Original papers

Bond strength of Biodentine to a resin-based composite at various acid etching times and with different adhesive strategies

Wytrzymałość wiązania bioaktywnego substytutu zębiny z żywicą kompozytową w różnym czasie wytrawiania kwasowego i przy zastosowaniu różnych technik adhezyjnych

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Abstract

Background. Biodentine® is a bioactive calcium silicate-based material, with better strength parameters, an easier application method and a shorter setting time than mineral trioxide aggregate (MTA). The bond strength between Biodentine and the composite material is essential for the durability of the layered restoration.

Objectives. The objective of this study was to evaluate the bond strength of Biodentine to a resin-based composite at various acid etching times and with different adhesive strategies.

Material and methods. In the 1st part of the experiment, the specimens were divided into 2 groups: the adhesive was applied in the total-etch (TE) and self-etch (SE) techniques. In the TE group, 37% orthophosphoric acid was applied after 30 s (TE 30) and 240 s (TE 240). In the SE group, the SE system was applied for 30 s (SE 30) and 240 s (SE 240). In the 2nd part, the SE systems Clearfil® SE Bond and Clearfil S3 Bond Plus were evaluated (the CSE and CS3 groups, respectively). In each group, the adhesive system was applied in 1 (the CSE 1 and CS3 1 subgroups) or 2 layers (the CSE 2 and CS3 2 subgroups). The specimens were subjected to a shear bond strength (SBS) test in a universal testing machine.

Results. Shear bond strength was higher after a prolonged etching procedure in the TE (TE 30: 2.51 MPa, TE 240: 9.39 MPa) and SE techniques (SE 30: 5.92 MPa, SE 240: 7.89 MPa). A statistically significant influence was detected in relation to 30 s of surface preparation time for Clearfil S3 Bond Plus ($p < 0.001$). Greater bond strength was revealed after the application of 1 layer of the Clearfil S3 Bond Plus single-bottle system (CS3 1: 6.42 MPa).

Conclusions. The SBS of Biodentine to the composite depends on both the etching time and the mode of application of the adhesive systems. Higher bond strength was obtained for the SE adhesive in a shorter application time.

Key words: shear bond strength, Biodentine, adhesive systems, calcium silicate cement

Słowa kluczowe: wytrzymałość wiązania na ścinanie, bioaktywny substytut zębiny, systemy wiążące, cement krzemianowo-wapniowy

Introduction

The goal of biological treatment is to maintain healthy, vital pulp and to promote its repair ability using bioceramic materials. One of the representatives of this new class of materials is Biodentine®, a tricalcium silicate bioactive cement with mechanical properties similar to those of healthy dentin.[1] Its microhardness as well as flexural and compressive strength allow us to name this material a 'dentin substitute'. Observations made in several studies suggest that Biodentine demonstrates better mechanical properties, easier application and a shorter setting time than previously used mineral trioxide aggregate (MTA).[2,3] Biodentine also has a broad-spectrum antibacterial effect and low cytotoxic activity.[4,5] Due to the brittleness and unsatisfactory esthetics of Biodentine, it is necessary to make the final filling with another restorative material, usually a composite one. The bond strength between Biodentine and the composite plays a vital role in achieving the final, lasting restoration, which depends to a large extent on the procedures associated with the application of the adhesive system.

There is a divergence of views about the time after which the bioceramic material should be covered with the composite and the appropriate adhesive strategy. Some authors recommend performing the final filling after at least 2 weeks, which allows Biodentine to obtain full maturity.[6,7] Others argue that the final reconstruction is possible immediately after the application of Biodentine.[8] There is also no consensus as to the type of the adhesive strategy which would give the highest bond strength between Biodentine and the composite. Some authors suggest the superiority of the self-etch (SE) systems over the total-etch (TE) technique,[9,10] while other publications indicate that the TE technique provides higher bond strength of the tested materials,[8,11] or that the choice of the adhesive strategy is irrelevant.[6,12]

The aim of the study was to evaluate the bond strength of Biodentine to a resin-based composite at various acid etching times and with different adhesive strategies.

