

ORIGINAL RESEARCH

Effect of different setting times of calcium silicate cements on the bond strength to paediatric restorative materials: an *in vitro* study

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Abstract

Background: This study aimed to evaluate the effect of cement maturation time on shear bond strength (SBS) between three calcium silicate-based cements and paediatric restorative materials. **Methods:** A total of 405 three-dimensional (3D)-printed resin blocks with standardised cavities (4 mm diameter, 2 mm depth) were filled with Biodentine, NeoMTA 2, or NeoPUTTY. A two-step self-etch adhesive system (Clearfil SE Bond 2) was then applied to the calcium silicate cement surfaces, and Dyract XP, Filtek Z550, or ACTIVA Kids BioACTIVE Restorative was placed after 3 min, 15 min, or 24 h using a Tygon tube with a 2-mm internal diameter to standardise the bonding area. Shear bond strength was measured after 24 h of storage at 37 °C and 100% humidity with a crosshead speed of 1 mm/min. Failure modes were examined stereomicroscopically, and selected interfaces were analysed by scanning electron microscopy (SEM). Data were analysed using three-way analysis of variance (ANOVA) with appropriate *post hoc* tests ($\alpha = 0.05$). **Results:** Setting time, cement type, and restorative material significantly affected bond strength ($p < 0.001$). SBS values ranged from 1.74 ± 0.49 MPa in the NeoPUTTY–Dyract XP group at 3 min to 11.55 ± 2.06 MPa in the Biodentine–ACTIVA Kids BioACTIVE Restorative group at 24 h. Bond strength increased progressively with longer setting times, with the lowest values observed at 3 min and the highest at 24 h. Biodentine generally showed higher bond strength than NeoMTA 2 and NeoPUTTY, while ACTIVA Kids BioACTIVE Restorative generally yielded the highest values among the restorative materials. Failure patterns shifted from predominantly adhesive failures at early time points to more mixed or cohesive failures with longer setting times. **Conclusions:** Bonding to calcium silicate-based cements is time-dependent. Within the limitations of this *in vitro* study, bond strength was influenced by setting time, cement type, and restorative material type.

Keywords

Calcium silicate-based cements; Shear bond strength; Biodentine; NeoMTA 2; ACTIVA Kids BioACTIVE Restorative

1. Introduction

Vital pulp therapies (VPTs) represent key biologically based approaches in paediatric dentistry for preserving the vitality of primary and immature permanent teeth. These treatments support pulp tissue healing, maintain tooth function, and, especially in young permanent teeth, allow continued root development [1].

Calcium silicate-based cements (CSCs) are among the most commonly used biomaterials in VPT. Mineral trioxide aggregate (MTA), a well-established CSC, has long been regarded as the “gold standard” because of its sealing ability, antibacterial properties, and potential to induce hard tissue formation [2, 3]. However, conventional MTA has disadvantages that may limit its clinical use, including handling difficulty, risk of

discoloration, and prolonged setting time [2]. A prolonged setting time may delay placement of the definitive restoration, require an additional appointment, and increase the risk of contamination and microleakage, thereby adversely affecting the success of VPT outcomes [4, 5].

To overcome these disadvantages, new-generation CSC systems have been developed. In this context, Biodentine (Septodont, France) has a wide range of indications due to its short setting time and favorable mechanical properties [6]. NeoPUTTY, developed by NuSmile (Houston, USA), is a premixed formulation, while NeoMTA 2 offers ease of use in clinical practice due to its optimized handling characteristics. These next-generation biomaterials represent contemporary and effective options, with reduced setting times and a lower risk of discoloration [7].

The long-term clinical success of VPT depends not only on the biological properties of the pulp-capping material but also on the quality of the interfacial bond formed with the definitive restorative material placed over it [8]. However, the mismatch between the hydrophilic nature and ongoing hydration of CSCs and the hydrophobic character of resin-based restorative materials may compromise interfacial bond strength [9]. The optimal adhesive protocol for bonding to CSCs remains a subject of debate [9–11]. In this context, both total-etch and self-etch strategies have been discussed in the literature. Although phosphoric acid etching used in total-etch systems may enhance micromechanical retention, it has also been reported to adversely affect the surface integrity of CSCs during early setting periods [12]. Therefore, self-etch adhesive systems are often preferred to preserve the surface characteristics of the bioceramic matrix. In addition to micromechanical retention, functional monomers such as 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) in some self-etch systems may contribute to interfacial stability through potential chemical interaction with calcium ions released from CSC surfaces [10, 11].

In paediatric dentistry, the choice of coronal restorative materials is made considering isolation conditions, patient compliance, and material-related mechanical properties. In this context, compomers such as Dyract XP are frequently preferred because of their fluoride release and ease of application, whereas nanohybrid composites such as Filtek Z550 are commonly chosen for their mechanical strength and aesthetic performance [13, 14]. In recent years, ACTIVA Kids BioACTIVE Restorative, described as a “bioactive” material, has attracted attention due to its potential to promote remineralisation [15]. One of the debated issues in clinical protocols is the timing of the definitive restoration following placement of the biomaterial. This issue is particularly relevant in paediatric patients, for whom limited cooperation may often necessitate completing treatment in a single visit. Although previous studies have investigated the bond strength of various restorative systems to CSCs, most have focused on later maturation stages, such as 24 h and beyond, when the material has largely completed its hydration process [8, 9]. However, data on the bonding performance of bioceramics such as NeoMTA 2 and premixed NeopUTTY at the clinically relevant time points of 3 and 15 min remain limited [16–18]. Furthermore, the interaction between ACTIVA Kids BioACTIVE Restorative, a material characterized by bioactive properties and ion release, and the dynamic hydration process of these bioceramics has not yet been investigated, particularly during the critical initial 15 min period.

Therefore, this study aimed to evaluate, under *in vitro* conditions, the shear bond strength between three different calcium silicate-based cements (Biodentine, NeoMTA 2, and NeopUTTY) and three paediatric restorative materials (Dyract XP, Filtek Z550, and ACTIVA Kids BioACTIVE Restorative) applied after different setting periods of 3 min, 15 min, and 24 h. The null hypothesis of the study was that there would be no statistically significant differences in shear bond strength values across setting time, cement type, and restorative material type.

2. Materials and methods

Ethical approval for this *in vitro* study was granted by the Afyonkarahisar Health Sciences University Non-Interventional Scientific Research Ethics Committee (Approval No: 2025/389). Sample size calculation was performed using G*Power software version 3.1.9.7 (Heinrich Heine University Düsseldorf, Düsseldorf, NRW, Germany), based on a similar reference study [19]. An a priori power analysis was conducted within the *F*-test family using the procedure for the planned full $3 \times 3 \times 3$ factorial ANOVA design. Assuming an effect size of $f = 0.25$, a significance level of $\alpha = 0.05$, and a power of 0.80, the minimum required sample size was determined to be 249 specimens. To compensate for possible technical losses, this number was increased by 50%, and the study was completed with a total of 405 specimens. Based on the final sample size, the achieved power of the study was 0.967. All experimental procedures were performed by a single operator (HEK). The materials used are presented in Table 1, and the study workflow is illustrated in Fig. 1.

