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# Bond Strength of a GPDM-Based Self-Adhesive Cement Compared to Conventional Resin Cements on 3D-Printed Crown Material

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# Abstract

Background: The clinical success of 3D-printed permanent restorations depends on the bond durability between the restorative material and resin cements. Comparative data on the performance of conventional and self-adhesive cements on these substrates after aging are limited. The purpose of this in vitro study was to evaluate the shear bond strength (SBS) and microtensile bond strength (μTBS) of four resin cements to a 3D-printed permanent crown material.

Methodology: The shapes of the restorative material used in the study were virtually created with dentalCAD software. For SBS test four circular cement layers were formed on each disc with a 3 mm diameter and 2 mm high transparent silicone index, each polymerized for 20 seconds, resulting in a total of 28 cement layers per group and 112 layers in total. For the SBS tester speed is set to 1 mm/s and for the μTBS test the tester is set to 0.5 mm/s. Fracture surfaces were examined with an electron microscope and stereomicroscope classified into three types; adhesive, mix, and cohesive. One-way ANOVA and Tukey HSD as a post-hoc test were used for statistical analysis.

Result: According to the Post-Hoc Tukey HSD test, significant differences were found between the adhesive resin cements for the SBS and μTBS ($p<0.05$). According to Levene's test and post-hoc Kolmogorov-Smirnov test, there is a statistically significant difference in fracture types ($p<0.05$).

Conclusion: According to the results, Nova Resin resin cement showed the highest bond value for SBS. For μTBS value, Nova Resin cement showed the lowest value among the groups.


# INTRODUCTION

Since the 1980s, CAD/CAM systems have been extensively utilised in dentistry.[1] With the advancement of technology, these systems have undergone continuous evolution. The implementation of CAD/CAM applications in dentistry was initially characterised by the utilisation of subtractive manufacturing techniques.[1,2] The advent of composite materials, accompanied by advancements in their composition, particularly in the domain of paediatric dentistry and the fabrication of single-unit and implant-supported restorations, marked a significant escalation in their utilisation.[3] The evolution of digital technology has precipitated a reduction in treatment duration, a decline in costs, and an enhancement in restoration precision.[1,5] The prevalence of intraoral scanners among practitioners can be ascribed to their ease of operation, their capacity for in-chair production, and the augmentation in measurement accuracy engendered by their extensive utilisation.[1,3]

The primary components of 3D-printed materials utilised for permanent crowns are 'urethane dimethacrylate' (UDMA) and 'ethoxylated bisphenol A dimethacrylate' (Bis-EMA). It is evident that resin-based luting cements demonstrate a significantly higher level of efficacy and durability in restoration procedures when compared to conventional cements.[5] These materials are particularly well-suited for adhesive cementation applications due to their compatibility with luting cement materials based on

resin.[2,6] Moreover, the utilisation of composite resin restorative material facilitates straightforward repairs, a consequence of the incorporation of composite resin components within 3D-printed resin materials.[3,5]

The adhesive molecule composition of composite resin cements is a primary classification criterion, dividing them into two categories: self-adhesive and conventional resin cements. The polymerization type constitutes a secondary classification criterion, dividing them into three categories: light-cure, chemical-cure, and dual-cure.[7] Self-adhesive resin cements can be used without applying a bonding agent to the tooth or material surface thanks to the adhesive compounds they contain. In conventional resin cements, the application of the bond to the surface is a prerequisite for successful adhesion. The utilisation of self-adhesive resin cements facilitates ease of isolation during both use and application.[7,8]

In this study, the bonding strength of a currently used dental material with resin cements was investigated, as has been previously done in laboratory studies. The advent of additive manufacturing techniques and resin materials in dentistry has precipitated a marked increase in the production speed of restorations, thereby reducing the time spent per patient.[5,10] Resin restorations produced with this technique have become increasingly prevalent among dentists, largely due to the advantages of additive manufacturing, such as material savings, the ability to produce in the desired form, and the possibility of bedside application.[7] One of the most significant advantages of resin materials in dentistry is their compatibility with composite-based resin cements utilised for cementation.[11] Consequently, the cementation success of inlays, onlays, partial or full crowns fabricated with permanent 3D-printed materials, and the durability of the restorations have been enhanced due to the adhesion to tooth tissue.[10,11]

The aim of this study was to compare the bond strength of four resin cement on 3D print crown resin material used for permanent crown restoration material using SBS and μTBS tests. The null hypothesis was that there is no statistically significant difference in bond strength between the resin cements.