Material and methods

The materials used in the study are presented in Table 1. The method of their application was consistent with the manufacturers' recommendations.

A total of 250 cylindrical acrylic blocks (Villacryl® IT; Zhermapol, Warszawa, Poland) with a central hole of 4 mm in both diameter and depth were prepared, and then filled with Biodentine (Septodont, Saint-Maur-des-Fossés, France) with slight excess. After 12 min (the Biodentine setting time), the surfaces of the samples were sanded wet with abrasive papers of 320 C and 400–600 C, and rinsed with water. All samples were stored in 0.9% NaCl. The study consisted of 2 parts.

In the 1st experiment, the specimens were divided into 2 groups depending on the adhesion system used: the TE technique – Adper® Single Bond (3M ESPE, Seefeld, Germany) (the TE group) or the SE technique – Clearfil® S3 Bond Plus (Kuraray Medical, Kurashiki, Japan) (the SE group). In the TE group, the samples were etched for 30 s (TE 30) and 240 s (TE 240), and then the Adper Single Bond adhesive system was applied. In the SE group, the SE adhesive Clearfil S3 Bond Plus was applied to the Biodentine surface for 30 s (SE 30) and 240 s (SE 240). The samples were dried with a gentle stream of air for 10 s

Table 1. Materials used in the study

Material	Product description	Composition	Manufacturer
Biodentine	bioactive dentin substitute	powder: tricalcium silicate, dicalcium silicate, calcium carbonate and oxide filler, iron oxide, zirconium oxide radiopacifier liquid: calcium chloride accelerator, hydrosoluble polimer water reducing agent	Septodont, Saint-Maur-des-Fossés, France
Adper Single Bond	etch-and-rinse dental adhesive	Bis-GMA, HEMA, dimethacrylates, methacrylate functional copolymer of polyacrylic and polyitaconic acids, ethanol, photoinitiator system, water	3M ESPE, Seefeld, Germany
Clearfil S3 Bond Plus	1-step SE adhesive system (single-bottle)	Bis-GMA, HEMA, hydrophilic aliphatic dimethacrylate, ethanol, sodium fluoride, colloidal silica, DL-Camphorquinone, 10-MDP, initiators, accelerators, water	Kuraray Medical, Kurashiki, Japan
Clearfil SE Bond	2 step SE adhesive system (2-bottle)	primer: 10-MDP, HEMA, hydrophilic dimethacrylate, N,N-diethanol-p-toluidin, camphorquinone, water bond: 10-MDP, HEMA, Bis-GMA, hydrophilic dimethacrylate, N,N-diethanol-p-toluidin, camphorquinone, colloidal silica, diethanol	Kuraray Medical, Kurashiki, Japan
Filtek Z250	microhybrid universal restorative	zirconia/silica, Bis-GMA, Bis-EMA, UDMA, TEGDMA	3M ESPE, St. Paul, USA
Blue Etch®	etching gel	36% orthophosphorid acid	Cerkamed, Stalowa Wola, Poland

Bis-EMA – bisphenol A ethoxylated dimethacrylate; Bis-GMA – bisphenol A glycidyl methacrylate; HEMA – 2-hydroxyethyl methacrylate; SE – self-etch; TEGDMA – triethylene glycol dimethacrylate; UDMA – urethane dimethacrylate; 10-MDP –10-methacryloyloxydecyl dihydrogen phosphate.

and polymerized for 20 s. In the control group, the Adper Single Bond system was used, which was applied without the prior etching of Biodentine.

In the 2nd part of the study, the specimens were also divided into 2 groups according to the SE system applied: the 2-bottle Clearfil SE Bond (Kuraray Medical) (the CSE group) or the single-bottle Clearfil S3 Bond Plus (the CS3 group). In each group, the adhesive system was applied in 1 layer (subgroups CSE 1 and CS3 1) or in 2 layers (subgroups CSE 2 and CS3 2). In the control group, a composite material was applied directly to Biodentine.

