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学 位 論 文 題 名

The effect of dentine surface preparation and reduced application time of adhesive on bond strength

(切削方法の違いと接着材処理時間の短縮が象牙質の接着強さに与える影響についての検討)

This study evaluated the effects of surface preparation and the application time of adhesives on the microtensile bond strengths (μ TBS) with universal adhesives.

Seventy-two extracted non-carious human third molars were used in this study. They were stored in an aqueous solution of 0.5% Chloramine-T at 4°C and used within 6 months after extraction. The teeth were collected under a protocol reviewed and approved by the university ethical committee (#2013-7). The teeth were abraded to expose mid-coronal dentine with a gypsum model trimmer under water coolant.

The teeth were randomly assigned into 12 experimental conditions ($n=5$ to μ TBS; $n=1$ to interfacial structure observation) according to: dentine surface preparation (SiC-prepared dentine vs. bur-cut dentine) and adhesive application time (manufacturer's instruction vs. shortened). These variables were tested for three adhesive systems: G-Premio Bond [GP, GC Corp., Tokyo, Japan], Clearfil Universal Bond [CU, Kuraray Noritake Dental Inc., Okayama, Japan], and Scotchbond Universal Adhesive [SB, 3M ESPE, St. Paul, MN, USA]. Occlusal dentine surfaces were prepared by using either 600-grit SiC paper (Sankyo-Rikagaku Co., Saitama, Japan) or tapered regular grit diamond bur (diamond point FG, #103R, Shofu, Kyoto, Japan). For SiC paper preparation, the surfaces were manually polished for 60 s under running water using a 600-grit SiC paper. In case of diamond bur, dentine surfaces were ground with the bur in a high-speed handpiece. For each surface preparation, half of the teeth received the adhesives applied according to manufacturer's instruction, and the other half received the adhesives applied under the shortened time. Each adhesive was dropped directly from the bottle on dentine, air dried immediately and then light cured. Two 2mm-thick layers of resin composite (Clearfil AP-X, Kuraray Noritake Dental Inc., Tokyo, Japan) were built-up on the bonded surface. Each layer was light cured for 20 s operating using a light curing device (Optilux 401, Demetron/Kerr, Orange, CA, USA) at ≥ 550 mW/cm².

After storage in 37°C water for 24 h, each bonded tooth was sectioned into beams (cross-sectional area approximately 1mm²) using an Isomet diamond saw (Isomet 1000, Buehler, Lake Bluff, Illinois, USA). For each tooth ($n = 5$), three beams from the central area were randomly selected for μ TBS, therefore resulting in a total of 15 beams to be tested. The beams were fixed to a Ciucchi's jig with cyanoacrylate glue (Model Repair 2 Blue, Dentsply-Sankin, Otahara, Japan) and subjected to a tensile force at a crosshead speed of 1 mm/min in a desktop testing apparatus (EZ test, Shimadzu, Kyoto, Japan). μ TBS was expressed in MPa, and data were analyzed by three-way ANOVA and Dunnett T3 tests ($\alpha=0.05$). The fractured specimens were determined using a scanning electron microscope (SEM, S-4000, Hitachi,

Tokyo, Japan). Specific features were further examined at 3000× and 10000×.

One tooth per group was bonded in the same way as described for the μ TBS test. The teeth were cut into slabs. Then, the slabs were prepared for SEM observation and then examined at 3000× magnification.

There were no pre-test failures in this study. Our results indicated that there were significant effects between adhesive vs surface preparation ($F=12.02$; $p<0.000$), and adhesive vs application time ($F=3.5$; $p=0.032$). There was no direct effect of surface preparation vs application time ($F=1.17$; $p=0.280$). The interaction of factors was significant ($F=10.006$; $p<0.000$)

Bond strengths were always significantly higher when the adhesives were bonded to 600-grit SiC paper-prepared surfaces compared to those of bur-cut dentine. The influence of application time was only observed for CU when bonded with SiC paper prepared dentine; and GP when bonded with the bur-cut dentine.

In general, the fracture modes were mainly categorized as mixed failure and adhesive failure. There was a clear tendency that more cohesive failures occurred with SiC prepared dentine. When adhesive failure areas were examined at higher magnifications (10000×), a high concentration of porosity was observed for both CU and GP adhesives, and at a lower degree for SB adhesive. More porosity and bigger pores appeared to be associated with the groups that were bonded with shortened application time and on bur-cut dentine.

In general, the hybrid layer was not distinct from the SEM images. Resin tags detected were short, sparsely distributed and only more distinct on surfaces prepared with SiC paper and preferably when the adhesive was applied according to the manufacturer's directions. Resin tags were either absent or appeared as very short projections and scarcely distributed along the observed area when the adhesives were bonded to bur-cut dentine.

From the results of this study, it might be concluded that 1) dentine surface preparation had an influence on the μ TBS. Smear layer from bur-cut dentine had an undesirable effect on all the three universal adhesives used in this study. 2) Application time had an impact on the adhesive performance. The shortened application time resulted in insufficient solvent evaporation and bonding mechanism which leads to lower bond strength for two out of three adhesives tested, depending on the type of surface preparation.