

# In vitro evaluation of shear bond strength of polymethyl methacrylate/montmorillonite modified Biodentine with dental resin composite

## Purpose

The aim of this study was to evaluate the bond strength between Biodentine, modified with polymethyl methacrylate/Montmorillonite nanoclay, and resin composite at different stages of Biodentine's setting time.

## Materials and Methods

Nanoclay was prepared and organo-modified with polymethyl methacrylate. The characterization of polymethyl methacrylate/Montmorillonite nanoclay, Biodentine, and modified Biodentine was assessed by X-ray diffraction analysis, Fourier-transform infrared spectroscopy, and scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy. A total of sixty acrylic molds were constructed; thirty specimens were filled with Biodentine, and the other thirty with nanoclay-modified Biodentine. Each group was subdivided according to different stages of Biodentine's setting time: 12 minutes, 2 hours, and 2 weeks. Universal adhesive, followed by flowable resin composite, was applied. The micro-shear bond strength was tested using a universal testing machine. Data were analyzed using one-way ANOVA followed by Tukey's post hoc test, in addition to two-way ANOVA. The significance level was set at  $p \leq 0.05$ .

## Results

The characterization results revealed the successful preparation of polymethyl methacrylate/Montmorillonite nanoclay and modified Biodentine. The micro-shear bond strength results showed that modified Biodentine had significantly higher micro-shear bond strength than unmodified Biodentine at 12 minutes. However, no statistically significant difference was found between the unmodified and modified Biodentine groups at 2 hours and 2 weeks.

## Conclusion




The incorporation of 10% modified nanoclay by weight into Biodentine could enhance the bond strength with resin composite when placed after 12 minutes of Biodentine's setting time.

**Keywords:** Biodentine, montmorillonite nanoclay, resin-modified nanoclay, micro-shear bond strength, pulp capping materials

## Introduction

Pulp capping materials have been used for pulp protection against chemical, thermal, and other noxious stimuli. They are placed as a protective layer on the floor of deep cavities or after traumatic exposure. These biomaterials should be bioactive to allow for the regeneration of dentin at the exposed pulp areas.

Calcium hydroxide ( $\text{Ca}(\text{OH})_2$ ) had been regarded as the gold standard for pulp capping due to its high pH which leads to stimulation of the pulp cells to form dentin bridge and its antibacterial effect. However,  $\text{Ca}(\text{OH})_2$  showed

Fagr Hassan Elmergawy<sup>1</sup> ,  
Ola M. Elborady<sup>2</sup> ,  
Dina M. Wahied<sup>1</sup> 

ORCID IDs of the authors: F.H.E. 0000-0003-1514-4851;  
O.M.E. 0000-0002-4168-5697; D.M.W. 0000-0002-8006-6901

<sup>1</sup>Dental biomaterials Department, Faculty of Dentistry,  
October University for Modern Sciences and Arts

<sup>2</sup>Institute of Nanoscience and Nanotechnology,  
Kafr El-Sheikh University, Qism Kafr El-Sheikh

Corresponding Author: Fagr Hassan Elmergawy

E-mail: fmergawy@msa.edu.eg

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high solubility, poor sealing ability, lack of adhesion with tooth structure, tunnel defects in the formed dentin bridge as well as necrosis and inflammation in pulp tissues. To overcome these drawbacks calcium-silicate based pulp capping materials have been introduced (1, 2). Mineral tri-oxide aggregate (MTA) is the first calcium silicate bioactive material introduced in 1993 (3). MTA is capable of apatite formation by either using calcium aluminates or calcium silicates. MTA exhibits a higher rate of clinical success compared to  $\text{Ca}(\text{OH})_2$ , owing to the formation of thicker and less porous dentin bridge with fewer signs of inflammation. Yet, MTA shows some disadvantages such as its long setting time, difficulty in handling, tooth discoloration, high cost as well as incompatibility with other dental materials when layered. (1, 3, 4)

