Bulgarian Chemical Communications, Volume 57, Special Issue E (pp. 23-28) 2025 DOI: 10.34049/bcc.57.E.SIMONS04

Adhesive bond between dentin and CROWNTEC with different printing orientations

M. Dimova-Gabrovska[1], Y. Brusarska[1], E. Kirilova[2*], S. Yanakiev[3], Z. Tomova[4], S. Rangelov[1]

[1]*Department of Prosthetic Dental Medicine, Faculty of Dental Medicine, Medical University – Sofia, 1 Sv. Georgi Sofiyski Str., Sofia 1431, Bulgaria*

[2]*Institute of Chemical Engineering, Bulgarian Academy of Sciences, Acad. G. Bontchev, Str., Bl. 103, 1113 Sofia, Bulgaria*

[3]*Medical College "Yordanka Filaretova" – Sofia, Medical University – Sofia, 3 Yordanka Filaretova Str., Sofia 1431, Bulgaria*

[4]*Department of Prosthetic Dental Medicine, Faculty of Dental Medicine, Medical University -Plovdiv, Hr. Botev Blvd. 3, 4000, Bulgaria*

Received: May 02, 2025; Revised: June 06, 2025

In prosthetic dental medicine, the success of treatment with fixed restorations depends on numerous key factors. Among them, achieving a strong and durable adhesive bond between the material and natural tooth tissues is of paramount importance for the longevity and effectiveness of any restoration. The present study aims to investigate, in a laboratory setting, the adhesive bond between the composite material CROWNTEC and the dentin of natural teeth using the RelyX Unicem adhesive system. The test specimens were evenly distributed into three groups according to the angle of the normal vector to the surface of the printing platform (angles of 0º, 45º, and 90º). The samples were bonded to previously prepared dentin plates. A universal testing machine MultiTest 2.5-i was used to conduct the tests with a shear bond strength testing fixture. From the conducted study, we established that the highest shear bond strength was recorded in group C - 3.000 MPa, followed by group A with a value of 2.4 MPa and group B with 2.0 MPa. In conclusion, it can be summarized that the best adhesive bond between the material and the dentin surface forms when the material layers of the test specimens are positioned horizontally during the printing, while the weakest adhesive bond forms when the material layers intersect at a 45º angle.

Keywords: additive manufacturing; adhesive bond; composites; shear bond strength.

INTRODUCTION

Technological advancements worldwide have a direct impact on treatment protocols in dental medicine [1-3]. Prosthetic dental medicine, a specialty fundamentally aimed at improving both function and aesthetics, is at the forefront of adopting innovative technologies [4-7].

Computer-Aided Design and Computer-Aided Manufacturing (CAD/CAM) is a technology created in the 1970s that has been repeatedly proven for the purposes of prosthetic dental medicine [8-12]. The system is based on digital scanning and designing of a virtual CAD model, followed by the construction of actual prosthetic restorations (CAM) [13-18]. Digital modeling is also the first step in 3D printing technology—a process increasingly used in the fabrication of removable and fixed prosthetic restorations [19-22]. "Additive manufacturing" is a term frequently encountered in scientific literature to denote this technology. The meaning of the term refers to the essence of the process—building a 3D model through layer-by-layer addition of material [8, 23, 24].

Materials used for additive manufacturing of prosthetic restorations must possess mechanical and physical properties close to those of the tissues they are meant to replace [25-28].

The specific needs of permanent restorations in prosthetic dentistry also necessitate the development of hybrid materials suitable for the additive manufacturing of permanent fixed restorations [5, 8, 19, 29]. Such a material was developed in 2022 by the company SAREMCO (Switzerland)—a light-curing hybrid composite with embedded ceramic particles in its composition: CROWNTEC (SAREMCO, Switzerland) [30, 31].

A crucial factor determining the success of any treatment involving crowns or bridgework is the adhesive bond between the restorative material and the dentin of natural teeth through the use of cement [14, 32].