2.1 Specimen preparation

To ensure standardised positioning of specimens within the universal testing machine (MOD Dental MIC-101, Esetron Smart Robotechnologies, Ankara, Turkey), 405 cylindrical resin blocks with a diameter of 15 mm and a length of 130 mm, featuring a central cavity measuring 4 mm in diameter and 2 mm in depth, were fabricated using a 3D printer (Sonic Mini 8K, Phrozen, Hsinchu City, Taiwan) and a polymethyl methacrylate (PMMA)-based 3D printing resin (NextDent, 3D Systems, Soesterberg, The Netherlands). To equilibrate moisture content and standardize initial conditions across all specimens, the resin blocks were stored in an incubator at 37 °C and 100% humidity for 24 h before the application of CSCs.

The resin blocks were randomly and equally divided into three main groups ($n = 135$ per group), according to the CSC applied: NeopUTTY® MTA, NeoMTA® 2, and Biodentine™. The calcium silicate-based materials were prepared in accordance with the manufacturers' instructions, as specified in Table 1, and condensed into the cavities. To ensure surface standardization and obtain a flat material surface, a glass slab was placed over the materials. The procedures related to the setting times and restorative application stages are detailed below.

2.2 Waiting-time protocol (3 min, 15 min, 24 h)

Following the placement of CSCs, the specimens were divided into three subgroups according to restorative application intervals of 3 min, 15 min, and 24 h. During the setting period, a moist cotton pellet was placed on the material surface of all specimens, and the specimens were stored at 37 °C and 100% humidity. At the end of the duration, the cotton pellet was removed, and restorative procedures were initiated.

TABLE 1. Commercial names and lot numbers, type/form, components, colour, manufacturer, and instructions for use of the materials used in the study.

Commercial names and lot numbers	Type/Form	Components	Manufacturer	Instructions for use
NeoMTA 2 Lot number: 2025031401	Calcium silicate-based root and pulp treatment cement/Powder–liquid gel	Powder: Tricalcium/dicalcium silicate, calcium oxide, tantalum oxide, and auxiliary metal oxides (Al_2O_3 , MgO , Fe_2O_3 , SiO_2), tricalcium aluminate. Liquid: Polymer-based carrier gel containing Ca^{2+}	Avalon Biomed Houston, TX, USA	1. Dispense 1 scoop (0.05 g or 0.1 g) of NeoMTA powder onto a glass slab or a non-absorbent pad. 2. Dispense one drop of NeoMTA Gel next to the powder. 3. Gradually add the gel to the powder. Mix by pressing the powder/gel mixture firmly against the glass slab, ensuring all powder is thoroughly wetted, and continue mixing until a putty consistency is achieved.
Biodentine Lot number: B32664B	Calcium silicate-based cement/Capsule: powder–liquid system	Powder: Tricalcium/dicalcium silicate, calcium carbonate, zirconium oxide, calcium oxide, iron oxide. Liquid: Soluble polymer–water system containing CaCl_2	Septodont; Saint-Maur-des-Fossés, France	1. Open the capsule and place it in the white capsule holder. 2. Detach the single-dose liquid container and tap the closed cap to ensure all liquid is inside. Add 5 drops from the container into the capsule. 3. Close the capsule. Place it in a mixing device operating at a 4000–4200 rpm and mix for 30 seconds.
Dyract XP Lot number: 2507000820	Polyacid-modified composite resin	UDMA, TEGDMA, TMPTMA-based dimethacrylate resins, camphorquinone, amine co-initiator, BHT and UV stabiliser, Sr-alumino-fluoro-silicate glass, SiO_2 , SrF , Fe_2O_3 , and TiO_2	Dentsply Sirona; Konstanz, Germany	1. Place the compule into the Compule Gun. 2. Remove the coloured cap. The compule can rotate 360° to ensure the correct entry angle into the cavity or onto the mixing pad. 3. Cure each layer using a suitable light-curing unit. For 2 mm thick increments, a polymerisation time of 10 seconds is recommended when using a light source with an output $\geq 500 \text{ mW/cm}^2$.
Filtek Z550 Lot number: 11225283	Nanohybrid composite resin	Bis-GMA/UDMA/Bis-EMA/TEGDMA/PEGDMA matrix + ZrO_2 – SiO_2 filler + photoinitiator system + TiO_2 – Fe_2O_3 pigments	3M ESPE; St. Paul, MN, USA	According to the manufacturer's instructions, the shade A2 restorative material is applied in 2.0 mm increments; and polymerization for 20 seconds is recommended with a light source of minimum 400 mW/cm^2 intensity, or for 10 seconds with a minimum intensity of 1000 mW/cm^2 .

TABLE 1. Continued.

Commercial names and lot numbers	Type/Form	Components	Manufacturer	Instructions for use
ACTIVA Kids BioACTIVE Restorative Lot number: 231231	Bioactive composite resin	Diurethane/polyacrylic acid-modified methacrylates, amorphous silica, NaF, and water	Pulpdent Corporation; Watertown, MA, USA	1. Place the ACTIVA tip into the preparation and allow the material to flow slowly. Keep the tip immersed in the material while moving it. Allow time for ionic activity to interact, integrate, and form chemical bonds with the dentine. Ensure no air bubbles remain. 2. Apply in increments ≤ 4 mm and polymerise for 20 seconds using a LED light-curing unit with an intensity ≥ 1000 mW/cm ² .
Clearfil SE Bond 2 Lot number: 000502	Two-step self-etch adhesive system	Primer: 10-MDP, HEMA, hydrophilic dimethacrylates, photoinitiator, and water. Adhesive: 10-MDP, HEMA, Bis-GMA, hydrophilic dimethacrylates, CQ, silanated colloidal silica, and initiator system	Kuraray Noritake Dental Inc.; Okayama, Japan	1. Apply the primer to the surface for 20 seconds. 2. Dry with mild air for 5 seconds. 3. After applying the adhesive, thin it with mild air for 5 seconds. 4. Light-cure for 10 seconds.

Al₂O₃, aluminium oxide; MgO, magnesium oxide; Fe₂O₃, iron oxide; SiO₂, silicon dioxide; Ca²⁺, calcium ion; CaCl₂, calcium chloride; UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; TMPTMA, trimethylolpropane trimethacrylate; BHT, butylated hydroxytoluene; UV, ultraviolet; SrF, strontium fluoride; TiO₂, titanium dioxide; Bis-GMA, bisphenol A-glycidyl methacrylate; Bis-EMA, bisphenol A ethoxylated dimethacrylate; PEGDMA, polyethylene glycol dimethacrylate; ZrO₂, zirconium dioxide; NaF, sodium fluoride; LED, light-emitting diode; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; HEMA, 2-hydroxyethyl methacrylate; CQ, camphorquinone; rpm, revolutions per minute.