# MATERIALS AND METHODS

Power analysis was performed a priori using G*Power (3.1.9.7; Heinrich-Heine-Universität Düsseldorf, Düsseldorf, Germany) software for a one-way ANOVA (Fixed effects, Omnibus). Based on a target effect size of Cohen's f = 0.40 (large), the minimum required sample size was calculated as n = 76 for both shear and tensile tests.The calculated statistical power was 0.82, which exceeds the generally accepted minimum threshold of 0.80.[33] Furthermore, the current sample sizes employed in the shear (n = 112) and tensile (n = 80) tests both surpassed the calculated minimum requirement. This demonstrates a high probability of detecting a significant effect with the existing parameters, confirming that the study is statistically robust and adequately powered.

The shapes of the restorative material used in the study for the SBS test were virtually created with the dentalCAD software ExoCAD (Align Technology, Exocad America Inc., USA) with 2mm thick and 20 mm

edge length square specimens. For the μTBS test, cylindrical specimens measuring 1 mm in diameter and 10 mm in length were designed using the ExoCAD software[3]. The resulting designs were subsequently fabricated with a permanent crown resin (Formlabs Inc., Somerville, USA) using a 3D printing device (Formlabs Inc., Somerville, USA)[4].

The adhesive cement groups were divided into four groups: i-Fix Duo(C1)(i-dental, LITHUANIA) and GC-LinkForce (C2)( GC Corp., JAPAN) for conventional dual-cure resin cements, G-Cem ONE (S1)(GC Corp., JAPAN) and Nova Resin (S2) (Imıcrly, TURKEY) for self-adhesive dual-cure resin cement (Fig. 1).

Surface preparation of the all specimen included 600 grit sanding with a water-cooled sander (Metkon Instruments Inc., Bursa, Turkey) followed by cleaning of surface residues with 96% isopropyl alcohol.[6] The materials used and their contents are shown in Table 1.

All samples were subjected to a one-year clinical thermal aging equivalent (SD Mechatronik THERMOCYCLR, Gmbh, Germany) with 5–55°C/10,000 cycles.[9,12]

# SBS Specimen Preparation and Test Application

For SBS test, Four circular cement layers were formed on each disc with a 3 mm diameter and 2 mm high transparent silicone index, each polymerized for 20 seconds. Bond application to the surface was made in groups C1 and C2, which are conventional resins, and G-Bond Premio application to the resin surface from groups S1 and S2 was not made because they are self-adhesive resin cements.a Consequently, a total of 112 cement layers were prepared, corresponding to 28 layers for each of the four cement groups. For the SBS test application, the speed of the universal testing machine (Instron, MIT, USA) was set to 1 mm/s.[14]

## μTBS Specimen Preparation and Test Application

For the μTBS test, the cylindrical specimens were manually aligned in an end-to-end configuration by a single operator. A uniform cement layer thickness was ensured by applying a constant load of 750 g during the light-polymerization process (20 seconds). A bonding agent G-Premio Bond was applied to the bonding surfaces exclusively for the conventional resin cement groups (C1 and C2), in accordance with the manufacturer's instructions. No bonding agent was used for the self-adhesive cement groups (S1 and S2). The prepared samples underwent thermal aging (SD Mechatronik THERMOCYCLR, Gmbh, Germany) with 5–55°C/10,000 cycles.[9,20]

For the μTBS test application, Bisco Microtensile Tester T-1010K (BISCO Inc. Schaumburg, IL, USA) tensile test device was set to 0.5mm/s.[15,16]

## Fracture Surface Analysis

Fracture surfaces were examined by electron microscopy (JEOL JSM 6060 LV,JEOL, Tokyo, Japan) and stereomicroscope (Leica MZ12; Wetzlar, Germany) was examined by a single operator with a magnification of x40. The results were divided into three types according to the amount of cement remaining on the surface of the material; Adhesive, Cohesive and Mix.

