stage, and the tooth was pressed in the acrylic till cementoenamel junction. After acrylic setting, the block was removed from the mold and checked carefully

Specimens preparation

The extracted caries-free human posterior teeth were preserved in distilled water. The crowns were polished with abrasive fluoride-free toothpaste with rotating brushes. Standardized class I cavities were prepared on the occlusal surface with 4mm mesiodistal width, 4mm buccolingual and 1.5mm depth using the digital caliper.

Each tooth was stabilized firstly by vice tool (a mechanical apparatus used to secure an object to allow work to be performed on it) to ensure standardization of the prepared cavities. Cylindrical fissure carbide bur (#H21, 204, size 010) revolving at the high-speed range was used under copious air-water spray coolant. The depth was adjusted by using stopper on the bur. Each cavity was measured with a William's graduated probe to ensure uniform size). Application of Adhesive system materials was subjected to thermo-cycling or not according to the grouping of specimens.

Restorative procedure:

The prepared cavities were air-water sprayed and then dried with an air syringe. The etchant gel was applied to the prepared enamel for 15-30 seconds and then applied to the prepared dentin for 10-15 seconds. All the etchant gel was rinsed with a vigorous water spray for at least 5 seconds. The different types of adhesive bonding agents were used to restore the prepared cavities, where the applicator brush was applied to enamel and dentin with a generous amount of adhesive bonding agent and agitated for at least 10 seconds. The adhesive layer was thinned with a weak stream of air to avoid its pooling and light-cured for 10 seconds at a light intensity of more than 500 $mW/cm^2$with the same curing unit. According to the manufacturer's instructions, the resin composite was packed by oblique incremental technique, so the maximum thickness of each increment was 4 mm & ended with feather edge at the occlusal surface, then light-cured. According to the manufactures instruction, the restorations were finished using diamond stone with small grit at high speed (100,000 rpm) after setting of material. The specimens were stored in an incubator at 37oC for one h after restoration to simulate the normal oral environment.

Thermocycling procedures:

Once the restorative procedures were completed, the specimen were randomly divided into two groups (n=60) according to whether thermocycling was conducted or not; the thermocycled group was divided into three subgroups according to the type of the bonding system (n=20). The thermal cycle procedure was 5000 cycles using Huber TC45 machine (SD Mechanotronik, thermocycler) (Model THE-1100). Each thermal cycling procedure was conducted between 5 and 55 ° C, whereas the dwell time was the 30s**.**This procedure was done at MSA University. [12].

Specimen preparation for the microtensile bond strength test

All teeth in different groups were examined to determine microtensile bond strength using the universal testing machine. Composite were sectioned using a diamond disc (Microdont, Sao Paulo, Brazil, n. 34570) at low-speed underwater cooling in a sectioning machine. Initially, the composite restoration was fixated with cyanoacrylate adhesive gel (Super Bonder Gel, Loctite Ltd., Sao Paulo, Brazil) on a metallic ˜ base attached to the sectioning machine. The specimen was positioned as perpendicular as possible in relation to the diamond disc of the machine. The first section, measuring approximately 1mm, was discarded if the possibility of excess or absence of composite at the interface might alter the results. This process was followed for the other two sections, and thus only the central specimens were used for the experiments. Six stick specimens (sp) were obtained from each block. The specimen had non-machined (non-trimmed) adhesive zones with bonded areas measuring approximately 1mm 2 and 10mm in length. The specimens were stored in water at 37 ◦C for 24 h before tests**.** [13] (13) Each sp was fixated with cyanoacrylate gel (Super Bonder Gel, Loctite Ltd., Sao Paulo, Brazil), keeping the adhesive zone free, ˜ to the rods of a device adapted for this test. The specimens were positioned parallel to the long axis of the device to reduce the bending stresses. The device was fixated in the universal testing machine (EMIC DL-1000, EMIC, Sao Jos˜edos´Pinhais, Brazil), as parallel as possible in relation to applying the tensile load, and testing was performed at a cross speed of 0.5mm/min−1. The bond strength was calculated according to the formula R = F/A, where "R" is the strength (MPa), "F" the load required for rupture of the specimen and "A" is the interface area of the specimen (mm^2), measured with a digital caliper before the test**.** [14]**.**

Results

Microtensile bond strength (MPa)

Current study was be conducted to evaluate microtensile bond strength of bulkfill resin composite to dentin using nanoclay modified adhesive system (with organic modifier and without organic modifier).