In all groups, both in the 1st and the 2nd part of the study, a light-cured composite material Filtek™ Z250 (3M ESPE, St. Paul, USA) was used. The material was applied through a canal of a non-translucent, cylindrical, silicone matrix of an internal diameter and height of 3 mm, and polymerized with 2-millimeter-thick layers. The light-emitting diode light-curing unit Demi® Plus (Kerr, Orange, USA) with an intensity of 1200 mW/cm^2 was used. The shear bond strength (SBS) of Biodentine to the composite was assessed by means of a universal testing machine Zwick/Roell Z020 (Zwick/Roell, Ulm, Germany) at the crosshead speed of 2 mm/min.

The experiment was performed by 2 operators; the first was responsible for the specimen preparation, the other for the testing. The data was subjected to statistical analysis. Average measures were calculated: mean (*M*) and measures of variation (standard deviation – *SD* and coefficient of variation). The Friedman analysis of variance (ANOVA) and generalized linear models in the multiagent system with flexible standard errors were used. The results of the relevant procedures were considered statistically significant if $p < 0.05$.

Results

The values of the SBS of Biodentine to the composite material depending on the time of etching and the adhesive system application techniques are summarized in Tables 2 and 3 and illustrated in Fig. 1–3.

The results of the 1st part of the study (Table 2, Fig. 1,2) indicate that the etching time had a significant effect on the bond strength both in the TE group ($p < 0.001$) and the SE group ($p = 0.033$). After the prolongation of the etching time, the bond strength increased in the case of the TE (from TE 30: 2.51 MPa to TE 240: 9.39 MPa) and SE techniques (from SE 30: 5.92 MPa to SE 240: 7.89 MPa). The differences between the 2 assessed adhesive systems were not statistically significant ($p = 0.257$), which was confirmed by means of the multivariate analysis. In the univariate analysis, SBS was higher after 30 s of application of Clearfil S3 Bond Plus ($p < 0.001$). However, there were no statistically significant differences in SBS between the TE system after the 240-second etching time and the SE system after 240 s of application ($p = 0.456$).

Table 2. Descriptive statistics of the shear bond strength (SBS) values T_{max} [MPa] with regard to the bonding system and different adhesive strategies

Group	Subgroup	Statistical parameter	
		M	*SD*
Adper Single Bond (TE)	TE 30	2.51	2.18
	TE 240	9.39	6.65
	overall	5.82	5.94
Clearfil S3 Bond Plus (SE)	SE 30	5.92	3.21
	SE 240	7.89	4.29
	overall	6.98	3.93
Control	–	6.89	2.97

M – mean; *SD* – standard deviation; TE – total-etch; 30, 240 – etching time [s].

Table 3. Descriptive statistics of the shear bond strength (SBS) values T_{max} [MPa] with regard to the type of the adhesive system and the number of layers of the adhesive system

Group	Subgroup	Statistical parameter	
		M	*SD*
Clearfil S3 Bond Plus (single-bottle)	CS3 1	6.42	4.60
	CS3 2	5.40	3.61
Clearfil SE Bond (2-bottle)	CSE 1	2.88	2.46
	CSE 2	1.38	0.67
Control	–	2.27	1.94

In the 2nd part of the study (Table 3, Fig. 3), no statistically significant differences were observed in the CS3 group between the CS3 1 and CS3 2 subgroups ($p = 0.550$), while in the CSE group, the difference between the CSE 1 and CSE 2 subgroups was statistically significant ($p = 0.039$; the Mann–Whitney *U* test). Higher SBS was observed after a single application of the Clearfil S3 Bond Plus system (CS3 1: 6.42 MPa). The application of the 2nd layer of the SE system did not increase the bond strength. The differences between the alternative surface preparation methods – relative to the control system – were highly statistically significant for both adhesive systems used in the experiment ($p < 0.001$; the Kruskal–Wallis test).

