

Research Article

SHEAR BOND STRENGTH OF DIFFERENT CALCIUM SILICATE BASED CEMENTS TO COMPOSITE AND COMPOMER

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ABSTRACT

Objective: Shear bond strength (SBS) of different calcium silicate cements (CSC) with different adhesive systems and restoratives was evaluated.,

Materials and methods: NeoMTA2, NeoPutty and TheraCalPT were used as CSC.120 acrylic blocks with a hole in the middle were prepared and divided into three groups (n=40) depending on the CSC used. CSCs were placed in the prepared cavities. All groups were divided into two subgroups and adhesives (Prime&BondNT) and Universal adhesive (Scotchbond) were applied. Then, composite (n=10) and compomer (n=10) were applied and polymerized. The prepared samples were kept at 37°C in a 100% humid environment for 24 hours and SBS tests were performed with an universal testing device. Fracture types were evaluated with SEM and stereomicroscope.

Results: TheraCalPT had statistically significant increased SBS values when compared to other materials (p<0.05). On the other hand, SBS values of NeoMTA and NeoPutty were insignificant (p>0.05). The difference between the adhesive systems and the restorative materials themselves was not significant (p>0.05).

Conclusion: The ease of use of TheraCal PT and its strong bonding ability with resin restorative materials may provide support for the idea that it is suitable for pulp therapies. However, since in vitro environmental conditions do not reflect intraoral conditions, it must be supported by clinical studies to understand the actual performance and clinical usability.

Keywords: NeoMTA, NeoPutty, TheraCal PT, composite, compomer, shear bond strength

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INTRODUCTION

Vital pulp treatment aims to eliminate microbial irritation by placing a impermeable dental biomaterial to protect the exposed dentin and pulp from external stimuli, through appropriate caries management, and thus prevent any new bacterial damage that may occur. The material to be placed on the pulp must be a biocompatible material that can maintain the vitality of the pulp, prevent bacterial leakage, be resistant to forces during restoration placement and function, and ensure the formation of a dentin bridge. In this way, it is aimed for the pulp to heal itself and maintain its vitality and function (1).

The search for an ideal pulp capping agent for vital pulp treatments has continued for many years, and in this process, calcium hydroxide, dental bonding agents, formocresol, ferric sulfate, mineral trioxide aggregate (MTA) and MTA-like biomaterials such as calcium silicate, calcium phosphate and calcium aluminate-based cements have been suggested (2).

Calcium hydroxide ($\text{Ca}(\text{OH})_2$) has been the first preferred material in vital pulp treatment for many years. The advantages of calcium hydroxide are its antibacterial properties, its effect on pulp healing and repair, its stimulation of dentin formation, its ability to activate alkaline phosphatase by neutralizing acid products, its low cost and ease of use (3). However, calcium hydroxide has some disadvantages such as high solubility, water absorption and dissolution in the presence of moisture, low modulus of elasticity, low compressive strength and low thermal conductivity, poor adhesion to dental structures, restorative materials and dentin, superficial pulp necrosis, tunnel defects in the dentin bridge and degradation after acid etching. Due to the inadequate physical properties of calcium hydroxides, it has brought about the need to investigate new materials that have better sealing properties, are easier to use, and can stimulate the complete dentinal bridge formation. For this reason, biomaterials have been developed as alternatives for direct pulp capping in the last two decades (4).

Calcium silicate-based materials have been considered the most suitable material for pulp capping as a surface-active hard tissue substitute due to its excellent bioactivity and biocompatibility. Calcium silicate-based materials began their development with Portland cement (PC) in 1878, and their use became widespread with the invention of MTA in the 1990s. Portland cement, which constitutes the main component of MTA,

contains tetracalcium aluminoferrite, dicalcium silicate, tricalcium aluminate, tricalcium silicate, and gypsum and bismuth oxide as a radiopaque substance. MTA is accepted as the gold standard material in vital pulp treatments due to its properties such as biocompatibility, bioactivity/biomineralization, low solubility and desirable biological properties such as hydrophilicity. Its antibacterial activity thanks to its high pH value, stimulation of reparative dentin formation, low solubility, high sealability, non-mutagenic, genotoxic, carcinogenic, biocompatible and bioactive material have made MTA an attractive material. In addition to these advantages, different MTA formulations have been developed due to the disadvantages of MTA such as long hardening time, difficulty of use, coloration and high cost (5).