Adhesive systems / thermocycling:

The microtensile bond strength was measured at different treatment groups and subgroups. Microtensile bond strength in Control Prime & Bond universal; Dellite-43 modified, and Dellite-LVF modified in both non-thermocycled and thermocycled groups were presented in (Tables 3,4).

The mean value for microtensile bond strength for Control Prime &Bond universal non-thermocycled (B0) specimens was 6.47±0.94 MPa (±SE). However, in thermocycled (B1) it was 12.77±1.35 MPa (Tables 3,4). Where, the difference between non-thermocycled and thermocycled was significant ($p<0.001$***), as revealed by independent samples t-test.

The mean value of microtensile bond strength in Dellite-43 modified in non-thermocycled (B0) specimens was 6.36±1.73 MPa and in thermocycled (B1)was 15.91±0.96 MPa (Tables 3,4). Where, the difference between non-thermocycled and thermocycled in microtensile bond strength was significant ($p<0.001$***), as revealed by independent samples t-test.

Furthermore, the mean value of microtensile bond strength in Dellite-LVF modified in non-thermocycled specimens was 5.40±0.84, however, in thermocycled was 11.58±1.64 MPa (Tables 3,4). Where, the difference between non-thermocycled and thermocycled was significant ($p<0.001$***), as revealed by independent samples t-test.

The differences in Microtensile bond strength between treatment groups and subgroups were assessed by Two-Way ANOVA at 0.05 level followed by Duncan's Multiple Range Test (DMRTs). The overall difference (corrected model), difference between adhesive systems, thermocycling and interaction between adhesive systems and thermocycling were highly significant difference as revealed by ANOVA.

Table (3)
Effect of different adhesive system (Control Prime &Bond universal; Dellite-43 modified, and Dellite-LVF modified) and thermocycling on Microtensile bond strength

Descriptive statics		Non-Thermocycled	Thermocycled	T-test	
		B0	B1	t-	p-value
Control (Prime&Bond universal)	A_1	6.47 ± 0.94d	12.77 ± 1.35b	-17.11	<0.001***

Dellite 43 modified	A_2	6.36 ± 1.73d	15.91 ± 0.96a	-21.55	<0.001***
Dellite LVF modified	A_3	5.40 ± 0.84e	11.58 ± 1.64c	-14.96	<0.001***
Total (average)		6.08 ± 1.17	13.42 ± 1.32	-21.71	<0.001***
ANOVA (1-way)	F-ratio	4.55	55.19		
	p-value	0.015*	<0.001***		
ANOVA (2-Way)	Df	F-ratio	*p*-value		
Corrected Model	5	218.7	<0.001***		
Intercept	1	6815.4	<0.001***		
Adhesive system (A)	2	42.1	<0.001***		
Thermocycling (B)	1	965.7	<0.001***		
Adhesive system x thermocycling	2	21.8	<0.001***		

* Significant at p<0.05; ** highly significant at p<0.01; *** very high significant at p<0.001

Table (4)
Descriptive statistic for Microtensile bond strength at different adhesive system and thermocycling treatments, (B0= Non-thermocycled; B1= thermocycled)

Descriptive statics	B	Mean	SD	SE	Min.	Max.
A1: Control (Prime & Bond universal)	B0	6.475	0.940	0.210	4.850	8.110
	B1	12.766	1.349	0.302	10.250	14.830
A2: Dellite 43 modified	B0	6.364	1.735	0.388	3.520	9.480
	B1	15.908	0.955	0.214	14.520	17.800
A3: Dellite LVF modified	B0	5.400	0.845	0.189	4.070	6.980
	B1	11.582	1.644	0.368	9.080	14.640

Thermocycling in Control Prime &Bond universal

The mean value of microtensile bond strength in Control Prime & Bond universal non-thermocycled specimens (±SE) of 6.47±0.21 MPa. However, in thermocycled it was (+SE) of 12.77±0.30 MPa.
The difference between non-thermocycled and thermocycled control Prime was highly significant revealed by independent samples t-test (t=-17.11; p<0.001***).

