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Immediate and delayed shear bond strength evaluation between root canal sealers and restorative materials: an experimental study

Amira Alghazaly[1], Mahir Mirah[1*] and Somaya Saleh[1,2,3]

Abstract

Background Several calcium silicate-based sealers have recently emerged in endodontics. This study aimed to compare the immediate and delayed shear bond strength between the bioceramic and calcium hydroxide-based sealers and different resin-based restorative materials.

Methods One hundred and twenty specimens with a 3-mm depth and a 3-mm diameter were prepared. They were evenly divided into two groups, the bioceramic sealer and calcium hydroxide-based sealer groups. Each primary group was subdivided into two subgroups based on the restorative material used; i.e., the flowable resin composite and resin-modified glass ionomer subgroups. Moreover, each subgroup was further divided into the restoration process's timing: either immediately post-sealing or delayed after setting the sealers for seven days. The mode of failure was assessed by stereomicroscopic examination.

Results The highest shear bond strength was found when the bioceramic sealer was used and restored with the flowable resin composite. The strengths were 8.45 (1.17) and 6.67 (1.60) megapascals (MPa) in the immediate and delayed restoration groups, respectively. In contrast, the lowest strength, 2.91 (1.22) MPa, was recorded when calcium hydroxide-based sealer was employed and restored after allowing the sealer to set completely with resin-modified glass ionomer. Notably, there were no cohesive fractures within the tested restorative materials. All observed fractures occurred within the sealer materials, at the interface of the sealer and restorative material, or in combination. Moreover, the most common failure was a mixed failure.

Conclusions When flowable resin composite was used immediately before complete setting, bioceramic sealers showed a higher bond strength than calcium hydroxide-based sealers.

Keywords Endodontic sealer, Bioceramics, Shear bond strength, Resin-modified glass ionomer, Resin composite

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Background

Effective sealing of the root canal system is essential to prevent bacterial leakage into the root canal which is the primary reason for treatment failure [1]. This can protect the root canal, system from reinfection and ensure the success of the treatment. To prevent this leakage, root canal obturation is performed to create a sufficient barrier between the restorative materials and the wall of the root canal. Sealers are key elements in the obturation process. To achieve an endodontic monobloc and provide a hermetic seal along the root canal wall, sealers should ideally adhere to the filling material and dentin while maintaining dimensional stability [2, 3]. Unfortunately, even a minimal shrinkage of the sealer can result in a gap at the interface between the sealer and the dentin or gutta-percha. This aperture provides sufficient space for bacteria to penetrate [4].

The first endodontic sealer introduced in clinical practice was a zinc oxide-eugenol based formula, widely used for its sealing ability, ease of use, and compatibility with other endodontic materials [5]. However, in 1984, Sealapex, a calcium hydroxide-based sealer, was introduced [6]. This new formulation had better biocompatibility and more effective antimicrobial properties because of a higher pH [7]. It could also induce hard tissue formation [8]. From its introduction until today, Sealapex is still in use because of its good properties, particularly its ability to stimulate hard tissue formation [9]. This makes it a reliable choice for endodontic applications.

The field of endodontics has witnessed the emergence of various calcium silicate-based (CSC) sealers in recent years, which is attributed to the superior sealing capabilities of CSC cement [10]. These materials seal the root canal through micromechanical interactions via tag-like structures and chemical bonding by forming a hydroxyapatite layer on the root dentin surface [11, 12]. These sealers also exhibit favorable mineralization activity, low shrinkage characteristics, high alkalinity, and biocompatibility [13]. In addition, premixed flowable formulations of these materials are available. Unlike other sealers, CSC materials are usable without the need for mixing, as moisture is essential for setting reactions. Recently, calcium silicate-based materials have been standardized under the name "bioceramics." Bioceramics can interact with tissues to promote growth or stimulate regeneration [14, 15]. Extensive documentation confirms positive contact between CSC materials and the periapical tissues without initiating inflammation or inducing foreign body reactions [16, 17].

