

# EVALUAREA ADERENȚEI DENTINARE A DOUĂ MATERIALE MODERNE PENTRU COAJUL PULPEI DENTARE

## DENTINE ADHESION EVALUATION OF TWO MODERN PULP CAPPING MATERIALS

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*This in vitro study aimed to evaluate and compare the dentine adhesion of two modern pulp capping materials: TheraCal LC (Bisco Inc. U.S.A.) and BioMTA (Cerkamed Poland). The samples have been prepared simulating in vivo conditions, in accordance with ISO/DIN standard protocols for this type of test. Twelve samples were prepared in this way, being preserved in good condition for each material in order to be investigated and the results interpreted. The values of the shear test, measured in kgf for each sample, have been recorded. A paired t-test was conducted to compare mean adhesion results in TheraCal and MTA groups. The results of our study showed a statistical significant difference between the measurements for TheraCal (M=1.27, SD=0.69) and MTA (M=0.22, SD=0.13); t(11)=4.80, p=0.0005. The results suggest that TheraCal has better adhesion properties than MTA.*

*Acest studiu in vitro și-a propus să evalueze și să compare aderența la dentină a două materiale moderne pentru coajul pulpei dentare: TheraCal LC (Bisco Inc. S.U.A.) și BioMTA (Cerkamed Polonia). Probele au fost preparate simulând condițiile in vivo, în conformitate cu protocoalele standard ISO / DIN pentru acest tip de test. În acest mod au fost preparate douăsprezece probe pentru fiecare material, fiind păstrate în stare bună pentru ca rezultatele să fie cercetate și interpretate. Au fost înregistrate valorile testelor de forfecare pentru fiecare probă, măsurate în kgf. Pentru compararea valorilor medii ale adeziunii pentru TheraCal și MTA a fost folosit testul statistic t. Rezultatele studiului nostru au arătat o diferență statistică semnificativă între măsurătorile pentru TheraCal (M = 1,27, SD = 0,69) și pentru MTA (M = 0,22, SD = 0,13); t (11) = 4,80, p = 0,0005. Rezultatele sugerează că TheraCal are proprietăți de aderență mai bune decât BioMTA.*

**Keywords:** dentine, pulp capping materials, adhesion, shear test

### 1. Introduction

Pulp capping is a technique used in dentistry to preserve dental pulp vitality, after being exposed, or nearly exposed. The dental pulp resides in a rigid chamber consisting of dentine, enamel and cementum, and providing strong mechanical support and protection from the microbial rich oral environment. However, if this rigid shell loses its structural integrity, the pulp is under the threat of the adverse stimuli from the mouth [1].

The treatment options for a pulp-exposed permanent tooth include direct pulp capping (DPC), pulpotomy, and pulpectomy. DPC is defined as "placing a dental material such as calcium hydroxide or mineral trioxide aggregate (MTA) directly on a mechanical or traumatic vital pulp exposure, thereby sealing the pulpal wound to facilitate the formation of reparative dentin and maintenance the vitality of the pulp [2].

The predictability of pulp capping treatment has been largely debated in the literature during

years, various parameters being implied in the wide range of success rate from 33.3% up to 92.2% [3,4]. The introduction of new materials (MTA, bioceramics or calcium silicate-based cements) and new techniques have uprighted the level of success rate in this treatment [5,6].

TheraCal LC is a light-curable resin-modified tricalcium silicate material (Bisco, Inc., Schaumburg, IL, USA) that is designed as a direct/indirect pulp capping material. It is composed of 45% Portland cement, 10% bismuth oxide, 5% fumed silica, and approximately 40% resin by weight [7,8]. The material might be very attractive for clinicians because of its easiness handling. Unlike other calcium silicate-based materials, TheraCal LC is resin-based and does not require any conditioning of the dentine surface. The material can be bonded with different types of adhesives directly after application. Due to its handling characteristics and chemical properties, there has been considerable research performed on this material since its launching [7].

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MTA was first developed as a dental root repair material [9,10]. It is used for creating apical plugs during apexification, repairing root perforations during root canal therapy, treating internal root resorption, root-end filling, as well as for pulp capping. Originally, MTA was dark gray in color, but nowadays white versions have been developed on the market. MTA powder is a mixture of a refined Portland cement, bismuth oxide and trace amounts of SiO<sub>2</sub>, CaO, MgO, K<sub>2</sub>SO<sub>4</sub>, and Na<sub>2</sub>SO<sub>4</sub>. MTA powder is mixed with supplied sterile water in a 3:1 powder/liquid. Hydrated MTA has an initial pH of 10.2, which rises to 12.5 three hours after mixing [11,12].

