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Evaluation of Microtensile Bond Strength Between Biodentine and Post Cement at Different Time Intervals

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Abstract

Objectives: To investigate the microtensile bond strength (μTBS) of various adhesive systems (etch & rinse, self-etch, and self-adhesive) for bonding MultiCore Flow and Biodentine at different time intervals.

Methods: Sixty pairs of 7x7x3 mm resin-based 3D-printed blocks with a 1x1 mm central tube were used in this study. One side of the blocks was filled with Biodentine, while another side was filled with MultiCore Flow. The materials were bonded using one type selected from these adhesive systems: ExciTE F DSC (etch & rinse), Multilink N (self-etch), or RelyX U200 (self-adhesive). Each group was subdivided into immediate and delayed groups (n=10). Specimens were subjected to μTBS testing, and failure modes were observed under a stereomicroscope. Two-way ANOVA was used to analyze the influence of time and adhesive system on μTBS.

Results: The results revealed that Multilink N group showed significantly higher μTBS in the immediate group compared to the delayed group (p=0.01). When comparing the materials, ExciTE F DSC performed significantly worse than Multilink N (p=0.02) and RelyX U200 (p=0.04) in the immediate group. The predominant failure modes observed under the stereomicroscope were mixed failure and cohesive failure within Biodentine.

Conclusions: Immediate placement of adhesives and MultiCore Flow over Biodentine showed higher microtensile bond strength than delayed placement. Overall, self-adhesive systems demonstrated high bond strength at both time intervals. Immediate bonding with self-adhesive systems may enhance the bond strength between Biodentine and MultiCore Flow in clinical practice, potentially leading to improved restoration longevity and reduced risk of failure.

Keywords: biodentine, cement, failure mode, microtensile bond strength, post

Introduction

Root canal treated teeth often have a significant loss of structural integrity. Consequently, after root canal treatment, it becomes necessary to place a post within the root canal as part of the restorative process. The primary purpose of a post is to serve as a retention for the core build-up material.[1] Additionally, a well-placed post contributes to a more intimate seal between the restorative material and the root canal. Studies conducted by Ray & Trope in 1995[2] and Tronstad *et al.,* in 2000[3] have demonstrated that the quality of root canal filling and restoration directly influences the long-term success rate of the treated tooth. Root canal filling materials, such as gutta-percha with sealer, are typically used in simple root canal treatment cases. However, more complex cases, including open apices and perforations, may require alternative materials. Hydraulic calcium silicate-based cements like Biodentine (Septodont, St. Maur-des-Fossés, France) have emerged as promising options for these situations. Biodentine possesses chemical and physical properties that make it an ideal material for root canal repair. It also demonstrates a short setting time and strong adhesive properties to dentin, making it a suitable restorative material for dentin.[4,5] Case reports have documented the successful use of Biodentine for apexification in a single visit, replacing the traditionally used MTA (mineral trioxide aggregate; Dentsply Tulsa Dental, Tulsa, OK, USA), which has a longer setting time.[6,7] Furthermore, a study by Yadaw *et al.,* in 2020[8] evaluated the single-visit obturation of necrotic immature permanent teeth with Biodentine and reported a 100% success rate at a 9-month follow-up.

Following endodontic procedures, resin composite core materials, MultiCore Flow (Ivoclar Vivadent, Schaan, Liechtenstein), are commonly used for post placement and core build-up. When placing a post within a root canal, an interface is created between two materials: hydraulic calcium silicate cement and post cement. Various adhesive systems are commonly used to bond these two surfaces. Contemporary adhesive systems include etch-and-rinse system such as ExciTE F DSC (Ivoclar Vivadent) and OptiBond FL (Kerr Corporation, Brea, CA, USA), self-etch system such as Multilink N (Ivoclar Vivadent) and Panavia V5 (Kuraray Noritake Dental Inc., Tokyo, Japan), and self-adhesive system such as RelyX U200 (3M ESPE, Deutschland GmbH, Neuss, Germany) and G-Cem LinkAce (GC Corporation, Tokyo, Japan). These adhesive systems have been widely used as post cements in clinical practice.