The effectiveness and strength of adhesion relate to the cement's ability to bond the dentin surface to the surface of the material, with the force required to break this bond referred to as shear bond strength [25, 26, 33]. Shear bond strength is a mechanical parameter that measures the loads needed to deform or destroy materials under the action of opposing forces. The test specimens are two objects adhesively bonded together [25].

* To whom all correspondence should be sent:
E-mail: *elisavetakirilova.iche.bas@gmail.com*

A key requirement for dental cements is their ability to create equally effective bonds both on enamel and dentin surfaces [30]. The structure of dentin in natural teeth varies greatly over time, influenced by factors such as the patient's age and the number and size of dentinal tubules, while the enamel surface remains homogeneous [35, 36]. Other factors affecting good adhesion between tooth dentin and restorative materials include excessive moisture or over-drying of the tooth after etching with phosphoric acid [37, 38].

The bond between dental structures and adhesive cements is achieved by the replacement of minerals from the hard-dental tissues with monomer molecules from dental cements [39].

Each type of material requires specific preparation of both the dentin surface and the material surface [33, 40-43]. Regarding resin-based materials such as CROWNTEC (SAREMCO, Switzerland), surface preparation usually involves sandblasting with aluminum oxide or chemical treatment with hydrofluoric acid followed by the application of a silane coupling agent. Hydrofluoric acid creates micro-retentions on the surface, thus increasing the bonding area between the material and the cement. This is also the standard protocol for glass-ceramic surface treatment, although for lithium disilicate, hydrofluoric acid application is not recommended above 4.9% concentration [15, 17, 44, 45].

The adhesion of metal-free restorations is achieved using composite cements, which may be self-curing, light-curing, or dual-curing. Composite cements form a strong chemical bond with natural dental structures, contributing to high shear bond strength [17, 46].

The adhesive cement 3M™ - RelyX™ U200 is divided into two components that are mixed immediately before use. The base paste contains methacrylate monomers with phosphoric acid groups, methacrylate monomers, silanized fillers, initiator components, and stabilizers. The catalyst paste also contains methacrylate monomers, alkaline fillers, silanized fillers, initiator components, stabilizers, and pigments. The cement is dual-curing, meaning the reaction starts either under light exposure or through the chemical reaction of the initiator [47]. Literature data indicate that this composite cement adheres better to tooth enamel than to dentin [47-50].

According to the scientific literature, enamel tissue exhibits better adhesion through various composite cements, while the strength of adhesive bonding to dentin remains a challenge [30, 34, 51].

AIM

The aim of the present study is to investigate, in a laboratory setting, the adhesive bond between test specimens produced from the composite material CROWNTEC (SAREMCO, Switzerland) and the dentin of natural teeth, using the RelyX Unicem (3M) adhesive system.

Null Hypothesis (H_0): Shear bond strength is comparable across all specimens' groups.

Alternative Hypothesis (H_1): Shear bond strength varies significantly among specimens' groups.

MATERIALS AND METHODS

For the purposes of this study, 45 test specimens were fabricated using a NextDent 5100 3D printer (NextDent, USA), based on digital light processing (DLP) technology, from the composite material CROWNTEC (SAREMCO, Switzerland), which contains ceramic fillers.

The specialized software "3D Sprint" was used for the design of the digital prototypes.

The samples were divided into three groups based on their spatial orientation during printing. The normal vector to the surface formed an angle with the printing platform surface as follows: 0º for Group A; 45º for Group B; and 90º for Group C.

The different spatial orientations during the production of the test specimens led to structural differences corresponding to the angles of 0º, 45º, and 90º.

Cylindrical test specimens were printed with these orientations, each with dimensions of 9 mm in diameter and 4 mm in height (Fig. 1A). After printing, each specimen was cleaned by soaking in 96% alcohol and brushing to remove any excess material. Final polymerization was carried out using a UV post-curing unit, the LC-3DPrint Box (NextDent, USA), for 30 min (Fig. 1B).