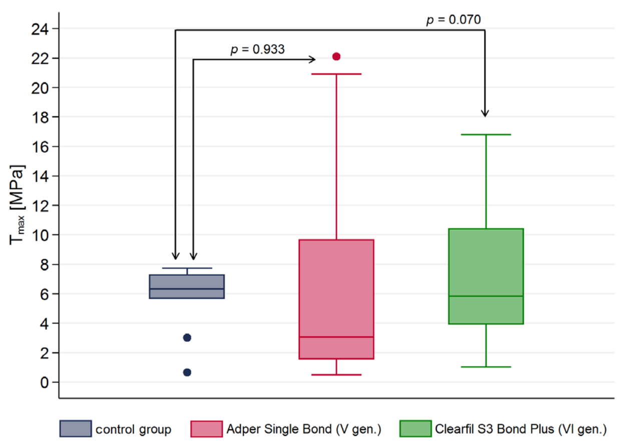

Fig. 1. Distribution of the shear bond strength (SBS) values in relation to the type of the bonding system and the control group

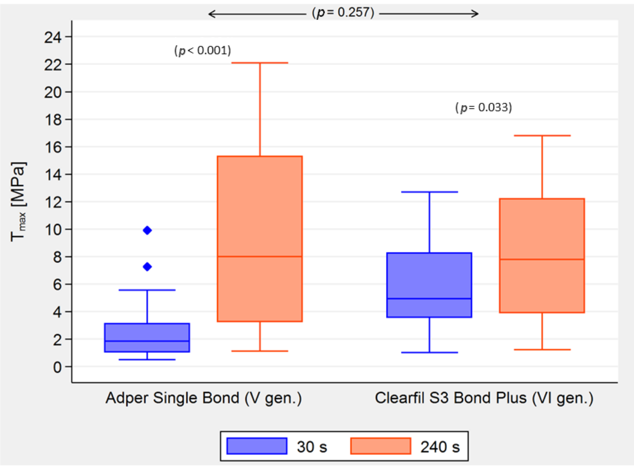

Fig. 2. Distribution of the shear bond strength (SBS) values in relation to the type of the bonding system and the application time

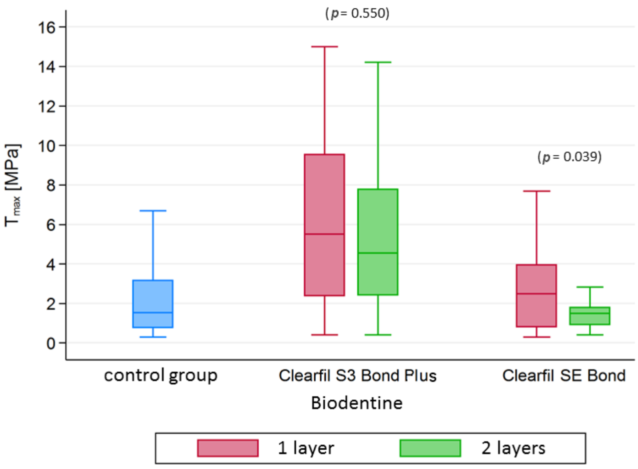

Fig. 3. Distribution of the shear bond strength (SBS) T_{max} [MPa], broken down by the type of the adhesive system and the number of layers of the adhesive system vs the control system

Discussion

During the reconstruction of lost tooth tissues, the most advantageous clinical variant is to carry out the entire procedure at one visit. The durability of such a reconstruction, apart from the materials used, is influenced by the quality and sufficiency of their bond. The use of Biodentine requires, as the manufacturer suggests, covering it with a final material after the prior use of an adhesive agent. For this reason, the aim of the present study was to evaluate the SBS of Biodentine to a composite material. The research concerned the assessment of the bond strength quality after the prolongation of the etching time or the application of the SE system as well as the comparison of SE systems with different clinical application protocols, applied in 1 or 2 layers.

