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Research Article

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Comparative evaluation of the bond strength of self-adhering and bulk-fill flowable composites to MTA Plus, Dycal, Biodentine, and TheraCal: an *in vitro* study

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No potential conflict of interest relevant to this article was reported.

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ABSTRACT

Objectives: This study aimed to compare the shear bond strength (SBS) of a self-adhering flowable composite (Dyad Flow) and a bulk-fill flowable composite (Smart Dentin Replacement [SDR]) to several pulp-capping materials, including MTA Plus, Dycal, Biodentine, and TheraCal.
Materials and Methods: Eighty acrylic blocks with 2-mm-deep central holes that were 4 mm in diameter were prepared and divided into 2 groups ($n = 40$ each) according to the composite used (Dyad Flow or SDR). They were further divided into 4 sub-groups ($n = 10$ each) according to the pulp-capping agent used. SBS was tested using a universal testing machine at a crosshead speed of 1 mm/min. Data were analyzed using 2-way analysis of variance. A p value of < 0.05 was considered to indicate statistical significance.
Results: A statistically significant difference ($p = 0.040$) was found between Dyad Flow and SDR in terms of bond strength to MTA Plus, Dycal, Biodentine, and TheraCal.
Conclusions: Among the 8 sub-groups, the combination of TheraCal and SDR exhibited the highest SBS.

Keywords: Biodentine; Bulk-fill composite; Dyad Flow; Mineral trioxide aggregate; SDR; Theracal

INTRODUCTION

Direct pulp capping involves sealing pulp that has been exposed due to trauma or through mechanical exposure with a cement that induces reparative dentin formation, shields the pulp from additional injury, and permits healing and repair [1]. Appropriate cements possess the properties of bioactivity (apatite formation), biointeractivity (ion release), and biocompatibility [2]. Traditionally, calcium hydroxide was used for this purpose since it is antimicrobial, has an alkaline pH, and stimulates mineralization [1]. Dycal (Dentsply Caulk, Milford, DE, USA), a calcium hydroxide material, contains sulfonamide, butylene glycol disalicylate, calcium phosphate, calcium tungstate, and oxides of zinc, iron, and titanium [2].

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However, the tunnel defects and microleakage documented with calcium hydroxide led to a decline in its use as a pulp-capping agent [1].

Calcium silicate-based materials, such as mineral trioxide aggregate (MTA), have become popular over the years. MTA Plus (Prevest Denpro, Jammu, India) consisting of tricalcium and dicalcium silicate, calcium aluminate, calcium sulfate, and bismuth oxide, is intended for direct pulp capping, perforation repair, apexification, and root-end filling [2,3]. The long setting time of MTA led to the introduction of materials such as Biodentine (Septodont, Saint-Maur-des-Fossés, France), which consists of tricalcium silicate, zirconium oxide, calcium carbonate, calcium chloride, and water. Biodentine, which is recommended as a dentin substitute for resin composite restorations, has good sealing ability, high compressive strength, a short setting time, biocompatibility, bioactivity, and remineralization properties [4,5]. Its setting time is as short as 9–12 minutes [2]. The main drawback of Biodentine is its weak micromechanical bonding to the restoration due to its water-based chemistry [5]. To overcome this, a light-curable resin-modified tricalcium silicate, TheraCal (Bisco, Inc., Schaumburg, IL, USA), consisting of 45% Portland cement, 10% bismuth oxide, 5% fumed silica, and approximately 40% resin by weight, was introduced. The rapid setting allows the treatment to be completed at the same appointment [5,6].

The bond strength between pulp capping material and the overlying restoration is vital for treatment success. A well-bonded adhesive joint between the restoration and the pulp capping agent can evenly spread stresses over the entire region of the bond [4]. Dyad Flow (Kerr, Orange, CA, USA) is a newly introduced, self-adhering flowable composite. Incorporation of an all-in-one bonding system into the composite eliminates the requirement for adhesive application, reducing chairside treatment time [4]. Bulk-fill flowable composites such as Smart Dentin Replacement (SDR) Flow (Dentsply DeTrey, Konstanz, Germany) can be placed with a thick bulk, instead of using the incremental placement technique, due to its higher translucency and greater depth of cure [6,7].

There is limited information in the literature about the bond strength of self-adhering flowable composites and bulk-fill composites to pulp-capping agents. Therefore, the aim of this study was to compare the shear bond strength (SBS) of a self-adhering flowable composite (Dyad Flow) and a bulk-fill flowable composite (SDR) to pulp-capping materials including MTA Plus, Dycal, Biodentine, and TheraCal, and to evaluate their bond failure mode under scanning electron microscopy (SEM).