Posts placed within root canals typically have a snug fit, limiting lateral movement. Therefore, the primary forces experienced by the restoration are vertical. These vertical forces, akin to tensile strength, may lead to debonding at the interface between Biodentine and post cement. Additionally, the setting reaction between Biodentine and the adhesive system may influence bond strength. Moreover, the material's stiffness can affect bond strength. Some studies have examined bond strength between Biodentine and adhesive systems, such as the study by Hardan *et al.,*[9] which found that both self-etch and total-etch strategies exhibited promising bonding performance with Biodentine. However, there is a lack of study on the microtensile bond strength of these materials, which should be elucidated. Therefore, this study addresses this gap by using standardized 3D-printed blocks to compare the microtensile bond strength of three different adhesive systems to Biodentine.

This study aimed to investigate the microtensile bond strength (μTBS) between Biodentine and MultiCore Flow using various types of adhesive systems at different time intervals.

Material and Methods

Sample size calculations was determined from a similar study using this formula (Figure 1).[10,11] With α level type I error at 0.05 and β level type II error of 0.20 for the study, a sample size of 10 were obtained for each group.

$$n/gr = \frac{2(Z_\alpha + Z_\beta)^2 \sigma^2}{(\mu_1 - \mu_2)^2}$$

Figure 1: The formula of calculating sample size.

Sixty sets of 3D-printed clear resin blocks (120 pieces of blocks and 60 pieces of covers) were meticulously designed using Google SketchUp 2020 software (Google LLC., Mountain View, CA) and printed using a 3D Printer called Pro 55 (SprintRay, Los Angeles, CA), with surgical guide resin, which utilized DLP processing. The transparency of the resin blocks facilitates complete light transmission, ensuring full curing and polymerization of the material. The manufacturer's guidelines were followed

to ensure precision and consistency in the fabrication process.

The block was designed with 7x7x3 mm^3 in size. These blocks featured a central tube with dimensions of 1x1 mm^2, extending along the length of the block. When paired, the central tubes from each block interconnected to form a continuous 14 mm long tube. To minimize material leaking, small gaps were incorporated between the "connecting end" of the blocks. The opposite end, referred to as the "application end", served as the entry point for introducing material into the block.

The cover was designed to prevent movement and stabilize a pair of blocks. It is 18x11x3 mm^3 in size. The cover had a 2-millimeter reinforcement on the edges of its width and length for added strength, while the base of the cover is 1-mm thick. The internal size with a dimension of 14x7x2 mm^3 were specifically tailored to accommodate the blocks securely. This would cause the blocks to protrude approximately 1 mm above the cover. The cover also featured a 3 mm^2 opening at each end, corresponding to the dimensions of the opening in the central tube of the blocks for material application. Additionally, a 3 mm^2 opening was located at the center of the base for observation during experiments and easy removal of the blocks (Figure 2).

Prior to use, each block was undergone thorough cleaning and subjected to an additional round of ultrasonic cleaning to ensure optimal sterility and cleanliness, thus maintaining the integrity of the experiment, and minimizing potential contamination. Then, the blocks were paired within the cover, with the connecting ends aligned towards each other. The connecting ends were ensured to fit snugly within the cover, forming a perfectly interconnected tube.

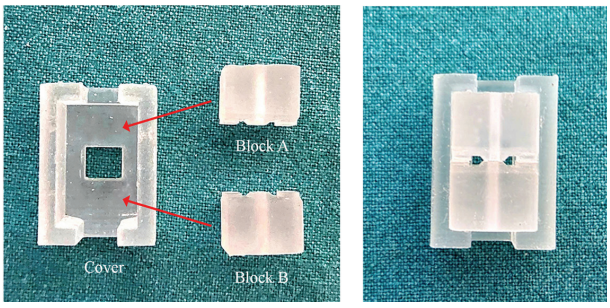

Figure 2: 3D-printed resin block and cover. (Left) Disassembled components, with arrows indicating the insertion of Block A and Block B into the cover. (Right) Assembled unit, showing the cover stabilizing and aligning the blocks for specimen preparation and testing.

