Original Article

Compressive Strength, Surface Roughness, and Surface Microhardness of Principle Tricalcium Silicate-Based Endodontic Cements after Universal Adhesive Application

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Abstract

Aims and Background: It was aimed to evaluate compressive strength (CS), surface roughness, and microhardness of mineral trioxide aggregate (ProRoot MTA) and Biodentine (BD) after adhesive application. **Materials and Methods:** Tests was carried out according to international ISO standard. ProRoot MTA and BD were prepared in Teflon molds according to manufacturer's instructions: n = 210 for CS; n = 210 for microhardness. Samples were incubated for 7 days at 37°C in 100% humidity. Surfaces were smoothed with up to 2000 grits of silicon-carbide sandpaper on abrasive device at 150 rpm, randomly divided into seven groups (n = 15). Clearfil Universal Bond, All Bond Universal, and Single Bond Universal (SBU) were applied in both total-etch and self-etch (SE) modes. Adhesives were applied according to manufacturers' recommendations (no adhesive used in control). CS was performed at speed of 1 mm/min, microhardness at 100 gr for 15 s. The surface roughness of the samples was analyzed with atomic force microscopy. Two-way analysis of variance and *post hoc* Tukey tests were used for the evaluation of the data. **Results:** Man CS and microhardness values between ProRoot MTA and BD were as follows: 24.9 N, 72.6 HV; 59.8 N, 59.0 HV, respectively. In CS, BD was higher than ProRoot MTA ($P < 0.05$). In other comparisons except for SBU SE group ($P < 0.05$), BD and ProRoot MTA showed similar results ($P > 0.05$). However, ProRoot MTA was found higher than BD regarding microhardness ($P < 0.05$). As a result of the adhesive application in both BD and ProRoot MTA groups, a decrease in surface roughness was observed compared to the control group. **Conclusion:** BD exhibited better results than ProRoot MTA regarding CS. However, ProRoot MTA was found to be more successful than BD in terms of microhardness. BD and ProRoot MTA showed similar physical properties in terms of surface roughness. To improve regenerative procedures, besides the selection of bioceramic cements, the interaction between cements and materials applied during coronal restoration should be considered.

Keywords: *Biodentine, compressive strength, mineral trioxide aggregate, regenerative endodontics, surface microhardness, surface roughness, tricalcium silicate-based cement*

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Introduction

Nowadays, it is known that regenerative treatments are gaining more importance in dental applications, especially in endodontics. Maintaining the viability of the pulp after the treatment of traumatic injuries or profound

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caries is the main goal of regenerative endodontic treatments. During these regenerative processes, especially in the double sealing method, there is a need for a biocompatible material that can be placed adjacent to the connective tissue, will not be affected by moisture and bleeding, and can stimulate bone formation in the damaged area. This material should also provide excellent sealing against coronal leakage after dental applications and should offer appropriate surface microhardness and compressive strength values.[1–3]

Mineral trioxide aggregate (MTA; ProRoot MTA; Dentsply Sirona Endodontics, Tulsa, OK, USA), which is a tricalcium silicate-based biocompatible endodontic repair material, is currently used as the gold standard in regenerative endodontic treatments due to its high tissue compatibility.[4] It also presents extremely good prevention of bacterial leakage.[5] When used in combination with a restorative agent, MTA stands out as an ideal material for repairing and maintaining the viability of the pulp tissue due to its biocompatibility and bacterial sealing properties. Thanks to these features, MTA can be placed adjacent to pulpal and periodontal connective tissues in regenerative treatments.[3] It has also been reported to contribute to the regeneration of cement,[6] harden in humid conditions,[7] not be affected by bleeding,[8] and prevent bacterial leakage.[9] Therefore, MTA has a wide range of dental applications such as vital pulp treatments, root perforation repair, and regenerative endodontic procedures. Despite these excellent properties of MTA, it has some difficulties in clinical applications. Commercial formulations of MTA require a long curing time, which limits the use of the material in the oral cavity.[10] Furthermore, the difficulty of condensation and the impracticality of its application make the clinical use of the material difficult.[5] It is very clear that the physical properties of the MTA need to be improved to simplify clinical use.

Biodentine (BD) (Septodont, Saint-Maur-des-Fossés, France) is a newly developed tricalcium silicate-based material. It has been developed for use in similar fields with MTA and has been shown to yield successful results in many studies.[11,12] It has also been reported that BD can be applied under dental fillings alone because it shows resistance similar to the mechanical properties of dental tissue[13,14] and it supports the dentin.[11] It is recommended to use this material in regenerative endodontic treatments.[15]

Although there is a great deal of research on MTA-adhesive applications, there is a scarcity of research evaluating BD-adhesive applications. In addition, studies conducted in general examined the shear bond strength between bioceramic cements and resins. It is also seen that there is not exactly consistency between the results. The quality of adhesion, the changes in the compressive strength, and the surface microhardness of BD and MTA following the application of the adhesive agents are issues that should be taken into consideration and more investigated.