The timing of restorative material application can be immediate or delayed, which is crucial for the treatment outcome. Immediate application can reduce the number of visits and provide immediate bonding between the sealer and restorative material [18]. Delayed application can increase the number of visits and the risk of contamination [19]. Some studies reported that SBS is higher in the immediate application of the restorative material [18, 20, 21], while others state the opposite [20, 21, 32]. However, this variation can attributed to the specific materials used, as some performed better in the immediate while others showed better results with delayed application.

New bioceramic products have entered the market with slight differences in formulation and manufacturer instructions. However, to our knowledge, no study has evaluated the interface between NeoSEALER® Flo and Sealapex or the flowable composite Tetric N-flow and Riva Light Cure™. If any of the tested materials exhibit superior bond strength, it could suggest a potential reduction in microleakage, thus lowering the risk of subsequent contamination of the pulp space.

This study aimed to determine the shear bond strength (SBS) of a bioceramic sealer (NeoSEALER® Flo) and a calcium hydroxide-based sealer (Sealapex) when paired with two resin-based restorative materials. The null hypothesis was that there was no significant difference between the tested groups.

Methods

Sample size calculations

The sample size was determined by power analysis conducted using G*Power version 3.1.9.7 [22, 23]. With a significance level (alpha) of 0.05, a power level of 0.95 (i.e., beta = 0.05), and an effect size of 0.822 based on a previous study, the minimum sample size required was found to be 40.

Sample grouping and the treatment procedure

A total of 40 acrylic blocks, each featuring three round spaces with a depth of 3 mm and a diameter of 3 mm, were prepared (Fig. 1), and each block was designed to accommodate up to three specimens. Sixty specimens were fabricated for each tested sealer.

All specimens were divided equally into two groups: Group I, bioceramic sealer (NeoSEALER® Flo; Avalon Biomed Inc, Houston, TX) and Group II, calcium hydroxide-based sealer (Sealapex; Kerr, Romulus, MI). Within both main groups, the specimens were restored with flowable resin composite (Tetric N-flow®, Ivoclar Vivadent, USA) and resin-modified glass ionomer RMGIC (Riva light cure®, SDI, Australia). The detailed compositions of each material used in the experiment are presented in Table 1.

Furthermore, it was further subdivided into two subgroups ($n = 15$ specimens for each subgroup) based on whether or not the restorative material was placed over sealers immediately (within 10 min after placement) or after a delay (seven days after placement). The study groups for the tested materials are illustrated in

Fig. 1 The mold used to prepare the specimens for the SBS experiment

Table 1 Composition of the bonding agent, sealers, and restorative materials used in the study

Product	Composition	Lot number
Universal Adhesive **Tetric N-bond universal**	Phosphoric acid acrylate, HEMA, Bis-GMA, urethane dimethacrylate, ethanol, film-forming agent, initiators, and stabilizers	Z030W1
Flowable composite Tetric N **flow**	Bis-GMA, UDMA, and TEGDMA	Z00Z1B
Resin-modified Glass Ionomer Cement **Riva Light Cure™**	Fluoroaluminosilicate glass powder. Polyacrylic acid, Tartaric acid, 2-Hydroxyethyle methacrylate. Dimethacrylate cross-linker Acidic monomer	J2102227
Bioceramic Sealer **(NeoSEALER® Flo)**	a dicalcium and tricalcium silicate (10% and 25%)-containing sealer containing calcium aluminate, tricalcium aluminate, and tantalite	2,022,111,102
Calcium Hydroxide Sealer **(Sealapex)**	Mixed sealer (polymethylsalicylate) Isobutyl salicylate 42.0 Calcium hydroxide 25.0 Barium sulfate 18.6 Zinc oxide 6.5 Titanium dioxide 5.1 Zinc stearate	8,572,714