# Statistical Analysis

SPSS 26 (IBM-SPSS Inc. Chicago, USA) software was used for statistical analysis. One-way ANOVA calculation and Tukey's HSD correction as post hoc correction were used in the evaluation of SBS and TBS analysis results. For fracture surface statistical analysis were performed with SPSS 26 (IBM-SPSS Inc. Chicago, USA) statistical data analysis program, with a Levene's test and Kolmogorov-Smirnov test as the post-hoc test.

## RESULTS

# SBS Test Results

Homogeneity results within and between groups showed homogeneous distribution within themselves ($p > 0.05$). However, according to the one-way ANOVA test, there was a significant difference between the groups ($p < 0.05$). Descriptive statistics including mean, standard deviation and sample size (n) are presented in Table 2. Tukey's HSD Post Hoc correction was applied to evaluate the differences between each group and the results are shown in Table 3. According to the Post Hoc Tukey HSD test, significant differences were found between G-Cem ONE(S1), Nova Resin(S2), i-Fix Duo(C1), and GC-Link Force(C2) ($p < 0.05$). When the mean differences and estimated marginal values for i-Fix Duo and G-cemONE cement were analyzed, it was found to be less resistant to fracture compared to the other two cements. Nova Resin and GC- LinkFORCE cements showed greater bond strength with no statistical difference between them ($p > 0.05$). There was no difference in bond strength between self-adhesive and conventional resin cement systems.

# µTBS Test Results

According to the Levene test and the homogeneity test results, the groups were homogeneously distributed among themselves, and variances are randomly distributed ($p > 0.05$). Descriptive statistics, including mean, standard deviation, and sample size (n), were used (Table 4). To test the significance between groups, a one-way ANOVA was performed as a parametric test and Tukey’s HSD Post-Hoc adjustment was used to evaluate the differences between each group.

The one-way ANOVA test indicated a significant difference in at least one group ($p < 0.05$). According to the Post-Hoc Tukey HSD test (Table 5), a significant difference was found when examining the mean differences and estimated marginal values for the S2 cement, it was observed to be less resistant in retention compared to the other three cements, indicating lower durability.

## Fracture Analysis Results

Fracture types were examined by electron and stereo microscope. As a result of the examination, the types were divided into 3 different classes as mix, adhesive and cohesive (Fig. 2). The types of SBS and μTBS fracture results are shown in graph of Fig. 3.

Using Levene's test, the p-value was determined to be 0.08 for μTBS and 0.37 for SBS. The test results showed that the groups were evenly distributed among themselves ($p > 0.05$). As a result of the Kolmogorov-Smirnov test, the p-value for SBS was 0.85, and the p-value for μTBS was 0.68. According to the test results, the groups had normal distribution.

A one-way ANOVA analysis revealed a statistically significant difference between the groups, with a p-value of 0.01 for SBS. Cohesive fracture type was found more than the other two types. There is no difference between mix and adhesive fracture types. The p-value for μTBS was calculated as 0.003 as a result of one-way ANOVA analysis. Mix type fracture was statistically significantly less than the other two types among all specimens. There was no difference between adhesive and cohesive fracture types ($p > 0.05$).

# DISCUSSION

Recent studies have shown that the use of 3D-printed dental resin materials as permanent tooth restorations is becoming increasingly widespread.[5,11] The most significant advantages of these smaterials include rapid production capability and their ability to minimize wear on opposing dentition.[17] Undoubtedly, one of the most critical stages in the success of a restoration is the cementation process.[16] The compositional similarity between dental resin crown materials and composite-based resin cements positively influences the adhesion of the restoration to tooth structures.[19] For this reason, in our in vitro laboratory study, we compared the shear bond strength (SBS) and microtensile bond strength (μTBS) values of four different composite-based adhesive cements applied to a 3D-printed resin material used for permanent crown fabrication.[15,18]

The null hypothesis was rejected as there was a statistically significant difference between the resin cements. S2 self-adhesive resin cement and C2 conventional resin cement, showed a higher bond strength value than the other two cements for the SBS test. For the μTBS value, S2 showed a lower value than the other three cements. There was no statistical difference between the other three cement groups. When the fracture types were analyzed, S2 cement showed the most cohesive type. This may be related to the low μTBS value and the physical strength of the cement may be weaker than the other three cements.