Universal adhesives or multimode adhesives are recently developed multipurpose adhesives that offer the opportunity for the clinician to choose the adhesive strategy that they desire. These adhesives can be used in either total-etch (TE) or self-etch (SE) modes, allowing the opportunity to create effective bonding to different tissues with a single adhesive material.[13] Regarding these materials, it is known that the Vitrebond copolymer, 10-Methacryloyloxydecyl dihydrogen phosphate (10-MDP) monomer, and silane technology can bind adhesives to different tissues and improve their performance.[16]

This study was planned based on the hypothesis that the possible forming hydrolytically stable salts 10-MDP—calcium (Ca) by binding the 10-MDP and Vitrebond copolymer to the calcium of MTA and BD and a possible chemical linkage between the MDP monomer and the alumina of the MTA.[17,18] The aim of the current study was to compare compressive strength, surface roughness, and surface microhardness values of MTA and BD following universal adhesive application to improve the clinical success rate in regenerative endodontic procedures. The H0 hypothesis of this study was that there would be no significant difference regarding compressive strength, surface roughness, and surface microhardness values between MTA and BD.

Materials and Methods

A total of 210 samples of MTA and BD were prepared with standard Teflon discs, for each compressive strength and surface microhardness tests. In the MTA group, the material was mixed using a 3:1 ratio of MTA powder to sterile water according to the manufacturer's recommendations and was applied to the Teflon mold using an MTA carrier (Dentsply Sirona Endodontics). The samples were covered with damp cotton until the setting process was completed. In the BD group, the BD capsules were mixed according to the manufacturer's instructions in a high-speed amalgamator for 30 s and were transferred to the Teflon molds. Following the preparation of cements, the samples placed in the molds were allowed to stand for 7 days at 37°C in 100% humidity; after which, the samples were gently removed from the molds. For each test, each main group ($n = 105$) was then randomly divided into seven groups, one of which was a control group. There were 15 samples in

each group. The adhesive systems were applied to each group according to manufacturer's recommendations, as described in Table 1, and no adhesive was applied to the control groups. The distribution of the groups was as follows: MTA main group (n = 105); include seven groups (n = 15): 1. Clearfil Universal Bond (CUB) TE, 2. CUB SE, 3. All Bond Universal (AU) TE, 4. AU SE, 5. Single Bond Universal (SBU) TE, 6. SBU SE, 7. control group. BD main group (n = 105); include seven groups (n = 15): 1. CUB TE, 2. CUB SE, 3. AU TE, 4. AU SE, 5. SBU TE, 6. SBU SE, 7. control group.

Compressive strength test

The international standard (ISO 9917-1:2007) was applied for the compressive strength test (International Organization for Standardization, 2007). A total of 210 samples (105 each for the MTA and BD main groups) were prepared according to each manufacturer's instructions, and they were placed in 4-mm diameter, 6-mm high specially manufactured Teflon molds with the help of an MTA carrier for ensuring that there were no air voids. During the sample preparation, both the upper and lower surfaces of the molds (for all the materials) were covered with plastic strips. Each material was set according to the manufacturer's recommendations [Table 2]. All of the samples were incubated in the molds in 100% humidity at 37°C for 7 days following the preparation.

The samples' lower and upper surfaces were sanded with 320, 600, 800, 1,200, 1,500, and 2,000 grit silicon carbide sandpaper, respectively, on an abrasive device (Mecatech Z34; PRESI, Eybens, France) under water cooling at 150 rpm for 30 s for each sanding sheet. At the end of the sanding process, all of the surfaces were standardized and smooth. Next, the adhesive systems were applied according to the manufacturers' instructions [Table 1]. The samples were then subjected to a compressive strength test, which was carried out at a speed of 1 mm/min on a universal testing machine (Autograph AGS-J, 5 kN; Shimadzu Corporation, Tokyo, Japan).

Microhardness test

For the microhardness test, the MTA (n = 105) and BD (n = 105) main groups (a total of 210 samples) were prepared at heights of 2 mm and diameters of 4 mm. The samples were incubated in 100% humidity at 37°C for 7 days. Then, the sanding processes and the adhesive applications were performed as described above. The microhardness measurements were performed at a 100-g load for 15 s using a Vickers tip in a microhardness testing machine (HMV-G; Shimadzu Corporation). Three measurements were taken from each sample surface for the microhardness test. The location of each point to be measured was chosen at least 1 mm^2 away from the others and the outer boundary of the sample. Then, the "pyramidal permissive" diagonal lines that were obtained were visualized using a digital camera (1192; Carl Zeiss Jena GmbH, Jena, Germany), and the values read under the microscope were converted to the Vickers HV) by calculating them using the HMV-G program on the computer to which the device was connected. The average of the three measured values was recorded as the surface HV of the relevant sample.