The 60 sets of blocks were equally divided into three groups based on the adhesive system used: ExciTE F DSC (Etch & rinse), Multilink N (Self-etch), and RelyX U200 (Self-adhesive). Each group was further subdivided into two subgroups according to the timing of adhesive material placement on Biodentine. In the immediate group, blocks were paired with MultiCore Flow immediately after Biodentine's initial setting (approximately 12 minutes). In the delayed group, blocks were paired with MultiCore Flow after a full 14-day Biodentine setting period.

Biodentine was carefully inserted into each individual block within a set. An endodontic plugger (RCP5/7; Hu-Friedy, Chicago, IL, USA) was used to compress the Biodentine within each block to a consistent thickness of approximately 2 mm. Finally, the paired resin blocks were combined to form the complete set.

Different adhesive systems were applied to Biodentine's interface according to their respective groups: ExciTE F DSC (etch-and-rinse), Multilink N (self-etch), and RelyX U200 (self-adhesive). For each adhesive system, the surface of the resin block was prepared according to the manufacturer's recommended protocol. MultiCore Flow was then applied to achieve a uniform 2-mm thickness to the prepared hole of the other resin block. This prepared block was then connected to the Biodentine block at the varying time intervals described earlier. Subsequently, the MultiCore Flow was light-cured through the resin block using Bluephase N® LED light-curing unit (Ivoclar Vivadent) at HIGH-mode for 20 seconds to ensure complete polymerization at the interface. Subsequently, the samples were incubated at 37°C and 99% humidity for 7 days before undergoing the μTBS test.

After a 7-day incubation, the prepared 3D-printed resin blocks were securely attached to the brass gripped testing fixtures using cyanoacrylate adhesive (Loctite 416; Henkel Corp. Connecticut, USA) to establish a firm connection for testing, The cover was removed prior to the test. Subsequently, μTBS was assessed using an Instron® 5566 universal testing machine (Instron Engineering Corporation, Norwood, MA, USA) at a crosshead speed of 1 mm per minute (Figure 3). The maximum force at failure was recorded in Newtons (N) and the μTBS values were calculated in megapascals (MPa; newton/mm^2) by dividing this force by the cross-sectional area of the bonded region (1 mm^2).

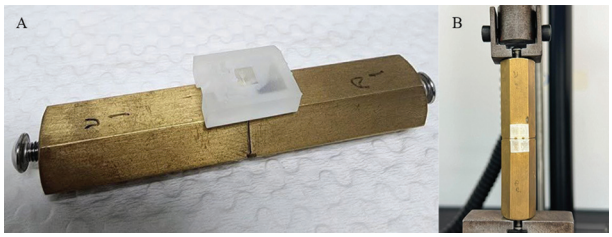

Figure 3: Specimen preparation and μTBS testing. (A), A 3D-printed resin block set (with bonded Biodentine and MultiCore Flow) was attached to the brass testing fixture using cyanoacrylate adhesive: (B), After attachment, the cover was removed, and the specimen was mounted in the universal testing machine for μTBS measurement.

The fractured surfaces of specimens were examined under a 40x stereoscopic microscope (Olympus Corp. Tokyo, Japan) to analyze and categorize into four types of failure modes according to the following criteria:

- Adhesive failure: This occurs entirely between the layers of Biodentine and MultiCore Flow.
- Cohesive failure in Biodentine: This occurs entirely in Biodentine.
- Cohesive failure in MultiCore Flow: This occurs entirely in MultiCore Flow
- Mixed failure: This involves fractures both in Biodentine and MultiCore Flow, as well as between the layers of Biodentine and MultiCore Flow.