One of the factors implied in pulp capping success rate procedure is the bond strength between the liner material and surrounding dentine walls. On this factor depends the possibility of a microleakage to the pulp exposed [13].

Although many clinical and randomized studies published during last years are comparing the properties of these two types of materials regarding tertiary dentinogenesis [6], there is few information available regarding their adhesion to dentine.

In accordance with these clinical findings, our study was developed to compare the dentine bond strength of two pulp capping materials: TheraCal LC (Bisco Inc. U.S.A.) and BioMTA (Cerkamed Poland). The null hypothesis was that there is no statistical difference between mean adhesion measurements of the tested materials.

## 2. Materials and methods

Twenty-four adhesive surfaces were investigated, 12 samples for each material taken into the study.

Human permanent teeth, including wisdom molars, with no indication of conservative treatment, were harvested by surgery from patients aged between 16 and 40 years. The teeth were without enamel dental caries surfaces and with the appearance of normal enamel, without mineralization defects, decalcification or cracks.

Immediately after extraction of teeth, their surface was washed under running water and cleaned of blood and soft tissue attachments. Immediately after extraction and purification, extracted teeth were kept for two weeks in bacteriostatic solution of 0.5% chloramine T at room temperature (23 ± 2) °C. Afterwards they were transferred to distilled water in accordance with DIN ISO 3696, grade 3, in a refrigerator (4 ± 2) °C [14]. The teeth were used in tests for a maximum of five weeks after extraction.

Each tooth has been prepared and subjected for specific investigations of detachment shear test, the radicular area being embedded into self-curing epoxy resin (Figure 1: a + b) [15].

After setting, each tooth was added to and maintained in a solution of distilled water for 72 hours, at room temperature [14].