Bond durability may be affected by the surface conditioning effect of calcium silicate cements.[13] Etching with 37% orthophosphoric acid is the first procedure in the TE technique before the insertion of a composite material that is to strengthen the bond between the materials and prevent microleakage. The examination of Biodentine under a scanning electron microscope confirmed the structural and chemical changes occurring after 20 s of etching with 37% orthophosphoric acid. According to previous research, the enhanced micromechanical connection could affect the bond strength of Biodentine with a resin composite material.[3] Kayahan et al. assessed the effect of the etching procedure on the compressive strength of calcium silicate materials: Angelus® MTA, ProRoot® MTA, the calcium-enriched mixture (CEM) cement, and Biodentine.[14] The study was carried out 7 days after the preparation of the materials. Biodentine turned out to be the material with the highest compressive strength ($p < 0.0001$) among all tested cements. The quality and durability of the bond between pulp capping and restorative materials is essential for the success of the treatment. As far as the choice of the adhesive strategy is concerned, prior, well-documented studies assessing the bond strength of a composite to MTA suggested the superiority of the TE technique over the SE systems.[15–17] However, reports on similar studies regarding Biodentine are ambiguous.

Cengiz and Ulusoy presented interesting results regarding the microshear bond strength (μSBS) of Biodentine and other material from the silicate-based group – TheraCal LC® to different glass-ionomer and composite materials, using the TE and SE techniques.[8] The authors noted higher μSBS in the groups in which they applied the Prime & Bond NT® TE adhesive system (13.99 MPa) or Scotchbond® Universal Adhesive (13.25 MPa) to Biodentine, while lower values were observed in the Biodentine–Clearfil SE Bond SE system (11.45 MPa). Similarly, Meraji and Camilleri assessed the SBS between Biodentine, TheraCal LC and Fuji IX®, but used different bonding systems in the TE (ExciTE® F) and SE techniques (AdheSE® One F).[11] In the group in which they used the SE primer, the composite resin was lost from all Biodentine samples during thermocycling, while higher and measurable values were noted in the group where they used the TE technique (4 MPa).

On the contrary, different conclusions were presented by Çolak et al.[9] They compared the SBS between a composite and Biodentine with different adhesives – TE (Prime & Bond NT) and 2 SE (Clearfil S3 Bond and Adper Prompt L-Pop®) in 2 periods: 9 min and 48 h after the preparation of Biodentine. The highest SBS values were observed in the group with the SE Clearfil S3 Bond, regardless of the time elapsed since its preparation (13.32 MPa after 9 min and 15.09 MPa after 48 h). Odabaş et al. used 3 adhesive systems for the study: the TE Prime & Bond NT, the 2-step SE Clearfil SE Bond and the 1-step SE Clearfil S3 Bond. The tests were carried out 12 min and 24 h after the preparation of the Biodentine material.[10] The highest bond strength (19.56 MPa) was found in the 2-stage SE system after 24 h. According to our study, the highest bond strength (5.92 MPa) was also noted for the SE system, but used in the 1-stage strategy, with the application time of 30 s. Odabaş et al. reported that the lowest values were recorded in the group where the TE system was used after 12 min,[10] which was confirmed by our earlier study[18] and the present one. Hashem et al. pointed out that the choice of the adhesive strategy is not that important for improving the reliability of the bond strength of a resin composite to Biodentine.[6]

As it can be seen, in the published literature there is no consensus as to the type of the adhesive system that would guarantee the highest bond strength. What is important is the time that has elapsed since the preparation of Biodentine, necessary to achieve the internal maturity of the material. Alsubait carried out a study to assess the compressive strength of Biodentine, ProRoot® MTA and the endosequence bioceramic root repair material-fast set putty (ERRM-FSP®) 24 h and 7 days after the preparation.[19] Significantly higher values of compressive strength were obtained after longer time (7 days), which could be related to the hydration reaction, crucial for binding materials based on calcium silicates. These conclusions coincide with the previous reports by Kayahan et al. on MTA, which showed that extending the time of binding of a bioactive material before its etching resulted in its optimal physical properties.[14] Similar observations were presented in the work by Tulumbaci et al., who evaluated the SBS of Biodentine and MTA to a composite (Filtek Z250), compomer (Dyract XP®) and the resin-modified glass-ionomer cement (RMGIC) 72 h after the preparation of materials.[20] The authors obtained significantly lower values of the SBS of Biodentine compared to MTA, which was explained by the incomplete maturation of the material. According to the manufacturer's recommendations, the Biodentine initial setting reaction takes approx. 12 min, but it can take from 2 to 4 weeks to achieve full maturation.[21] Similar conclusions were also formulated by Deepa et al., who obtained in their study the lowest SBS values in the Biodentine–composite group, evaluated immediately after 12 min, in comparison with the TheraCal LC–composite and RMGIC–composite groups.[22]