In the present study, an investigation was conducted into the values for the SBS test, and it was established that S2 and C2 cements exhibited higher values with regard to connection strength in comparison to C1 and S1 cements. The investigation revealed no statistically significant variation in

bond strength values among the adhesive connection systems. The highest bond value of S2 cement may be attributable to the increase in bond strength of the carboxylic and phosphate groups of GDPM and 4-META monomer in its content. In their study, Kodeira et al. reported that the bond strength increased as a result of the formation of a strong phosphate bond with the resin through the phosphate group contained in the GDPM monomer.[20] Calamita et al. reported that the binding strength of 10-MDP and GDPM functional monomers on zirconia increased and strong chemical bonds were formed between the phosphate groups of both monomers. It is hypothesized that this leads to an increase in the chemical bond strength of the 10-MDP in the G-Premio Bond content and the GDPM monomers in the Nova RESIN self-adhesive resin cement content.[21] The higher bonding strength of Nova RESİN self-adhesive resin cement compared to other adhesive cements may be attributable to the presence of these monomers in the content.

The structure of the C2 conventional resin cement contains similar molecules Bis-EMA as 3D-print permanent resin material. Since the structures of these resin-based ceramic materials are similar, it is possible that the bond strength of C2 cement to this resin material decreased less after thermal cycling than other cements, suggesting that C2 cement has stronger bond strength. In a study, SBS test results after G-Premio Universal adhesive and C2 adhesive cement application on Cerasmart 270 hybrid ceramic material show similar values to our study.[22] Daneshpooy et al. compared Self-etch and etch&rise systems in their study, and as a result, no significant difference was found in terms of microtensile bond strength to the composite surface.[23] In our study, we applied G-Premio universal adhesive to the resin material surface without applying it and found the connection values (MPa) to be compatible with previous studies.[18,19,23]

As a result of the μTBS test, the connection force of S2 cement was found to be statistically significantly lower than S1, C1, C2 cements. When the fracture types are examined, it is likely that the high cohesive fracture of S2 cement is due to the physical property of the cement rather than suggesting that the bond strength may be low. In addition, thermal aging application adversely affects the physical properties of cement.[9,11,12,23] Feng et al. reported that the physical strength of the resin decreased significantly with the increase in the filler content as a result of 5000 thermal cycles and 60-day water immersion tests of composite resins to which they added glass flake filler and polyurethane dimethacrylate, a hydrophilic monomer.[24]

In their study, Toida et al. emphasize the relationship between μTBS value and hardness, emphasizing the importance of evaluating both micro- and nano-scale mechanical properties to better understand the behavior of resin cements. The combined use of nano and micro indentation techniques provides a comprehensive framework for evaluating material performance in μTBS testing.[31] In accordance with the previous study, the resin cement we used had a filler ratio of 70% S2 self-adhesive resin cement is the resin that contains the most filler. In this regard, the effect of cement contents on the physical strength of the resin needs to be investigated further.

The present study has revealed no statistically significant difference between the bond strength of self-adhesive resin cement and conventional composite resin cement. A substantial number of preceding studies in the relevant literature have found no discrepancy between resin cements with two discrete adhesive systems.[4,7,15,27] In a meta-analysis study, Alverange et al. reported that the failure rates of self-adhesive resin cements were not different from the failure rates of conventional adhesive agents in the cementation of indirect single tooth restorations.[30]

Electron microscopy is the most effective method used to evaluate the fracture types, residual cement and surface changes on the material surfaces.[25] In many studies, stereromicroscop and SEM are used especially in the evaluation of the effect of pickling and sandblasting processes on the surface.[26] However, stereomicroscopes with magnification up to x100 can be used to classify the residual cements remaining on the surface after bond strength tests.[27] In this laboratory study, stereomicroscopy and SEM were used to examine the cement surfaces.

Nagaoka et al. after examining the surface of zirconia disks with and without 10-MDP acidic monomer by stereomicroscopy at x40 magnification, showed that the acidic monomer-containing adhesive caused 50% more cohesive fracture on the surface and as a result, the adhesive bond resistance increased.[27] Since S2 resin cement contains acidic monomer (GPDM), we think that it causes more cohesive fracture types in the fracture surface examination. In their study, Elsaka et al. stated that the cohesive fracture type was appropriately correlated with high connection values (MPa).[28] Piwowarczyk et al. reported that the cohesive fracture type may be found to be higher in relation to the higher joint strength before and after thermal aging.[29] In line with previous studies, the higher occurrence of cohesive fractures in SBS and TBS tests when the fracture type analysis is examined shows that the bonding force to the resin material surface is acceptable in the two different types of adhesive cement used.

