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The micro-shear bond strength of new endodontic tricalcium silicate-based putty: An in vitro study

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Abstract

This study aimed to compare in vitro micro-shear bond strength (μSBS) of three different endodontic tricalcium silicate-based materials in contact with a bulk-fill resin-based composite. Thirty cylindrical resin blocks with a hole in the centre (2 mm in depth and 4 mm in diameter) were manufactured with a 3D printer and divided into three groups ($n = 10$), depending on the calcium silicate cement used: light curing TheraCal LC (Bisco, Schaumburg, IL, USA), liquid–powder NeoMTA 2 (NuSmile Avalon Biomed, Bradenton, FL, USA) and putty NeoPutty (NuSmile, Houston, TX, USA). Each sample was stored for 24 h at 37°C and 100% humidity. Then, after adhesive placement, the restorative material Filtek bulk-fill (3 M ESPE, St. Paul, MN, USA) was placed over the capping material using cylindrical plastic capsules (2 mm height and 2 mm) and polymerised for 20 s. Specimens were then tested in a universal testing machine for the compression load resulting in the μSBS. The data were compared with the one-way ANOVA (Welch) and the Tamhane test. The mean value was significantly higher in the TheraCal LC group than in the other two groups ($p < 0.05$). There was no significant difference between NeoMTA 2 and NeoPutty groups ($p > 0.05$). The majority of failure modes for all groups were cohesive within biomaterial. Using TheraCal LC in the pulp capping procedure can result in higher bond strength values to the tested bulk-fill resin-based composite than NeoMTA 2 and NeoPutty.

KEYWORDS

adhesion of biomaterial, NeoMTA 2, NeoPutty, pulp capping material, TheraCal LC

INTRODUCTION

Pulp-capping procedure may be defined as the process of covering the decalcified dentin tissue or the exposed vital dental pulp, due to iatrogenic or traumatic injuries, with a biomaterial and stimulating the formation of tertiary dentin by odontoblasts to prevent the development of pulpal and periapical pathoses [1]. Biomaterials stimulate the pulp organ to form tertiary dentin by placing them directly on the pulp tissue or indirectly over the remaining dentin. In addition, other properties expected from pulp capping biomaterials can be listed as follows: adhesion to restorative material, radiopacity, bactericidal/bacteriostatic property, good physical/biochemical property and good shear bond strength (SBS) [2].

Mineral trioxide aggregate (MTA) was the first calcium silicate-containing biomaterial used in dentistry [3]. In addition to being developed as a root-end filling material, MTA is also used in pulp capping, apexogenesis, pulpotomy, repair of root perforation, internal/external

resorption, root canal filling and apexification procedures [4, 5, 6]. This material has excellent impermeability, stimulating hard tissue formation, antibacterial activity, calcium hydroxide releasing, low solubility and setting in a wet environment, even in the presence of blood [7, 8, 9, 10]. Despite all these advantages, its manipulation is difficult, and the setting time is long. It can also cause discoloration of the tooth tissue [5, 9, 11].

According to the information given by the manufacturer, the problem of tooth discoloration was solved in NeoMTA 2 (NuSmile Avalon Biomed, Bradenton, FL, USA) by adding tantalite instead of bismuth oxide as a radiopacifier. This material is a calcium silicate-based biomaterial developed for vital pulp treatment in primary and permanent teeth. It triggers the healing process by stimulating hydroxyapatite in dentin. NeoMTA 2 has immediate wash-out resistance and has low water solubility (<3%). Depending on the clinical case (retrograde filling, pulp capping, etc.), the consistency can be adjusted by mixing different powder–gel ratios [12].

To reduce the effect of hand mixing on the setting reaction and simplify clinical application, premixed calcium silicate cements have been developed as an alternative to conventional powder–liquid cements [13]. When the lid is opened for reuse, the shelf-life of cement in powder–liquid form, which is exposed to atmospheric moisture, is shortened. The manufacturer of NeoPutty (NuSmile, Houston, TX, USA) suggests that the experimental putty has overcome this problem. Unlike NeoMTA 2, NeoPutty is formulated with a water-free organic liquid. When applied, it hardens with water from the apical, dentin tubules or pulp [12]. In one of the few NeoPutty studies in the literature, NeoPutty material was more biocompatible and have a longer shelf-life than EndoSquence BC putty [14].