AFM analysis

AFM images were taken from a total of six samples, one from each of the most successful groups [Picture 1]. The samples were prepared with the help of Teflon molds [Table 2] and adhesive systems were applied according to the relevant group [Table 1], as described before. Then, the surface topography measurements of the samples were performed with the Nanosurf Easyscan 2 Controller AFM device using non-contact mode. The images obtained as 10 × 10 μm were created using a special software (Nanoscope v616r1, Veeco Metrology Inc., Santa Barbara and WSxM 4.0 Develop 11.1, Nanotec Electronica SL, TreaCantas, Spain) were analyzed.

Statistical analysis

The data obtained from the experiments were evaluated using the Statistical Package for the Social Sciences version 17 (SPSS Inc., Chicago, IL, USA). The data sequence obtained was first checked using the Shapiro–Wilk's test for the normal distribution. A two-way analysis of variance was used to evaluate all of the groups together, and a Tukey test was used for the *post hoc* comparison. Moreover, the one-way analysis of variance was used to determine the differences between the groups. After viewing the homogeneity of the variances with Levene's test, the groups were compared in binary using the *post hoc* Tukey test to determine the difference. Then, each group was compared with a two-Sample t-test in terms of the data from the equivalent group in the other main group. The confidence interval was assumed to be 95% during all of these analyses.

Results

Compressive strength test

The compressive strength results are summarized in Figure 1, and significant differences were found within the MTA main group ($P < 0.05$).

The statistical ordering of the groups for the MTA main group was as follows: AU SE = CUB SE = SBU

Table 1: Adhesive system, content, and, according to the instructions of the manufacturers, the application modes: self-etch and total-etch

Adhesive System (Manufacturer/Lot Number)	Material Composition	Self-Etch	Total-Etch
Single Bond Universal (3MDeutschland GmbH, Neuss, Germany/3300767)	1) MDP phosphate monomer 2) Dimethacrylate resin 3) Vitrebond copolymer 4) Filler 5) Ethanol 6) Water 7) Silane 8) Catalyst	1) Apply adhesive and rub it in for 20 s with a disposable applicator. 2) Direct a gentle stream of air over the liquid for about 5 s until it no longer moves and the solvents were evaporated completely 3) Light cure for 10 s.	1) Apply for 15 s 35% phosphoric acid gel and allow to react 15 s. 2) Rinse thoroughly with water and dry with water-free and oil-free air or with cotton pellets; do not over dry. 3) Apply the adhesive in self-etch mode.
All Bond Universal (Bisco Dental Products, Schaumburg, IL, USA/1700005544)	1) Acid: 32% phosphoric acid, benzalkonium chloride. 2) Adhesive: MDP, 5-15 wt%, Bis-GMA, 30-60 wt%, HEMA, 5-15 wt%, Ethanol, water, 10-40 wt%, Photocatalyst.	1) Apply two separate coats of adhesive scrubbing the preparation with a micro brush for 10-15 s. per coat. Do not light cure between coats. 2) Evaporate the excess solvent for at least 10 s until there should be no visible movement of the adhesive and uniform glossy appearance on the surface. 3) Light cure for 10 s.	1) Apply acid for 15 . 2) Rinse thoroughly. Remove the excess water by blotting the surface with an absorbent pellet of high volume evacuation for 1-2 s, leaving the preparation visibly moist. 3) Apply the adhesive in self-etch mode.
Clearfil Universal Bond (Kuraray, Noritake Dental Inc., Okayama Japan/000017)	1) 10-Methacryloyloxydecyl dihydrogen phosphate (MDP) 2) Bisphenol A diglycidyl methacrylate (bis-GMA) 3) 2-Hydroxyethyl methacrylate (HEMA) 4) Hydrophilic aliphatic dimethacrylate 5) Colloidal silica 6) Silane coupling agent 7) DL-Camphorquinone 8) Ethanol, water.	1) Apply the adhesive to the entire cavity and rub it in for 10 s. 2) Dry the entire cavity wall sufficiently by blowing mild air for more than 5 s until the bond does not move. 3) Light cure at a density of more than 1500 mW/cm^2 for 5 s.	1) Apply phosphoric acid to the entire cavity, leave it in place for 10 s, then rinse and dry. 2) Apply the adhesive in self-etch mode.