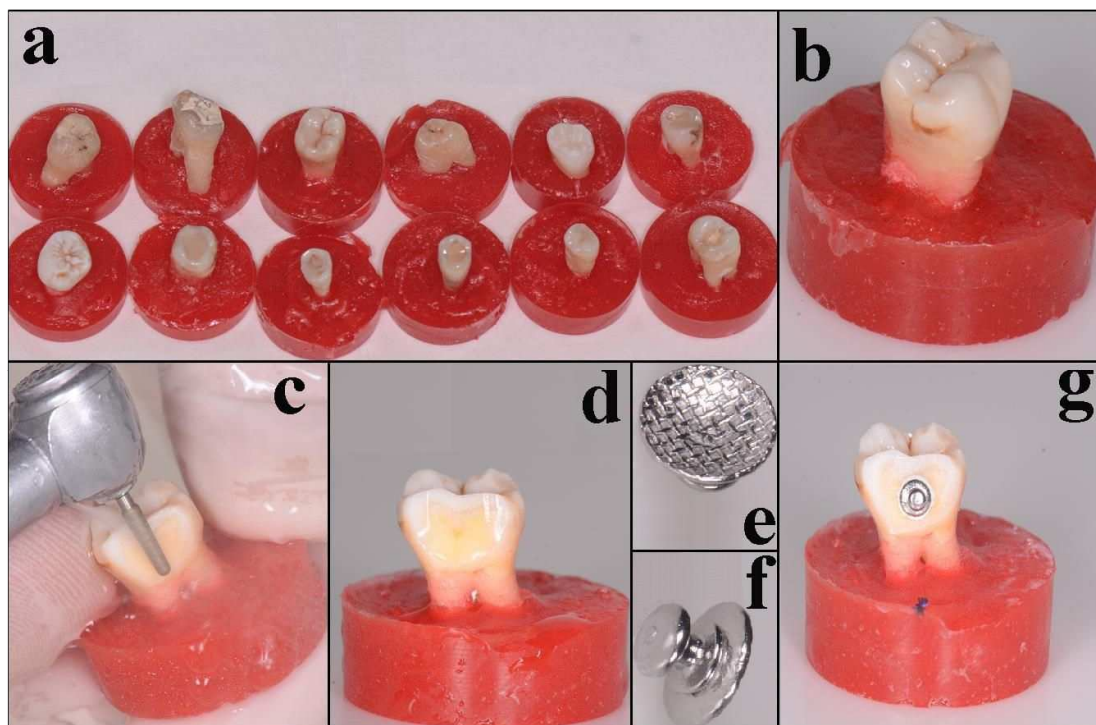


Fig. 1 - a + b: Samples preparation / *Prepararea probelor*  
 c + d: Dentin surface preparation / *Prepararea suprafeței dentinare*  
 e + f: Orthodontic button used for shear test / *Butonul ortodontic folosit pentru testul de forfecare*  
 g: Sample prepared for share test / *Probă preparată pentru testul de forfecare*

In order to reduce the morphological variables due to different quality and quantity of teeth mineralized tissues upon adhesion force, two opposite adhesion dentin surfaces (buccal and oral) were then prepared on each tooth. Dentine surface was exposed using the same protocol represented by high speed diamond bur rotation (200,000 rpm) having continuous irrigation, mimicking real clinical conditions (Figure 1: c + d).

Same surface standardized orthodontic buttons (FlexMedics, USA) with 3 mm diameter and 4.71 mm<sup>2</sup>, were applied on the dentine surfaces of each tooth, using TheraCal and BioMTA, respectively (Figure 1: e + f).

An example of sample preparation is shown in Figure 1g. The steps for mounting the device on the dentine surface of each tooth have been realized in accordance with manufacturer indications regarding manipulation, armamentarium, working and setting time being strictly complied. After setting, each assembly tooth-capping material-button was added to and maintained in a solution of distilled water, at room temperature.

At 24 hours after fixation, each assembly tooth-capping material-button system was prepared and subjected for specific investigations of detachment shear test. The mechanical tests were made at room temperature and the "shear type test" performed on a Multi-Functional Bond Tester Condor 70 device (XYZTEC) having an excursion of the action element of 1mm/min (Figure 2: a + b).

The values of the shear test, measured in kgf for each sample, have been recorded (Figures 3 and 4).

A paired *t*-test was conducted to compare mean adhesion results in TheraCal and MTA groups. We performed statistical analyses using Stata/IC 16 (StataCorp, College Station, TX, USA).

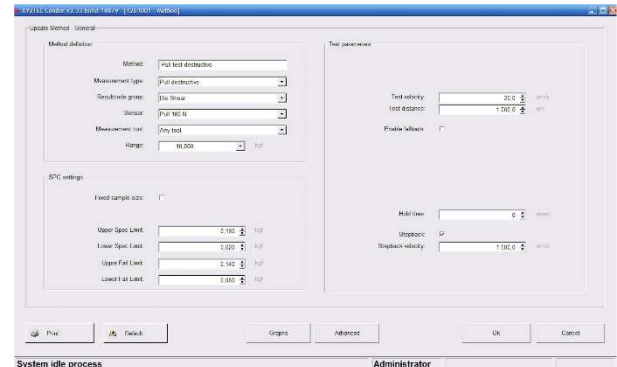


Fig. 3 - Digital recordings / Înregistrări electronice

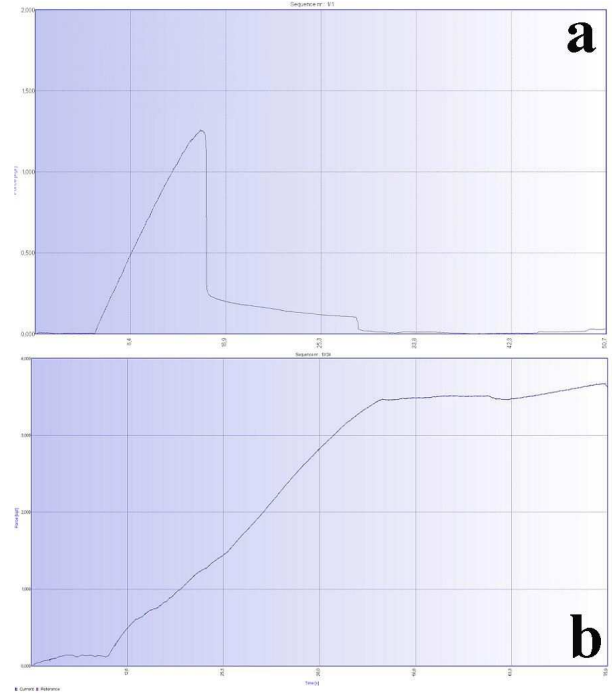


Fig. 4 - a: Graphic force/time until shear TheraCal sample / Dependența forță / timp până la forfecarea probei de TheraCal  
- b: Graphic force/time until shear BioMTA sample / Dependența forță / timp până la forfecarea probei de BioMTA

