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# The physical characteristics of resin composite–calcium silicate interface as part of a layered/laminate adhesive restoration

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## ABSTRACT

**Objectives.** To compare in-vitro micro-shear bond strengths ( $\mu$ SBS) of resin composite to calcium silicate cement (Biodentine™) vs. glass ionomer cement vs. resin modified glass ionomer cement (RM-GIC) using an adhesive in self-etch (SE)/total etch (TE) mode after aging three substrates and bond and characterizing their failure modes.

**Methods.** Resin composite was SE/TE bonded to 920 standardized disks of Biodentine™, GIC & RM-GIC. Dividing samples into two groups, the first underwent early ( $t=0$  min, 5 min, 20 min, 24 h) or delayed ( $t=2$  wk, 1 month, 3 months, 6 months) substrate aging before bonding and  $\mu$ SBS ( $t=24$  h) testing. In the second, adhesive was applied after either early ( $t=5$  min) or delayed ( $t=2$  wk) substrate aging and then tested after bond aging ( $t=2$  wk, 1 month, 3 months, 6 months). The failure modes were identified using stereomicroscope. SEM images of selected samples were analyzed.

**Results.** No significant differences were observed between (SE)/(TE) bonding modes ( $P=0.42$ ). With substrate aging, a significant reduction in  $\mu$ SBS occurred between early and delayed time intervals for Biodentine™ ( $P=0.001$ ), but none for the GIC/RM-GIC ( $P=0.465$ ,  $P=0.512$  respectively). With bond aging, there was no significant difference between time intervals for all groups, except at 6 months for the GIC ( $P<0.05$ ). Modes of failure were primarily cohesive within all the substrates (68.82%) followed by adhesive failure at the resin–substrate interface (21.71%).

**Significance.** Biodentine™ is a weak restorative material in its early setting phase. Placing the overlying resin composite as part of the laminate/layered definitive restoration is best delayed for  $>2$  wk to allow sufficient intrinsic maturation to withstand contraction forces from the resin composite. A total-etch or self-etch adhesive may be used.

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## 1. Introduction

The operative treatment of deep carious lesions approaching the pulp and the related histopathological dentin–pulp complex changes pose a significant challenge as an increased risk of pulp exposure reduces the overall predictability and long term success [1–3]. Treatment modalities must aim to maintain pulp vitality using a minimally invasive, tooth preserving approach. With the introduction of a calcium silicate material Biodentine™ (Septodont, St Maure des Fosses, France) to the market, the clinical procedure has been somewhat simplified by combining its therapeutic pulp capping capabilities with its ability to be used simultaneously as a provisional bulk restorative material. However, because Biodentine™ is exposed to wear under load with time and its relatively poor esthetics, a second, overlaid restoration of resin composite is required to provide mechanical strength, wear resistance, and improved esthetics of the definitive restoration [4].

The quality and durability of the adhesive bond between Biodentine™ and the resin composite is of clinical significance with regards to the longevity and predictability of the final laminate restoration. The durability of this bond may be affected by the type of adhesive used (self-etch vs. etch and rinse adhesives). To date, there is no published information on the effect of the bonding technique on the bond strength of Biodentine™ to resin composite. As Biodentine™ has a similar chemical composition to MTA, hydration of Biodentine™ should resemble that of MTA. Therefore, it is assumed that when Biodentine™ is exposed to a low pH such as that of phosphoric acid etch, this could affect the chemical setting of Biodentine™ by disrupting the hydration of tricalcium silicates resulting in weakening of the setting material's microstructure [5–7]. Milder etching for a shorter time period may cause selective loss of matrix around the crystalline structures with minimal loss of cement, exposing these crystalline structures and hence encouraging successful adhesion through micro-mechanical retention [6].

Currently, placing the veneering restoration is a 2-stage clinical procedure, completed ideally within 6 months of placing the initial Biodentine™ bulk restoration, as per manufacturer's recommendations. However, investigating the potential for bonding the veneering restoration at the same visit as placing the Biodentine™ is worthwhile as this would be easier and less time consuming, eliminating the need to bring the patient back for a second visit.

There are many methods used to assess interfacial bond strength between dissimilar restorative materials. Statically, they can be measured using a macro- or micro-test depending on the area of the tested interface [8]. The micro-shear test was used in this investigation allowing simpler specimen preparation with a reduced risk of specimen preparation damage. It eliminates the need to section specimens to obtain sticks or hour-glass specimens which is required for other tests such as the micro-tensile test [9–11]. Indeed this is necessary with Biodentine™ which is brittle in thin cross section and must be used in bulk to avoid damage to the Biodentine™ samples.

The aim of this in-vitro study was to determine the micro-shear bond strength ( $\mu$ SBS) of a resin composite (N'Durance, Septodont, Louisville, USA) to Biodentine™ using

a self-etch adhesive (Scotchbond™ Universal, 3M ESPE, USA) compared to glass ionomer cement (GIC) (Fuji IX™ GP, GC Corporation, Tokyo, Japan) and resin modified glass ionomer cement (RM-GIC) (Fuji II LC, GC corporation, Tokyo, Japan), which are materials that have similar clinical applications to Biodentine™ in terms of being used as provisional bulk restorative materials in deep cavities. The study also aimed to compare the use of the self-etch adhesive in a self-etch mode (SE) and a total-etch (TE) mode while aging the substrates and aging the bond at different time intervals and to identify the specific modes of failure. The null hypothesis was that there is no difference in the  $\mu$ SBS within each substrate (Biodentine™, GIC, and RM-GIC) and when comparing between them using the self-etch and total etch techniques at the different time intervals.

## 2. Materials and methods

The materials used are summarized in Table 1. Nine hundred and twenty disks of Biodentine™ ( $n=320$ ), Fuji IX™ (control) ( $n=320$ ), and Fuji II LC™ (control) ( $n=280$ ) were fabricated by mixing each material according to the manufacturer's instructions and condensing them into a standardized  $3 \times 4$  mm cylindrical plastic polymer mold. A glass slab was placed on top of the mold so that all the materials set against a smooth surface to ensure standardization of the sample surface. The samples were divided into two main groups. In the first group, the effect of aging the substrate (Biodentine™, GIC and RM-GIC) on the micro-shear bond strength ( $\mu$ SBS) was investigated. In the second group, the effect of aging the bond on the  $\mu$ SBS was investigated.

The first group ( $n=440$ ) which investigated the effect of aging the substrate on the  $\mu$ SBS was subdivided into the following:

1. Aging each substrate for “early” time intervals ( $t=0$  min,  $t=5$  min,  $t=20$  min,  $t=24$  h) following which the same adhesive was applied in either SE or TE mode. The first time interval represents the application of the adhesive immediately after setting of each material as stated in the manufacturer's instructions, from the start of mixing. For RM-GIC there were only two time intervals ( $t=0$  min, and  $t=24$  h) as the material was photo-cured on command.
2. Aging the substrate for a “delayed” time interval ( $t=2$  wk,  $t=1$  month,  $t=3$  months,  $t=6$  months) following which the same adhesive was applied in either SE or TE mode.

The bonded samples in both groups were stored in distilled water for 24 h before being subjected to  $\mu$ SBS testing.

The second group ( $n=480$ ) which investigated the effect of aging the adhesive bond on the  $\mu$ SBS was subdivided into the following:

1. Aging the substrate for 5 min following which the same adhesive was applied in either SE or TE mode and the bond then aged in distilled water before testing ( $t=2$  wk,  $t=1$  month,  $t=3$  months,  $t=6$  months).
2. Aging the substrate for 2 wk following which the adhesive was applied in either SE or TE mode and the bond aged for

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