Table 2: The calcium silicate-based endodontic cements used in this study, their compositions, and the application steps, according to the manufacturers' instructions

Endodontic Cements (Manufacturer/Lot Number)	Material Composition	Application Steps
Mineral Trioksit Agregat (Proroot MTA (Dentsply Tulsa Dental, USA/0000155583)	Tricalcium silicate, bismuth oxide dicalcium silicate, tricalcium aluminate, calcium sulfate dehydrate, or gypsum	Gradually incorporate the liquid into the cement. Mix the material with the liquid for about 1 min to ensure all powder particles are hydrated.
Biodentine (Septodont, Saint-Maur- des-Fosses Cedex France/B21745)	Powder: Tricalcium silicate, dicalcium silicate, calcium carbonate and oxide, iron oxide, and zirconium oxide Liquid: Calcium chloride and hydrosoluble polymer	Open a capsule and pour 5 drops of liquid from a single-dose container into the capsule. Close and place the capsule on an amalgamator at a speed of 4000-4200 rpm. Mix for 30 s.

TE = AU TE, SBU SE = CUB TE = control, and CUB TE = SBU SE = AU TE. For the BD main group was as follows: control = CUB SE = SBU SE = AU TE = AU SE, SBU TE < CUB SE = control, CUB TE < SBU SE = CUB SE = control, and SBU TE = CUB TE.

The findings obtained as a result of the binary comparisons among the main groups were as follows: the BD showed a significantly higher compressive strength value when compared to the MTA ($P < 0.05$). However, there were no significant differences between the BD and MTA main groups in the bilateral comparisons of the CUB TE, AU SE, CUB SE, SBU TE, and AU TE groups ($P > 0.05$). Finally, the BD SBU SE group exhibited a significantly higher

compressive strength value than that of the MTA SBU SE group ($P < 0.05$) [Table 3].

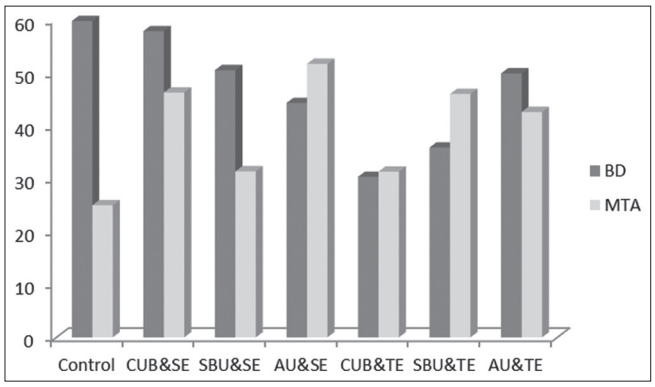


Figure 1: The compressive strength test results of the tested materials

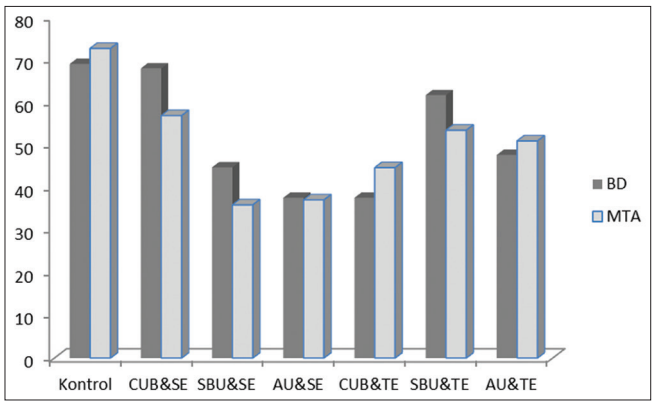


Figure 2: The microhardness test results of the tested materials

Microhardness test

The microhardness results are summarized in Figure 2. Significant differences were found in the MTA main group ($P < 0.05$).