Second generation calcium silicate based materials were introduced in an attempt to overcome the drawbacks of MTA, among which is the Biodentine. (4) Biodentine is a repair material that was introduced in 2011 as a bioactive dentin substitute due to its resemblance to dentin regarding its mechanical properties. Biodentine is composed of tri-calcium and di-calcium silicates, calcium carbonate, in addition to iron oxide and zirconium oxide (5). Biodentine has an accelerated setting time and higher viscosity, in addition to its easier manipulation, and less tooth discoloration than MTA. Moreover, it shows very promising bioactivity which comes mainly from being formed of tri-calcium silicate and calcium carbonate matrix along with zirconium oxide and iron oxide (6, 7). Owing to its promising properties, it has not been used only as a pulp capping material but also used as a retrograde filling, perforation repair, treatment of immature necrotic teeth, pulpotomy, and apexification (7, 8). However, one of the main drawbacks of Biodentine is the poor bond strength with the overlying resin composite restoration due to its water based chemistry which affects the micromechanical retention with overlying resin composite, impairing the longevity of the final restoration (3).

Nanoclays are promising nanoparticles that attracted many researchers due to their biocompatibility, good mechanical properties as well as high abundance (9). Montmorillonite (MMT) is a type of nanoclay that is formed of aluminosilicate sheets of approximately one nanometer thickness that are stacked over one another forming complex crystallites (10). Organo-modification of nanoclays involves the grafting of bulkier organic polymers onto MMT sheets to increase its compatibility with resin based materials.

To our knowledge, no previous studies have been done to modify Biodentine with resin or nanoclay in an attempt to increase bond strength with resin composite. Thereby, the aim of this study was the addition of polymethyl methacrylate modified MMT nanoclay to Biodentine and to evaluate its effect on shear bond strength with resin composite restoration over different stages of Biodentine setting time. The null hypothesis states that there would be no statistically significant effect of adding organo-modified nanoclay on shear bond strength between Biodentine and overlaid composite at different stages of setting time.

## Materials and Methods

### Materials

Materials used for the synthesis of MMT modified with polymethyl methacrylate (PMMA/MMT), as well as Bioden-

tine, universal adhesive system and flowable resin composite are listed in Table 1.

**Table 1.** Materials used in this study, their chemical composition and manufacturer.

Material	Chemical composition	Manufacturer
Tetraethyl orthosilicate (TEOS)	$(\text{C}_2\text{H}_5\text{O})_4\text{Si}$	Alpha Chemika (India)
Aluminum nitrate	$\text{Al}(\text{NO}_3)_3$	Alpha Chemika (India)
Magnesium nitrate	$\text{Mg}(\text{NO}_3)_2$	Alpha Chemika (India)
Calcium nitrate	$\text{Ca}(\text{NO}_3)_2$	Sigma-Aldrich (Germany)
Sodium nitrate	$\text{NaNO}_3$	LOBA Chemie (India)
Acrylamido-methyl Propane sulfonic acid (AMPS)	$\text{C}_7\text{H}_{13}\text{NO}_4\text{S}$	Acros organics (USA)
Ammonium persulfate (APS)	$(\text{NH}_4)_2\text{S}_2\text{O}_8$	LOBA Chemie (India)
Methyl methacrylate	$\text{C}_4\text{H}_6\text{O}_2$	LOBA Chemie (India)
Carbon tetrachloride	$\text{CCl}_4$	Acros organics (USA)
Ethanol	$\text{C}_2\text{H}_5\text{OH}$	LOBA Chemie (India)
Methanol	$\text{CH}_3\text{OH}$	LOBA Chemie (India)
Biodentine	Tri-calcium silicate, di-calcium silicate, calcium carbonate, iron oxide and zirconium oxide	Septodont (France)
All bond universal adhesive	Bis-GMA, 10-MDP, HEMA, ethanol and water	Bisco (USA)
Flowable resin composite	Bis-GMA and TEGDMA Methacrylate-based nanohybrid resin composite with 76wt% filler loading	VOCO GmbH. (Germany)

### PMMA/MMT nanoclay preparation

PMMA/MMT was prepared following a method mentioned in a previous study (10), where TEOS,  $\text{Al}(\text{NO}_3)_3$ ,  $\text{Mg}(\text{NO}_3)_2$ ,  $\text{Ca}(\text{NO}_3)_2$ , and  $\text{NaNO}_3$  were used as sources of  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{MgO}$ ,  $\text{CaO}$ , and  $\text{Na}_2\text{O}$  respectively. MMT nanoclay was first synthesized from sol-gel technique, where a solution of 57%  $\text{SiO}_2$ , 33%  $\text{Al}_2\text{O}_3$ , 5.4%  $\text{MgO}$ , 4%  $\text{CaO}$  and  $\text{Na}_2\text{O}$  was mixed in 250ml of acidified distilled water of pH 2. Afterward, 26.26g

of TEOS and 30 ml of ethanol were added to the solution, and mixed till a powder is precipitated. The formed powder was washed with distilled water using a centrifugal machine, dried at 80°C for 24hrs and ground into fine powder.