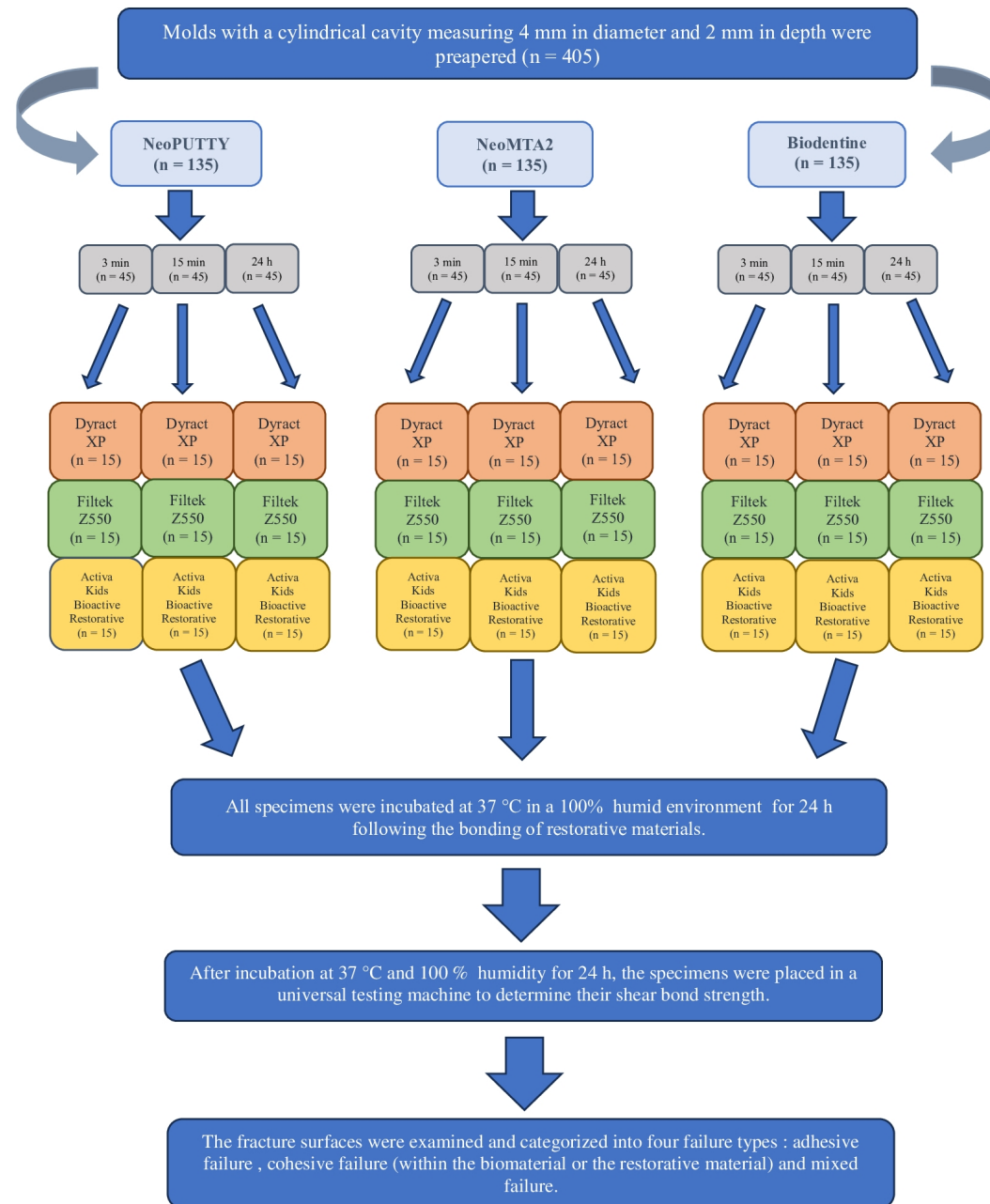


FIGURE 1. Study workflow and experimental design.

2.3 Preparation of restorative materials

A two-step self-etch adhesive system (Clearfil SE™ Bond 2, Kuraray Noritake Dental Inc., Okayama, Japan) was applied to the CSC surface, restricted to the bonding area designed for restorative material application, according to the manufacturer's instructions (Table 1). The adhesive material was polymerised using a light-emitting diode (LED) light-curing unit (VALO™, Ultradent Products Inc., South Jordan, UT, USA) in standard mode at 1000 mW/cm². A flexible transparent polymer tube (Tygon® S3 E-3603, Product Code: ACF1S1502, Saint-Gobain Performance Plastics, Charny, France; distributed by Interlab AS, Istanbul, Turkey) with an internal diameter of 2 mm, an external diameter of 4 mm, and a height of 2 mm was used to standardize the bonding area.

2.4 Application of the restorative materials

All restorative materials used in the study, including Dyract XP, Filtek Z550, and ACTIVA Kids BioACTIVE Restorative, were carefully placed into the tubes to prevent air-bubble formation. Dyract XP and ACTIVA Kids BioACTIVE Restorative were applied using their manufacturer-specific application guns with mixing tips, whereas Filtek Z550 nanohybrid composite was placed using a hand instrument. All restorative materials were polymerised using the same LED light-curing unit (VALO™, Ultradent Products Inc., South Jordan, UT, USA) according to the manufacturer's instructions (Table 1). Following polymerization, all specimens were incubated at 37 °C and 100% humidity for 24 h. After the storage period, the tubes were carefully removed using a sharp scalpel.

2.5 Shear bond strength testing

The specimens were placed in a universal testing machine (MOD Dental MIC-101, Esetron Smart Robotechnologies, Ankara, Türkiye) to determine shear bond strength. A shear load was applied at a crosshead speed of 1 mm/min until failure using a knife-edged apparatus positioned parallel to the interface adjacent to the base of the restorative cylinder (Fig. 2). The measured force values were recorded in Newtons (N). Because the bonding area was standardised using a Tygon tube with an internal diameter of 2 mm, the bonding area was calculated as 3.14 mm² (πr^2 with a radius of 1 mm), and the bond strength values were expressed in megapascals (MPa) by dividing the force values by this area.

2.6 Failure mode analysis

Following shear bond strength (SBS) testing, the fractured surfaces of all specimens were examined using an operating microscope (Zumax® OMS 3200 PRO, Zumax Medical Co., Ltd., Suzhou, China) at $\times 24$ magnification. Failure modes were evaluated by two blinded researchers (İHÇ, HEK), and inter-observer agreement was assessed using Cohen's kappa coefficient. Failure modes were classified as adhesive failure, cohesive failure within the CSC, cohesive failure within the restorative material, and mixed failure.