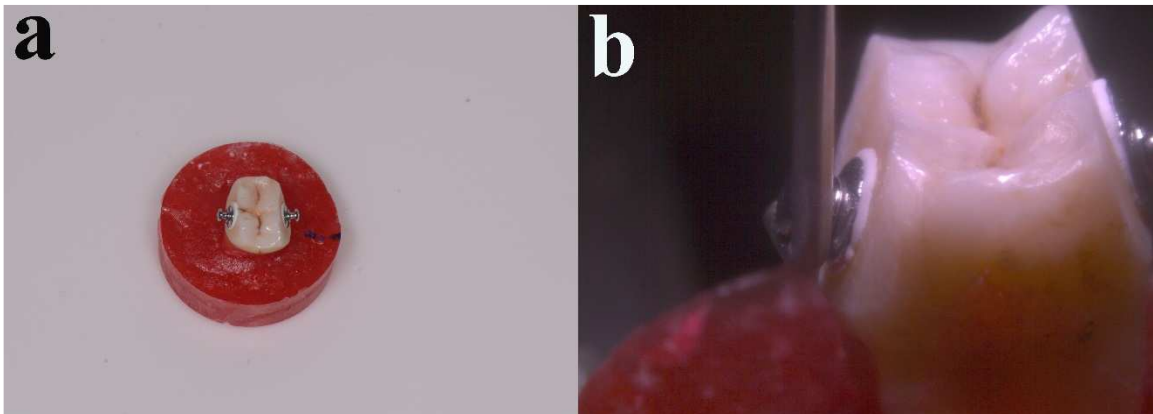


Fig. 2 (a + b) - Shear test performed with Condor 70 Shear Test – HYZTEC device / Testul de forfecare realizat cu ajutorul aparatului de testare Condor 70 Shear Test.



### 3.Results and discussion

All samples, except one, showed higher values of the shear test for TheraCal LC, as compared to MTA (Table 1). The behavior of these two materials under tension was different (Figure 5).

There was a statistically significant difference between the measurements for TheraCal (M=1.27, SD=0.69) and MTA (M=0.22, SD=0.13);  $t(11)=4.80$ ,  $p=0.0005$  (Figure 6). The null hypothesis was thus rejected.

Even though there are many researches evaluating biological and clinical aspects of pulp capping materials, only few are addressed to conditions influencing the microleakage that may occur around these materials and dentine tissues. Not only the bond strength between pulp capping material and the overlying restoration is vital for treatment success [16], but also, in our opinion, the bond strength between pulp capping material and dentine walls surrounding the exposed pulp. Different types of tests, such as shear, tensile, micro-shear, and micro-tensile have been performed to measure the bond strength and clinical performance of different types of dental materials. Our research was performed for the shear test evaluation of the adhesion between dentine and two pulp capping materials proved with good clinical performances.

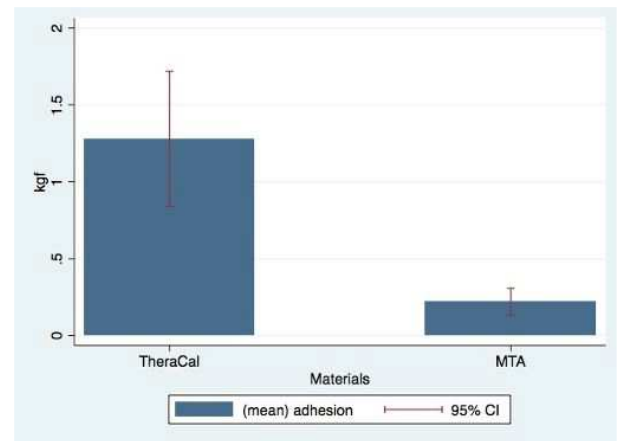


Fig. 6 - The mean values of measurements / Valorile medii ale măsurătorilor.