The results were presented as mean ± standard deviation (SD) and were subjected to statistical analysis using SPSS 25.0 software (SPSS Inc, Chicago, IL, USA). The μTBS values were assessed for normal distribution using the Shapiro-Wilk test. A two-way analysis of variance (ANOVA) was conducted to assess the presence of significant differences in μTBS values between the groups.

Results

The microtensile bond strength testing

In the immediate group, Multilink N showed the highest μTBS values (18.81±6.61 MPa) compared to RelyX U200 (17.95±4.13 MPa) and ExciTE F DSC (10.66±4.07 MPa). However, in the delayed group, Multilink N showed the lowest μTBS values (6.95±3.76 MPa) compared to ExciTE F DSC (8.39±1.60 MPa) and RelyX U200 (12.26±6.34 MPa).

Within each material group, the delayed subgroups consistently showed lower μTBS values than the immediate subgroups. However, statistical analysis revealed a significant time-dependent effect only in the Multilink N group (p=0.01). Moreover, when comparing material groups, ExciTE F DSC revealed significantly worse than Multilink N (p=0.02) and U200 (p=0.04) in the immediate group. On the contrary, no significant differences were observed among the materials in the delayed group (p>0.05) (Figure 4).

Failure mode distribution

The most common failure modes observed were mixed failures and cohesive failures in the Biodentine in

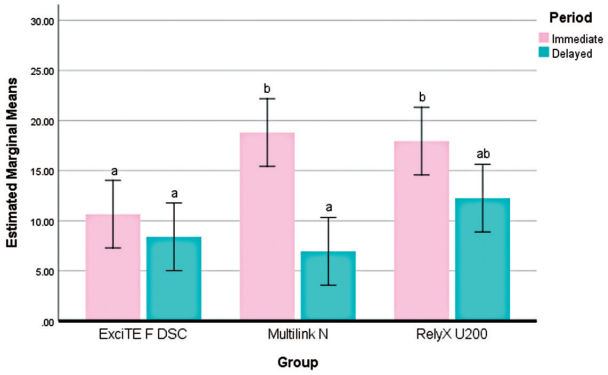

Figure 4: The bar graph of microtensile bond strength by groups (n=10/group). Same letter means no statistically significant difference and different letters means statistically significant difference among the experimental groups.

Table 1: Comparison of microtensile bond strength between different adhesives at different sealer application times.

Group (MPa)	Immediate	Delay
Multilink N	18.81±6.61	6.95±3.76
RelyX U200	17.95±4.13	12.26±6.34
ExciTE F DSC	10.66±4.07	8.39±1.60

Table 2: Effect sizes (Cohen's d) comparing microtensile bond strength between immediate and delayed groups for each adhesive.

Adhesive system	Multilink N	RelyX U200	ExciTE F DSC
Effect size	0.74	0.47	0.34

Table 3: effect sizes (Cohen's d) comparing microtensile bond strength between adhesives at immediate and delayed time points.

Adhesive Comparison	Immediate	Delay
Multilink N/RelyX U200	0.16	1.02
Multilink N/ExciTE F DSC	1.48	0.50
RelyX U200/ExciTE F DSC	1.78	0.84

Table 4: Percentages of failure modes among experimental groups.

Groups	Types of failure (%)	Adhesive	Cohesive in Biodentine	Cohesive in MultiCore Flow	Mixed failure
ExciTE F DSC	Immediate	20	30	10	40
	Delayed	10	40	0	50
Multilink N	Immediate	20	40	0	40
	Delayed	30	30	0	40
RelyX U200	Immediate	0	50	0	50
	Delayed	10	30	0	60