PMMA was grafted onto MMT nanoclay via in-situ graft polymerization methodology adopted from Atai *et al* (11, 12) (Figure 1). An aqueous solution of distilled water containing 0.5 wt% MMT was prepared and kept at 50°C for 12 hours with continuous stirring using a magnetic stirrer. Then 2.5g of AMPS (2-acrylamido-2methyl-1 Propane sulfonic acid) was added to the aqueous solution and mixed for 2hrs. Afterward, 2g of the initiator (Ammonium persulfate), in addition to 50ml of methacrylic acid and 2ml of CCl<sub>4</sub> (the chain transfer agent) were added to the solution. The temperature was then elevated to 70°C to allow for full polymerization and gel formation. Precipitation was done by dropping the formed gel into methanol solution. The precipitate was washed using distilled water and ethanol by centrifugal machine. Purification of the powder was performed by the dialysis method adopted from Sample-Lord and Shackelford (13). The prepared sample powder was then dried and ground into fine powder and stored in a sealed container till use.

#### Sample size estimation

Statistical power analysis was performed to determine the sample size using Power and Sample Size Calculation Software (Version 3.1.2, Vanderbilt University, Nashville, Tennessee, USA). Sample size was calculated to be 10 for each experimental condition (n=10) with power value of 90% and type I error probability of 0.05.

#### Specimens preparation

A total of sixty acrylic circular molds with a depth of 2mm and internal diameter of 10mm were prepared using self-cured acrylic resin (Acrostone Cold Cure Acrylic Resin, Acrostone Co., England). Thirty acrylic molds were fully filled with unmodified Biodentine (Biodentine™, Septodont, Saint-Maur-des-Fossis, Creteil, France). Samples were stored at 37°C and 100% humidity in an incubator at three time intervals (12min for initial setting time, 2hrs and 2 weeks for final setting time). Another thirty acrylic molds were packed with

10wt% PMMA/MMT modified Biodentine, stored at the same condition and aging periods (12min, 2hrs and 2 weeks).

Unmodified Biodentine was prepared according to the manufacturer's instructions, where the powder was mixed with five drops of Biodentine liquid in an amalgamator for 30 seconds. The prepared Biodentine paste was packed into the acrylic molds using a plastic condenser, then a glass slab was used to gently press over the Biodentine to create a smooth surface. Biodentine modified with 10% PMMA/MMT by weight was prepared. Samples of both unmodified and modified Biodentine were stored in an incubator as mentioned above before the application of adhesive bonding agent. After each incubation period, specimens were randomly selected to apply the adhesive bonding agent and resin composite build up.

A universal adhesive (All bond universal, Bisco, USA) was applied in a self-etch mode using a bristle brush on the surface of each tested material, rubbed for 20 seconds and dried by gentle air for 5 seconds. Before light curing, five transparent cylindrical shaped silicon tubes of 1mm internal diameter and 2mm length were placed on the surface of each tested specimen and then the adhesive resin was polymerized by a light cure unit (VivaDent Bluephase, Ivoclar, USA) for 20 seconds. Flowable resin composite (Polofil NHT Flow, VOCO GmbH, Germany) was carefully injected in a inside each tube in a single bulk to avoid air bubbles formation and light cured. Polyethylene tubes were then removed by a sharp razor blade. Specimens were stored for 48 hours at 37°C at a relative humidity of 100% before testing.