TheraCal LC has a composite structure, and theoretically, the bonding mechanism of composites to calcium in pulp-capping agents is comparable with mecano-chemical adhesion to hard dental tissues [17]. The second material taken into the study, BioMTA, consists of a cement powder which is mixed with a liquid (mainly purified water). This is why BioMTA is a cement which sets especially by aggregation of its particles and does not have a chemical reaction for setting, like most of so called “biodentine” materials do. For these materials, the setting time

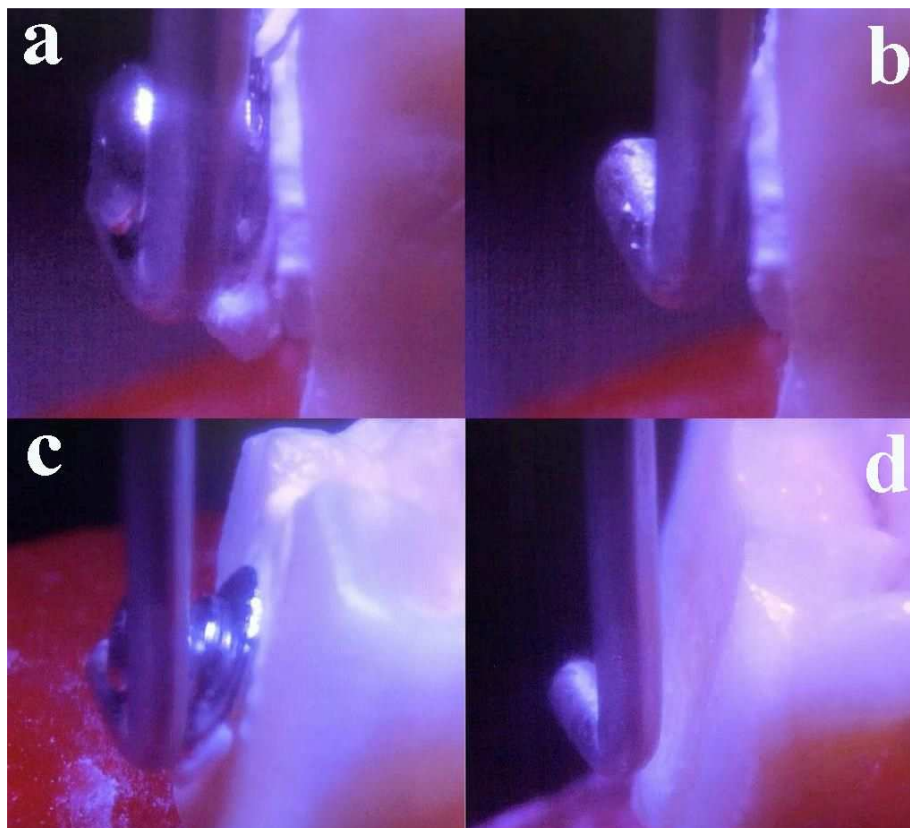


Fig. 5 - a + b: Shear test of TheraCal sample (a – peak moment; b – after shear) / Testul de forfecare pentru mostra de TheraCal (a – momentul de vârf; b – după forfecare)  
 c + d: Shear test of BioMTA sample (a – peak moment; b – after shear) / Testul de forfecare pentru mostra de BioMTA (a – momentul de vârf; b – după forfecare)

Table 1

Shear test results for Theracal LC and BioMTA adhesion to dentine  
 Rezultatele testelor de forfecare pentru adeziunea la dentină a celor două materiale: Theracal LC și BioMTA

Sample no	Theracal LC (kgf)	BioMTA (kgf)
1	1.262	0.329
2	0.069	0.368
3	1.393	0.107
4	1.138	0.273
5	0.528	0.075
6	0.579	0.155
7	0.991	0.444
8	2.466	0.025
9	1.167	0.356
10	2.170	0.300
11	1.847	0.113
12	1.733	0.100

is 12 min, and is much lower than that of BioMTA (120 min) or other regular MTAs (180 min) [18,19].

Both materials contain silicates, but BioMTA seems to display more mineral phases. While TheraCal LC has a surface smoother after setting, probably due to the resin phase from its composition, BioMTA's surface remains rough due to higher mineralization phase. Regardless different compositions, both materials have bioactive properties, which would develop apatite on their surfaces, after immersion in a solution similar to the human physiological environment [20].