For the test procedure, dentin plates (Fig. 2) with a thickness of 2.5 mm were prepared from natural extracted teeth with intact crowns, using a microtome. The dentin plates were embedded in epoxy resin.

The bonding of the test specimens to the dentin blocks was carried out using the composite cement RelyX Unicem (3M) according to the following protocol:

A. B.

Figure 1. Printing and post-curing of the test specimens.

Figure 2. Dentin plates.

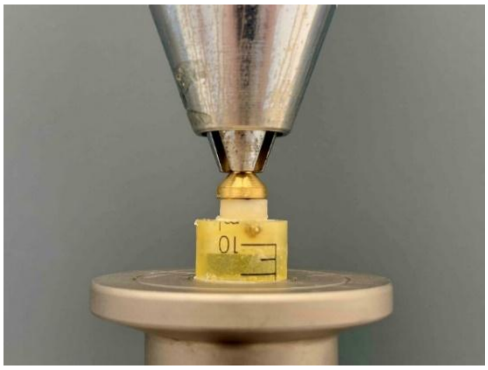

Figure 3. Fixation by applying a weight of 50 N.

- The surface of the specimens was abraded by sandblasting with aluminum oxide (Al_2O_3) particles with a size of 110 μm.
- The specimens were cleaned with alcohol and dried.
- The dentin surface was rinsed and air-dried.
- The required amount of composite cement was applied onto the prepared specimens.
- The specimens were initially fixed to the dentin blocks.
- A universal testing machine for physical-mechanical testing (MultiTest 2.5-i) was used to apply a weight of 50 N during fixation (Fig. 3).
- After the application of the weight to the specimens fixed to the dentin blocks, light polymerization was performed for 5 sec to achieve primary curing and to remove the excess material.
- Final light polymerization was applied for 20 sec.

The shear bond strength tests were conducted using a universal testing machine for physical-mechanical testing (MultiTest 2.5-i) (Fig. 4). The device consists of a monolithic construction with an integrated lead screw drive, to which a strain gauge load cell is attached, measuring the force applied at one end. Depending on the attachments used, the system allows testing of materials under tension, compression, bending, and shear.

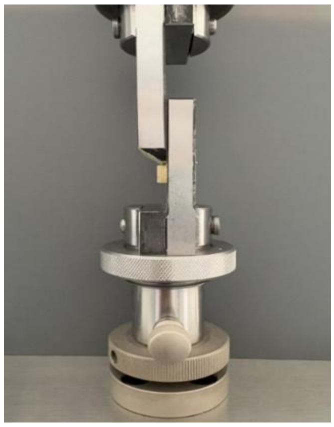

Figure 4. Shear bond strength testing using the MultiTest 2.5-i universal testing machine.

The attachment used for shear bond strength testing consists of two metal plates sliding against each other in a single plane. One plate serves as a "frame" with a pentagon-shaped opening, while the other, known as the "blade," is beveled. The test specimens are fixed at three support points within the stationary frame, with the plane of the cemented bond positioned precisely at the boundary between the "blade" and the "frame" (i.e., between the two metal plates). The movement of the blade is preprogrammed; during the test, the blade encounters a resistive force, which is continuously and automatically recorded until failure of the material occurs.

Statistical methods used

• Descriptive statistics:
- Mean (Average) – a measure of central tendency;
- Median – a measure of the middle value;
- Standard deviation (SD) – a measure of dispersion;
- Lower control limit (LCL) and upper control limit (UCL) – the boundaries within which the true mean of the population lies;
- Absolute values (N) – a measure of the number of cases;
- Minimum and maximum values – the smallest and largest observed measurements.
  - Hypothesis testing:
- Nonparametric test for differences among "k" independent samples (Kruskal-Wallis test);
- Post-hoc test to determine between which of the "k" independent groups the significant differences occur.