#### Chemical analysis and phase identification

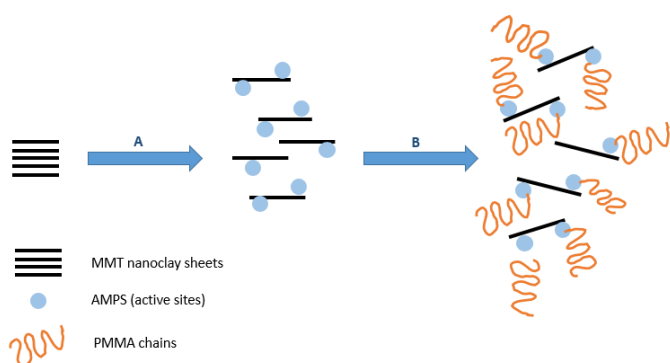
Chemical analysis of PMMA/MMT nanoclay as well as Biodentine before and after modification with PMMA/MMT was done by Fourier Transform Infrared Spectroscopy (FTIR) (IRAffinity-1S.SHIMADZU, Japan), with a wavelength of 4000 to 400 cm<sup>-1</sup>. Phase identification was tested by X-ray diffraction analysis (XRD) (PXRD-6000 SCHIMADZU, Japan) with a voltage of 40KV and a current of 30mA with CuK $\alpha$  radiation ( $\lambda=1.54056\text{\AA}$ ).

#### Scanning electron microscope (SEM) and energy dispersive X-Ray spectroscopy (EDX) assessment

Surface morphology of Biodentine before and after modification with PMMA/MMT was performed by SEM (Quanta FEG-250, FEI, USA) coupled with energy dispersive X-Ray spectroscopy (EDX) for elemental composition analysis. Disc shaped specimens (4mm in diameter and 2 mm in height) were mounted on stubs, gold coated using a vacuum sputter coater and their upper surfaces were examined by the SEM.

#### Micro-shear bond ( $\mu$ SBS) strength testing

All tested samples were subjected to shear bond strength by a Universal testing machine (Model 3345; Instron Industrial Products, Norwood, USA) with a load cell of 5 kN. Samples were mounted to the lower fixed compartment of testing machine and load was applied at resin-liner interface using a metallic loop attached to the upper movable compartment of testing machine operated at crosshead speed



**Figure 1.** Schematic representation of grafting of PMMA onto MMT nanoclay sheets; step (A) represents swelling behavior of nanoclay sheets in distilled water in the presence of AMPS. Step (B) represents graft polymerization of PMMA chains in the presence of the initiator and chain transfer agent.

of 0.5 mm/min. The load required to de-bonding was recorded in Newtons.

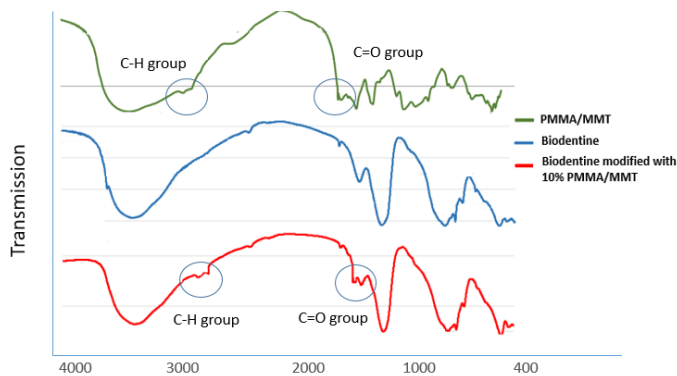
### Statistical analysis

Mean values and standard deviation for each group were tested for normality using Shapiro-Wilk tests. One-way ANOVA then Tukey's post hoc test were used to evaluate the effect of unmodified and PMMA/MMT modified Biodentine within each setting time interval and the effect of setting time intervals within each group on micro-shear bond strength test. A two-way ANOVA test was used to test the interactions between the tested variables. The significance level was set at  $P \leq 0.05$ . Statistical analysis was performed with (IBM SPSS Statistics Version 23 Armonk, NY, USA) for Windows.

## Results

### FTIR analysis results

Results of FTIR of PMMA/MMT showed peaks corresponding to aluminum silicates and magnesium silicates at 447  $\text{cm}^{-1}$  and 537  $\text{cm}^{-1}$  respectively (Figure 2). In addition, bands of aluminum oxide and silicon oxide were aligned at 900  $\text{cm}^{-1}$  and 1045  $\text{cm}^{-1}$  respectively (14-18). Bands were detected at the regions of 2900 that were attributed to C-H bonds of the organic modifier PMMA. Another band was observed at approximately 1700  $\text{cm}^{-1}$  which is associated to the stretching of the carbonyl group (C=O) belonging to PMMA (19-23).