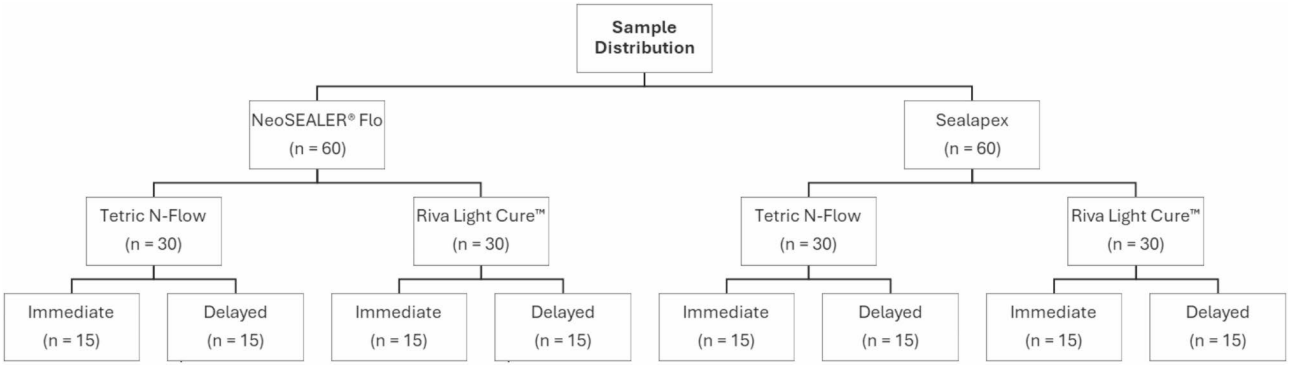

Fig. 2 Diagram illustrating the sample distribution

Fig. 2. Per the manufacturer's instructions, the Sealapex sealer was prepared and placed in a designated cavity within the acrylic block. NeoSEALER® Flo was placed directly within the cavity of the acrylic block. Thereafter, each sealer was subjected to a process of condensation followed by flattening with a glass microscopic slide. This ensures that the sealer adapts optimally to the acrylic block and forms a flat surface.

For the delayed placement restorative groups, the acrylic blocks were carefully stored in an incubator (GI2

So-Low Cincinnati, OH, USA) at a consistent temperature of 37℃ and 100% humidity for seven days to ensure the complete setting of the sealers before restoration. However, the restoration process was initiated immediately after sealer placement in the immediate restoration groups.

A translucent polyethylene mold with a round space of 2 mm depth and 3 mm diameter was precisely positioned over the tested sealer after the application of the adhesive Tetric N-bond universal for both the immediate and delayed groups. Subsequently, either flowable resin composite (Tetric N-flow®) or RMGIC (Riva Light Cure®) was carefully injected into the mold, which was covered with a Mylar strip. The material was flattened and the excess was removed by pressing it with a glass microscopic slide (Fig. 3). The restorations were light cured using an LED polymerization unit, Bluephase C8 (Ivoclar Vivadent Inc., Amherst, NY, USA), with a black 10-mm light probe at a high power of 800 mW/cm^2 for 40 s. The curing tip was maintained perpendicular to the mold surface and centered directly over the material with no distance between the probe and the material.

Following the restorative procedure, all specimens were stored in an incubator for 72 h at a temperature of 37℃ with 100% humidity before the start of the SBS experiment.

The specimens were mounted in an Instron testing machine (model no.8500, Illinois Tool Works Inc., Norwood, MA, USA) with the crosshead perpendicular and flush with the restoration interface and sealer material (Fig. 4). The specimens were loaded at a crosshead speed of 1 mm/min using a knife-edge blade. The SBS was calculated as MPa by applying the following formula:

$$Stress\ (MPa) = \frac{Force\ (N)}{Bonding\ Area\ (mm^2)}$$

All fractured and debonded surface specimens were examined using a Stereomicroscope (SZX16 Olympus, Tokyo, Japan) at a magnification of 40× to identify and categorize the mode of failure into one of the following categories: adhesive, cohesive, or mixed.

Statistical analysis

Statistical analyses were performed using SPSS (version 26, Inc., Chicago, IL, USA). The Shapiro–Wilk test was used to assess the normality of data distribution. The homogeneity of variances was tested using Levene's test. All quantitative data were normally distributed and had homogenous varience, meeting the assumptions for further analyses. Data were analyzed using the three-way ANalysis Of VAriance. The threshold for statistical significance for all tests was set at $P<0.05$.