Thermocycling in Dellite-43 modified

The mean value of microtensile bond strength in Dellite-43 modified in non-thermocycled was 6.08±0.17 MPa, however, in thermocycled it was 15.91±0.21 MPa. The difference between thermocycled and Non-thermocycled Dellite-43 modified were highly significant revealed by independent samples t-test (t-statistic=-21.55; p<0.001***)

Thermocycling in Dellite-LVF modified

The mean microtensile bond strength in non-thermocycled Dellite-LVF modified groups 5.40±0.19, however the thermocycled was 11.58±0.37**.** The difference between non-thermocycled and thermocycled Dellite-LVF were highly significant revealed by independent samples t-test ($t=-14.96$; $p<0.001$***).

Microtensile bond strength at different adhesive system

Figure (1) represents the microtensile bond strength in thermocycled, non-thermocycled and both in the three tested adhesive system.
The overall mean value for microtensile bond strength in all groups either non-thermocycled or thermocycled was 9.75 ± 4.12 MPa. Furthermore, the overall mean value of microtensile bond strength or Control Prime &Bond universal, Dellite 43 modified, and Dellite LVF modified was 9.62±3.39, 11.14±5.03, and 8.49±3.39 MPa (±SD); respectively.
The overall difference between non-thermocycled and thermocycled was significant ($p<0.001$***), as revealed by independent samples t-test. Moreover, the overall differences between groups adhesive systems were significant ($p=0.015$*).

Discussion

Continuous development of materials provides a wide range of new dental materials with improved mechanical and physical properties and biological compatibility for various clinical applications. However, despite these advances, the need still exists for biomaterials demonstrating high biocompatibility, antimicrobial effects and ideal mechanical properties [15]. The purpose of this study was to evaluate Microtensile bond strength of bulk fill resin composite to human dentine of extracted teeth using nanoclay modified adhesive system (with and without organic modifier) and to assess the pulp response in deep dentine cavities in permanent posterior teeth of dogs.

Resin-based composite (RBC) materials are increasingly being used for the restoration of posterior teeth. The increasing demand for aesthetic, tooth-colored restorations coupled with the patient's concerns regarding the use of mercury containing restorations, has driven a surge in the use of RBC materials. In order to simplify and speed-up the placement of large posterior RBCs, manufacturers have produced a range of materials which can be placed in single or deeper increments, known as bulk-fill RBCs. Over a relatively short period of time many bulk-fill RBCs have been marketed quoting increment depths between 4-10 mm. The placement of these larger increments of RBC may reduce the time needed when placing posterior restorations and thereby reduce technique sensitivity [16].

Bulk fill composites have been introduced to facilitate the easier placement of direct resin composite in deeper cavities, Bulk-fill composites exhibited adequate bond strength to dentin and can be considered as restorative material of choice in posterior stress bearing areas [17].

The use of nanoparticles has become a significant area of research in dentistry. Nanoparticles are widely used as reinforcing fillers in restorative materials, since they have a strong impact on properties when incorporated in smaller amounts in comparison with macro/micro fillers. Besides this, because of their small sizes, nanoparticles are able to penetrate into the tooth micropores, providing a bond layer with improved properties [18].

Nanomaterials have engaged response from the scientific world in recent decades due to their exceptional physical and chemical properties counter to their bulk. They have been widely used in a polymer matrix to improve mechanical, thermal, barrier, electronic and chemical properties. In rubber nanocomposites, nanofillers dispersion and the interfacial adhesion between polymer and fillers influences the composites factual properties [19].

Nano-montmorillonite (bentonite) is the most common member of smectite clay family usually referred as "nanoclay". Nanoclay consists of layer made of an inner octahedral sheet of alumina or magnesia sandwiched between two tetrahedral sheets of silica. Organo-clay produced by treatment of clay minerals such as bentonite and montmorillonite with organics have a lot of applications in many fields due to their specific active sites and attractive adsorptive properties. On the other hand, the study of Na montmorilloniteclay modification by using different types of alkylammonium salts indicated that surface modification has impact on interlayer space between the silicate layers as well as on cation exchange capacity of the clay [20]**.** This was with agreement with many studies that found that Incorporation of the modified nanoclay provided a dentin bonding system with higher shear bond strength. The dispersion stability of the modified nanoparticles in the dilute adhesive was also increased more than 40 times in comparison with the pristine nanoclay [21].