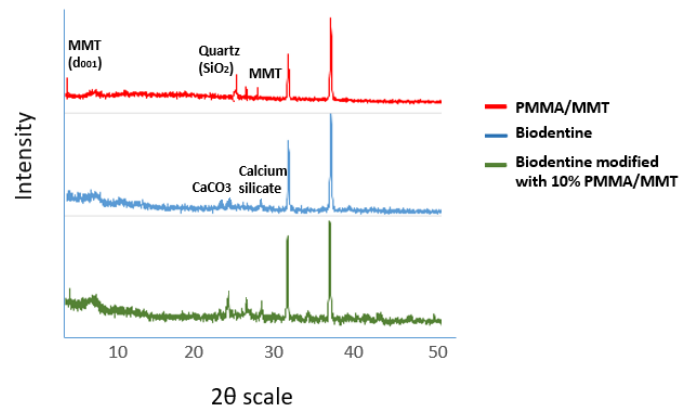
**Figure 2.** FTIR analysis of PMMA/MMT nanoclay, Biodentine and PMMA/MMT modified Biodentine.

FTIR analysis of Biodentine and modified Biodentine revealed bands at 500  $\text{cm}^{-1}$  that corresponded to silica vibrations. Broad bands at 1400 and 1600  $\text{cm}^{-1}$ , as well as bands at 870, and 700  $\text{cm}^{-1}$  attributed to carbonate groups were also presented (24, 25) Modified Biodentine showed new bands corresponding to C-H and C=O of PMMA.

### XRD analysis results

XRD results of PMMA/MMT (Figure 3) showed the characteristic peak of MMT (d001) at  $2\theta=7.4^\circ$  (26). Typical peaks of quartz were also detected at  $2\theta=25.5^\circ$  (27). XRD results of Biodentine showed peaks corresponding to calcium carbonates and calcium silicates at approximately  $2\theta=24.5^\circ$  and

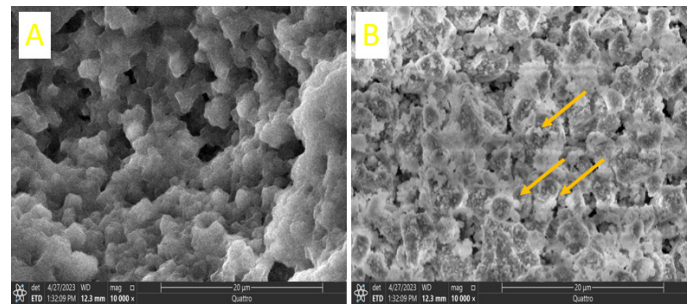
$28^\circ$  respectively (28). However, Biodentine modified with PMMA/MMT showed peaks corresponding to MMT(d001) at  $2\theta=7.7^\circ$ . As well as peaks of quartz at approximately  $2\theta=25.5^\circ$ .



**Figure 3.** XRD patterns of PMMA/MMT nanoclay, Biodentine and PMMA/MMT modified Biodentine.

### Scanning electron microscopy

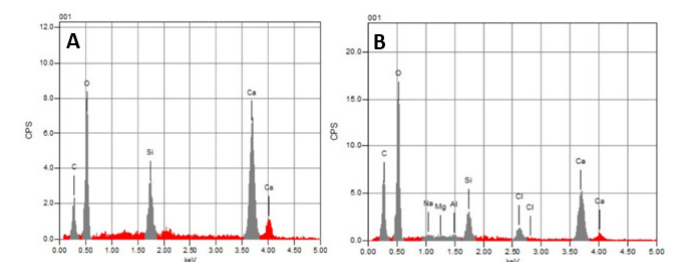
SEM images of Biodentine (Figure 4A) showed Biodentine crystals with large spaces between them, while Biodentine modified with PMMA/MMT (Figure 4B) showed nanoclay particles (yellow arrows) on the surface of Biodentine and in between the crystals.



**Figure 4.** SEM images (10000X) of A: Biodentine, B: PMMA/MMT modified Biodentine.

### EDX analysis results

EDX analysis (Figure 5-A) revealed the typical composition of Biodentine with high percentages of calcium (50.21%), oxygen (36.77%), carbon (6.75%) and silicon (6.25%), whereas in EDX analysis of PMMA/MMT modified Biodentine, aluminum (0.48%), sodium (0.23%) and magnesium (0.07%) attributed to MMT nanoclay were detected as shown in (Figure 5-B).