Compared to MTA (Angelus Soluções Odontológicas, Londrina PR, Brazil) and Biodentine (Septodont, Saint Maur des Faussés, France), TheraCal LC (Bisco Inc., Schaumburg, IL, USA) is a light-curing calcium silicate-based biomaterial with less microleakage/solubility, good sealing ability and more calcium ion release [15]. Permanent restoration can be done immediately, using its light-curing [15, 16]. Regarding the bond strength of restorative materials on pulp capping agents, the bonding procedure (self-etch or etch and rinse) and the appropriate timing for permanent restoration are controversial [17, 18]. Although the American Association of Endodontists (AAE) recommends a 3 to 4mm glass ionomer layer on the biomaterial by the 'Clinical Considerations for Regenerative Procedure' [19], many studies are carried out on the bond strength of the restorative material placed directly on the biomaterial [2, 9, 10].

While the interfacial bond failure between the adhesive and the adherend is an adhesive failure, if an adhesive layer remains on both surfaces as a result of fracture, it is defined as a cohesive failure [20]. Therefore, the shear bond strength (SBS) to the biomaterial and permanent restoration and failure modes are essential in constructing permanent restoration, which is also important to success in endodontics. While studies are investigating the SBS of TheraCal LC to restorative material in the literature [2, 21, 22, 23], there is no study on this subject with NeoPutty and NeoMTA 2. Therefore, this study aimed to evaluate the micro-SBS (μSBS) of the calcium silicate-based TheraCal LC, NeoMTA 2 and NeoPutty biomaterials to the bulk-fill composite resin. The tested null hypothesis was that no statistically significant differences may be found among the three tested silicate cements regarding the adhesive μSBS value.

MATERIALS AND METHODS

The sample size calculation performed with G Power software (Heinrich Heine University, Düsseldorf, Germany) indicated that the sample size for each group must be a minimum of five resin blocks [18]. A total of 30 cylindrical resin blocks measuring 35 mm height and 25 mm diameter with a hole in the centre of the resin block (2 mm in depth and 4 mm in diameter) were manufactured with a 3D printer and divided into three groups ($n = 10$), depending on the calcium silicate cement used: TheraCal LC, NeoMTA 2 and NeoPutty. According to the manufacturer's instructions, each material was mixed and placed in the resin block. The materials were levelled using a mixing spatula to be flush with the surface of the resin block. The base of each resin block was submerged in water so that the cotton pellet was moistened. Before the bonding procedure, each sample was allowed to be set in a humidor at 37°C and 100% humidity for 24 h.

The samples' surface was air-dried and the Bond Force II™ adhesive bottle system (Tokuyama Dental, Tokyo, Japan) was applied according to the manufacturer's instructions. No acid etching was performed before bonding system application in any of the study groups. Lastly, the restorative material Filtek bulk-fill (3M ESPE, St. Paul, MN, USA) was centrally placed over the capping material using cylindrical plastic capsules with 2 mm height and 2 mm of internal diameter and polymerised for 20 s using a light-emitting diode (LED) curing unit (DTE Lux, E-Guilin Woodpecker Medical Instrument Co. Ltd., Guangxi, China). All the samples were stored at 37°C with 100% humidity for 24 h, before proceeding to the μSBS tests.

Each sample was set up in a universal testing machine (Instron, Shimadzu Corp., Chiyoda-Ku, Tokyo, Japan), and the shear mode was selected. First, the compression

load resulting in the SBS was performed parallel and close to the adhesive interface. Then, a chisel-shaped rod applied the shear force at a crosshead speed of 1 mm/min, up to bond disruption. The SBS values, calculated through the quotient between the peak break force (N) and the cross-sectional area of the bonded interface (3.14 mm^2), are expressed in megapascals (MPa) (1 MPa = 1 N/mm^2).