Nano-sized montmorillonite has been successfully used as a drug carrier system solubility enhancer with cyclodextrin use as catalyst inorganic synthesis, food additive for prevention of aflatoxicosis, fabrication of scaffold for biomedical use, as adsorbent for non-ionic, anionic, cationic dyes and metal ions antibacterial activity in dental infection and in food packaging material, [22] and to increase its poor mechanical stability of hydrogels used for wound dressings [23]. Therefore, bulk fill composite resins modified with montmorillonite nanoparticles adhesive were chosen to be investigated in this study.

Microtensile bond strength is an important mechanical property since the bond strength between restorative material and tooth structure is so important to reach a durable restoration. Considering the recent attention to low shrinkage composite resins, evaluation of micro tensile bond strength of these materials would be valuable [24]**.**

Microtensile bond strength (μTBS) test was introduced in 1994. Since then, it has been utilized profoundly across many bond strength testing laboratories, making it currently one of the most standard and versatile bond strength test. Although it is a static and strength-based method, together with the morphological and spectroscopic investigations, it has been contributing immensely in the advancement of dentin adhesive systems. μTBS test has a greater discriminative capability than the traditional macro-shear bond test [25]. In the present study the microtensile bond strength of bulk fill resin composite to human dentin without thermocycling was higher than when it was used [26].

In-vitro models are essential for providing an insight into the mechanisms of biodegradation due to the complexity of intra-oral conditions [27] for this reason Standardized class I cavities were prepared on occlusal surface of the extracted caries-free human molar teeth with 4mm mesiodistal width, 4mm buccolingual and 1.5mm depth using the digital caliper. Cylindrical fissure carbide bur (#H21, 204, size 010) revolving at high-speed range was used under copious air water spray coolant. Therefore, selecting an appropriate bur and its speed may improve bonding for adhesive systems, although crosscut fissure carbide burs produced high bond strengths at either speed used [28]. The depth was adjusted by using stopper on the bur. Each cavity was measured with a William's graduated probe to ensure uniform size). The prepared cavities were air –water sprayed and then dried with air syringe. The different types of adhesive bonding agents were used for restoration of the prepared cavities. According to the manufacture's instruction, the restorations were finished using diamond stone with small grit at high speed (100,000 rpm) after setting of material to obtain a restoration that has good contour, occlusion, healthy embrasure forms and a smooth surface [29]. The specimens were stored in an incubator at 37oC for 1 h after restoration to simulate the normal oral environment [13].
Regarding this study, the effect of bulk-filling on the Bonding efficacy in Occlusal Class I Cavities was evaluated, so they found that bulk-fill composite has a great impact on bonding to cavity-bottom dentin [30]. Bulk-fill composites exhibited adequate bond strength to dentin and can be considered as restorative material of choice in posterior stress bearing areas. [31].

Once the restorative procedures were completed the specimen were randomly divided into 3 groups (n=40) according to the type of the bonding system and each group was divided into 2 subgroups (n=20) according to whether thermocycling was conducted or not. The thermal cycle procedure was 5000 cycles. Each thermal cycling procedure was conducted between 5 and 55 ° C, whereas the dwell time was 30s, this study found that the interaction effect revealed significant influence of thermocycling (TC) aging [12].

Regarding this study, Thermocycling the composite substrates resulted in the lowest repair bond strength. This was with agreement with different study [14]. According to the results of the study, Microtensile bond strength ranged in all examined adhesive systems (Control Prime & Bond universal; Dellite-43 modified, and Dellite-LVF modified) show better results in non-thermocycling than thermocycling, this reveals a highly significant difference in Microtensile bond strength between non-thermocycled and thermocycled. This was with agreement with [26] which found that the adhesive tensile strength showed a reduction with the addition of nanoclay at elevated temperatures.

This study was in disagreement with that study which found that the thermocycling did not enhance chemical or mechanical degradation of the bonds produced by a two-step self-etch and a three-step etch and rinse adhesive to dentin. The one-step self-etch adhesive tested was not able to withstand polymerization shrinkage stress, nor thermo-cycling, when applied in class-I cavities [32].

Resin composite with alkaline fillers was characterized by good μTBS and marginal integrity with weak antibacterial effect [33]. That is why in this study, Microtensile bond strength of bulk fill resin composite to dentine using nanoclay modified adhesive system (with organic modifier and without organic modifier) was evaluated. Meanwhile the pulp responses in deep dentine cavities were assessed.
And this was with disagreement with that study which found that Silorane-based resin composite as opposed to methacrylate-based resin composite and layering placements in contrast to bulk filling method had higher Microtensile bond strength. [34].