Ethical approval

The research protocol received ethical approval from the ethics committee of the College of Dentistry, Taibah University with reference no. TUCDREC/240,523/ASAAlghazaly.

Results

The results of the SBS test are presented in Table 2.

The SBS of Tetric N-Flow composite with Sealapex when it was placed immediately and delayed was 4.91 MPa and 3.27 MPa, respectively. In addition, the values when NeoSEALER® Flo was used instead of Sealapex; increased to 8.45 MPa and 6.67, respectively, making it the highest in all tested groups. For Riva LCB, the SBS with Sealapex was 5.03 MPa in the immediate subgroup

Fig. 3 The prepared specimens before incubation

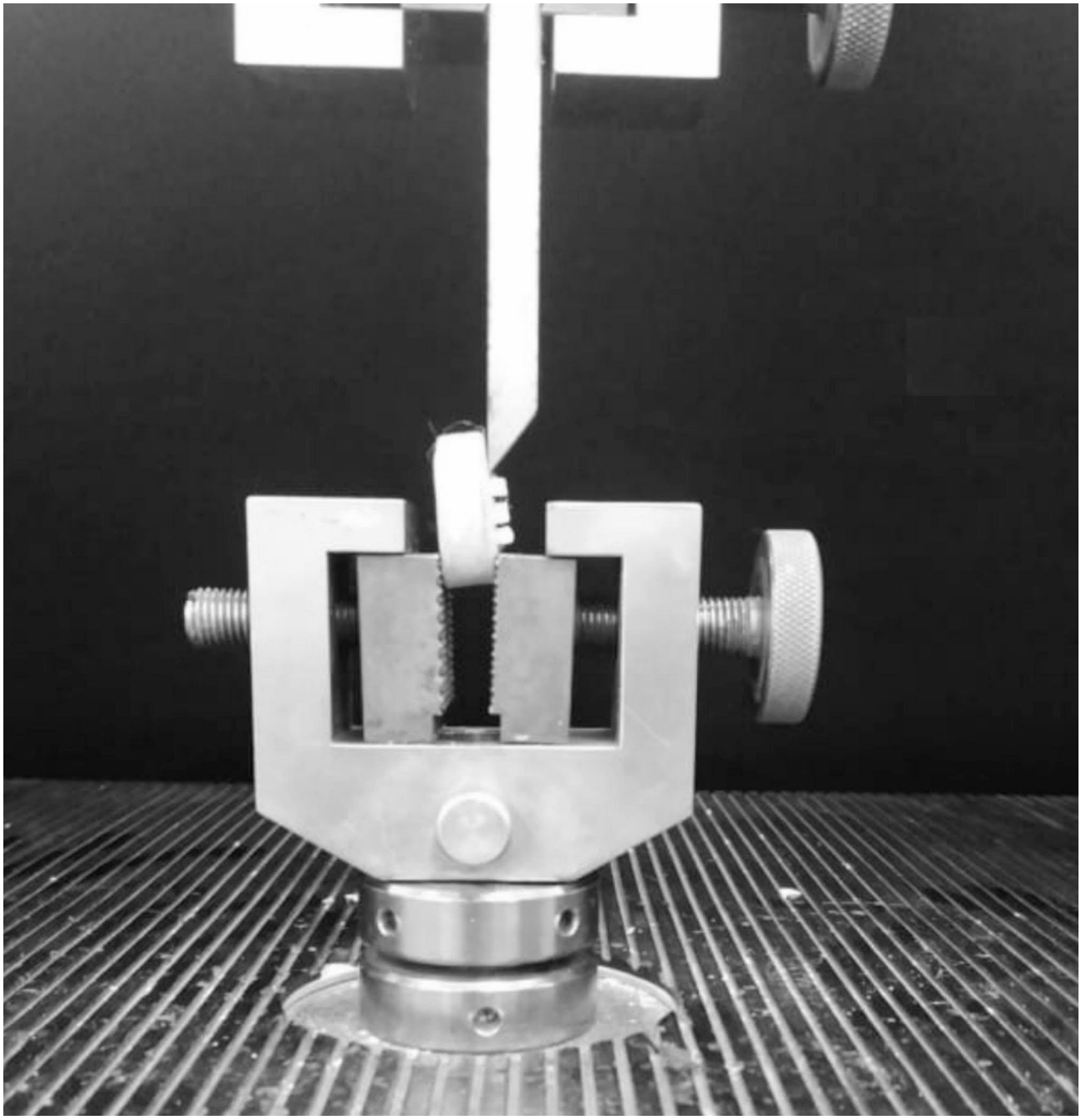

Fig. 4 The mold mounted in the Instron instrument

Table 2 Mean and SD of SBS (in MPa) for the two sealers bonded with various materials

	Shear Bond Strength			
Sealer	**Restoration**			
	Tetric N-Flow		**Riva Light Cure™**	
	Immediate (mean ± SD)	**Delayed (mean ± SD)**	**Immediate (mean ± SD)**	**Delayed (mean ± SD)**
NeoSEAL-ER® Flo	8.45 ± 1.17	6.67 ± 1.60	6.89 ± 1.74	4.60 ± 2.56
Sealapex	4.91 ± 0.60	3.27 ± 1.22	5.03 ± 0.66	2.91 ± 1.16

and 2.91 in the delayed subgroup. On the other hand, when paired with NeoSEALER® Flo, the SBS values were 6.89 MPa in the immediate subgroup and 4.6 MPa in the delayed subgroup.

Overall, no cohesive fractures were observed within the tested restorative materials. All fractures occurred within the sealer materials, at the interface of the sealer and restorative material, or as a combination of the two. Specifically, in the NeoSEALER® Flo groups (regardless of the restorative materials), 40% of the specimens displayed