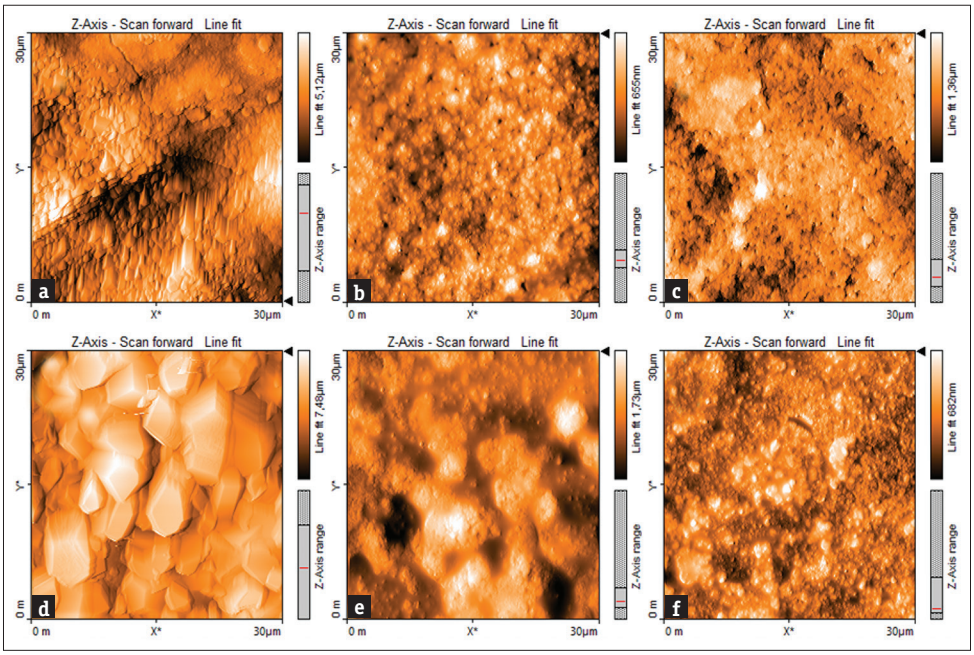
The statistical ordering of the groups according to the microhardness test for the MTA main group was as follows: control > CUB SE > SBU TE > AU TE > CUB TE > SBU SE = AU SE. For the BD main group was as follows: control > CUB SE > SBU TE > AU TE > SBU SE > CUB TE = AU SE.

The findings obtained as a result of the binary comparisons between the BD and MTA main groups were as follows: the difference between the control groups was significant ($P < 0.05$). No significant differences were found between the BD and MTA main groups when comparing the AU SE, SBU SE, and CUB SE groups ($P > 0.05$). However, the differences between the AU TE, SBU TE, and CUB TE groups were significant ($P < 0.05$) [Table 4].

AFM analysis

The decrease in surface roughness was remarkable when the control sample and the other two samples were compared in MTA main group. On the other hand, it has been seen that the surface roughness of the SBU TE group containing phosphoric acid was less than the CUB SE group containing weak acid.

According to the results observed in the BD main group, similar to the MTA main group, the surface roughness in



Picture 1: The atomic force microscopy images taken from the most successful groups. a) Biodentine control group b) Biodenitne-CUB SE group, c) Biodentine-SBU TE group d) ProRoot MTA control group, e) ProRoot MTA-CUB SE group, f) ProRoot MTA-SBU TE group. CUB: Clearfil Universal Bond; SE: self-etch; TE: total-etch; MTA: mineral trioxide aggregate; SBU = Single Bond Universal

Table 3: The mean values and standard deviations of the ProRoot mineral trioxide aggregate and Biodentine main groups according to the compressive strength test results

Groups	Main Groups		*P*
	ProRoot MTA	Biodentine	
Control	24.97±4.66^{a,X}	59.81±18.13^{b,XZ}	*P*<0.05
CUB TE	31.332±9.07^{a,XZ}	30.34±6.71^{a,Y}	*P*>0.05
CUB SE	46.29±13.13^{a,Y}	57.89±17.00^{a,X}	*P*>0.05
AU TE	42.61±11.19^{a,YZ}	49.91±16^{a,XY}	*P*>0.05
AU SE	51.71±11.92^{a,Y}	44.34±15.95^{a,XY}	*P*>0.05
SBU TE	46.01±9.12^{a,Y}	35.90±14.99^{a,YT}	*P*>0.05
SBU SE	31.39±11,87^{a,XZ}	50.51±19.94^{b,XT}	*P*<0.05
P	*P*<0.05	*P*<0.05	

Mean values±Std. Dev. values are presented. Different superscript lowercase letters in the same line indicate a significant difference between ProRoot MTA and Biodentine main groups as a result of binary comparisons; different superscript capital letters in the same column indicate a significant difference as a result of comparison between groups within each main group. CUB: Clearfil Universal Bond; SE: self-etch; TE: total-etch; MTA: mineral trioxide aggregate; SBU=Single Bond Universal; AU: All Bond Universal

Table 4: The mean values and standard deviations of the ProRoot mineral trioxide aggregate and Biodentine main groups according to the microhardness test results

Groups	Main Groups		*P*
	ProRoot MTA	Biodentine	
Control	72.61±1.38110^{a,X}	69.00±0.71^{B,X}	*P*<0.05
CUB TE	44.65±1.12616^{a,U}	37.58±0.61^{B,T}	*P*<0.05
CUB SE	56.88±1.49260^{a,Y}	67.96±1.16^{a,Z}	*P*>0.05
AU TE	50.92±1.75447^{a,T}	47.66±0.94^{B,Y}	*P*<0.05
AU SE	37.09±0.88094^{a,Z}	37.57±0.81^{a,T}	*P*>0.05
SBU TE	53.45±0.69987^{a,T}	61.66±1.12^{B,V}	*P*<0.05
SBU SE	35.93±0.83229^{a,Z}	44.74±0.63^{a,U}	*P*>0.05
P	*P*<0.05	*P*<0.05	