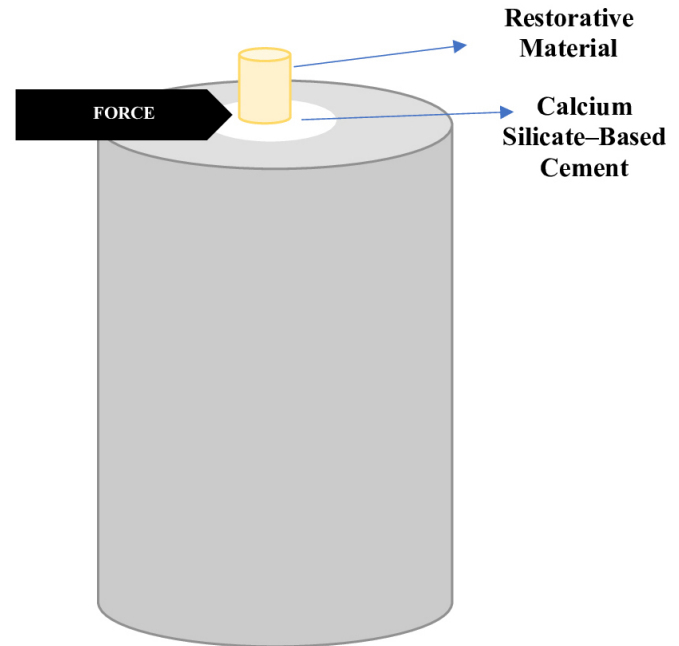


FIGURE 2. Shear bond strength test set-up.

2.7 Scanning electron microscopy (SEM) analysis

A total of 27 specimens were subjected to SEM analysis, with one representative specimen selected from each experimental subgroup. The selected specimens were those that best represented the predominant failure pattern in their respective groups. The specimens were mounted on aluminum stubs using adhesive tape and coated with 5 nm of gold in a LEO 1430 VP scanning electron microscope (Carl Zeiss, Oberkochen, BW, Germany). A single operator scanned the entire specimen surface and evaluated the samples at an accelerating voltage of 15 kV and $\times 1000$ magnification.

2.8 Statistical analysis

Data were analysed using SPSS version 22.0 (IBM SPSS Statistics; IBM Corp., Armonk, NY, USA). Normality was assessed using the Kolmogorov-Smirnov test, and homogeneity of variances was evaluated using the Levene test. The main effects of time, cement type, and restorative material, as well as their interactions, were analyzed using three-way ANOVA. For significant results, simple-effects analyses were performed, and pairwise comparisons were conducted using Tukey honestly significant difference (HSD) or Games-Howell tests, depending on variance homogeneity. The significance level was set at $p < 0.05$.

3. Results

According to the three-way ANOVA results, the main effects of setting time, cement type, and restorative material on bond strength were statistically significant ($p < 0.001$). The three-way interaction (Time $\times$ Cement $\times$ Restorative material) was not statistically significant ($p = 0.054$); however, all two-way interaction effects were statistically significant (Time $\times$ Cement: $p = 0.038$; Time $\times$ Restorative material: $p < 0.001$;

Cement $\times$ Restorative material: $p = 0.009$) (Table 2). Comparisons related to these two-way interactions are presented in Tables 3 and 4.

In the Dyract XP group, bond strength varied significantly among cement types at 24 h ($p = 0.018$); however, *post-hoc* analyses revealed no significant pairwise differences between the cement groups. In the Filtek Z550 group, significant differences were identified among the cements at the 3 min and 24 h time points ($p < 0.001$), with the highest bond strength observed in the Biodentine group and the lowest values in the NeoPUTTY group at both time points. In the ACTIVA Kids BioACTIVE Restorative group, significant differences were found among the cements at all time points ($p < 0.05$). Regarding the effect of setting time, bond strength increased significantly with increasing setting time across all restorative material and cement groups; the lowest values were obtained at 3 min, and the highest values were recorded at 24 h ($p < 0.001$) (Table 3).

In the NeoPUTTY and NeoMTA 2 groups, the restorative material type significantly influenced bond strength at the 15 min and 24 h intervals ($p < 0.05$). In both cement groups, the highest bond strength was achieved with ACTIVA Kids BioACTIVE Restorative, whereas the lowest values were recorded in the Dyract XP group. Specifically, at 24 h, Dyract XP demonstrated significantly lower bond strength compared to Filtek Z550 and ACTIVA Kids BioACTIVE Restorative ($p < 0.001$). In the Biodentine group, bond strength exhibited significant differences among restorative materials at all time points ($p < 0.001$), with ACTIVA Kids BioACTIVE Restorative yielding the highest values and Dyract XP yielding the lowest values across all measurements. Regarding the effect of setting time, bond strength increased significantly across all cement–restorative material combinations as the setting time increased; the lowest values were obtained at 3 min, and the highest values were recorded at 24 h ($p < 0.001$) (Table 4).

Inter-observer agreement for the failure mode evaluation was high (Cohen's kappa = 0.87). No cohesive failure within the restorative material was observed in any group or at any time point. Adhesive failures predominated at 3 min; however, their frequency decreased at 15 min, accompanied by increases in cohesive failures within the CSC and Mixed failures. By 24 h, adhesive failures declined to their lowest level, and the majority of failures consisted of cohesive failures within the

CSC and mixed failures (Table 5). Representative operating microscopic images of the observed failure modes are presented in Fig. 3, and SEM images of representative fracture surfaces are presented in Fig. 4.

4. Discussion

Because CSCs continue to undergo setting and maturation after placement, the bond formed over them may be influenced by the timing of the restorative application. Therefore, bond quality depends not only on the restorative material used but also on the stage of cement maturation at the time of restoration. In the present study, the null hypothesis was rejected, as SBS was significantly affected by setting time, cement type, and restorative material type. This finding is particularly important in paediatric dentistry, where the timing of restorative treatment is often determined by clinical conditions.

In clinical practice, treatment must often be completed in a single visit due to limited patient cooperation in paediatric populations [20]. Therefore, the 3 min and 15 min intervals were selected in the present study to simulate a single-visit clinical approach, whereas 24 h was included as a commonly used reference time point in the literature. In addition, because acid etching may adversely affect the surface integrity of immature CSC, a two-step self-etch adhesive containing 10-MDP (Clearfil SE Bond 2) was preferred to avoid aggressive surface alteration during early maturation while maintaining a standardized bonding protocol [10–12].