NeoMTA 2 (NuSmile Avalon Biomed, Bradenton, FL, USA) is a bioactive material that stimulates hard tissue formation by releasing Ca^{+2} and OH^- ions, like calcium hydroxide and other calcium silicate-containing cements, and provides antibacterial activity at alkaline pH. It triggers the healing process by stimulating hydroxyapatite in dentin (5, 6).

To reduce the effect of hand mixing on the setting reaction and to simplify clinical application, premixed calcium silicate cements have been developed as an alternative to traditional powder-liquid cements. Premixed tricalcium silicate and dicalcium-based NeoPUTTY is used as a retrograde filling material in vital pulp procedures, apexification, root resorption and perforation repair (6). NeoPUTTY, which is easy to use and has high bioactivity, is a resin-free bioceramic. The material stimulates the formation of hydroxyapatite by releasing Ca^{+2} and OH^- ions from the surface. It has an alkaline pH when applied, thus providing antibacterial activity (7).

Because MTA and similar hydraulic calcium silicate-based materials exhibit a water-based chemical structure and therefore create insufficient micromechanical adhesion to the overlying resin restoration, new light-curing resin-modified tricalcium silicate materials are being formulated for pulp treatment procedures. For this purpose, TheraCal PT (ThPT; Bisco Inc., Schaumburg, IL, USA), a new dual-cured resin modified calcium silicate material designed for pulpotomy, has recently been introduced into clinical use with its biocompatibility, radiopacity, ease of use and calcium release feature (8).

Versatile universal adhesive (UA) systems introduced to the market are designed to provide bonding to tooth structures with both etch-and-rinse (ER) and self-etch (SE) techniques.

Glass ionomer cements and resin modified glass ionomers are frequently used for permanent restorations to be applied after vital pulp treatment, especially in pediatric dentistry, due to their chemical adhesion to dental tissues, fluoride release properties and the fact that they do not require much sensitivity in their application. However, in cases where chewing forces are intense or aesthetics are important, composite and compomer resins are the first choice (9).

Our research; It aims to comparatively evaluate the shear bond strength (SBS) of three different calcium silicate containing vital pulp treatment materials, with two different adhesive systems, to composite and compomer restorative materials frequently used in the clinic.

The null hypotheses of this research are (a) the bond strength of TheraCal PT, a resin-containing material, is significantly higher than the other two materials, (b) there is no significant difference in bond strength between NeoMTA 2 and NeoPUTTY, (c) the bond strength of groups using the ER adhesive system is significantly different from groups using the universal adhesive system, (d) there is no significant difference between the bond strength of the materials to the composite and the bond strength to the compomer.

MATERIALS AND METHODS

Materials Used

In our research, 3 different biomaterials (Group 1: NeoMTA 2, Group 2: NeoPUTTY, Group 3: TheraCal PT), 2 different restorative materials (compomer and composite resin) and one self-etch and one universal adhesive material were used.