It is important to consider some of the limitations of the current study, such as its inability to replicate the natural biological oral environment and conditions. The presence of acid produced by intraoral enzymes, bacterial end products, and thermal and chemical changes made it impossible to fully replicate the oral environment. In addition, only four different resin cement and a single 3d-printed resin crown material were used. Long-term clinical studies should be used for the final performance evaluation of materials. After thermal cycles from alternative resin systems, further research is needed to evaluate the binding strength of materials and cement materials. The results of clinical trials will be the main determining factor in this regard.

# CONCLUSION

In the context of the present study, the following results were obtained following the thermal cycling of the bonding strength values of the four resin cements used to the 3D-printed permanent crown resin.

It is evident that the utilization of both the GPDM acidic phosphate monomer, a constituent of the NovaResin Self-adhesive resin cement, and the GC-LinkFORCE conventional resin cement with G-Premio

universal adhesive application, serves to enhance the resistance of these cements to restorative incoming shear forces. This is attributed to the high bond strength of these cements to the 3D-Print resin crown material.

When the fracture types are included in the analysis, there is a high probability that a connection exists between the cohesive fracture of NovaResin cement and the lowest TBS value between the hydrophilic dimethacrylate it contains and the high filler ratio. Consequently, this aspect must be given due consideration when undertaking restorations involving short crown lengths, where the ability to resist tensile forces assumes greater significance.

# Declarations

**Author Contribution**

Murat ŞENOL,DDS, Assistant Prof.

manuscript writer and reviewer, design of laboratory research ,Literature review for article writing, Statistic analysis

Elif Aslı KÖROĞLU,PhD

Manusciript reviewer, conducting laboratory experiments , Literature review for article writing

**Conflict of Interest Statement**

There is no conflict of interest between the authors.

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# Tables

Table 1
Table showing the materials used in the study.

| Materials | Manufacturer | Group | Compositions |
|---|---|---|---|
| Permanent Resin Material and 3D-Printer | Formlabs Inc., Somerville, USA | Methacrylic acid ester-based resin | Organic Matrix: ≥ 50%– < 75% wt. Bis-EMA, 4,4'-isopropylidenediphenol, Diphenyl (2,4,6-trimethylbenzoyl) phosphine oxide <3%<br><br>Inorganic Filler: silanized dental glass (30%–50% wt.) |
| G-Cem ONE (S1) | GC Corp., JAPAN | Self-adhesive composite resin cement | Hydroxy-1,3 dimethacryloxypropane, UDMA, 6-tert-butyl-2,4-xylenol, phenyl(2,4,6- trimethylbenzoyl)phosphine oxide, $Ti_2O$ |
| NovaResin (S2) | Imıcrly, TURKEY | Self-adhesive composite resin cement | Hydrophoblic dimethacrylates (matrix) and acid acrylates,<br><br>glycerol phosphate dimethacrylate (GPDM),<br><br>4-methacryloxyethyl trimellitate anhydride (4-META)<br><br>The filler (more than 70% w/w) is barium glass, silicon dioxide,<br><br>fluoro-alumino silicate, catalysts, and stabilizators |
| i-Fix Duo(C1) | i-dental, LIETUVA | Conventional resin-based adhesive cement | Dental glass grinded based on silica and barium 50–70%, methacrylate mixture<br><br>30–50%, conditioner 1–5%, silicon dioxide 1–5%, initiator < 1%, opacifier < 1%,<br><br>accelerator 1%, inhibitor < 1% |
| GC-LinkForce (C2) | GC Corp., JAPAN | Conventional resin-based adhesive cement | Bis-GMA, Bis-MEPP, UDMA, dimethacrylate, barium glass form (300 nm), reaction initiators, Colorants |
| G-Premio | GC Corp., JAPAN | Universal adhesive | 10-MDP,4-META, MDTP, 4-MET, BHT, acetone, water, dimethacrylate monomer, photoinitiator, silica filler; pH: 1.5 |

Table 2
Descriptive statistics such as sample size (n), mean and standard deviation values (MPa) for the SBS test.