In the dental literature, both macro-scale tests (*e.g.*, SBS and tensile bond strength (TBS)), and micro-scale (*e.g.*, μ SBS, μ TBS) bond strength tests are commonly used to evaluate the bond strength. Although micro-tests provide measurements from limited bonding areas, they have disadvantages related to technique sensitivity during specimen preparation and a high risk of pre-test failure [21]. The low cohesive strength and structural heterogeneity of CSCs during the early setting phase may compromise specimen integrity in μ TBS tests. Therefore, the SBS test was selected in the present study to ensure higher reproducibility and methodological standardization.

Standardized 3D-printed resin blocks were used in this study to reduce the anatomical variability inherent in natural teeth and to ensure uniform specimen geometry across all groups. Although natural tooth tissues provide the most clinically relevant substrate, structural differences such as tubule density and

TABLE 2. Effects of setting time, cement type, and restorative material on bond strength.

Effect	Type III SS	df	Mean Square	F	p
Time	2667.668	2	1333.834	853.518	<0.001
Cement	197.902	2	98.951	63.319	<0.001
Restorative material	391.266	2	195.633	125.185	<0.001
Time $\times$ Cement	16.039	4	4.010	2.566	0.038
Time $\times$ Restorative material	186.695	4	46.674	29.867	<0.001
Cement $\times$ Restorative material	21.521	4	5.380	3.443	0.009
Time $\times$ Cement $\times$ Restorative material	24.132	8	3.017	1.930	0.054

SS, sum of squares; df, degrees of freedom; MS, mean square. *p*-values were obtained from three-way ANOVA (Type III SS). Statistical significance was set at $p < 0.05$.

TABLE 3. Time-dependent changes in bond strength for each CSC according to restorative material.

Restorative Material	CSC	Time		
		3 min (Mean $\pm$ SD)	15 min (Mean $\pm$ SD)	24 h (Mean $\pm$ SD)
Dyract XP				
	NeoPUTTY	1.74 $\pm$ 0.49 ^{Aa}	3.10 $\pm$ 0.65 ^{Ab}	5.53 $\pm$ 0.72 ^{Ac}
	NeoMTA 2	1.80 $\pm$ 0.65 ^{Aa}	2.94 $\pm$ 1.53 ^{Ab}	5.33 $\pm$ 1.15 ^{Ac}
	Biodentine	2.16 $\pm$ 0.95 ^{Aa}	4.17 $\pm$ 1.68 ^{Ab}	6.84 $\pm$ 1.84 ^{Ac}
	<i>p</i>	0.732	0.108	0.018
Filtek Z550				
	NeoPUTTY	1.83 $\pm$ 0.57 ^{Aa}	3.73 $\pm$ 0.93 ^{Ab}	8.06 $\pm$ 0.94 ^{Ac}
	NeoMTA 2	1.92 $\pm$ 0.72 ^{Aa}	3.78 $\pm$ 0.81 ^{Ab}	9.65 $\pm$ 1.68 ^{Bc}
	Biodentine	3.71 $\pm$ 0.85 ^{Ba}	4.46 $\pm$ 1.44 ^{Aa}	10.57 $\pm$ 1.54 ^{Bb}
	<i>p</i>	<0.001	0.423	<0.001
ACTIVA Kids BioACTIVE Restorative				
	NeoPUTTY	2.30 $\pm$ 0.63 ^{Aa}	4.30 $\pm$ 1.12 ^{Ab}	8.81 $\pm$ 1.33 ^{Ac}
	NeoMTA 2	2.40 $\pm$ 0.47 ^{Aa}	4.55 $\pm$ 1.38 ^{Ab}	10.35 $\pm$ 2.08 ^{ABc}
	Biodentine	3.77 $\pm$ 1.08 ^{Ba}	6.88 $\pm$ 1.82 ^{Bb}	11.55 $\pm$ 2.06 ^{Bc}
	<i>p</i>	<0.001	<0.001	0.003

Data are presented as mean $\pm$ standard deviation (SD).

p: The *p*-value is the one-way ANOVA *p*-value for the overall comparison between groups at each point.

Different uppercase letters in the same column indicate statistical differences between CSCs, and different lowercase letters in the same row indicate statistical differences between time points ($p < 0.05$).

Tukey HSD or Games-Howell post-hoc tests were used for pairwise comparisons.

Bond strength is expressed in MPa.

CSC, Calcium silicate-based cement.

TABLE 4. Time-dependent changes in bond strength for each restorative material according to cement type.

CSC	Restorative Material	Time		
		3 min (Mean $\pm$ SD)	15 min (Mean $\pm$ SD)	24 h (Mean $\pm$ SD)
NeoPUTTY				
	Dyract XP	1.74 $\pm$ 0.49 ^{Aa}	3.10 $\pm$ 0.65 ^{Ab}	5.53 $\pm$ 0.72 ^{Ac}
	Filtek Z550	1.83 $\pm$ 0.57 ^{Aa}	3.73 $\pm$ 0.93 ^{ABb}	8.06 $\pm$ 0.94 ^{Bc}
	ACTIVA Kids BioACTIVE Restorative	2.30 $\pm$ 0.63 ^{Aa}	4.30 $\pm$ 1.12 ^{Bb}	8.81 $\pm$ 1.33 ^{Bc}
	<i>p</i>	0.066	0.012	<0.001
NeoMTA 2				
	Dyract XP	1.80 $\pm$ 0.65 ^{Aa}	2.94 $\pm$ 1.53 ^{Ab}	5.33 $\pm$ 1.15 ^{Ac}
	Filtek Z550	1.92 $\pm$ 0.72 ^{Aa}	3.78 $\pm$ 0.81 ^{ABb}	9.65 $\pm$ 1.68 ^{Bc}
	ACTIVA Kids BioACTIVE Restorative	2.40 $\pm$ 0.47 ^{Aa}	4.55 $\pm$ 1.38 ^{Bb}	10.35 $\pm$ 2.08 ^{Bc}
	<i>p</i>	0.087	0.015	<0.001
Biodentine				
	Dyract XP	2.16 $\pm$ 0.95 ^{Aa}	4.17 $\pm$ 1.68 ^{Ab}	6.84 $\pm$ 1.84 ^{Ac}
	Filtek Z550	3.71 $\pm$ 0.85 ^{Ba}	4.46 $\pm$ 1.44 ^{Aa}	10.57 $\pm$ 1.54 ^{Bb}
	ACTIVA Kids BioACTIVE Restorative	3.77 $\pm$ 1.08 ^{Ba}	6.88 $\pm$ 1.82 ^{Bb}	11.55 $\pm$ 2.06 ^{Bc}
	<i>p</i>	<0.001	<0.001	<0.001

Data are presented as mean $\pm$ standard deviation (SD).

p: The *p*-value is the one-way ANOVA *p*-value for the overall comparison between groups at each point.

Different uppercase letters in the same column indicate a statistically significant difference between restorative materials, and different lowercase letters in the same row indicate a statistically significant difference between time points ($p < 0.05$).

Tukey HSD or Games-Howell post-hoc tests were used for pairwise comparisons.