Table 1. Materials used in the study

Material	Content	Type / Application Method	Manufacturer
NeoMTA 2	Di and Tricalcium silicate / Tantalum oxide / tricalcium aluminate	Powder-liquid gel	NuSmile, Houston, TX, USA
NeoPutty	Di and Tricalcium silicate / calcium aluminate / Tantalum oxide / Tricalcium aluminate / calcium sulfate / proprietary organic liquid and stabilizers	Premixed paste	NuSmile, Houston, TX, USA
TheraCal PT	Base: Silicate glass mixed cement / polyethylene glycol / dimethacrylate / BisGMA / barium zirconate Catalyst: Barium zirconate / ytterbium fluoride / initiator	Syringe	Bisco Inc., Schaumburg, IL, USA
Prime&Bond NT	Di and Trimethacrylate Resins / PENTA /Bis-GMA/ Nanofillers - Amorphous Silicon Dioxide / Photoinitiators / Stabilizer / Cetylamine Hydrofluoride / Acetone	Etch&Rinse Adhesive	Dentsply, Caulk, Germany
Scotchbond Universal	10-MDP monomer / dimethacrylate resins / HEMA / Methacrylate modified polyalkenoic acid copolymer / Filler / Ethanol,Water / Initiators / Silane	Universal Adhesive	3M ESPE Dental Products, St. Paul, MN, USA
Compoglass F Compomer	UDMA / Polyethylene glycoldimethacrylate / CADCADMA / Silanized mix oxide / Ytterbium trifluoride / Ba-Al-Fluorosilicatecam / (silanized) Catalysts / Stabilizers / Pigments	Refill compomer/ gun application	Ivoclar Vivadent, Schaan, Liechtenstein
3M ESPE Filtek Z550 composite	Bis-GMA, UDMA, Bis-EMA / PEGDMA / TEGDMA / Zirconia and Silica fillers	Nanohybrid composite	3M ESPE, St. Paul, MN, USA

Preparation of Samples

For the SBS test, 120 acrylic blocks with cylindrical cavities (depth: 2 mm, diameter:4 mm) were prepared. NeoMTA 2 was prepared according to the manufacturer's instructions and placed in the space in the middle of the acrylic molds. NeoPUTTY was placed into the molds with appropriate hand tools. In order for the hardening reaction to occur, the molds where these two materials were applied were covered with moist cotton pellets. In accordance with the manufacturer's recommendations, it was kept at 37°C, 100% humidity for 48 hours. TheraCal PT was applied to the molds and hardened by irradiation with the Elipar™ Deepcure-L Light Device for 20 seconds. Before the adhesive application, the biomaterials were left for at least 5 days without any force to complete their hardening.

Each biomaterial group was divided into 2 subgroups for adhesive application. After acid application, Prime&Bond NT adhesive agent was applied to 20 of them according to the manufacturer's instructions. 3M Scotchbond universal adhesive agent was applied and polymerized to the other 20 according to the manufacturer's instructions.

A cylindrical polyethylene mold (diameter: 2 mm, height:2 mm) was used to place the restorative materials to be applied on the biomaterials. Each biomaterial sample was divided into 2 groups, with 10 samples in each group, according to the restorative material to be applied; Ivoclar Compoglass F compomer was placed in one group and 3M Filtek Z550 composite was placed in the other group and polymerized by irradiating with a LED light device for 20 seconds.

Evaluation of Shear Bond Strength

To measure SBS values, the samples were fixed on the universal testing device (LF Plus, LLOYD Instruments, Amatek Inc, UK). An approach speed of 1 mm/min was applied and waited until rupture occurred. Later; The test was stopped automatically and the results were calculated by the computer in Newtons. The values obtained on the test device were converted to MPa after being saved to the computer. After fracture, the fracture surfaces of all samples were examined with a stereomicroscope (SMZ 800, Nikon, Tokio, Japan) under 25x magnification. After examination, the fracture types of the samples were determined and recorded. Five of the broken samples from each biomaterial group were examined at various magnifications on the Tescan Mira3 SEM device.

Statistical Evaluation

To analyze data SPSS22.0 (SPSS, Inc., Chicago, IL, America) program was used. Since parametric test assumptions could not be fulfilled in the evaluation of the data (Shapiro-Wilk Test), Kruskal-Wallis Test was used when comparing measurements obtained from more than two independent groups. When the significance decision was made as a result of the analysis, the Mann-Whitney U Test was used to indicate which group the difference originated from, and the Mann-Whitney U Test was used when comparing the measurements obtained from two independent groups. Our data were stated in the tables as arithmetic mean, median, standard deviation, minimum value, maximum value, and the error level was taken as 0.05.