**Figure 5.** EDX analysis of A: Biodentine, B: PMMA/MMT modified Biodentine.



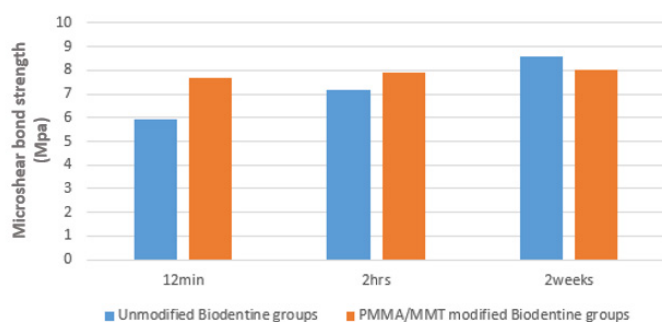
### Micro-shear bond strength ( $\mu$ SBS) results

Statistical analysis of micro-shear bond strength results (Figure 6 and Table 2) revealed significantly lower  $\mu$ SBS values (MPa) for the unmodified Biodentine group at 12 minutes compared to 2 weeks where  $p < 0.001$ . However, no statistically significant difference was found between 12 minutes with 2 hours and between 2 hours and 2 weeks within unmodified Biodentine groups. No statistically significant differences were found between PMMA/MMT modified BD groups between the three-time intervals. Pairwise comparisons showed significant difference between unmodified and modified Biodentine groups at 12 minutes, where ( $p=0.002$ ), however, no statistically significant difference was found between unmodified and modified Biodentine groups at 2 hours and 2 weeks, where ( $p=0.176$  and  $0.289$ ) respectively. The results of two-way ANOVA showed that type of Biodentine exhibited a statistically significant effect on micro-shear bond strength at  $p=0.0463$ . While different setting time intervals showed a statistically significant effect at  $p < 0.001$ . Moreover, the interaction between the two tested variables showed statistically significant effect at  $p=0.0137$ .

**Table 2.** Micro-shear bond strength ( $\mu$ SBS) data in MPa for tested groups.

Groups	12 min	2 hours	2 weeks	p value
Unmodified Biodentine	5.95 <sup>a</sup> ±1.35	7.19 <sup>ab</sup> ±1.54	8.59 <sup>b</sup> ±0.88	<0.001
PMMA/MMT modified Biodentine	7.67 <sup>a</sup> ±0.98	7.92 <sup>a</sup> ±1.28	8.02 <sup>a</sup> ±0.97	0.756
P-value	0.002	0.176	0.289	

Different letter within each row indicates significant difference by Tukey's post hoc test ( $P$ -value<0.001).



**Figure 6.** Bar chart representing micro-shear bond strength of the different tested groups.

## Discussion

Biodentine is a tri-calcium silicate based bioactive material that was advertised in markets as "Bioactive dentin substitute" since it has mechanical properties comparable to that of dentin. Moreover, it has a superior ability to stimulate the odontoblasts differentiation leading to the formation of reparative dentin, excellent bioactive property as well as biocompatibility. However, Biodentine has water-based chemistry which has greatly compromised the micromechanical bond with overlying resin composite (29-31). To overcome

this drawback, polymethyl methacrylate (PMMA) modified Montmorillonite (MMT) nanoclay (PMMA/MMT) was used in this study to modify Biodentine in an attempt to improve its bond strength with resin composite due to the good chemical interaction between PMMA and Bis-GMA in resin composite matrix (32).

MMT was synthesized by sol-gel technique due to its ability to control the structural morphology of the final product, utilize relatively low temperature and produce very fine powder with high chemical homogeneity (33, 34). Then PMMA was incorporated inside MMT using in-situ graft polymerization method (Figure 1) which was reported to be an easy and reliable technique that allows the penetration of polymeric chains into nanoclay sheets and also promotes covalent bonding between polymer and nanoclay (35, 36).

FTIR analysis for PMMA/MMT (Figure 2) was in agreement with that reported in literature; where peaks contributing to distinctive functional groups of Si-O-Al, Si-O-Mg as well as Si-O-Si of the tetrahedral layer of MMT were observed (37, 38). The appearance of C-H band as well as C=O band could further confirm the success of organo-modification and incorporation of PMMA into nanoclay sheets (38, 39). Bands of functional groups containing carbon were detected in FTIR analysis of PMMA/MMT modified Biodentine suggesting the success of incorporation of PMMA/MMT into Biodentine.