The fracture surfaces were evaluated by a single operator using a dental operative microscope (OMS 2380, Zumax, Suzhou, China) under ×19.8 magnification. The fracture pattern was classified as follows: (a) adhesive fracture (failure between the biomaterial and the restorative material with no resin remnants), (b) cohesive fracture within the biomaterial, (c) cohesive fracture within the restorative material, (d) mixed fracture (comprises both adhesive and cohesive fracture; Figure 1).

Data were analysed with SPSS 21.0 Software (IBM Corp, Armonk, NY). The data showed non-normal distribution according to the Shapiro–Wilk test. Natural log (LN) transformation was applied, and normality was achieved. Since the variances were not homogeneous according to the Levene's test, the data were compared with the one-way ANOVA (Welch) test. Multiple comparisons were made using the Tamhane test. The significance level was taken as $p < 0.05$.

RESULTS

The descriptive statistics in each group were listed in Table 1. When the data were analysed with the LN transformation form, it was determined to be a difference between the groups ($p = 0.003$). While the mean LN value was 3.07 in the TheraCal LC group, it was 2.03 in the NeoMTA 2 group and 2.36 in the NeoPutty group. As a result of the Tamhane post hoc test, the mean value was significantly higher in the TheraCal LC group than in the other two groups ($p < 0.05$). There was no significant difference between NeoMTA 2 and NeoPutty groups ($p > 0.05$).

The fracture patterns were given in Table 2. Most of the observed failure modes in all groups were cohesive failures within the bioceramic material. Adhesive and mixed failures were registered only in one sample in the TheraCal LC group. In the NeoMTA 2 group, cohesive failure within the restorative material was observed in three samples.

DISCUSSION

Calcium hydroxide is the most commonly used liner material; however, tunnel defects, dissolution over time and insufficient adhesion to dentin [2] have led researchers to use different biomaterials. Various commercial

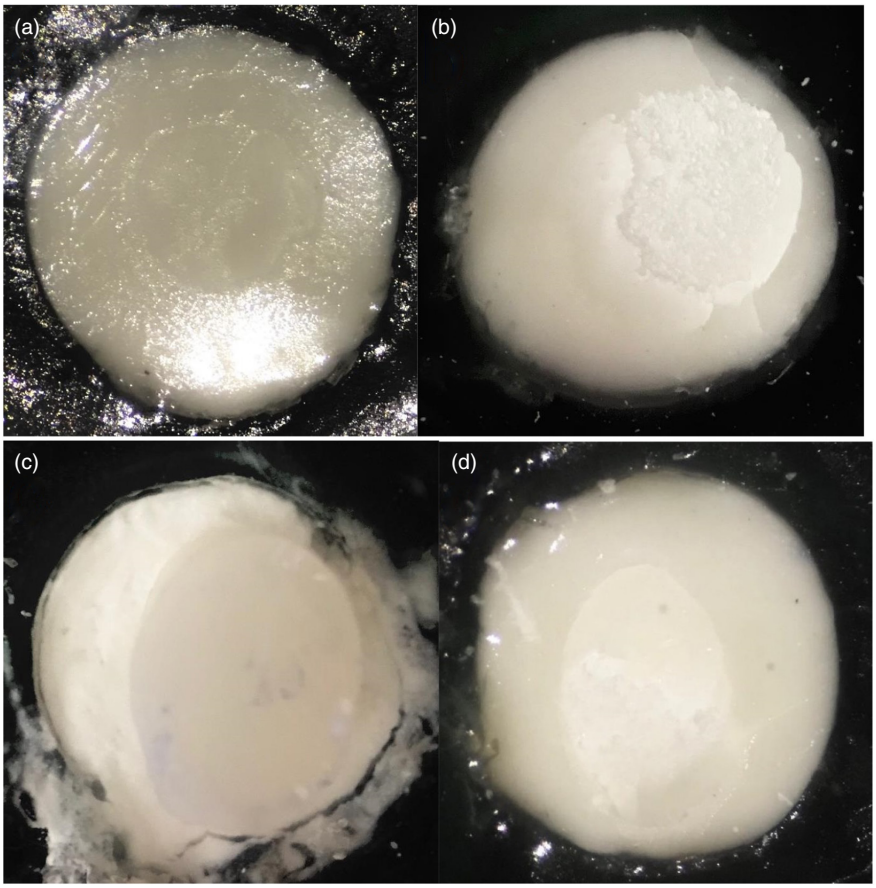

FIGURE 1 Images of failure modes. Adhesive fracture (a), cohesive fracture within the biomaterial (b), cohesive fracture within the restorative material (c), mixed fracture (d)