MATERIALS AND METHODS

Eighty cylindrical acrylic blocks were prepared using copper ring molds (2 cm in height and 2 cm internal diameter). A hole was drilled in the center of each block (diameter 4 mm and 2 mm in depth). These 80 blocks were divided into the following groups:

Group I: Dyad Flow ($n = 40$)

- Sub-group I-a ($n = 10$): MTA Plus + Dyad Flow
- Sub-group I-b ($n = 10$): Dycal + Dyad Flow
- Sub-group I-c ($n = 10$): Biodentine + Dyad Flow
- Sub-group I-d ($n = 10$): TheraCal + Dyad Flow

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Group II: SDR ($n = 40$)

- Sub-group II-a ($n = 10$): MTA Plus + SDR
- Sub-group II-b ($n = 10$): Dycal + SDR
- Sub-group II-c ($n = 10$): Biodentine + SDR
- Sub-group II-d ($n = 10$): TheraCal + SDR

MTA Plus, Dycal, and Biodentine were placed in their respective blocks after mixing according to the manufacturer's instructions. TheraCal was inserted directly in 2 increments of 1 mm each into its corresponding blocks. Each increment was polymerized for 20 seconds using a light-emitting diode (LED) curing unit (Unicorn Denmart, New Delhi, India). All 80 blocks were coded and incubated at 100% humidity and 37°C for 72 hours.

After incubation, Dyad Flow was dispensed directly over the capping agents in the group I blocks in 2 increments of 1 mm each, through a dispensing tip, using a plastic cylinder (2 mm height and 2 mm diameter). Each increment was light cured for 15–20 seconds using the LED unit. In the group II blocks ($n = 40$), OptiBond all-in-one self-etch adhesive (Kerr) was applied in 2 increments, each of which was light-cured for 20 seconds using the LED unit. SDR was then placed over the capping agents using a plastic cylinder (2 mm height and 2 mm diameter) and light-cured for 20 seconds using the LED unit. All the specimens were incubated at 100% humidity and 37°C for 24 hours.

The specimens were mounted in a universal testing machine (Instron-1195, Instron, Norwood, MA, USA) and subjected to a shearing force using a knife-edge blade at a crosshead speed of 1 mm/min. The load at failure was recorded in newtons (N), and the bond strength was calculated in megapascals (MPa) by dividing the load at failure by the adhesive surface area (mm^2).

All specimens were subjected to gold sputtering using a plasma sputtering coater (MTI Corporation, Richmond, CA, USA), followed by bond failure mode evaluation under SEM (Quanta 200, FEI, Thermo Fisher Scientific, Waltham, MA, USA). The mode of bond failure was categorized as adhesive, cohesive, or mixed.

Data were analyzed using SPSS version 24.0 (IBM Corporation, Armonk, NY, USA). A p value < 0.05 was considered to indicate statistical significance. Descriptive and analytical statistics were done. Two-way analysis of variance (ANOVA) was used to evaluate the interactions of bonding performance. *Post hoc* analysis was done using the Tukey honest significant difference test.

RESULTS

Two-way ANOVA showed statistically significant differences in the bonding performance ($p = 0.040$) between the Dyad Flow and SDR composite resins used with 4 pulp capping materials (**Table 1**). The distribution of failure modes (**Table 2**) of the specimens after the SBS evaluation were characterized as adhesive, cohesive, or mixed. The MTA Plus specimens did not exhibit any instances of mixed failure. However, Dycal, Biodentine, and TheraCal displayed all 3 failure modes.

Table 1. Comparison of shear bond strength (MPa) between Dyad Flow and Smart Dentin Replacement (SDR) with different pulp-capping agents

Variables	Number	Dyad Flow	SDR	p value*
MTA Plus	10	1.81 ± 1.90	4.05 ± 2.92	0.040†
Dycal	10	1.07 ± 0.54	2.68 ± 1.66	
Biodentine	10	2.05 ± 1.11	4.63 ± 1.90	
TheraCal	10	4.70 ± 2.33	9.79 ± 2.52	
Total	40	2.41 ± 2.09	5.29 ± 3.51	

Values are presented as mean ± standard deviation.
*The p value derived from 2-way analysis of variance; †Significant at $p < 0.05$.

Table 2. Distribution of bond failure mode between Dyad Flow and Smart Dentin Replacement (SDR) with different pulp-capping agents ($n = 10$)

Composites	Pulp-capping agents	AF	CF	MF
Dyad Flow	MTA Plus	1	9	0
	Dycal	6	4	0
	Biodentine	4	2	4
	TheraCal	3	7	0
SDR	MTA Plus	0	10	0
	Dycal	1	7	2
	Biodentine	0	7	3
	TheraCal	1	6	3

AF, adhesive failure; CF, cohesive failure; MF, mixed failure.