All hypothesis testing was performed with a significance level (alpha) set at 5%.

The statistical analyses were carried out using IBM SPSS Statistics 26, and graphical representations were prepared with Excel 2010.

RESULTS

The present study included a total of 45 test specimens, evenly distributed into three groups (A, B, and C), according to the angle of the normal vector to the surface of the printing platform (angles of 0°, 45°, and 90°, respectively). The shear bond strength of the specimens was measured in megapascals (MPa).

The measured average shear bond strength of the tested samples was 1.293 MPa, with a standard deviation of ±0.951 MPa.

The lowest recorded shear bond strength was 0.100 MPa, and the highest was 3.000 MPa, both values observed in the specimens from Group C. In Group A, the highest recorded value was 2.400 MPa, while in Group B it was 2.000 MPa.

Half of the specimens had a shear bond strength below 1.100 MPa, while the other half exhibited higher values (Table 1).

Upon examining and comparing the shear bond strength among the different groups of specimens, the results showed that the highest shear bond strength was observed in Group C (1.840 MPa ±1.093 MPa), while the lowest was recorded in Group B (0.780 MPa ±0.718 MPa). Group A exhibited a mean shear bond strength of 1.260 MPa, with a standard deviation of ±0.728 MPa.

Table 1. Summary of the statistical characteristics of the sample. Unit of measurement: MPa

	Shear bond
Mean	1.293
Median	1.100
Std. dev.	0.951
Min	0.100
Max	3.000
LCL/UCL	1.015/1.571
№	45

The differences between the groups are graphically presented (Fig. 5).

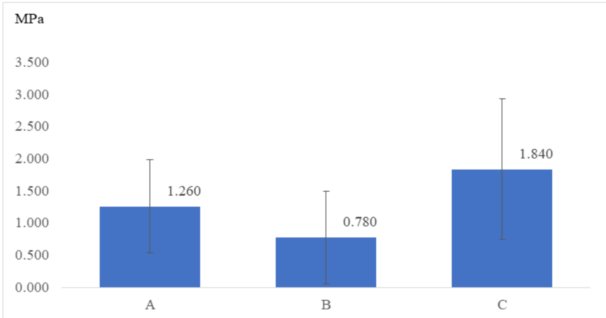

Figure 5. Differences in shear bond strength between the groups (MPa).

Table 2. Results from the test of differences in shear bond strength between the three groups.

Test group / Char. under test	Chars.	Groups under test A	B	C
Shear bond	Mean	1.260 ^{AB}	0.780 ^{A}	1.840 ^{B}
	SD	±0.728	±0.718	±1.093
	N	15	15	15
Kruskal-Wallis test	P-value	P=0.018		

The significance level obtained from the test [p = 1.8%] is lower than the accepted risk of error of 5%. Therefore, the alternative hypothesis is accepted, stating that there is a statistically significant difference in the shear bond strength of the test specimens between the groups. To determine more specifically between which groups the differences are significant, a post-hoc test (Least Significant Difference test) was performed. This analysis revealed that a significant difference exists between the shear bond strength of Group B and Group C, with the strength being higher in favor of the specimens from Group C. This conclusion can be stated with 95% confidence. The difference between

Groups A and C is also very close to being statistically significant, but it reaches significance only at a 10% risk of error.

DISCUSSION

The present study clearly demonstrates a marked dependence between the orientation of the test specimens relative to the printing platform and their shear bond strength. Dividing the specimens into three groups based on the angle of the normal vector (0°, 45°, and 90°) allowed for tracking the influence of the printing angle on the mechanical properties of the material.