| Cement | Mean(MPa) | Std. Deviation |
|---|---|---|
| C1 | 7,6469 | 3,8773 |
| C2 | 15,7342 | 4,8362 |
| S1 | 9,2579 | 5,7925 |
| S2 | 19,5493 | 7,3531 |

Table 3
Table showing the comparative analysis of the SBS groups to which Post Hoc Tukey HSD test was applied.

| Tukey HSD *(SBS)* | Q statistic | p-value | |
|---|---|---|---|
| C1 – S1 | 1,4988 | 0,6915419 | p > 0.01 |
| C1 – S2 | 11,0497 | 0,0010053 | ** p < 0.01 |
| C1 – C2 | 7,6233 | 0,0010053 | ** p < 0.01 |
| S1 – S2 | 9,3551 | 0,0010053 | ** p < 0.01 |
| S1 – C2 | 6,1245 | 0,0010053 | ** p < 0.01 |
| S2 – C2 | 3,4264 | 0,0792 | p > 0.01 |

Table 4
Descriptive statistics such as sample size (n), mean and standard deviation values (MPa) for μTBS test result.

| Cement | Mean (MPa) | Std. Deviation |
|---|---|---|
| C1 | 81,46 | 4,1395 |
| C2 | 83,68 | 5,0752 |
| S1 | 78,78 | 4,4573 |
| S2 | 56,63 | 3,7624 |

Table 5
These µTBS post-hoc tests would likely identify which of the pairs of treatments are significantly differerent from each other.

| Tukey HSD (µTBS) | Q statistic | p-value | |
|---|---|---|---|
| C1 – S1 | 0,6679 | 0,8999947 | p > 0.01 |
| C1 – S2 | 5,5057 | 0,0011866 | **p < 0.01 |
| C1 – C2 | 0,5021 | 0,8999947 | p > 0.01 |
| S1 – S2 | 4,8378 | 0,0054538 | **p < 0.01 |
| S1 – C2 | 1,17 | 0,8211001 | p > 0.01 |
| S2 – C2 | 6,0077 | 0,0010053 | **p < 0.01 |

# Figures

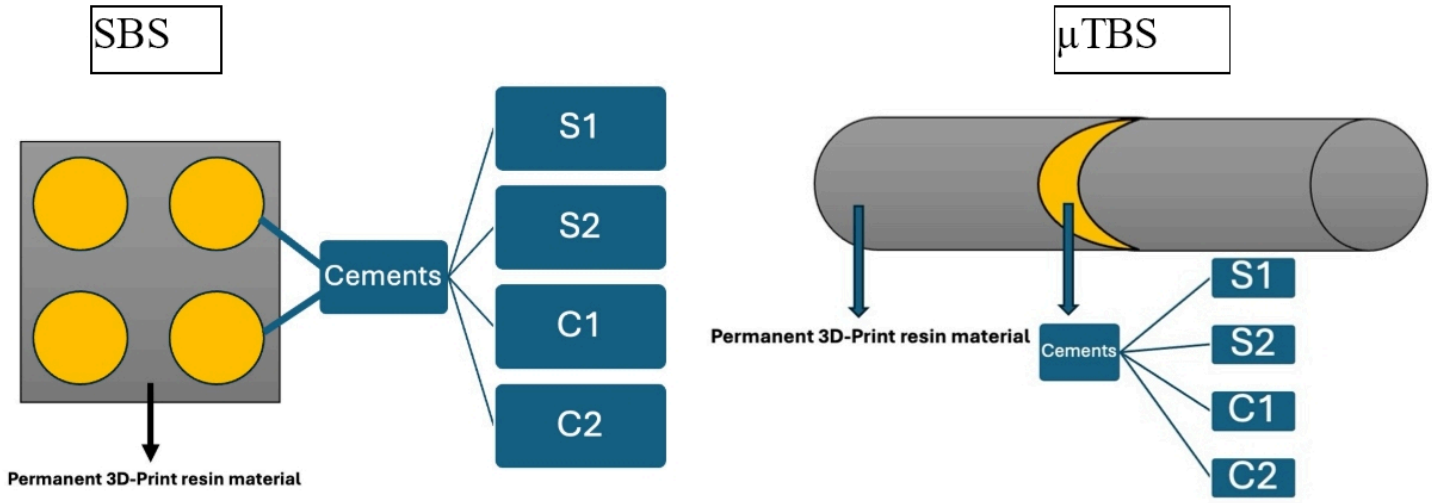



**Figure 1**

Schematic view of adhesive cements applied on restorative resin material SBS and µTBS tests . (S1: GcemONE, S2: NovaResin, C1: I-Fix DUO, C2: GC-LinkFORCE)