RESULTS

The mean SBS values of the groups are shown in Table 2. According to these results, TheraCal PT showed significantly higher values than the NeoMTA 2 and NeoPUTTY groups ($p < 0.05$). No statistically significant difference was detected between NeoMTA 2 and NeoPutty groups ($p > 0.05$).

Table 2. Mean and std. dev of shear bond strength of the groups without subdividing them

Groups	Mean±Std. Deflection
Group 1 (NeoMTA2)	13.51±4.88
Group 2 (NeoPUTTY)	11.58±6.93
Group 3 (TheraCalPT)*	31.90±9.57*

Among all groups, the highest average SBS value was in the TheraCal PT-UA-compomer group (34.93 ± 8.53 MPa), and the lowest average SBS value was in the NeoPUTTY-UA-compomer group (8.00 ± 6.30 MPa) was observed (Table 3).

When all groups were compared in terms of bonding strength to the composite and compomer, the difference between the groups was found to be insignificant ($p > 0.05$). When all groups were compared in terms of bond strength of UA and SE adhesive systems, the difference between the groups was found to be insignificant ($p > 0.05$) (Table 3).

Table 3. Mean and std. dev. values of shear bond strength for subgroups

Groups		Mean±Std. Dev	
		Composite	Compomer
NeoMTA2	ER	14.59±4.75	15.16± 3.93
	UA	12.80 ± 5.69	11.48 ± 4.86
NeoPUTTY	ER	12.92±7.03	13.63± 6.25
	UA	11.76 ± 7.68	8.00 ± 6.30
TheraCalPT*	ER	31.63±7.58*	29.02± 12.83*
	UA	32.03 ± 9.11*	34.93 ± 8.53*

When the types of ruptures between the biomaterial and the restorative material were evaluated, it was seen that all (100%) of the fractures were cohesive ruptures within the biomaterial. In the SEM analysis

performed on randomly selected broken sample surfaces from biomaterial groups, only the material surface could be examined because all the breaks were cohesive within the material. It was observed that TheraCal PT (Figure 3) had a more homogeneous structure and its surface was more regular and less porous than the surface of NeoPUTTY (Figure 2) and NeoMTA 2 (Figure 1). The structure of NeoMTA 2 was seen to be more hollow and heterogeneous than NeoPUTTY.