The highest mean shear bond strength was recorded in Group C (90°) - 1.840 MPa ±1.093 MPa. This result is likely due to the horizontal orientation of the structural layers located at the surface bonded to the dentin blocks. In Group B (45°), the lowest mean shear bond strength was observed - 0.780 MPa ±0.718 MPa, which may be explained by the less favorable arrangement of the material layers at a 45° angle, leading to a weaker adhesive bond. Group A (0°) demonstrated an intermediate mean shear strength - 1.260 MPa ±0.728 MPa - suggesting that a vertical spatial orientation of the layers is the least favorable for achieving an effective adhesive bond.

Both the lowest (0.100 MPa) and highest (3.000 MPa) individual values among all tested specimens were recorded in Group C. This variability may be attributed to technological factors during the manufacturing process or the presence of microdefects in the material's structure.

The result of the hypothesis test (p = 1.8%) is below the accepted significance level of 5%, which necessitates the rejection of the null hypothesis and acceptance of the alternative hypothesis—that is, there is a statistically significant difference in shear bond strength among the groups. This difference was specifically established between Groups B and C, in favor of Group C, indicating that specimens with horizontally oriented layers demonstrate superior mechanical properties, with 95% confidence. Although a difference between Groups A and C also exists, it would only reach statistical significance at a risk level higher than 10%.

The results of the laboratory study regarding the influence of this spatial orientation during printing on the shear strength are in accordance with those reported by Khanlar *et al.* [52] The results of their study confirm that the horizontal orientation of the layer (90°) leads to a higher shear strength, which corresponds to the values observed in Group C at the core of our study. In [52], lower values for shear strength are observed at angles of 0° and 45°, confirming our hypothesis that the structure of the layer of the material is a key factor for the adhesive bond.

In the review article by Liang *et al.* [53] regarding the influence of parameters on printing, including spatial orientation, the authors emphasized that optimization of the layers is of key importance to achieve better mechanical quality of the material, which confirms the conclusion of the study.

CONCLUSION

The spatial orientation of the test specimens relative to the printing platform of the 3D printer has an influence on the mechanical properties of the material, particularly on shear bond strength. The results from the conducted laboratory tests support the recommendation that, during the digital design and fabrication of constructions made from the CROWNTEC material (SAREMCO, Switzerland), zones subjected to high mechanical loads should have a horizontally oriented structural layer arrangement.

The long-term clinical success of permanent fixed prosthetic restorations is directly dependent on the quality of the adhesive bond between the restorations and the dental tissues. Effective adhesion not only contributes to the stability and durability of prosthetic structures but also reduces the risk of micro-movements and the development of secondary caries, thereby ensuring long-term oral health for patients.

Acknowledgement: *The data presented in this study are subject to investigation in the Research Project Competition "Grant-2024," entry number 132/29.05.2024, at the Medical University - Sofia.*

REFERENCES

1. D. Shopova, D. Bakova, S. Yordanova, M. Yordanova, T. Uzunov, *Appl. Sci.*, **13**, 4335 (2023).
2. Z. Tomova, Y. Zhekov, G. Alexandrov, A. Vlahova, E. Vasileva, *Aust. Dent. J.*, **68**, 294 (2023).
3. M. Dimitrova, A. Vlahova, R. Kazakova, B. Chuchulska, M. Urumova, *J. IMAB - Annu. Proc. (Sci. Pap.)*, **29**, 4939 (2023).
4. H. Hubálková, I. Linetskiy, *Prague Med. Rep.*, **107(2)**, 149 (2006).
5. M. Dimova-Gabrovska, Y. Brusarska, *Sci. Works Union Sci. Bulg.-Plovdiv Ser. G Med. Pharm. Dent. Med.*, XXXI, **118** (2024).
6. M. Dimova-Gabrovska, T. Uzunov, A. Gusiyska, D. Shopova, I. Taneva, I. Gerdzhikov, S. Rangelov, *Appl. Sci.*, **14(9)**, 3923 (2024).
7. J. P. M. Tribst, G. K. R. Pereira, C. J. Kleverlaan, *J. Clin. Med.*, **13(5)**, 1225 (2024).
8. A. Vlahova, S. Zlatev, CAD/CAM technologies in prosthetic dentistry, 2021.