Mean values±Std. Dev. values are presented. Different superscript lowercase letters (^{a,b}) in the same line indicate a significant difference between ProRoot MTA and Biodentine main groups as a result of binary comparisons; different superscript capital letters (^{X,Y,Z,T,U,V}) in the same column indicate a significant difference as a result of comparison between groups within each main group. CUB: Clearfil Universal Bond; SE: self-etch; TE: total-etch; MTA: mineral trioxide aggregate; SBU=Single Bond Universal; AU: All Bond Universal

the control sample decreased with the application of the adhesive agent. However, unlike the MTA main group, it was observed that the roughness values of the SBU TE group containing strong acid and the CUB SE group containing self-weak acid were close to each other.

Discussion

The current study was planned based on the following two hypotheses: It may create a possible chemical linkage between the alumina in the structure of ProRoot MTA and the MDP monomer contained in universal adhesives, and also the universal adhesives' 10-MDP and Vitrebond copolymers bind to the calcium present in the structure of ProRoot MTA and BD to form hydrolytically stable salts. Based on these hypotheses, compressive strength and surface microhardness results were investigated following the universal adhesive application of ProRoot MTA and BD. According to the results of this study, the H0 hypothesis was rejected.

The endodontic cements used during regenerative endodontic treatments will get in contact with moist environments.[18] In addition, since it is thought that one of the basic physical properties of hydraulic cements is to meet the chewing forces, the compressive strength test was preferred in this study. According to the results obtained in this study, the BD control group showed a significantly higher compressive strength value than that of the MTA control group, and the BD SBU SE group value was significantly higher than the MTA SBU SE group value. These results could be attributed to the enhanced strength due to the low water/cement ratio used in BD. Kayahan *et al*.[18] researched whether the acid application differs in terms of the compressive strength in the ProRoot MTA, BD, MTA Angelus, and calcium-enriched mixture cements. They found that the acid application significantly reduced the compressive strengths of the Angelus MTA and CEM cement, but the ProRoot MTA and BD did not show significant differences in the compressive strength values. Similarly, in the current study, an acid application was performed on the TE groups, and there were no significant differences between the TE groups of the BD and ProRoot MTA main groups. Elnaghy[19] investigated the effects of an acidic media on the compressive strengths of the BD and MTA at different pH values, and they found that the BD had higher compressive strengths than the MTA at the different pH values. Since the etching and rinsing procedures in the TE adhesive systems resulted in the preferential dissolution and detachment of filler particles from the ProRoot MTA, this process could have caused the degradation of the cement surface and reduced the compressive strength of the materials. In the study by Butt *et al*.,[20] the compressive strength of the BD was found to be higher than that of the MTA. Similar to these studies, the results of the present study also showed that the BD control group had a higher compressive strength value than that of the MTA control group. However, the MTA Angelus was used in the studies by Elnaghy[19] and Butt *et al*.,[20] it is believed to have a lower compressive strength value due to the absence of the calcium sulfate dihydrate required for the formation of ettringite crystals when compared to the ProRoot MTA.[18] Additionally, it has been reported that the high compressive strength values observed in the BD group may be related to the absence of alumina

in the BD, which is believed to increase the fragility of the hardened material and cause it to weaken.[20] Smaller particle size and uniform BD structural components may play roles in the stronger BD-adhesive connection. In a recent review, it was reported that the presence of finer particle size, use of zirconium oxide as radiopacifier, purity of tricalcium silicate, absence of dicalcium silicate, and the addition of calcium chloride and hydrosoluble polymer could be attributed to the enhanced physical and biologic properties of Biodentine.[15] Based on the results of these studies, it can be assumed that the clinical use of BD seems to be more advantageous in terms of its compressive strength.