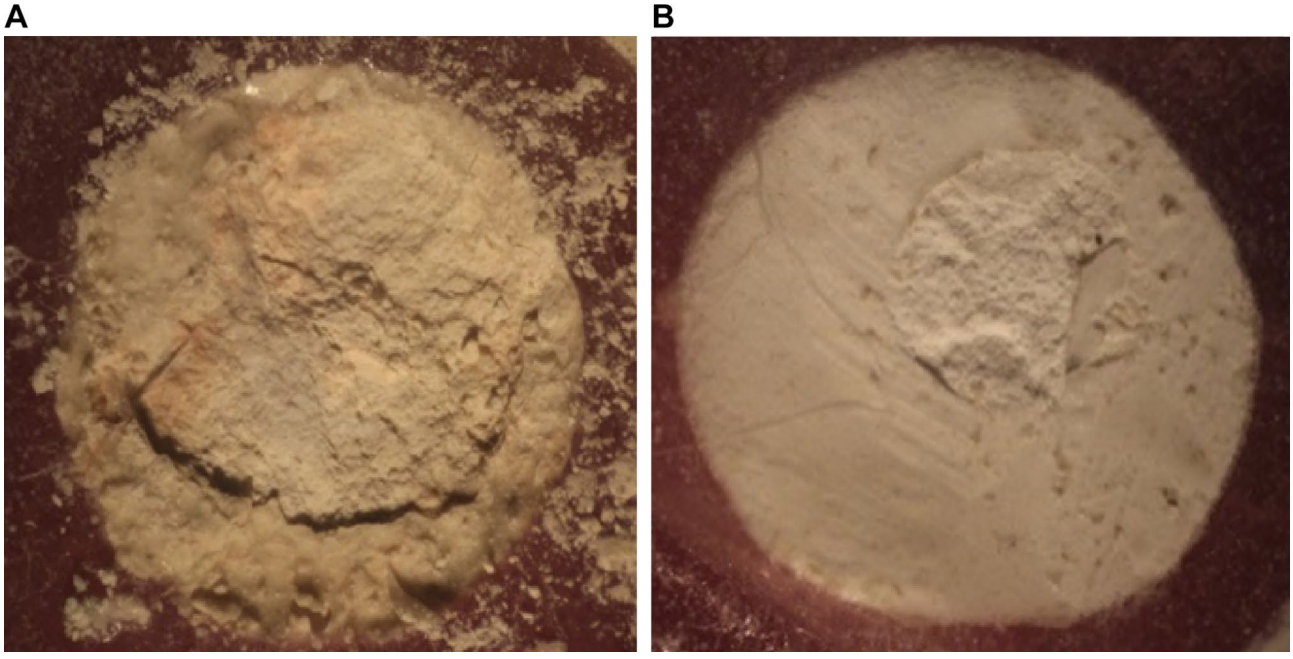

Fig. 5 Stereomicroscopic images representing modes of failure. (**A**) Cohesive failure in Sealapex specimen, (**B**) mixed failure in NeoSEALER® Flo specimen

Table 3 Three-ANOVA for shear bond strength values

Parameter	Sum of squares	Mean square	f-value	*p*-value	Partial eta squared (95% CI)
Restorative material	0.27	0.27	0.09	0.766	0.001 (0.000:0.027)
Sealer	469.26	469.26	154.79	< 0.001*	0.580 (0.481:0.650)
Time	104.35	104.35	34.42	< 0.001*	0.235 (0.128:0.338)
Restorative * sealer	4.00	4.00	1.32	0.253	0.012 (0.000:0.064)
Restorative * time	11.35	11.35	3.74	0.056	0.032 (0.000:0.102)
Sealer * time	5.68	5.68	1.87	0.174	0.016 (0.000:0.074)
Restorative * sealer * time	10.62	10.62	3.50	0.064	0.030 (0.000:0.098)
Error	339.54	3.03			

*significant ($p < 0.05$), eta < 0.02 - Very small, 0.02 <= eta < 0.13 – Small, 0.13 <= eta < 0.26 – Medium, eta >= 0.26 – Large

mixed failures, 30% exhibited adhesive failures, and 30% showed cohesive failures. Conversely, for the Sealapex sealer, 50% of the specimens experienced mixed failures, 30% displayed cohesive failures within the sealer, while 20% showed adhesive failures. Figure 5 Shows stereomicroscopic images demonstrate the cohesive failure in a Sealapex specimen and a mixed failure in a NeoSEALER® Flo specimen.

The statistical analysis is presented in Table 3. Results showed the type of restorative material had no significant effect on bond strength ($p = 0.766$). They also showed NeoSEALER® Flo achieve significantly higher bond strength values than Sealapex ($p < 0.001$). In addition, they showed bond strength values measured immediately to be significantly higher than those measured after storage ($p < 0.001$). All interactions were not statistically significant ($p > 0.05$).

Discussion

Proper cleaning, shaping, and creation of an apical seal on root canal walls is essential for successful endodontic treatment [22]. Despite the materials and obturation methods employed, root canal fillings exposed to saliva are at risk of contamination, which can lead to coronal leakage and negatively impact the outcome of endodontic treatment. Although apical leakage significantly contributes to endodontic failure, techniques aimed at establishing successful coronal sealing following root canal filling have garnered increased attention [19]. This study highlights this important aspect, especially considering the use of various sealers and restorative materials.

Resin materials have gained popularity in restorative dentistry because of their promising esthetic results [11]. The success of any dental restoration depends on proper bonding between the CSC cement and resin components [20]. The bond strength is the most commonly used parameter to assess the adhesive properties of restorative

materials [20, 24]. However, NeoSEALER® Flo must be covered with a restorative substance after its application to prevent bacterial penetration and withstand dislodging forces to ensure a proper seal [15]. The most commonly used restorative materials are resin composites, and the performance of these restorations is significantly influenced by the quality of the bond between the sealer and composite. The bond strength between the resin composite and sealer is a critical clinical factor that determines the efficacy of these treatments [14, 20, 24].