9. W. Grzebieluch, P. Kowalewski, D. Grygier, M. Rutkowska-Gorczyca, M. Kozakiewicz, K. Jurczyszyn, *Materials*, **14(17)**, 4919 (2021).
10. S. Yanakiev, S. Velikov, N. Kostova-Kamburova, M. Moskova, E. Micheva, *Health Sci.*, **1–2(049–050)**, 195 (2023).
11. S. Yanakiev, S. Velikov, N. Kostova-Kamburova, M. Moskova, E. Micheva, *Health Sci.*, **3–4(047–048)**, 48 (2022).
12. M. Dimitrova, M. Corsalini, R. Kazakova, A. Vlahova, G. Barile, F. Dell'Olio, Z. Tomova, S. Kazakov, S. Capodiferro, *J. Compos. Sci.*, **6(7)**, 201 (2022).
13. G. Vico, A. B. Rossi, M. R. Rodrigues, A. L. B. Almeida, A. D. Costa, A. F. Silva, *Oral Implantol. (Rome)*, **1(3–4)**, 104 (2008).
14. M. J. Calheiros-Lobo, T. Vieira, R. Carbas, L. F. M. da Silva, T. Pinho, *Materials*, **16(8)**, 2996 (2023).
15. A. Wiegand, L. Stucki, R. Hoffmann, T. Attin, B. Stawarczyk, *Clin. Oral Investig.*, **19(8)**, 2007 (2015).
16. N. Krajangta, A. Klaisiri, S. Leelaponglit, N. Intralawan, P. Tiansuwan, N. Pisethsalasai, *Dent. Mater. J.*, **43**(3), 386 (2024).
17. A. Mine, M. K. Sato, T. O. Honda, N. Ueda, S. Ueno, *J. Dent. Sci. Rev.*, **55(1)**, 41 (2019).
18. P. Suksuphan, N. Krajangta, P. P. Didron, T. Wasanapiarnpong, T. Rakmanee, *J. Prosthodont. Res.*, **68**(2), 23 (2023).
19. I. Katreva, T. Dikova, M. Abadzhiev, T. Tonchev, D. Dzhendov, M. Simov, S. Angelova, D. Pavlova, M. Doychinova, *Scr. Sci. Med. Dent.*, **2(1)**, 7 (2016).
20. B. Yang, S. Wang, G. Wang, X. Yang, *J. Mech. Behav. Biomed. Mater.*, **124**, 104859 (2021).
21. H. Cai, X. Xu, X. Lu, M. Zhao, Q. Jia, H.-B. Jiang, J.-S. Kwon, *Polymers (Basel)*, **15(10)**, 2405 (2023).
22. D. Shopova, M. Yordanova, S. Yordanova, *Eur. J. Dent.*, **16**, 815 (2022).
23. I. Taneva, 3D-Printed Splints for the Prevention of Complications in Bruxism - Experimental and Clinical Study, Sofia, 2022.
24. D. Shopova, M. Yordanova, S. Yordanova, *Open Access Maced. J. Med. Sci.*, **10**, 6 (2022).
25. I. Anastassov, Y. Yoshida, T. Dragev, Dental Materials, 2013.
26. J. Manappallil, Basic Dental Materials, 2015.
27. O. E. Pecho, R. Ghinea, E. A. N. do Amaral, J. C. Cardona, A. Della Bona, M. M. Pérez, *Dental Materials*, **32(5)**, e105 (2016).
28. X. Z. Liu, P. J. Lu, Y. Wang, *J. Peking Univ. Health Sci.*, **40(6)**, 654 (2008).
29. M. Dimitrova, A. Vlahova, Y. Kalachev, S. Zlatev, R. Kazakova, S. Capodiferro, *Polymers (Basel)*, **15(23)**, 4525 (2023).
30. Permanent 3D printed crowns CROWNTEC, https://saremco.ch/en/products/crowntec.