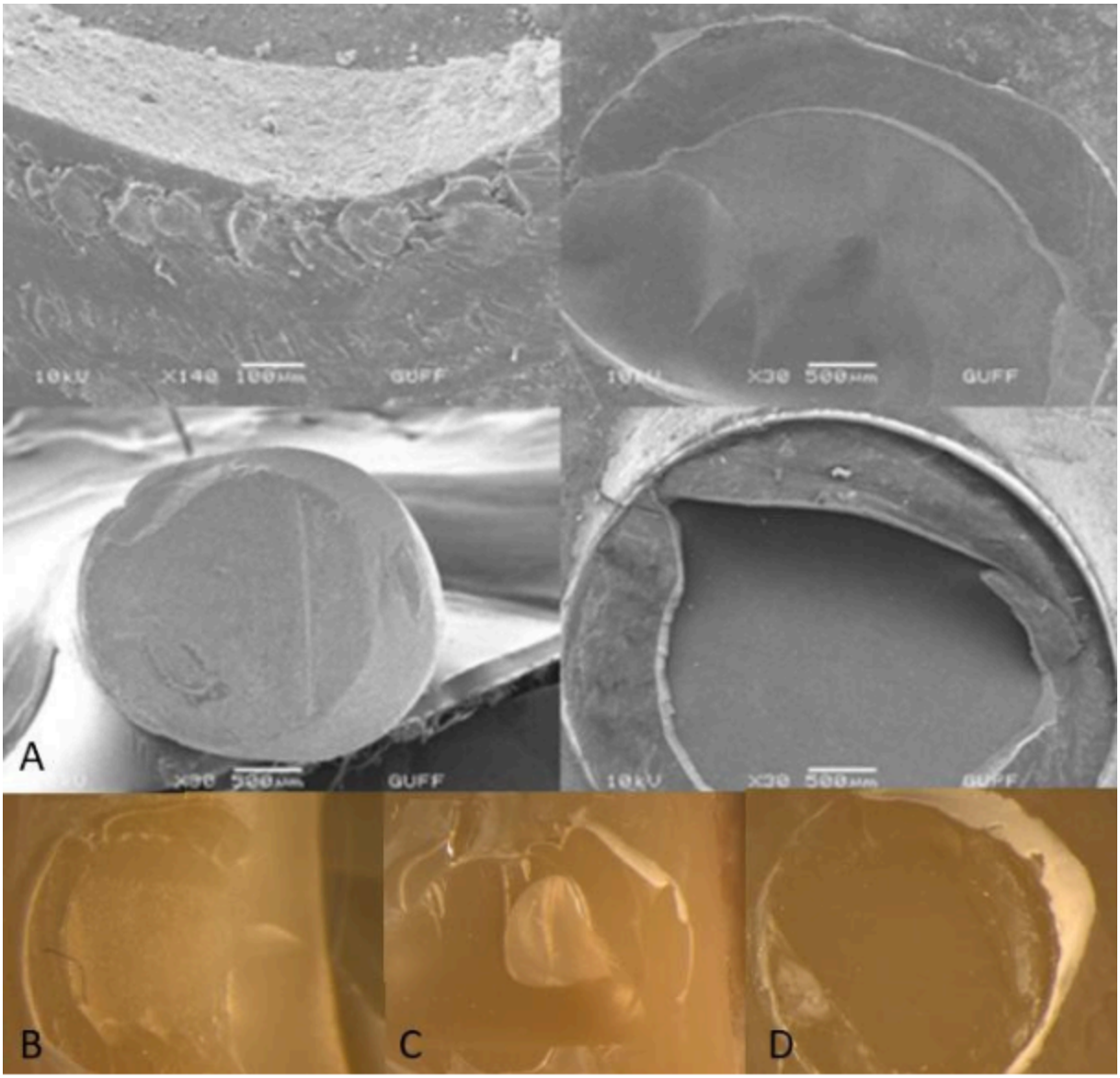



**Figure 2**

Scanning electron microscope images of SBS and μTBS test surfaces (A) and stereomicroscope images of cohesive (B), Mix (C), Adhesive (D).