Bond strength is expressed in MPa.

CSC, Calcium silicate-based cement.

TABLE 5. Fracture types in different groups.

CSC	Time	Restorative Material	Fracture Types			
			Adhesive	Cohesive failure within the restorative material	Cohesive failure within the CSC	Mixed
NeoPUTTY						
3 min		Dyract XP	13	0	0	2
		Filtek Z550	12	0	1	2
		ACTIVA Kids BioACTIVE Restorative	11	0	1	3
15 min		Dyract XP	9	0	2	4
		Filtek Z550	8	0	3	4
		ACTIVA Kids BioACTIVE Restorative	8	0	2	5
24 h		Dyract XP	4	0	6	5
		Filtek Z550	3	0	8	4
		ACTIVA Kids BioACTIVE Restorative	2	0	8	5
NeoMTA 2						
3 min		Dyract XP	12	0	1	2
		Filtek Z550	11	0	1	3
		ACTIVA Kids BioACTIVE Restorative	10	0	2	3
15 min		Dyract XP	6	0	2	7
		Filtek Z550	5	0	3	7
		ACTIVA Kids BioACTIVE Restorative	4	0	3	8
24 h		Dyract XP	3	0	8	4
		Filtek Z550	2	0	10	3
		ACTIVA Kids BioACTIVE Restorative	1	0	11	3
Biodentine						
3 min		Dyract XP	8	0	2	5
		Filtek Z550	9	0	2	4
		ACTIVA Kids BioACTIVE Restorative	8	0	3	4
15 min		Dyract XP	5	0	4	6
		Filtek Z550	4	0	5	6
		ACTIVA Kids BioACTIVE Restorative	3	0	4	8
24 h		Dyract XP	3	0	9	3
		Filtek Z550	2	0	11	2
		ACTIVA Kids BioACTIVE Restorative	1	0	12	2

CSC, Calcium silicate-based cement.

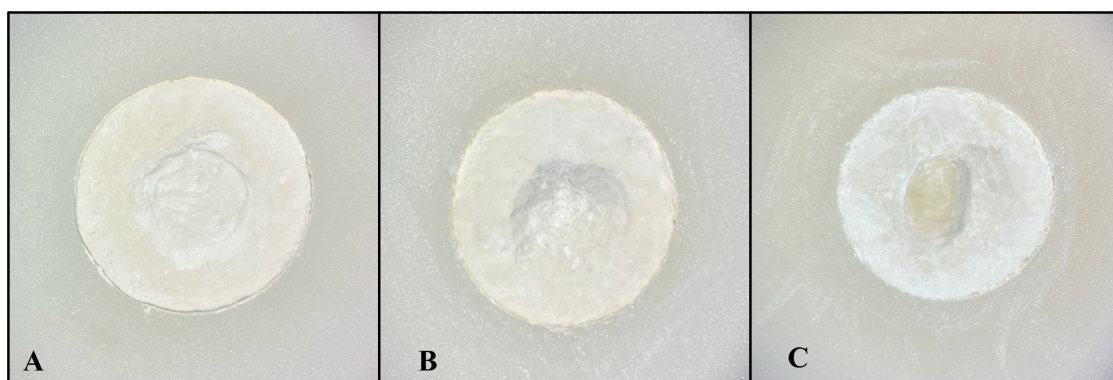


FIGURE 3. Representative operating microscopic images of failure modes after SBS testing. (A) Adhesive failure. (B) Cohesive failure within the calcium silicate cement. (C) Mixed failure.