Our study was with agreement with these studies as they found that the bond strength values decreased with the thermomechanical cycling. Bond strength of bulk-fill composite was negatively influenced by the depth of dentin and thermomechanical loading [35]. Thermocycling significantly reduced the cohesive strength of the composite resins up to 65 % [36]**.**

The search for dental materials that are biocompatible with the dentin–pulp complex, in addition to having adequate physical, mechanical and esthetic properties, has guided the development of several commercial products. Most of these dental materials have to contact or interact with connective tissue and fluids, in such way that the selection of a new material must consider not only the mechanical and physical properties but also its biological compatibility. Failure of application of an experimental material occurs when it triggers a specific inflammatory reaction resulting in damage to the body tissue. Consequently, the biocompatibility of experimental dental materials as well as their adequate physical and mechanical properties are requirements necessary to recommend them for clinical application [37]**.**

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List of Figures

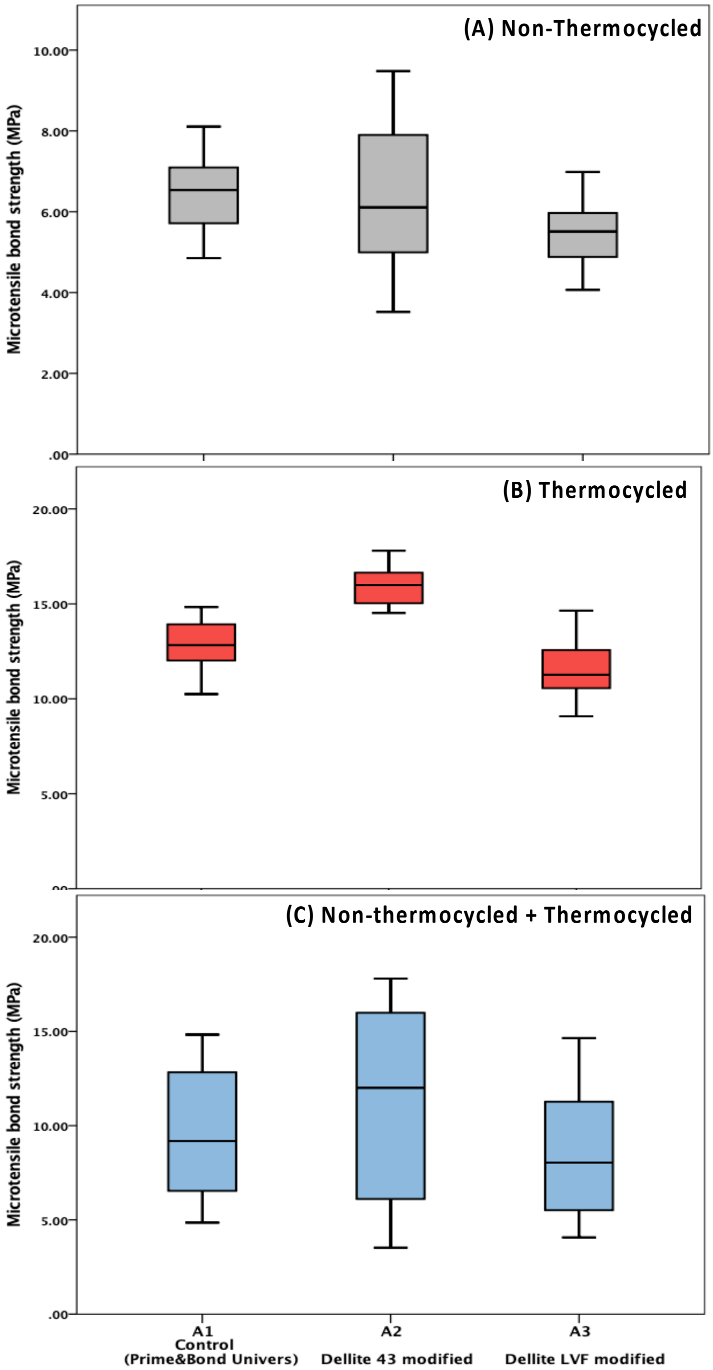

Figure 1: Box plot presenting the Microtensile bond strength at different group and thermocycling