31. Instruction for Use I saremco print - CROWNTEC I US-version, https://saremco.ch/en/pages/document/gebrauchsanweisungen.
32. C. D'Arcangelo, L. Vanini, M. Casinelli, M. Frascaria, F. De Angelis, M. Vadini, M. D'Amario, *Compend. Contin. Educ. Dent.*, **36(8)**, 570 (2015).
33. R. Vladova, T. Petrova, E. Kirilova, B. Boyadjiev, A. Apostolov, W. Becker, A. Moravski, *Acta Polytechnica CTU Proceedings*, **50**, 94 (2024).
34. J. Perdigão, *J. Dent. Sci. Rev.*, **56(1)**, 190 (2020).
35. D. Damyanova, I. Georgieva, *Am. J. Eng. Res.*, **6**, 130 (2017).
36. I. Georgieva, D. Damyanova, M. Miteva, *Int. J. Sci. Res.*, **6**, 566 (2017).
37. D. McLean, E. Meyers, V. Guillory, K. Vandewalle, *Oper. Dent.*, **40(4)**, 410 (2015).
38. D. Damyanova, *Int. J. Eng. Res. Appl.*, **6**, 68 (2016).
39. B. Van Meerbeek, M. Yoshida, H. Inoue, H. Van Landuyt, F. Fredrickson, *Oper. Dent.*, **28(3)**, 215(2003).
40. Y. Nagasawa, Y. Eda, H. Shigeta, M. Ferrari, H. Nakajima, Y. Hibino, *Odontology*, **110(1)**, 70 (2022).
41. Z. Zhao, Q. Wang, J. Zhao, B. Zhao, Z. Ma, C. Zhang, *Front. Mater.*, **7**, 615225 (2021).
42. T. Petrova, *Bulg. Chem. Commun.*, **55(3)**, 349 (2023).
43. D. Damyanova, R. Andreeva-Borisova, *Int. J. Public Health Sci.*, **10**, 241 (2021).
44. S. Duma, N. Ilie, *J. Funct. Biomater.*, **13(4)**, 217 (2022).
45. A. Chin, M. Ikeda, T. Takagaki, T. Nikaido, A Sadr, Y. Shimada, J. Tagami, *Materials*, **14(22)**, 7058 (2021).
46. A. Heboyan, A. Vardanyan, M.I. Karobari, A. Marya, T. Avagyan, H. Tebyaniyan, M. Mustafa, D. Rokaya, A. Avetisyan, *Molecules*, **28(4)**, 1619 (2023).
47. Instruction for Use, RelyX U200 Self-Adhesive Resin Cement, https://multimedia.3m.com/mws/media/1278181O/3m-relyx-u200-and-ultimate-clicker-easy-use-guide-for-zirconia-and-glass-ceramic-crowns.pdf
48. J. Fehrenbach, E. A. Münchow, C. P. Isolan, L. P. Brondani, C. D. Bergoli, *Int. J. Adhes. Adhes.*, **107**, 102863 (2021).
49. R. F. Rodrigues, C. M. Ramos, P. A. S. Francisconi, A. Flávia, S. Borges, *J. Prosthet. Dent.*, **113(3)**, 220 (2015).
50. T. Rojpaibool, C. Leevailoj, *J. Prosthodont.*, **26(2)**, 141 (2017).
51. M. Le, E. Papia, C. Larsson, *BDJ Open*, **10(1),** 44 (2024).
52. L. Nasiry Khanlar, M. Revilla-León, A.B. Barmak, M. Ikeda, Q. Alsandi, J. Tagami, A. Zandinejad, *J. Prosthet. Dent.,* **129(5)**, 788 (2023).
53. X. Liang, B. Yu, Y. Dai, Y. Wang, M. Hu, H.-J. Zhong, J. He, *Materials*, **18(10)**, 2202 (2025).