New bioceramic materials recently released offer various benefits. They improve clinical efficiency through significantly shorter setting periods and enhanced handling properties, allowing for the immediate insertion of the final restoration upon completion of regenerative procedures. Bonding operations in this study were performed immediately after the specified setting time by the manufacturers to replicate common clinical applications. This investigation particularly focused on evaluating the SBS between materials used in regenerative endodontic procedures. The concept is that minimizing leakage by improving the bond between components placed coronal to the regenerating canal space could reduce the risk of recontamination within the regenerating pulp space. We acknowledge that the intricate relationship between SBS and microleakage is essential; however, this study does not attempt to evaluate microleakage. Previous research investigated the correlation between microleakage and bond strength or clinical performance, sparking considerable debate over the findings [25].

In our method, we investigated the SBS of the specimens when fabricated immediately and delayed after seven days. Immediate measurement provides insight into the initial adhesive performance of the material, while delayed measurement can assess the long-term stability, which is critical for the evaluation of the material's durability [26, 27]. Additionally, certain materials, such as composite, may require some time, from 24 h to months, for the maturation process to complete [28]. The delayed measurement captures the SBS after this maturation process [27]. In general, the values obtained in the immediate period are higher compared to those evaluated at the delayed period, as the material initially forms a stronger bond, which weakens over time [29]. Evaluating SBS immediately and delayed can help understand the effectiveness of the material at different stages [30].

The storage of the specimens in an incubator at 37 °C for 72 h is known to have significant functions in the experiment. It is used based on previous protocols in researching dental materials whereby the restorative materials should be set and hardened fully [31]. This duration enables the chemical reactions in the components to go to completion as the composite reaches its sustainable physical and mechanical characteristics [32]. The incubator has a proper temperature of 37 °C and proper humidity of 100%, which would be similar to the oral cavity [33]. The recreation of such conditions leads to more practical results, providing similar conditions to the oral environment [30]. In addition, this period establishes the light-cured bond stability of the sealer to the restorative material before proceeding with other tests. This stability is important for assessing the SBS without the interference from the setting and factors that are transient in nature [34]. In this way, the researchers are sure that after incubation, the specimens have been set, the conditions in the laboratory are appropriate, and all results are accurate and can be repeatable.

Numerous studies have investigated the bonding of restorative materials to bioceramics, identifying it as a critical therapeutic concern [26, 27, 35]. In one study by C Retana-Lobo et al. [35], they measured the SBS for AH Plus™, EndoSequence® BC Sealer™, ProRoot® ES, and BioRoot™ RCS on natural teeth. They found that the highest SBS was with BioRoot™ RCS, with a value of around 5.576 MPa. However, the lowest was EndoSequence® BC Sealer™ with a value of around 3.267 MPa. Both were considered bioceramic sealers; the NeoSEALER® Flo has higher values than all the sealers in their study when measured with Tetric N-Flow restorative material. However, when it was measured with Riva Light Cure™, the value was lowered to 4.60 MPa, which is lower than BioRoot™ RCS and higher than EndoSequence® BC Sealer™. This can be because of one of two reasons: the composition of each material can be different than the other, and the other reason is that the SBS of the sealer depends on the material they are using with the sealer. In their study, they used natural teeth, and in our study, we used two different restorative materials.

In another study by KA Hursh, TC Kirkpatrick e al. [36], they measured the bond strength of White ProRoot MTA, Biodentine, EndoSequence RRM Fast Set Putty, and NeoMTA when they bonded with learFil DC Core Plus. The SBS was 4.47 MPa for the EndoSequence RRM Fast Set Putty, and the highest was 7.96 MPa for the White ProRoot MTA, which is in the range of the NeoSEALER® Flo in our study.

However, to the best of our knowledge, no study has examined the materials used in this study. When selecting bioceramic materials, the bond strength between the coronal restoration material and the bioceramic should be considered [37]. Adhesive failure in this study was defined as a failure occurring at the sealer/adhesive interface, even with minimal adhesive resin visible on the sealer substrate. Cohesive failure was noted only when it occurred within the resin composite, RMGIC, or sealer substrate. Failure was classified as mixed if both cohesive and adhesive failures were observed.