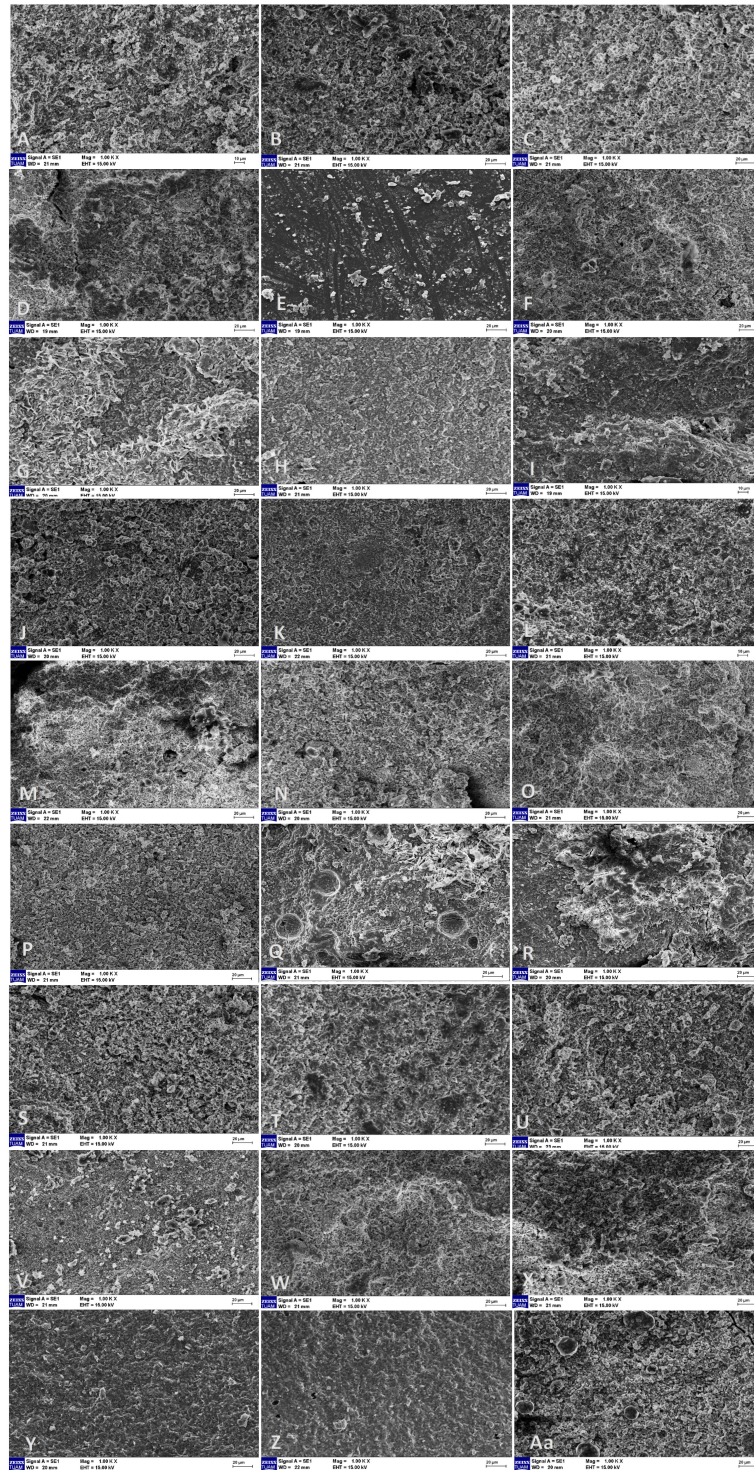


FIGURE 4. SEM images of fracture surfaces after SBS testing. (A) Dyract XP + NeoPUTTY, 3 min; (B) Filtek Z550 + NeoPUTTY, 3 min; (C) ACTIVA Kids BioACTIVE Restorative + NeoPUTTY, 3 min; (D) Dyract XP + NeoMTA 2, 3 min; (E) Filtek Z550 + NeoMTA 2, 3 min; (F) ACTIVA Kids BioACTIVE Restorative + NeoMTA 2, 3 min; (G) Dyract XP + Biodentine, 3 min; (H) Filtek Z550 + Biodentine, 3 min; (I) ACTIVA Kids BioACTIVE Restorative + Biodentine, 3 min; (J) Dyract XP + NeoPUTTY, 15 min; (K) Filtek Z550 + NeoPUTTY, 15 min; (L) ACTIVA Kids BioACTIVE Restorative + NeoPUTTY, 15 min; (M) Dyract XP + NeoMTA 2, 15 min; (N) Filtek Z550 + NeoMTA 2, 15 min; (O) ACTIVA Kids BioACTIVE Restorative + NeoMTA 2, 15 min; (P) Dyract XP + Biodentine, 15 min; (Q) Filtek Z550 + Biodentine, 15 min; (R) ACTIVA Kids BioACTIVE Restorative + Biodentine, 15 min; (S) Dyract XP + NeoPUTTY, 24 h; (T) Filtek Z550 + NeoPUTTY, 24 h; (U) ACTIVA Kids BioACTIVE Restorative + NeoPUTTY, 24 h; (V) Dyract XP + NeoMTA 2, 24 h; (W) Filtek Z550 + NeoMTA 2, 24 h; (X) ACTIVA Kids BioACTIVE Restorative + NeoMTA 2, 24 h; (Y) Dyract XP + Biodentine, 24 h; (Z) Filtek Z550 + Biodentine, 24 h; (Aa) ACTIVA Kids BioACTIVE Restorative + Biodentine, 24 h.

mineral content may influence bond strength measurements. Therefore, a standardized 3D-printed model was preferred to provide more controlled conditions for evaluating the effects of setting time and material combinations on bond strength.

Adequate bonding of the restorative material to the bio-ceramic cement is essential for maintaining a long-term seal and minimising microleakage. Although no definitive threshold for bond strength exists in the literature, clinically acceptable values have been reported to be approximately 9–12 MPa, whereas values of 17–20 MPa may be required to maintain interfacial integrity against polymerization shrinkage stresses [18, 22]. However, these values should be interpreted as approximate reference ranges rather than absolute thresholds, particularly for calcium silicate-based cements. In the present study, mean SBS values were generally observed to remain below these reference ranges, even in the 24 h delayed restoration groups.

In the present study, bond strength increased with longer waiting times across all cement-restorative material combinations, with the lowest values at 3 min and the highest values at 24 h. This pattern may be explained by progressive maturation of CSCs, as the initially porous and mechanically weak surface becomes more cohesive over time [23]. The accompanying shift in failure mode distribution, from predominantly adhesive failures at 3 min toward more cohesive and mixed failures at later time points, further supports the development of a more stable bonding interface as cement maturation proceeds.

The present findings are largely consistent with the literature. Previous studies have reported that delayed restoration protocols over Biodentine exhibit higher SBS values compared to early-stage applications [10]. Similarly, for NeoMTA 2 and NeoPUTTY materials, delayed restorations (7 days) have been reported to significantly improve bonding performance compared to early-stage applications (14 min) [18]. Conversely, several studies have reported that the setting time does not result in statistically significant differences in bond strength [16, 24].

The findings of the present study are partially consistent with those of Celiksoz and Irmak (2024), who reported significantly lower bond strength values at 3 min for Biodentine and RetroMTA [16]. However, while their study demonstrated similar bonding effectiveness at 12 min and 24 h, a continuous increase in SBS was observed in the present study as maturation progressed toward 24 h. This discrepancy may be attributed to methodological differences, including the variations in the restorative material sets used and the selection of a 15 min interval instead of 12 min.

Similarly, immediate and 7-day delayed restorations were compared using Biodentine, TotalFill BC Putty, and an experimental bioactive cement named pulp capping material (PCM; Coltène/Whaledent, Altstätten, Switzerland), and no significant difference was reported between immediate and delayed groups when restorations were performed using Clearfil SE Bond and SDR [24].

In the present study, CSC type influenced bond strength, with the highest mean SBS values observed in the Biodentine group, followed by NeoMTA 2 and NeoPUTTY. This pattern may be related to the presence of calcium chloride in the Biodentine liquid, which may accelerate hydration and

promote earlier surface stabilization. As a result, the adhesive system may interact with a more mechanically stable surface even at an early stage. In contrast, premixed materials such as NeoPUTTY, which rely largely on environmental moisture for setting, may exhibit lower early cohesive strength and more variable surface properties, contributing to the lower SBS values [25].

Several studies have reported that Biodentine may exhibit higher bond strength compared to other CSCs. For example, in a study evaluating the bonding of different bioceramics to dual-cure composite, Biodentine yielded higher SBS values than NeoMTA [26]. Similarly, a recent study indicated that Biodentine demonstrated higher bonding performance compared to NeoMTA Plus, and that surface preparation methods significantly influenced bond strength [27]. However, other studies suggest that the cement type may not have a decisive effect on bond strength. A study published in 2023 evaluating the μ SBS of various CSCs to bulk-fill composite reported no significant difference between Biodentine and NeoMTA 2 [28]. This finding contrasts with the higher SBS values observed in the Biodentine group in the present study. This discrepancy may be attributed to differences between μ SBS and SBS testing methods, particularly regarding bonding area and stress distribution, as well as differences in adhesive strategies and surface preparation protocols. Furthermore, evaluation of early time points (3 and 15 min) using Clearfil SE Bond 2 may have enhanced the detection of differences related to initial surface stability and setting kinetics.

Another *in vitro* study comparing self-adhering flowable composite and bulk-fill flowable composite on various pulp capping materials reported the highest SBS value for the NeoMTA Plus and SDR combination [29]. This result suggests that bonding performance may depend significantly not only on the cement type but also on the restorative material type and the adhesive strategy employed. The improved management of polymerisation stress by bulk-fill materials such as SDR, along with the protocol-related difference, may explain the favourable outcome observed for NeoMTA Plus. In contrast, the present study used Clearfil SE Bond 2 and evaluated early time points, which may have increased sensitivity to the initial surface characteristics of the cements.