XRD results of PMMA/MMT exhibited the characteristic peaks of MMT ( $d_{001}$ ), quartz and Montmorillonite which were in accordance with the chemical structure reported in literature(40) (Figure 3). Peaks of MMT ( $d_{001}$ ) and quartz were also observed in the XRD pattern of PMMA/MMT modified Biodentine confirming the incorporation of PMMA/MMT into Biodentine. FTIR and XRD findings were further justified by SEM images and EDX analysis. SEM shows clusters of PMMA/MMT nanoclay on modified Biodentine surface (Figure 4-B). Moreover, the EDX analysis confirmed the presence of aluminum, sodium and magnesium in modified Biodentine which are the main constituent elements of MMT, thus confirming the incorporation of PMMA/MMT in Biodentine (Figure 5-B).

Biodentine has different stages for setting, the initial setting stage is when the tri-calcium silicate reacts with water forming calcium silicate gel and calcium hydroxide and it takes nearly 12 minutes, then afterward nucleation and expansion of this gel over the tri-calcium silicate occur filling the spaces between tri-calcium silicate till crystallization of the hydrated calcium silicates gel. Crystallization continues till complete maturation of the cement occurs which might take 12 weeks up to 1 month (7, 41).

The literature was unclear regarding the time after which resin composite should be placed over Biodentine. Some studies suggested immediate placement of resin composite after Biodentine setting (42-44). However, one study claimed that resin composite should be placed after 2 hours of Biodentine setting and they suggested that the changes in Biodentine became minimal after nearly 120 minutes (8). Other studies reported that final composite restoration should be placed at least 14 days after setting to allow sufficient maturation of Biodentine (7, 45-47). Accordingly, the time intervals chosen in this study for placement of resin composite and testing the bond strength were; after 12 minutes (as claimed by the manufacturer), 2 hours and 2 weeks.

Regarding the type of adhesive systems used with Biodentine, some studies suggest the advantage of using etch and rinse methodology over self-etch (48, 49). However, other studies reported that self-etch strategies showed higher bond strength (42). While other studies reported that the choice of the adhesive strategy doesn't affect the bond strength (45, 50-52). In this study, universal bonding in self-etch mode was used to simplify the application process and reduce the technical errors (53).

Micro-shear bond strength (SBS) test was performed to evaluate the bond strength between Biodentine and resin composite because of its simplicity, ease of specimen preparation and lower incidence of pretest failure. Micro-shear mode testing was employed in this study rather than macro-shear testing as it was reported that conventional macro shear bond test resulted in non-uniform stress distribution with heterogeneous stress patterns when assessed by finite element stress analysis (54). Acrylic resin molds were used as it is an easy and fast way for standardization and central holes with 10 mm × 2 mm were done allowing better retention of the overlying resin composite (45).

The null hypothesis in this study was rejected as there was a statistically significant difference between the shear bond strength of the modified Biodentine and the unmodified one after 12 minutes of Biodentine setting (as shown in Figure 6 and Table 2). Addition of PMMA modified nanoclay to Biodentine might have increased the bond strength with resin composite, as PMMA chains within the nanoclay could interact with BisGMA of resin composite matrix creating stronger network with the monomers in resin composite.

Besides, the incorporation of nanoclays in between Biodentine crystals could act as crack deflectors resisting crack propagation, thus increasing the bond strength (55). Moreover, the use of self-etch adhesives with freshly mixed Biodentine was reported to cause deeper penetration of the adhesive system into the unset Biodentine leading to stronger micro-mechanical retention (46). This was in agreement with previous studies that recommended immediate placement of resin composite restoration on fresh Biodentine (42-44, 56).

The results also revealed that unmodified Biodentine group after 2 weeks showed significantly higher shear bond strength than after 12 minutes. This could be attributed to the fact that calcium hydroxide released during the hydration setting reaction of Biodentine could react chemically with 10-MDP monomers present in self-etch adhesives, thereby enhancing their chemical bonding. Moreover, the initial water-based nature of Biodentine could have a detrimental effect on the bond strength when resin composite is placed immediately. This was in accordance with other studies that suggested that the bond strength continues to increase by time and it should not be placed immediately after setting (7, 45-47). However, no significant difference was found between different setting times of PMMA/MMT modified Biodentine. This could be due to the incorporation of PMMA/MMT that increased the initial bond strength compared to unmodified Biodentine as mentioned above and changed the mode of interaction with resin composite.