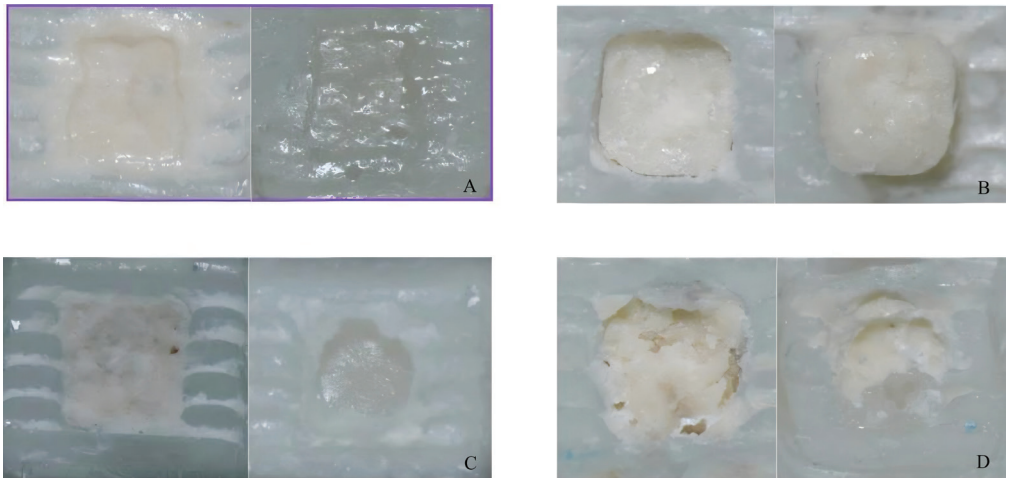

Figure 5: Representative images of the different failure modes observed after microtensile bond strength testing. (A), Adhesive failure at the Biodentine-MultiCore Flow interface: (B), Cohesive failure within the Biodentine material: (C), Cohesive failure within the MultiCore Flow material: (D), Mixed failure.

all experimental groups (Table 4). The different characteristics of failure modes observed are shown in Figure 5.

Discussion

Microtensile bond strength test is a widely used method to evaluate the bond strength between composite materials and dentin. It has also been applied to assess the bond strength between acrylic teeth and denture bases.[12] In this research, we modified the μTBS technique for a novel application in the field of endodontics, specifically focusing on bond strength between endodontic materials like Biodentine and MultiCore Flow with different adhesive systems. This model allows for a standardized and quantitative evaluation of bond strength used in endodontics. The results of this study will contribute to the development and optimization of bonding protocols for endodontic ceramic materials.

In this study, customized 3D-printed resin blocks were developed with a block and cover system aimed at reducing bias. However, further validation is required to confirm their efficacy and reproducibility. Despite the implementation of meticulous protocols, the potential for operational errors to impact on the findings cannot be eliminated. Moreover, μTBS testing may not fully represent clinical performance due to the complexities of the oral environment. Future investigations could benefit from exploring alternative methods, such as tensile tests, which are generally less sensitive and may provide more reliable results.

The observed differences in bond strength between adhesive systems can be attributed to their varying interaction mechanisms with Biodentine. This study found that the etch-and-rinse system, ExciTE F DSC, exhibited lower immediate bond strength, suggesting that acid-etching may not create optimal surface conditions for micro-retention on Biodentine's surface, potentially due to differences in its microstructure compared to dentin. The self-adhesive system (RelyX U200) demonstrated superior immediate

bond strength compared to ExciTE F DSC, possibly due to the chemical interaction of its functional monomers with Biodentine's components. While Multilink N (self-etch) showed high initial bond strength, its significant decrease over time suggests potential hydrolytic degradation at the interface, a known concern with some self-etch adhesives. According to a study by Odabas *et al.,* in 2013[13] studies on various bonding systems for composite resin restorations to Biodentine at different time points have shown that etch-and-rinse systems exhibited a decrease in shear bond strength, which is consistent with this study. The ExciTE F DSC group, one of the etch-and-rinse systems, demonstrated the lowest shear bond strength compared to other groups in the immediate group. Our findings revealed that the self-adhesive system provided superior bond strength compared to etch-and-rinse adhesives system at all time points, particularly in the immediate bonding group. This is consistent with previous studies[14] although the delayed bonding group exhibited some variability. These findings suggest that the acid-etching process, typically known to induce surface porosity and thereby enhancing micro-retention and bond strength, may not facilitate comparable micro-retention in Biodentine, nor may it substantially improve bond strength between the two materials. Alternatively, it is possible that surface porosity from acid etching did not occur, or that the etching duration was either too brief or too prolonged. These results indicate that while the choice of bonding system can influence initial bond strength, other factors, such as clinical variables and material properties, may also contribute to the long-term performance of composite restorations bonded to Biodentine.