DISCUSSION

Numerous tests—including shear, tensile, micro-shear, and micro-tensile tests—are performed to gauge the bond strength and clinical performance of composite resin. In this study, we utilized SBS testing as a reliable and practical method [8].

Dyad Flow, a self-adhering flowable composite with an all-in-one bonding system, combines the properties of flowability and self-adhesion, eliminating the need for etching, priming, and bonding prior to its placement [4,9]. SDR is a bulk-fill flowable composite composed of urethane dimethacrylate (UDMA), triethyleneglycol dimethacrylate (TEGDMA), and ethoxylated bisphenol-A-dimethacrylate (EBPDMA). Higher translucency and light transmittance allow it to be cured in thick, 4- to 5-mm bulks, without altering the curing time or light intensity. The polymerization modulator embedded in the resin backbone imparts flexibility and lowers the polymerization shrinkage stress compared to conventional flowable, nano, hybrid, or even silorane-based composites [6,10]. Dyad Flow bonds to the tooth via micro-mechanical etching, facilitated by its low pH and chemical interactions between the phosphate groups in the monomer and the calcium ions in the tooth [9,11]. Theoretically, the bonding mechanism of composites to calcium in pulp-capping agents is presumed to be comparable to their bonding mechanism to calcium in the tooth structure [12].

In the present study, statistically significant differences ($p < 0.05$) were found when comparing the SBS of Dyad Flow and SDR to various pulp-capping agents. SDR had a higher SBS of 5.29 ± 3.51 MPa, whereas the SBS of Dyad Flow was 2.41 ± 2.09 MPa (**Table 1**). Dyad Flow, a self-adhering composite, did not require any adhesive application. However, in the SDR group, we used the OptiBond all-in-one self-etch adhesive. OptiBond was preferred since it incorporates the same adhesive technology as Dyad Flow [9]. These self-etch systems simultaneously incorporate an acidic and hydrophilic monomer, eliminating the need for rinsing after etching [13]. Some studies have demonstrated that as a self-adhering flowable composite, Dyad Flow displayed lower bond strength to enamel and dentin than a

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conventional flowable composite and several all-in-one adhesives [9,11]. Peterson *et al.* [14] also concluded that self-adhesive composites presented significantly lower bond strengths than conventional composites applied with etch-and-rinse or self-etch adhesives. Altunsoy *et al.* [4], however, reported no significant differences between the SBS of Dyad Flow and X-tra base (Voco GmbH, Cuxhaven, Germany) to MTA, a calcium-enriched mixture, and Biodentine. Although OptiBond and Dyad Flow incorporate the same adhesive technology for bonding to the tooth structure, their bond strengths vary. A contributing factor to the low bond strength of self-adhesive flowable composites may be the lack of compression forces/pressure during placement, which is crucial to prevent open spaces on the interface, thereby affecting the longevity of the resin [15]. Other reasons could include their greater viscosity, absence of solvent, poorer wettability, and lower monomer penetration [11]. The presence of a solvent (ethanol, acetone, or water), lower viscosity, better wettability, and a stronger mechanical retention enhance the bond strength with OptiBond [16]. The interaction of self-etch adhesives with dentin is also reliant on their acidity and aggressiveness [17]. The pH of Dyad Flow is 1.9 and that of OptiBond is 1.7 [11]. Its lower pH may contribute to the higher bond strength of OptiBond than Dyad Flow. However, Shin *et al.* [18] reported that the pH of the adhesive was not a key contributor to the superior bond strength of composite resin to MTA.

Among the 4 capping agents used in our study, TheraCal and Dycal showed the highest (9.79 ± 2.52) and lowest (1.07 ± 0.54) SBS, respectively. A significant factor contributing to successful pulp therapy is the ability of the material to release and diffuse calcium ions into the dentin and surrounding structures [3]. The lower SBS of Dycal can be explained by its tendency to release fewer calcium ions than calcium silicate-based materials [2,3]. In the present study, Biodentine showed a higher SBS than MTA Plus. Tulumbaci *et al.* [19] reported that MTA bonded better to compomer and composite than Biodentine. However, Cantekin and Avci [6] showed that Biodentine displayed a higher SBS to methacrylate-based composites than MTA. Kaup *et al.* [20] also stated that the adhesion of Biodentine to dentin surfaces appeared to be superior to that of MTA. The higher SBS of Biodentine than MTA may be because its smaller particle size enhances its micromechanical interlocking. Furthermore, the presence of calcium chloride in Biodentine improves its resistance to displacement, thereby increasing its bond strength [20]. Several studies have shown that TheraCal exhibited a higher SBS to composites than MTA and Biodentine [21-23]. The high bond strength of TheraCal may be credited to its hydrophilic resin-based methacrylate monomer that promotes chemical adhesion and creates a strong interface between TheraCal and the bonding adhesive [21,23-25]. In contrast, the lack of resin content in MTA Plus, Dycal, and Biodentine indicates that their bond to composite is purely micromechanical, occurring via the penetration and interlocking of the adhesive systems into surface pores and irregularities due to their chemical composition [12,24]. In the present study, the MTA Plus and Biodentine specimens were stored for 72 hours to allow for complete setting. For MTA Plus and Biodentine, 70% of setting can be achieved after approximately 55 minutes and 9 minutes, respectively [2]. The desired sealability of MTA can be achieved after 72 hours. Altunsoy *et al.* [4] reported that the SBS of Biodentine increased after 24 hours. In contrast, the benefit of TheraCal application is the immediate placement of the final restoration [5].