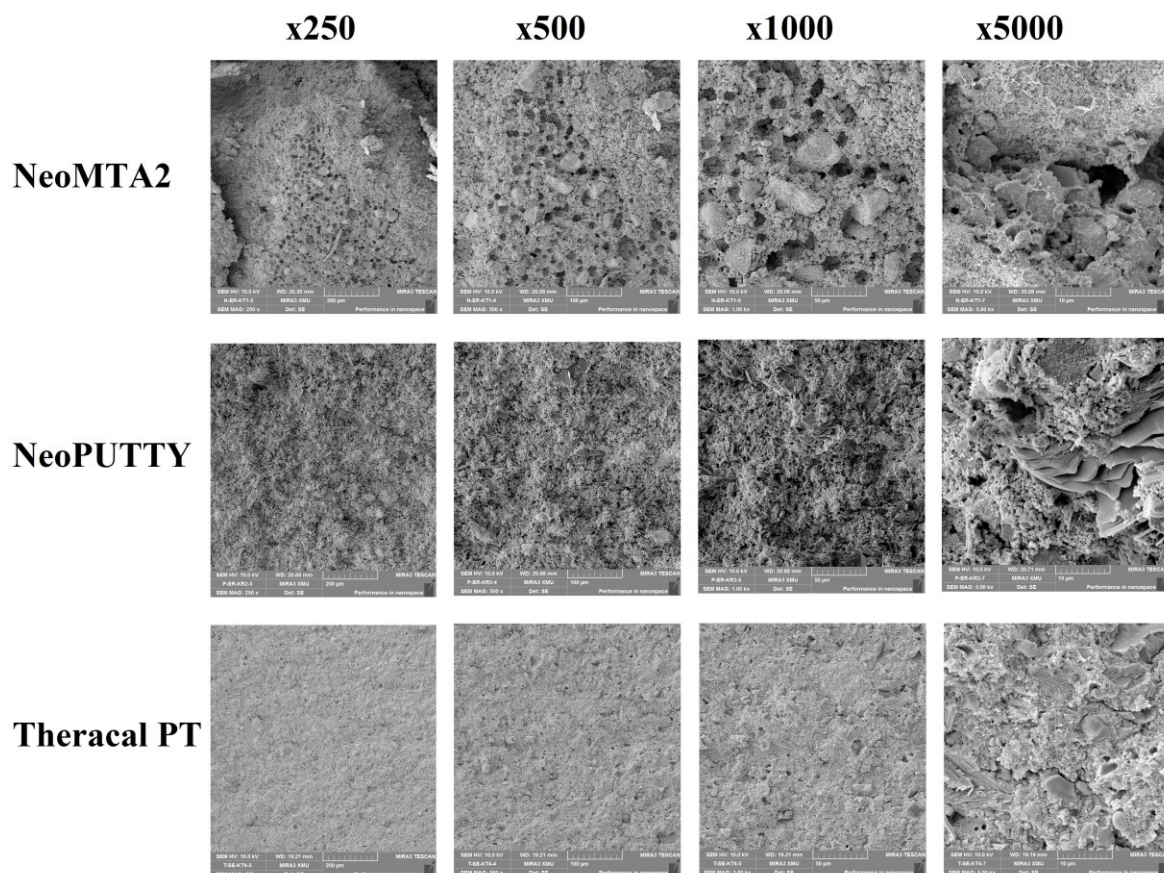


Figure 1. SEM images of NeoMTA 2, NeoPUTTY and TheraCal PT (250x, 500x, 1000x, 5000x)

DISCUSSION

Vital pulp therapy (VPT) procedure is a treatment performed by removing carious and infected tissue and placing a protective, healing, biocompatible and good covering material on the healthy and vital pulp. For the success of the treatment, a permanent restoration must be placed immediately after the treatment to

ensure and maintain good coverage. In addition, in reducing bacterial microleakage and in the long-term success of vital pulp treatment, the bond strength between the pulp material and the restorative material must be high (10).

Biomaterials containing calcium silicate have come to the fore with their advantages such as better physical properties and higher clinical success, and with the discovery of MTA, their use for many treatments and VPT has become widespread (11). The good covering ability of MTA, which is a biocompatible material with its high impermeability, low solubility, high alkaline structure, antibacterial activity and dentinogenesis properties, has a significant impact on the success of VPT. However, MTA has disadvantages such as long hardening time, difficult manipulation, and high cost. In addition to these disadvantages, problems such as being difficult to remove after hardening and the negative effect of etching on the bonding of MTA to increase the adhesion of resin-containing filling materials have led to the development of different MTA formulations (12).

NeoMTA 2, one of these developed materials, has a structure that is resistant to washing, unlike MTA, it is more radiopaque and contains tantalum oxide instead of bismuth oxide for radiopacity. The fine particle structure of the powder and the gel form of the liquid facilitate the manipulation of the material and provide comfortable working (13).

Pre-prepared calcium silicate cements have been developed as an alternative to traditional powder liquid cements in order to eliminate the mixing process of powder liquid materials, reduce their effect on the hardening reaction, facilitate their use and provide a homogeneous structure (14). Since NeoPUTTY, a premixed cement, is formulated with an organic liquid that does not contain water, its manufacturer reports that it is minimally affected by air humidity and has a longer shelf life than its counterparts (15). NeoPUTTY, which has a non-sticky, firm consistency, is resistant to washing, which creates an advantage in that there is no problem in contact with water after it is applied to the cavity (16).