In previous studies, surface microhardness values of tricalcium silicate-based cements significantly decreased after being exposed to low pH.[19,21–25] Based on these results reported in the previous studies, the surface microhardness of ProRoot MTA and BD was decided to investigate after adhesive application regarding recommended "double-seal" method[1,2] during regenerative endodontic procedures. Vickers surface microhardness is defined as the resistance to plastic deformation of the material surface after indentation or penetration. The surface microhardness value of healthy dentin has been reported to range from 60 to 90 HV.[26] According to the microhardness test results, the ProRoot MTA control group (72.62 ± 1.38 HV) showed a significantly higher microhardness value than the BD control group (69 ± 0.72 HV). The ProRoot MTA and BD showed microhardness values close to that of dentin according to the results of the current study. Unlike the results of this study, Kaup *et al*.[26] found that the microhardness value was higher in the BD control group (62.35 ± 11.55 HV) than in the ProRoot MTA control group (26.93 ± 4.66 HV). While the BD surface HV was similar between the two studies, there was about a three-fold difference in the surface HV of the MTA. This difference between the results may have been related to the fact that the microhardness test was performed 1 day after the hardening of the materials in the study by Kaup *et al*.,[26] but it was performed 7 days after the setting in the present study. Elnaghy[19] investigated the effects of acidic media on the surface microhardness of the BD and MTA at different pH values and found that the MTA exhibited lower microhardness values than the BD at the different pH values. The reason for this may have been related to the fact that the MTA Angelus used in the study by Elnaghy[19] was different in terms of its structural content from the ProRoot MTA used in this study. ProRoot MTA consists of 75% portland cement, 20% bismuth oxide, and 5% calcium sulfate dehydrate. MTA Angelus contains 80% portland cement and 20% bismuth oxide, but it does not contain calcium sulfate, which shortens the setting time. Similar to the results of the present study, Majeed and AlShwaimi[27] and Caronna *et al*.[28] found that the ProRoot MTA showed high microhardness values at significant levels when compared to the BD. In examining the results of the present study in TE groups of ProRoot MTA and BD, microhardness values were decreased according to the control groups, and these results were in resemblance with previous studies.[19,21–25]

According to the AFM images, in the ProRoot MTA main group, the decrease in surface roughness is remarkable when the control group is compared with CUB SE and SBU TE groups. On the other hand, the surface roughness of the SBU TE group containing phosphoric acid is less than the CUB SE group containing weak acid. It can be concluded that the phosphoric acid in the SBU TE group is more corrosive than the CUB SE group. In accordance with the results observed in the BD main group, the roughness in the control group was reduced by the application of the adhesive agent, similar to the ProRoot MTA main group. However, unlike the ProRoot MTA main group, the roughness values of the strong acid-containing SBU TE group and the weak acid-containing CUB SE group were similar. It can be said that, depending on the content of the adhesive agent applied, the corrosive effect of phosphoric acid decreases.

The limitation of the present study is the low ability to imitate the clinical conditions physiologically, as it is an *in vitro* study on materials. Future *in vitro* studies should be planned with a chewing simulator in a human dental tissue model with oral conditions such as the presence of saliva, body temperature, etc., The clinical significance to be derived from the findings of this study is as follows: The use of BD can provide more successful results when treating an area that requires intense compressive strength such as the furcal area of the molar teeth. In treatments where there are fewer requirements for compressive strength, such as the double seal method or direct pulp capping, where the adhesion with the coronal restoration becomes more important, the high surface microhardness value may be the reason for the preference for MTA. And also the decrease observed especially in the TE groups compared to the control groups should be taken into attention clinically. For more reliable results, the long-term outcomes of clinical studies are needed. The studies to be planned in the light of the results obtained from the current study will be a guide for increasing the success rate of regenerative endodontic treatments and providing a better treatment quality for patients.

Conclusions

Within the limitations of this *in vitro* study, the following conclusions can be drawn from the results: According to the results obtained from the study, BD was found to be more successful in terms of compressive strength; MTA was found to be more successful in terms of surface microhardness. BD SBU TE, BD CUB SE, and control groups exhibited sound dentin-like microhardness values. After TE adhesive applications, the compressive strength and microhardness values decreased in both BD and MTA groups.

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Ethical approval

This article does not contain any studies with human participants or animals performed by any of the authors.

Informed consent

For this type of study, formal consent is not required.

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Conflicts of interest

There are no conflicts of interest.