After the SBS analysis of the specimens, their failure modes were evaluated under SEM (**Figure 1**), and recorded as adhesive (2 flat surfaces, failure at the capping agent-composite interface), cohesive (failure within the capping agent or composite), or mixed (combination of adhesive and cohesive). The dominant failure mode was cohesive. Adhesive failure

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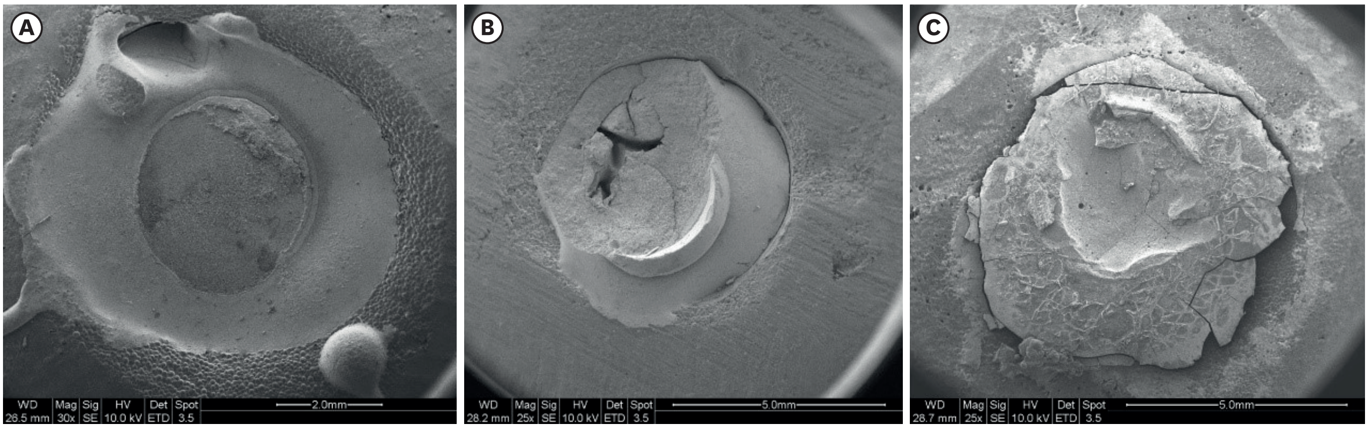

Figure 1. Scanning electron micrographs of bond failure modes. (A) Representative image of adhesive failure; the specimen was from the Dycal + Dyad Flow group. (B) Representative image of cohesive failure; the specimen was from the TheraCal + Smart Dentin Replacement (SDR) group. (C) Representative image of mixed failure; the specimen was from the Biodentine + SDR group.

indicates absence of a strong bond at the capping agent–composite interface. Cohesive failure indicates a lower compressive strength compared to other materials [25]. However, this does not reflect the true interfacial bond strength between the adhesive resin and the pulp capping material [4,25]. It is stated that the bond is acceptable when fracture occurs within the material rather than at the bonded interface (*i.e.*, cohesive failure rather than adhesive failure). Consequently, it can be said that pulp-capping materials may present a higher SBS to composites when cohesive failure is absent.

CONCLUSIONS

Within the limitations of this study, it was observed that both self-adhering flowable composite and bulk-fill flowable composite showed the highest SBS with TheraCal, followed by Biodentine, MTA Plus, and Dycal. The bulk-fill flowable composite bonded better than the self-adhering flowable composite in comparisons using the same pulp-capping agent.

Thus, it is concluded that TheraCal might be favored as a pulp-capping agent due to its higher SBS. Between the 2 types of composites, bulk-fill flowable composite might be preferred over self-adhering flowable composite for use over pulp-capping agents.

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