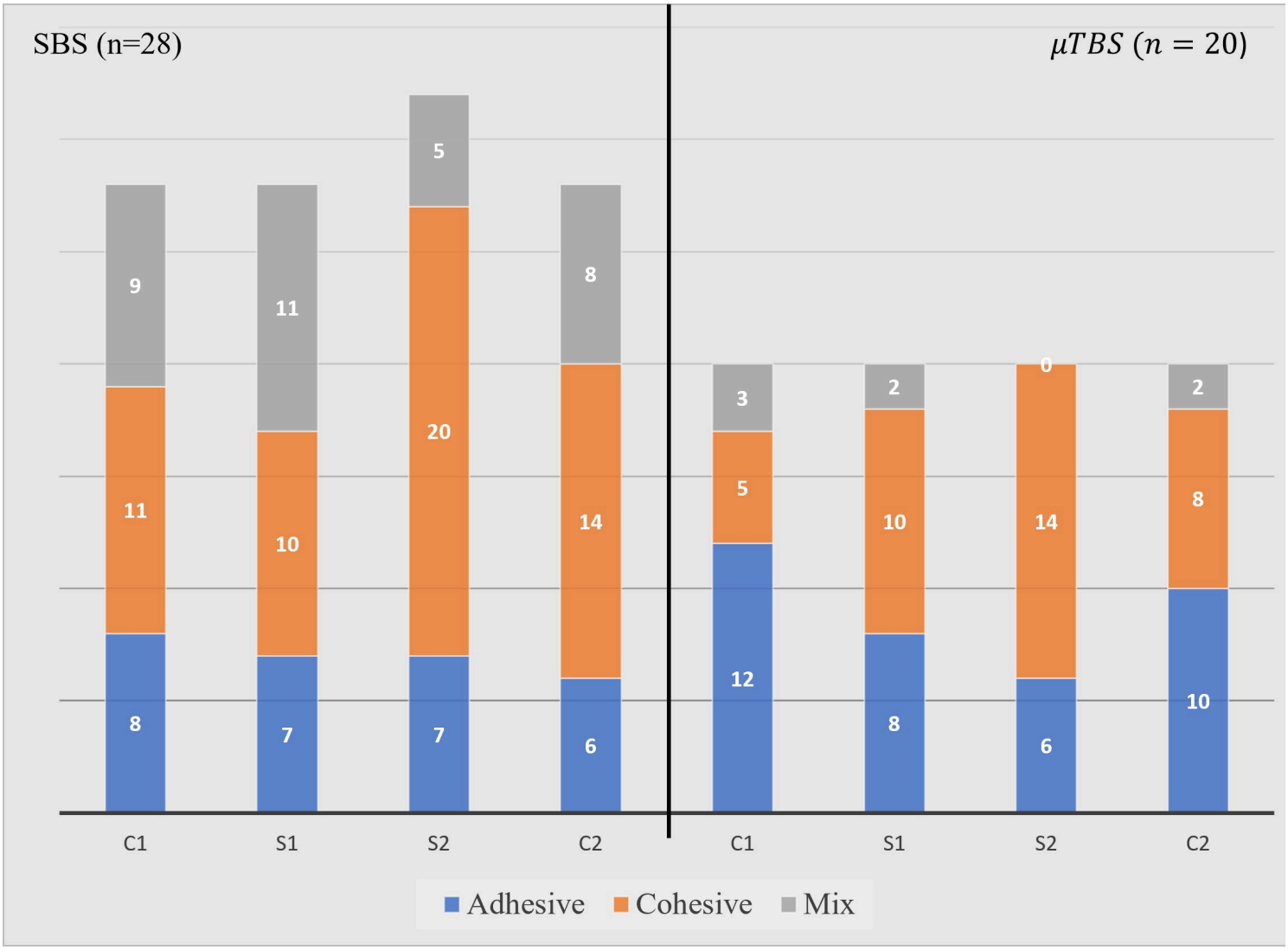



**Figure 3**

Fracture types of SBS and μTBS values (S1: GcemONE, S2: NovaResin, C1: I-Fix DUO, C2: GC-LinkFORCE).