Besides a fast setting time and a high compressive strength, any material used for capping should also have the ability to create a bond to the dentine, and to the overlaying restorative materials, glass ionomer cements and composites. A material used as a cap, base or base build-up should provide an adequate seal, be able to prevent leakage and remain in place under dislodging forces, such as chewing pressure or the application of other restorative material, thus having adhesive properties to dentine [19].

The Condor device used in the present study has advantages for obtaining of high-performance shear test results, because of the possibility to elect the speed and time of the force that is applied onto the sample [21]. The comparative analysis of the graphics force/time until shear from our study showed for almost all samples in case of TheraCal a cohesive fracture and a completely different behavior to shear-test registered by BioMTA samples. In this later cases, the fracture curves were more suggesting an adhesive failure. The comparative analysis of the shear tests of the two materials was presented in Figure 4. BioMTA was cleaned from the dentine surface almost entirely. Further scanning electron-microscopy evaluations can establish the degree of materials clearance from the exposed dentine surfaces.

Other studies have been realized to

investigate the adhesion of TheraCal LC to different adhesive systems or composite restorative materials. All of restorative materials have presented a good adhesion behavior of this material [22,23]. It has been recently suggested the possibility to improve the adhesiveness of this type of resin modified tricalcium silicate material by adding acidic monomer [24]. However, the balance between achieving a better adhesion and raising the level of acidic monomer should be further investigated, because minimally toxic concentrations of acidic monomer can promote a pulpal inflammatory response and suppress odontoblastic differentiation [25].

#### 4. Conclusions

The results of our studies suggest that TheraCal has better adhesion properties than BioMTA. Further researches should be conducted in order to analyze the way these two materials obtain the adhesion to dentine surface (e.g. morphological analyses of the surfaces).

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#### REFERENCES

- [1] Z. Mohammadi, S. Shalavi, M. K. Soltani, Mineral trioxide ag-gragate (MTA)-like materials: an update review. The Compendium of Continuing Education in Dentistry, 2014, **35**, 557–561.
- [2] C. Yu, P. V. Abbott, An overview of the dental pulp: its functions and responses to injury, Australian Dental Journal, 2007, **52**(1 Suppl), S4-16.
- [3] American Association of Endodontists, Glossary of endodontic terms, 2003.
- [4] A. S. Al-Hiyasat, K. M. Barrieshi-Nusair, M. A. Al-Omari, The radiographic outcomes of direct pulp-capping procedures performed by dental students: a retrospective study, Journal of American Dental Association, 2006, **137**(12), 1699-1705.
- [5] T. M. Auschill, N. B. Arweiler, E. Hellwig, A. Zamani-Alaei, A. Sculean, Success rate of direct pulp capping with calcium hydroxide [Article in German], Schweizer Monatsschrift für Zahnmedizin, 2003, **113**(9), 946-952.