TABLE 1 Mean bond strength values of the tested groups following the SBS test (Mpa)

Pulp capping material	*N*	Mean ± SD[1]	Mean ± SD[2]	*p*
TheraCal LC	10	23.32 ± 9.15[a]	3.07 ± 0.45	**0.003***
NeoMTA 2	10	12.17 ± 11.2[b]	2.03 ± 1.06	
NeoPutty	10	11.37 ± 4.63[b]	2.36 ± 0.39	

* and Bold indicates One-way ANOVA (Welch), Mean and standard deviations with the same superscript were not statistically different.

[1]Original value.

[2]LN transformation.

TABLE 2 The fracture pattern of the tested groups following the SBS test

Pulp capping material	N	Adhesive	Cohesive (failure within bioceramic material)	Cohesive (failure within restorative material)	Mixed
TheraCal LC	10	1	8	–	1
NeoMTA 2	10	–	7	3	–
NeoPutty	10	–	10	–	–

modifications and preparations of calcium silicate-based liners are available in dentistry. After powder–liquid preparations (ProRoot MTA, MTA Angelus, MM-MTA, Ortho MTA, MTA HP, NeoMTA 2, Biodentine, etc.), it started to be used in putty putties in endodontics (NeoPutty, EndoSequence BC RRM putty). The high SBS between calcium silicate-based cements and restorative materials provides better bonding and adhesion between the two interfaces while reducing microleakage [24, 25]. Many studies in the literature examine the SBS of calcium silicate-based biomaterials and restorative filling material or adhesive material [2, 9, 10, 18, 21, 23, 26].

The manufacturer listed the advantages of NeoPutty over NeoMTA 2 as follows: It does not need mixing; it is more radiopaque and the syringe system provides an even unit dose dispensing [12]. Despite these advantages, the present study found that NeoPutty was not superior to Neo MTA 2 in terms of SBS. The TheraCal LC had higher SBS values with respect to these two biomaterials. In this case, the study's results require rejecting the null hypothesis that there is no difference in the SBSs between biomaterials and resin composite when using different biomaterials.

A study reported that the moistened pellet should remain on liquid–powder MTA for 24 h to optimise flexural strength [27]. However, in the studies, there is no consensus about the waiting time of the biomaterial in a humid environment before application of restorative material. Keeping in an incubator period for MTA varies between 24, 48, 72, 96 h and 7 days [9, 10, 17, 18, 27]. In the present study, NeoMTA 2 and NeoPutty were kept in an incubator for 24 h at 100% humidity to allow complete hardening of the materials before application of the bulk-fill resin composite. Although there were no data on the timing of the composite material applied on TheraCal LC, TheraCal LC samples were also kept in the incubator for 24 h to ensure standardisation. In a time-dependent study using human teeth, the SBS of TheraCal LC to the composite was evaluated immediately after placement and showed better SBS (17.51 Mpa) than Biodentine [2]. In the present study, the μSBS of TheraCal LC was 23.32 MPa. This value was higher than previously reported. This may be due to the fact that the material was maintained in an incubator and resin blocks were used.

TheraCal LC has been introduced as a resin-modified Portland cement-based light-cure MTA allowing immediate placement of the final restoration [21, 28, 29]. This biomaterial consists of a combination of HEMA/TEGDMA-based resin and calcium silicate powder. A chemical bond is formed due to the copolymerization between the unreacted methacrylate groups of the HEMA monomer in the oxygen inhibition layer of the material and the methacrylate groups of the composite resin. In this case, a strong connection interface is formed [21, 30]. Many studies showed that TheraCal LC has high SBS to composite resin or glass ionomer cement than Biodentine [2, 21, 23] and MTA [18, 22, 31]. In the current study, the mean μSBS values of TheraCal LC, Neo MTA 2 and NeoPutty were 23.32 Mpa, 12.17 Mpa and 11.37 Mpa, respectively, significantly better for TheraCal LC. The reason may be the chemical bond formed by TheraCal LC with the composite resin as a result of copolymerization. However, since SBS studies have not been performed previously using NeoMTA 2 and NeoPutty materials, further studies with these capping materials are needed.