On the contrary, different results comparing the SBS of 3 different liner flowable composites to MTA and Biodentine at 3 different time intervals were published by Schmidt et al.[23] The tests were carried out 3 min, 15 min and 2 days after mixing the materials based on calcium silicate. The obtained results did not confirm the increase of SBS, a longer waiting time after mixing did not increase the adhesion and there were no statistically significant differences between the groups assessed. It is difficult to comment on the presented results, because in our study, Biodentine was evaluated after 12 min, which is in accordance with the waiting period suggested by the manufacturer, taking into account the use of a tricalcium silicate cement in the clinical procedure.

The discrepancies in the data from other studies may also result from the type of the adhesive system used. Neelakantan et al. used 3 different adhesive systems: a 1-stage TE system (Prime & Bond NT), a 2-stage SE system (AdheSE) and the 1-step SE system Clearfil S3 Bond.[24] Investigating the combination of MTA and a composite material, they obtained the highest SBS for the group in which they applied a single-step SE adhesive system. The authors paid particular attention to 2 parameters: pH and the type of the solvent. The pH value for the 2-stage SE AdheSE system is 1.4, while for the single-step SE system Clearfil S3 Bond – 2.0. Their results coincide with the reports by Bayrak et al. and Inoue et al., and confirm that low pH of the adhesive system translates into lower SBS.[25,26] Shin et al., assessing the bond strength of MTA and a composite with 4 adhesive systems, among them AdheSE and Clearfil SE Bond, with a pH of 1.4 and 1.9, respectively, noted the highest bond strength for AdheSE single-step system with lower pH, thus questioning the importance of pH as a dominant contributor to the superior bond strength.[13]

In the aforementioned study, Çolak et al. used SE systems with different functional monomers and different pH (Clearfil S3 Bond and Adper Prompt L-Pop).[9] Clearfil S3 Bond, pH 2.7, is a mild SE agent that guarantees greater bond strength when compared with Adper Prompt L-Pop, pH 0.8. The authors also concluded that the 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) acid monomer contained in this system could affect bond strength, since it can bind chemically to the calcium ions present in Biodentine, creating chemical adhesion and improving micromechanical attachment. However, they did not record statistically significant differences in SBS measured at different time intervals.

In our study, higher SBS was obtained after a single application of Clearfil S3 Bond Plus in comparison to Clearfil SE Bond (6.42 MPa vs 2.88 MPa). In addition, this SE adhesive allowed higher strength in the 30-second application time compared with the TE system evaluated at the same time (5.92 MPa vs 2.51 MPa). It should also be emphasized that the Clearfil S3 Bond Plus system used in this study, with the highest SBS, contains water and ethanol as the solvent. This may favor better wettability of a cement based on silicates and create its effect on bond strength.

The composition of Biodentine is similar to that of MTA. The hydration of both materials is similar, hence it may be assumed that the exposure of Biodentine to low pH, e.g., when applying orthophosphoric acid, may affect the chemical binding process by disrupting the hydration of tricalcium silicates and adversely affecting its microstructure. Milder etching performed in a shorter period, according to Kayahan et al., may cause less selective loss of matrix and better visibility of the crystalline structures, thus encouraging successful adhesion through micromechanical retention.[14]

Conclusions

Within the limitations of the study, it can be concluded that the SBS of Biodentine to a composite material depends on the time of etching and on the application of the adhesive strategies. There has been found an increase in bond strength after the prolongation of the etching time with respect to the TE and SE techniques. The SE bonding system allowed for greater SBS in a shorter application time. In the assessment of the impact of the number of self-adhesive layers, greater bond strength was obtained after a single application of the 1-bottle system; the application of the 2nd layer did not affect the bond strength between Biodentine and the composite.

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