When comparing between different time point, The bar graph (Figure 4) shows that the immediate groups exhibited higher bond strength compared to the delayed groups in all experimental conditions. Our results are at odds with those reported by Odabas *et al.,*[13] which reported an increase in shear bond strength when bonding was delayed for 24 hours. They attributed this to the polymerization shrinkage of composite resins, which can induce tensile stresses on the unset Biodentine, leading to interfacial failure. However, our study used MultiCore Flow, a dual-cure material. Odabas *et al.*[15] used Clearfil Majesty (Kuraray Noritake Dental Inc., Okayama, Japan), a nanohybrid composite. While Clearfil Majesty is a high-quality material, studies have indicated it can generate relatively high polymerization stress. Multi Core Flow, in contrast, has been shown to exhibit a lower degree of conversion compared to some light-cured resins[16] and its polymerization stress has been reported as 10.9 MPa, within the range of many resin composites.[17] This lower polymerization stress likely reduces the tensile forces at the Biodentine-adhesive interface, potentially mitigating the negative impact of immediate bonding observed by Odabas *et al.*[13] Despite this, the long-term decrease in bond strength, especially with Multilink N, suggests that factors beyond initial shrinkage stress, such as hydrolytic degradation, significantly influence bond durability. Another possible explanation for the higher bond strength in the immediate groups compared to the delayed groups is the setting reaction between Biodentine and MultiCore Flow, which may have enhanced the interfacial bond. Further studies are warranted to investigate this hypothesis.

A key limitation of this study was the relatively small sample size (n=10) used for microtensile bond strength testing. This small sample size has several implications. First, it increases the risk of committing Type II errors (false negatives). Second, microtensile bond strength measurements are inherently susceptible to high variability, influenced by factors such as specimen geometry, adhesive application, and inherent material properties. A sample size of ten may be insufficient to adequately represent the full range of this variability, obscuring the true distribution of bond strengths. Furthermore, the limited sample size reduces the external validity of our findings. Finally, with a small sample, the results are more susceptible to being skewed by outlier values. Anomalous bond strengths, arising from premature failures during specimen preparation or inconsistencies during testing, can exert a disproportionately large influence on the overall statistical analysis. Furthermore, while the specific bond strength values obtained in this study may not be directly transferable to other commercially available materials, the relative performance of the tested groups could offer valuable insights for clinicians when selecting materials. While this *in vitro* study provides valuable insights into μTBS of endodontic materials, further research is essential to bridge the gap to clinical outcomes. Studies replicating the oral environment, including thermocycling to simulate temperature fluctuations, could offer a more accurate assessment of long-term bond stability. Additionally,

investigating the chemical interactions between endodontic and restorative materials and potential changes within these materials during the setting time could offer a deeper understanding of the bonding mechanisms. Such knowledge could play a critical role in developing improved material formulations and enhancing their clinical performance.

Conclusions

The immediate placement of adhesives on Biodentine demonstrated superior microtensile bond strength than the delayed placement. The self-adhesive system consistently had strong bond strengths at both time intervals. It is suggested that immediate placement and use of self-adhesive system may enhance the bond strength between Biodentine and MultiCore Flow in clinical applications.

Conflicts of Interest

The authors declare no conflict of interest.