In a study examining the SBS of TheraCal LC, ProRoot MTA and Biodentine with composite resin, glass ionomer cement and resin-modified glass ionomer cement, the resin composite was superior to the other two glass ionomer cements [21]. The bulk-fill resin-based composite material is recommended for posterior applications as a time-saving material. The manufacturer specified composite layer increment depth ranging from 4.56 to 4.24 mm in the A1-C2 colour scale [32]. Rosatto et al.

stated that the bulk-fill composites showed lower cuspal stress, shrinkage stress and higher fracture resistance [30]. Raina et al. observed that the bulk-fill composite (SDR) bonded better than the self-adhesive flowable composite (Dyad Flow) for the same pulp capping materials and the TheraCal LC showed higher μSBS with these two materials compared to other pulp capping materials [33].

Marto et al. investigated the SBS of the resin composite, which was applied with different surface treatments, due to repair with the same resin composite. They compared Tokuyama Bond Force II and Gluma Self Etch and obtained the highest SBS values in all the groups in which they used Tokuyama Bond Force II (4.7 and 5.4 Mpa) [34]. The reason for obtaining high Mpa values in the present study may be the Tokuyama Bond Force II system used. In a study examining the SBS of composite resin to TheraCal LC and Angelus MTA with different adhesive systems, TheraCal LC had the highest bond strength values, regardless of the adhesive agents tested. In addition, the authors emphasised that acid etch application affects the surface micromorphology of both materials [22]. In the present study, TheraCal LC showed the highest μSBS values. However, the SBS may vary with the use of different bonding systems and surface treatments. Therefore, further studies are needed on the use of these materials with different bonding systems.

The cohesive failure indicates that the materials have reached the maximum strength in the adhesiveness. Therefore, it is a preferred type of fracture. However, the failure mode is not a criterion for measuring the success of the adhesion. The ultimate strength of a joint is a more important criterion than the mode of joint failure [20]. When the fracture types between the biomaterial and restorative material were examined, more cohesive failure was observed in all groups, which was consistent with other studies [9, 21, 33]. This indicates that there was strong bond strength between the biomaterials and the restorative materials. In the present study, cohesive failure within the restorative material was found in three specimens in the NeoMTA 2 group. Although the bond strength of NeoMTA 2 in itself seems better than the other two groups according to the failure type, the μSBS test does not support this finding. Although it is not statistically significant, considering the SBS test of NeoMTA 2 and this material's failure mode, it can be mentioned that the strength of the material is better than NeoPutty. In addition, cohesive failure within the biomaterial was observed in all samples in the NeoPutty group.

There are also SBS studies in the literature using real human teeth instead of resin or acrylic blocks [2, 35]. Bond strength is affected by dentin water content, the presence or absence of a smear layer, dentin permeability and the relationship of dentinal tubules to the surface [36]. The limitations of this study can be listed as follows: SBS test was performed on resin blocks instead of human teeth; the surfaces of all biomaterials were prepared flat; and therefore, the effect of dentin or caries affected dentin on bonding could not be evaluated.

CONCLUSION

The findings exhibit that the μSBS of NeoPutty and NeoMTA 2 to bulk-fill composite resin was similar. TheraCal LC had better μSBS to bulk-fill composite resin than other biomaterials. More research is needed to understand the bonding mechanism of bulk-fill resin composite systems to NeoMTA 2 and NeoPutty.

AUTHOR CONTRIBUTION

All authors have contributed significantly, and all authors are in agreement with the manuscript.

CONFLICT OF INTEREST

The author declares that they have no conflict of interest.

ETHICS STATEMENT

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

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