- [6] S. Linu, M. S. Lekshmi, V. S. Varunkumar, V. G. Sam Joseph, Treatment outcome following direct pulp capping using bioceramic materials in mature permanent teeth with carious exposure: A pilot retrospective study, *Journal of Endodontics*, 2017, **43**(10), 1635-1639.
- [7] A. C. Didilescu, C. M. Cristache, M. Andrei, G. Voicu, P. Perlea, The effect of dental pulp-capping materials on hard-tissue barrier formation: A systematic review and meta-analysis, *Journal of American Dental Association*, 2018, **149**(10), 903-917 e4.
- [8] N. Z. Arandi, T. Rabi, TheraCal L C: From biochemical and bioactive properties to clinical applications, *International Journal Of Dentistry*, 2018, **2018**:3484653.
- [9] K. Cantekin, S. Avci, Evaluation of shear bond strength of two resin-based composites and glass ionomer cement to pure tricalcium silicate-based cement (Biodentine(R)), *Journal of Applied Oral Science*, 2014, **22**(4), 302-306.
- [10] S. J. Lee, M. Monsef, M. Torabinejad, Sealing ability of a mineral trioxide aggregate for repair of lateral root perforations, *Journal of Endodontics*, 1993, **19**(11), 541-544.
- [11] J. Camilleri, F. E. Montesin, K. Brady, R. Sweeney, R. V. Curtis, T. R. Ford, The constitution of mineral trioxide aggregate, *Dental Materials*, 2005, **21**(4), 297-303.
- [12] J. Camilleri, The chemical composition of mineral trioxide aggregate, *Journal of Conservative Dentistry*, 2008, **11**(4), 141-143.
- [13] Z. Mohammadi, S. Shalavi, M. K. Soltani, Mineral trioxide aggregate (MTA)-like materials: an update review, *Compendium of Continuing Education in Dentistry*, 2014, **35**(8), 557-561, quiz 62.
- [14] L. Daniele, Mineral Trioxide Aggregate (MTA) direct pulp capping: 10 years clinical results, *Giornale Italiano di Endodonzia*, 2017, **31**(1), 48-57.
- [15] International Organization for Standardization, Water for analytical laboratory use—Specification and test methods, ISO Geneva, Switzerland, 1987.
- [16] DIN Deutsches Institut für Normung e. V. DIN 13990-2: 2009-05, dentistry—test methods for shear bond strength of adhesives for orthodontic attachments—part 2: bonding of the entire bonding system attachment-adhesive-enamel 26, 2009.
- [17] A. Raina, A. Sawhny, S. Paul, S. Nandamuri, Comparative evaluation of the bond strength of self-adhering and bulk-fill flowable composites to MTA Plus, Dycal, Biodentine, and TheraCal: an in vitro study, *Restorative Dentistry and Endodontics*, 2020, **45**(1), e10.
- [18] N. Sultana, R. R. Nawal, S. Chaudhry, M. Sivakumar, S. Talwar, Effect of acid etching on the micro-shear bond strength of resin composite-calcium silicate interface evaluated over different time intervals of bond aging, *Journal of Conservative Dentistry*, 2018, **21**(2), 194-197.
- [19] J. Camilleri, F. Sorrentino, D. Damidot, Investigation of the hydration and bioactivity of radiopacified tricalcium silicate cement, Biodentine and MTA Angelus, *Dental Materials*, 2013, **29**(5), 580-593.
- [20] M. Kaup, C. H. Dammann, E. Schafer, T. Dammaschke, Shear bond strength of Biodentine, ProRoot MTA, glass ionomer cement and composite resin on human dentine ex vivo. *Head and Face Medicine*, 2015, **11**, 14.
- [21] G. Voicu, A. C. Didilescu, A. B. Stoian, C. Dumitriu, M. Greabu, M. Andrei, Mineralogical and microstructural characteristics of two dental pulp capping materials, *Materials (Basel)*, 2019, **12**(11), 1772.
- [22] D. I. Ciocan, D. Stanciu, M. A. Popescu, F. Miculescu, I. Plotog, G. Vârzaru et al., Electron microscopy analysis of different orthodontic brackets and their adhesion to the tooth enamel, *Romanian Journal of Morphology and Embryology*, 2014, **55**(2 Suppl), 591-596.
- [23] H. Alzraikat, N. A. Taha, D. Qasrawi, M. F. Burrow. Shear bond strength of a novel light cured calcium silicate based-cement to resin composite using different adhesive systems, *Dental Materials Journal*, 2016, **35**(6), 881-887.
- [24] V. L. Deepa, B. Dhamaraju, I. P. Bollu, T. S. Balaji, 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, *Journal of Conservative Dentistry*, 2016, **19**(2), 166-170.
- [25] Y. K. Kim, M. H. Hong, T. Y. Kwon. Dentin bonding of TheraCal LC calcium silicate containing an acidic monomer: An In Vitro Study, *Materials (Basel)*, 2020, **13**(2), 293.
- [26] E. C. Kim, H. Park, S. I. Lee, S. Y. Kim. Effect of the acidic dental resin monomer 10-methacryloyloxydecyl dihydrogen phosphate on odontoblastic differentiation of human dental pulp cells, *Basic and Clinical Pharmacology and Toxicology*, 2015, **117**(5), 340-349.

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## ANIVERSĂRI

### 20 septembrie (1459) – Ziua orașului București



La această dată se împlinesc 561 de ani de la prima atestare documentară a existenței Orașului București, într-un hrisov emis de cancelaria voievodului Vlad Țepeș. În ordine cronologică, orașul București a devenit treptat, în secolele următoare, cea de a patra capitală a Țării Românești a Munteniei (dupa Câmpulung, Curtea de Argeș și Târgoviște) și prima capitală a României Mari după Războiul de Întregire Națională din 1916 – 1918.

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