References

1. Henry PJ, Bower RC. Post core systems in crown and bridgework. Aust Dent J. 1977;22(1):46-52.
2. Ray HA, Trope M. Periapical status of endodontically treated teeth in relation to the technical quality of the root filling and the coronal restoration. Int Endod J. 1995;28(1):12-8.
3. Tronstad L, Asbjørnsen K, Døving L, Pedersen I, Eriksen HM. Influence of coronal restorations on the periapical health of endodontically treated teeth. Endod Dent Traumatol. 2000;16(5):218-21.
4. Han L, Okiji T. Bioactivity evaluation of three calcium silicate-based endodontic materials. Int Endod J. 2013; 46(9):808-14.
5. Lucas CP, Viapiana R, Bosso-Martelo R, Guerreiro-Tanomaru JM, Camilleri J, Tanomaru-Filho M. Physicochemical properties and dentin bond strength of a tricalcium silicate-based retrograde material. Braz Dent J. 2017;28(1): 51-6.
6. Nayak G, Hasan MF. Biodentine-a novel dentinal substitute for single visit apexification. Restor Dent Endod. 2014;39(2):120-5.
7. Tolibah YA, Kouchaji C, Lazkani T, Ahmad IA, Baghdadi ZD. Comparison of MTA versus Biodentine in apexification procedure for nonvital immature first permanent molars: a randomized clinical trial. Children (Basel). 2022;9(3): 410. doi: 10.3390/children9030410
8. Yadav A, Chak RK, Khanna R. Comparative evaluation of mineral trioxide aggregate, biodentine, and calcium phosphate cement in single visit apexification procedure for nonvital immature permanent teeth: a randomized controlled trial. Int J Clin Pediatr Dent. 2020;13(Suppl 1):S1-s13.
9. Hardan L, Mancino D, Bourgi R, Alvarado-Orozco A, Rodríguez-Vilchis LE, Flores-Ledesma A, *et al.* Bond strength of adhesive systems to calcium silicate-based materials: a systematic review and meta-analysis of *in vitro* studies. Gels. 2022;8(5): 312. doi: 10.3390/gels8050311.
10. Rosner B. Fundamentals of biostatistics: 7th ed. Boston : Brooks/Cole, Cengage Learning, 2011; 2011.
11. Mustafa RM, Al-Nasrawi SJH, Aljdaimi A. The effect of biodentine maturation time on resin bond strength when aged in artificial saliva. Int J Dent. 2020;2020. doi: 10.1155/2020/8831813.
12. Pashley DH, Carvalho RM, Sano H, Nakajima M, Yoshiyama M, Shono Y, *et al.* The microtensile bond test: a review. J Adhes Dent. 1999;1(4):299-309.
13. Odabaş ME, Bani M, Tirali RE. Shear bond strengths of different adhesive systems to biodentine. Sci World J. 2013;2013:626103. doi: 10.1155/2013/626103.
14. Hashem DF, Foxton R, Manoharan A, Watson TF, Banerjee A. The physical characteristics of resin composite-calcium silicate interface as part of a layered/laminate adhesive restoration. Dent Mater. 2014;30(3):343-9.
15. Yamamoto T, Kubota Y, Momoi Y, Ferracane JL. Polymerization stresses in low-shrinkage dental resin composites measured by crack analysis. Dent Mater. 2012; 28(9):e143-9.
16. Alshabib A, Jurado CA, Azpiazu-Flores FX, Aldosary K, Tsujimoto A, Algamaiah H. Mechanical properties and degree of conversion of resin-based core build-up materials and short fiber-reinforced flowable resin-based composite. Dent Mater J. 2024;43(3):453-9.
17. Domarecka M, Szczesio-Włodarczyk A, Krasowski M, Fronczek M, Gozdek T, Sokołowski J, *et al.* A comparative study of the mechanical properties of selected dental composites with a dual-curing system with light-curing composites. Coatings. 2021;11(10):1255.