SBS measures the maximum stress that the bonding layer can withstand, assessing the integrity of the materials. High bond strength values indicate better interface bonding and reduced microleakage [38]. It is important to note that the stress level required for crack propagation can be significantly higher than the average or nominal value. Because of variations in specimen preparation, storage conditions, loading techniques, and bonding substrate, the nominal bond strength cannot be used to calculate failure stress. Bond strength estimates for specific types of cement can also vary significantly between studies. According to the findings of this study, none of the materials exhibited optimal SBS. In addition, earlier studies showed variations in specimen size dimensions. For example, M Altunsoy et al. [39] used specimens with a 3×1.5 mm central preparation, while K Cantekin and S Avci [27] used 5×2-mm specimens. ME Odabaş et al. [16] and H Çolak et al. [17] used 4×2-mm preparations. After reviewing these studies, we chose a 3×2-mm specimen size for the current investigation to enhance the retention of filling materials. Despite differences in the central preparation dimensions, the dimensions of the restorative materials placed over the sealer were consistent in all previous experiments.

In this investigation, the group in which NeoSEALER® Flo was used exhibited the strongest bonding when applied immediately with the Tetric N-Flow material. This was followed by groups in which NeoSEALER® Flo was applied either immediately or later with Riva Light Cure. The highest SBS value recorded may be attributable to the sealer's inherent self-adhesive properties. This sealer generates hydroxyapatite with dentin, thereby establishing a chemical connection [40]. Its low contact angle and hydrophilic nature also allow it to spread quickly, fostering adaptation and a robust hermetic seal [25]. These characteristics align with those of previous research comparing bioceramic-based sealers to other classes of sealers [25, 35, 41, 42].

The second objective of the investigation was to employ a Stereomicroscope for a detailed examination of the samples to determine the mode of failure. Cohesive failure was a significant mode within both sealers, accounting for 50% of the fractures in Sealapex and 40% in NeoSEALER® Flo. This might be due to the uniformity of the materials, ease of application and mixing, and consistent set times. The distribution of failure locations exhibited a noticeable pattern: it was primarily cohesive within NeoSEALER® Flo or mixed within the Sealapex sealer, suggesting a durable interaction. This occurs when the material itself fractures before the bond between NeoSEALER® Flo and the resin.

However, this study has some limitations. The specimens were created flat with a single interface between the restorative material and the bioceramic-based sealer, not evaluating the impact of dentin on bonding. In addition, the in vitro settings may not accurately simulate the dynamic conditions of the oral environment. Given these limitations, further clinical research is recommended to explore the effects of aging and cyclic loading during mastication on the bonding interface between these materials within the oral cavity. This would help inform the understanding of how the materials respond to the functional environment in the mouth.

The findings of this study should be interpreted cautiously because of the aforementioned limitations.

Conclusions

Upon immediate application, the group using NeoSEALER® Flo along with the Tetric N-Flow restorative material exhibited the strongest bonding. Conversely, the group using Sealapex and Tetric N-Flow demonstrated the weakest bond strength. Clinically, it is recommended to immediately place Tetric N-Flow restorative material over NeoSEALER® Flo sealer to achieve higher bond strength.

Abbreviations

AS	AS
MPa	Megapascals
SBS	Shear bond strength
SEM	Scanning electron microscopy

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Not applicable.

Author contributions

AA conceptualized the study and contributed to methodology and preparation of original draft. SS contributed to data curation, writing, and original draft preparation. MM performed formal analysis and contributed in writing, reviewing, and editing. All authors read and approved the final manuscript.

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Data availability

Shear bond strength data that support the findings of this study have been deposited in OSF with the following link https://doi.org/10.17605/OSF.IO/MXRB9.

Declarations

Ethics approval and consent to participate

Not applicable.

Consent for publication

Not applicable.

Competing interests

The authors declare no competing interests.

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