In the present study, restorative material type was a determinant of bond strength to CSCs. A consistent ranking was observed across all time intervals; the highest mean SBS values were obtained in the ACTIVA Kids BioACTIVE Restorative group, followed by Filtek Z550 and Dyract XP, respectively. The higher bond strength values observed with ACTIVA Kids BioACTIVE Restorative across all time intervals may be attributed to the hybrid structure of the material and its potential chemical affinity with bioceramics [30]. While the hydrophilic nature of CSCs and high surface moisture during the early hydration phase restricts the bonding of traditional hydrophobic restorative material, the moisture-tolerant bioactive matrix of ACTIVA Kids BioACTIVE Restorative provides more effective interfacial wettability.

It is speculated that ionic interaction between the acid-modified methacrylates in the restorative material and free Ca^{2+} ions released from the CSC surface may contribute

to chemical integration alongside micromechanical retention. This mechanism may enhance interfacial stability; however, further chemical analyses are required to confirm this mechanism.

Composite resins with higher resin content are known to demonstrate improved compatibility with adhesive systems and enhanced interfacial stability [31]. Nevertheless, their chemical interaction potential with CSCs remains more limited compared to that of bioactive hybrid restorative materials, which may explain the lower SBS values compared with ACTIVA Kids BioACTIVE Restorative. Similarly, compomers have been reported to possess lower resin content and reduced wettability properties, which may negatively affect interfacial stability by limiting effective mechanical and chemical interaction with CSCs [13].

The present study findings are consistent with several previous studies. For example, in a study conducted on ProROOT MTA, ion-releasing materials such as Cention N and ACTIVA Kids BioACTIVE Restorative demonstrated higher SBS than compomers [32]. Similarly, bioactive materials have been reported to show high bonding performance with CSCs, even under different experimental protocols and Activa formulations (ACTIVA BioACTIVE Restorative) [19]. Furthermore, composite resin restorations applied to Biodentine have been reported to produce stronger and more stable bonds than compomers and some other restorative materials [33], possibly due to improved adhesive infiltration into surface microporosities and formation of a more homogeneous hybrid layer.

However, some studies report findings that contrast with the present results. Certain investigations have shown that ACTIVA BioACTIVE Restorative exhibited significantly lower bonding performance than that of the composite resin groups [27]. This difference may be explained by protocol-related differences, including the use of total-etch adhesive systems, which may alter the surface chemistry of CSCs following acid application. The 10-MDP-containing self-etch approach used in the present study may have preserved surface integrity more effectively, thereby improving bonding performance. Furthermore, some studies have reported no statistically significant differences between composite resin and compomer restorations bonded to white MTA [34]. These differences may be related to variations in adhesive generation, cement maturation time, surface preparation procedures, and testing protocols.

The distribution of failure modes (Table 5) was generally consistent with the SBS findings. The absence of cohesive failure within the restorative material suggests that failure occurred predominantly at the interface or within the cement component. A significant shift in failure patterns over time was observed; adhesive failures predominated at 3 min, reflecting weak early interfacial bonding, whereas intra-cement cohesive and mixed failures increased at later time points, indicating improved interfacial stability during cement maturation. By 24 h, adhesive failures declined to their lowest level, and failure patterns predominantly towards intra-cement cohesive/mixed characters; this shift indicates that the bond strengthened over time, causing failure to occur more within the cement itself and qualitatively supporting the measured increase in SBS.

This study has several limitations. First, the *in vitro* design

did not allow full simulation of intraoral biological and mechanical conditions, such as saliva contamination, pH fluctuations, and masticatory forces, which limits the generalisability of the findings. Second, although the application intervals of 3 and 15 min were selected to simulate a single-visit approach, all specimens were stored for 24 h after restoration placement before SBS testing under standardized conditions. Therefore, the present findings reflect the bond strength of an interface that was restored at an early stage but allowed to mature for 24 h before testing, rather than its immediate resistance to functional loading. This factor should be considered when interpreting the results in the context of single-visit clinical protocols. Third, the 3D-printed resin blocks used for specimen standardization do not reproduce the hydrophilic tubular structure of natural dentine or pulp fluid dynamics, which may influence the early maturation of CSCs, and the behaviour of adhesives on these surfaces may differ from clinical conditions. Fourth, because only one adhesive system (Clearfil SE Bond 2) was evaluated, the findings of the present study cannot be directly generalised to other bonding strategies, including universal and total-etch adhesive systems. Furthermore, the study focused on physical bond strength measurements and topographical evaluations; however, the absence of chemical interface analyses, such as Fourier-transform infrared (FTIR) or Raman spectroscopy, means that the proposed chemical interaction between the bioactive materials and CSCs remains hypothetical and requires further investigation. Finally, because the evaluation was limited to 3, 15 min, and 24 h time points, later time points such as 6 to 12 h or 7 days were not assessed; therefore, the full spectrum of maturation kinetics could not be comprehensively evaluated.

5. Conclusions

Within the limitations of this *in vitro* study, setting time, the type of CSC, and the type of restorative material significantly influenced the SBS. Across all CSC–restorative material combinations, SBS increased as the waiting period extended, with the lowest values observed at 3 min and the highest values at 24 h. Overall, Biodentine exhibited higher SBS, whereas NeoPUTTY was associated with lower values. Among the restorative materials, ACTIVA Kids BioACTIVE Restorative exhibited the highest SBS, whereas Dyract XP showed significantly lower performance, particularly on NeoPUTTY and NeoMTA 2 at 24 h. Failure mode analysis demonstrated a predominance of adhesive failures at early time points, followed by a shift toward intra-cement cohesive and mixed failures over time, supporting the progressive improvement of interfacial stability during CSC maturation. Clinically, delaying restoration until a later maturation stage may be advantageous whenever possible, and the use of restorative materials with higher bonding capacity to CSCs may improve outcomes. Future studies should investigate the early interfacial bonding behaviour of moisture-dependent premixed bioceramics using clinically relevant experimental models, such as dentin-disc systems with simulated pulpal pressure, and evaluate whether delayed restoration remains advantageous under such conditions.

AVAILABILITY OF DATA AND MATERIALS

The datasets generated and/or analysed during the current study are available from the corresponding author upon reasonable request.

AUTHOR CONTRIBUTIONS

HEK—performed the experimental procedures. İHÇ—performed statistical analysis and interpreted the data; critically revised the manuscript. İHÇ and HEK—conceived the idea and designed the study; drafted the manuscript. Both authors revised and approved the final version of the manuscript.

ETHICS APPROVAL AND CONSENT TO PARTICIPATE

Ethical approval for this study was granted by the Afyonkarahisar Health Sciences University Non-Interventional Scientific Research Ethics Committee (Approval No: 2025/389), which confirmed that no informed consent was required due to the absence of human subjects or biological samples.

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CONFLICT OF INTEREST

The authors declare no conflict of interest.

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