References

1. Hursh KA, Kirkpatrick TC, Cardon JW, Brewster JA, Black SW, Himel VT, *et al*. Shear bond comparison between 4 bioceramic materials and dual-cure composite resin. J Endod 2019;45:1378-83.
2. Law AS. Considerations for regeneration procedures. J Endod 2013;39:S44-56.
3. Kayahan MB, Nekoofar MH, Kazandağ M, Canpolat C, Malkondu O, Kaptan F, *et al*. Effect of acid-etching procedure on selected physical properties of mineral trioxide aggregate. Int Endod J 2009;42:1004-14.
4. Mcdonald RE, Avery DR, Dean JA. Treatment of Deep Caries, Vital Pulp Exposure, and Pulpless Teeth. 9th ed. Missouri: Mosby Elsevier; 2011.
5. Parirokh M, Torabinejad M. Mineral trioxide aggregate: A comprehensive literature review –Part I: Chemical, pysical, antibacterial properties. J Endod 2010;36:16-27.
6. Alhadainy HA. Root perforations. A review of literature. Oral Surg Oral Med Oral Pathol 1994;78:368-74.
7. Karabucak B, Li D, Lim J, Iqbal M. Vital pulp therapy with mineral trioxide aggregate. Dent Traumatol 2005;21:240-3.
8. Tunca YM, Aydin C, Ozen T, Seyrek M, Ulusoy HB, Yildiz O. The effect of mineral trioxide aggregate on the contractility of the rat thoracic aorta. J Endod 2007;33:823-6.
9. Fuks AB. Current concepts in vital primary pulp therapy. Eur J Paediatr Dent 2002;3:115-20.
10. Torabinejad M, Hong CU, McDonald F, Pitt Ford TR. Physical and chemical properties of a new root-end filling material. J Endod 1995;21:349-53.
11. Odabas ME, Bani M, Tirali RE. Shear bond strengths of different adhesive systems to Biodentine. ScientificWorldJournal 2013;2013:626103.
12. Sultana N, Nawal RR, Chaudhry S, Sivakumar M, Talwar S. Effect of acid etching on the micro-shear bond strength of resin composite-calcium silicate interface evaluated over different time intervals of bond aging. J Conserv Dent 2018;21:194-7.
13. Koubi G, Colon P, Franquin JC, Hartmann A, Richard G, Faure MO, *et al*. Clinical evaluation of the performance and safety of a new dentine substitute, Biodentine, in the restoration of posterior teeth -a prospective study. Clin Oral Investig 2013;17:243-9.
14. Raskin A, Eschrich G, Dejou J, About I. *In vitro* microleakage of Biodentine as a dentin substitute compared to Fuji II LC in cervical lining restorations. J Adhes Dent 2012;14:535-42.
15. Rajasekharan S, Martens LC, Cauwels RGEC, Anthonappa RP, Verbeeck RMH. Biodentine™ material characteristics and clinical applications: A 3 year literature review and update. Eur Arch Paediatr Dent 2018;19:1-22.
16. Deepa VL, Dhamaraju B, Bollu IP, Balaji TS. Shear bond strength evaluation of resin composite bonded to three different liners: TheraCal LC, Biodentine, and resin-modified glass ionomer cement using universal adhesive: An *in vitro* study. J Conserv Dent 2016;19:166-70.
17. Colak H, Tokay U, Uzgur R, Uzgur Z, Ercan E, Hamidi MM. The effect of different adhesives and setting times on bond strength between Biodentine and composite. J Appl Biomater Funct Mater 2016;18:14:e217-22.
18. Kayahan MB, Nekoofar MH, McCann A, Sunay H, Kaptan RF, Meraji N, *et al*. Effect of acid etching procedures on the compressive strength of 4 calcium silicate-based endodontic cements. J Endod 2013;39:1646-8.
19. Elnaghy AM. Influence of acidic environment on properties of biodentine and white mineral trioxide aggregate: A comparative study. J Endod 2014;40:953-7.
20. Butt N, Talwar S, Chaudhry S, Nawal RR, Yadav S, Bali A. Comparison of physical and mechanical properties of mineral trioxide aggregat and Biodentine. Indian J Dent Res 2014;25:692-7.
21. Deepthi V, Mallikarjun E, Nagesh B, Mandava P. Effect of acidic pH on microhardness and microstructure of theraCal LC, endosequence, mineral trioxide aggregate, and biodentine when used as root repair material. J Conserv Dent 2018;21:408-12.
22. Bolhari B, Nekoofar MH, Sharifian M, Ghabrai S, Meraji N, Dummer PM. Acid and microhardness of mineral trioxide aggregate and mineral trioxide aggregate-like materials. J Endod 2014;40:432-5.
23. Mohebbi P, Asgary S. Effect of pH on physical properties of two endodontic biomaterials. J Conserv Dent 2016;19:212-9.
24. Wang Z, Ma J, Shen Y, Haapasalo M. Acidic pH weakens the microhardness and microstructure of three tricalcium silicate materials. Int Endod J 2015;48:323-32.
25. Namazikhah MS, Nekoofar MH, Sheykhrezae MS, Salariyeh S, Hayes SJ, Bryant ST, *et al*. The effect of pH on surface hardness and microstructure of mineral trioxide aggregate. Int Endod J 2008;41:108-16.
26. Kaup M, Schafer E, Dammaschke T. An in vitro study of different material properties of Biodentine compared to ProRoot MTA. Head Face Med 2015;2:11:16.
27. Majeed A, Alshwaimi E. Push-Out bond strength and surface microhardness of calcium silicate-based biomaterials: An in vitrostudy. Med Princ Pract 2017;26:139-45.
28. Caronna V, Himel V, Yu Q, Zhang JF, Sabey K. Comparison of the surface hardness among 3 materials used in an experimental apexification model under moist and dry environments. J Endod 2014;40:986-9.