Resin modified calcium silicate cements have been developed to eliminate the disadvantages of MTA and similar hydraulic cements, such as their inability to provide adequate adhesion with the placed resin restoration and the delay of the permanent restoration to complete the hardening of some MTA derivatives.

TheraCal PT, which the manufacturer states that it is designed for pulpotomy, is a resin-modified calcium silicate material and thanks to its dual cure nature, it prevents the problem of insufficient polymerization due to application thickness. The fact that this material contains resin provides better adhesion with resin restorations and has a positive effect on sealing (8).

The long-term success of VPT depends on many features of pulp capping agents, as well as providing good sealing, strong adhesion with the permanent restoration, and thus high sealing. This adhesion strength is routinely determined by shear bond tests (12).

When the literature information on the SBS of calcium silicate-based biomaterials was examined, it was seen that there were few studies (15, 17) on TheraCal PT, NeoMTA 2, NeoPUTTY. In our study, TheraCal PT showed high bond strength in both adhesive systems and restorative materials. NeoPUTTY exhibited the lowest average shear bond value in all groups.

Yavuz et al. (17) evaluated the SBS of three different calcium silicate cements (TheraCal PT, NeoPUTTY, Biodentine) with three different universal adhesive systems and reported that TheraCal PT showed the highest values and NeoPUTTY showed the lowest values. Özata et al. (15) in their study with TheraCal LC, NeoMTA 2 and NeoPUTTY; They reported the average bond strengths as TheraCal LC (23.32 Mpa), NeoMTA 2 (12.17 Mpa) and NeoPUTTY (11.37 Mpa), respectively. Ipek et al. (18) evaluated the bond strength of Biodentine, MTA Repair HP and NeoPUTTY to root dentin and stated that NeoPUTTY showed low bond values. Ipek et al. (19), in another study, evaluated the bond strength of NeoPUTTY, Biodentine and MTA Cem LC to two different bulk fill composites with and without fiber content and reported that NeoPUTTY showed the lowest bond values in both composite groups. Alqahtani et al. (20) evaluated the immediate and delayed (7 days) bonding of 4 different biomaterials (NeoPUTTY, NeoMTA 2, TotalFill BC RRM™ and ProRoot MTA) to resin-modified glass ionomer cement and flowable composite. They reported that ProRoot MTA (14.65 MPa) showed the highest average bond strength in delayed bonding in flowable composite groups, and NeoPUTTY (8.03 MPa) showed the lowest average bond strength. Şişmanoğlu et al. (21) in their study investigating the bond strength of five calcium silicate cements (MTA Angelus, Biodentine, NeoMTA, TheraCal LC, Well Root ST) to the composite with ER and SE adhesive systems, reported that the highest bond strength values

belonged to TheraCal LC, which is a resin-containing material. Akbıyık et al. (22) reported that resin-containing coating agents (Calcimol LC, ApaCal ART, TheraCal LC) showed higher bond strength than others in the shear bond test they conducted with different pulp capping materials (TheraCal LC, Dycal, ProRoot MTA, Calcimol LC, Biodentine, ApaCal ART) and different adhesive systems. It was found that the resin-containing biomaterials exhibited the highest bond strengths in these studies and that TheraCal PT, a resin-containing dual cure material, showed the highest bond strengths in our study. Thus, our first hypothesis regarding TheraCal PT was accepted. The success of TheraCal PT in the SBS test is thought to be due to the resin monomer in its forming a strong adhesion by establishing a chemical bond with the adhesive resin (17).

When the bond strength studies in the literature about the NeoPUTTY group, which showed the lowest SBS values in our research, were examined, it was reported that the NeoPUTTY material showed low bond strength, consistent with our study (17-19). When applied clinically, NeoPUTTY is a material that hardens by absorbing moisture and water from the dentinal tubules and surrounding tissues. The low binding values of NeoPUTTY in in vitro studies can be considered as the inability to achieve ideal hardening because it cannot find sufficient moisture and water when placed in acrylic molds (17). Further in vitro and in vivo studies are necessary which tests the binding success of NeoPUTTY.