It is worth noting that Theracal LC (a resin modified light-cured calcium silicate base material) showed higher bond strength to resin composite compared to Biodentine. This was attributed to the similarity between the resin chemistry

of Theracal and resin composite promoting true chemical adhesion and creating strong interface (57).

The current study findings are of important clinical relevance; as strong bond between resin composite and underlying base material is essential to increase the longevity of the final restoration and enhance its stability and performance. However, the current study has some limitations; this is an in vitro study with limited sample size and the simulation of in vivo conditions is yet unsolved. The concentration of nanoclay used was 10% by weight only, yet the effect of different concentrations needs to be examined. Moreover, the effect of modified Biodentine on other properties such as solubility, bioactivity and water sorption are of great importance and it is recommended to be considered in further researches. Evaluation of the biological properties and assessment of the biocompatibility of the nanoclay modified Biodentine should be taken in consideration in further researches. Bond strength durability after different aging conditions was not considered in this study and it is also recommended to be investigated in further studies.

## Conclusion

Within the limitations of this study, it was concluded that incorporation of 10% PMMA/MMT by weight to Biodentine improved shear bond strength with resin composite when placed after 12 minutes of Biodentine setting time.

**Türkçe özet:** Polimetil Metakrilat/Montmorillonit Modifiye Biodentine'nin Dental Rezin Kompozit ile Kayma Bağlanma Dayanımının İn Vitro Değerlendirilmesi. Amaç: Bu çalışmanın amacı, polimetil metakrilat/montmorillonit nanokil ile modifiye edilen Biodentine ile rezin kompozit arasındaki bağlanma dayanımını, Biodentine'in farklı sertleşme aşamalarında değerlendirmektir. Gereç ve Yöntemler: Nanokil hazırlandı ve polimetil metakrilat ile organik olarak modifiye edildi. Polimetil metakrilat/montmorillonit nanokilin, Biodentine'in ve modifiye Biodentine'in karakterizasyonu; X-ışını kırınım analizi, Fourier dönüşümlü kızılötesi spektroskopisi ve enerji dağılımlı X-ışını spektroskopisi ile eşleştirilmiş taramalı elektron mikroskobu kullanılarak yapıldı. Toplam altmış akrilik kalıp hazırlandı; otuz örnek Biodentine ile, diğer otuz örnek ise nanokil ile modifiye Biodentine ile dolduruldu. Her grup, Biodentine'in sertleşme sürecinin farklı aşamalarına göre üç alt gruba ayrıldı: 12 dakika, 2 saat ve 2 hafta. Evrensel adeziv uygulandıktan sonra akışkan rezin kompozit yerleştirildi. Mikro-kayma bağlanma dayanımı, üniversal test cihazı kullanılarak test edildi. Veriler tek yönlü ANOVA ve ardından Tukey'nin post hoc testi ile analiz edildi; ayrıca iki yönlü ANOVA uygulandı. Anlamlılık seviyesi  $p \leq 0,05$  olarak belirlendi. Bulgular: Karakterizasyon sonuçları, polimetil metakrilat/montmorillonit nanokilin ve modifiye Biodentine'in başarılı bir şekilde hazırlandığını ortaya koydu. Mikro-kayma bağlanma dayanımı testleri, modifiye Biodentine'in 12. dakikada, modifiye edilmemiş Biodentine'e kıyasla anlamlı derecede daha yüksek bağlanma dayanımına sahip olduğunu gösterdi. Ancak, 2 saat ve 2 hafta grupları arasında istatistiksel olarak anlamlı bir fark bulunmadı. Sonuç: Biodentine'e ağırlıkça %10 oranında modifiye nanokil eklenmesi, Biodentine'in sertleşme sürecinin 12. dakikasında uygulandığında, rezin kompozit ile bağlanma dayanımını artırabilir. Anahtar Kelimeler: biodentine, montmorillonit nanokil, rezin-modifiye nanokil, mikro-kayma bağlanma dayanımı, pulpa örtüleme materyalleri

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