When studies evaluating the bonding of biomaterials with adhesive systems were examined, different results were found. While there are studies reporting that MTA shows more successful results with ER adhesive systems (23-25), there are also studies reporting that the etching process weakens the physical properties of MTA and reduces its bonding strength (26-28). In our research, the bond strength averages of NeoMTA 2 and NeoPUTTY's ER adhesive applied samples were higher than the bond strength averages of UA applied samples; It was observed that the average of UA values of TheraCal PT groups was higher than the average of ER adhesive values.

There are a few studies comparing the bond strength of calcium silicate-containing materials to compomer and composite (25, 29, 30). Keleş et al. (29) reported in their study that composite resin showed significantly higher bond strength than compomer, while Tunç et al. (25) and Tulumbacı et al. (30) reported that there was no significant difference between the two restorative materials in their studies. In our study,

when composite resin and compomer were compared in all NeoMTA 2, NeoPUTTY and TheraCal PT groups, no statistically significant difference was seen between the two restorative materials ($p>0.05$). Therefore, our hypothesis regarding composite and component materials was accepted.

In our research, cohesive type rupture was observed in the biomaterial in all materials. For this reason, when classifying our rupture types, we classified them as deep cohesive rupture and superficial cohesive rupture. We think that another reason for the cohesive rupture in this biomaterial is the non-homogeneous stress distribution. The majority of the ruptures observed in the NeoPUTTY material were observed as superficial cohesive ruptures. We attribute the cause of superficial cohesive ruptures to the low bonding values of the material itself and the inability to complete the hardening reaction. In addition, the fact that NeoMTA 2 and NeoPUTTY materials exhibit lower values and more superficial ruptures compared to TheraCal PT may be due to the fact that MTA is water-based and has a particulate, granular and brittle structure. We think that the reason why deep cohesive breaks are mostly observed in TheraCal PT samples and SBS values are higher than previous studies may be due to the difference in surface preparation and environmental conditions. Deep breaks suggest that the bonds within the material are also strong.

When the SEM images were examined, it was seen that the surfaces of TheraCal PT and NeoPUTTY were more regular and homogeneous than the surface of NeoMTA 2. It is thought that this is because manual mixing cannot provide sufficient homogeneous mixing.

One of the limitation of this study is that in vitro environmental conditions do not reflect intraoral conditions and in vivo studies are necessary to see these effects on bonding strength values. Another limitation of this study was that one of the tested material "TheraCal PT" is a light cured material, but there is no other material that can be light cured in order to compare as calcium silicate based material.

CONCLUSION

According to the findings of our research, it is thought that TheraCal PT, a resin-containing material, can prevent treatment failure due to microleakage thanks to its high bond strength to resin restorations, and its use can be recommended. However, since our study only tests the mechanical and physical properties of

the material, more biological and histological in vitro and in vivo studies are needed for this material to be placed on the pulp. Although the average SBS values of NeoPUTTY and NeoMTA 2 materials are below the clinically accepted SBS value of 17-20 MPa (31, 32), since in vitro environmental conditions do not reflect intraoral conditions, they need to be supported by in vivo studies to understand the real performance and clinical usability of the materials.

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None

Authorship contributions

Contributed to conception FO and FKA, design FO and FKA, collecting data FKA, statistical analysis FKA, data acquisition and interpretation FO, SEM analysis FKA, writing some parts of manuscript FKA, writing and editing of the manuscript FO.

Data availability statement

The data that support the findings of this study are available from the corresponding author, [FO], upon reasonable request.

Declaration of competing interest

The authors deny any conflicts of interest related to this study.

Ethics

Cumhuriyet University Clinical Research Ethic Committee (2021-11/10).

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