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学位論文内容の要旨

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学　位　論　文　題　名

Long-term dentin bonding performance of HEMA-free and HEMA-containing universal adhesives restored with resins loaded or not with bioactive particles

（生体活性粒子添加型および非添加型レジンと HEMA 含有および非含有ユニバーサル接着材における長期象牙質接着性能について）

キーワード（５つ）Universal adhesive, S-PRG, HEMA, physical and mechanical properties, bond strength.

Universal adhesives (UAs), distinguished by versatility, require a complex composition that integrates hydrophobic and hydrophilic monomers, silane, and water for effective bonding interfaces. Confronting the challenge of blending adhesive compounds, incorporating 2-hydroxyethyl methacrylate (HEMA) enhances stability and homogeneity, albeit with considerations such as challenges during air-drying and increased water sorption. The adjustment of HEMA concentration has been explored to mitigate these concerns and improve the longevity of composite restorations. Bioactivity is a critical and controversial property in dental restorative materials. Following the FDI statement (2023), the bioactivity concept can encompass ion glass particles like S-PRG filler, recognized for their ability to release multiple ions, following the chemical mechanism of action. Attempting to unite the advantages of the UAs and properties of S-PRG fillers, new resin-based products have been developed and lack investigation. Therefore, this study aimed to investigate the effects of restorative resin composites (RCs) containing or not S-PRG filler on dentin bond strengths of HEMA-free and HEMA-containing UAs over 6 months. Two two-step adhesives served as reference groups. Also, the materials' water sorption, solubility, and conversion degree were investigated.

Flat dentin surfaces were initially treated with three UAs with different HEMA concentrations: Beautifil Bond Xtreme (0%HEMA-BBX), experimental adhesive 1 (5%HEMA-EBX1), experimental adhesive 2 (10%HEMA-EBX2), and two self-etch adhesives: FL-BONDII (S-PRG filler containing- FBII) and experimental adhesive (EFB1). Then, the specimens were

restored by Beautifil Flow Plus (S-PRG filler containing BFP) and one experimental RC with silica-based filler (EBP2). The micro-tensile bond strength (μTBS) and fracture modes were evaluated after 24 hours and 6 months of water storage. All tested materials' water absorption (WS) and solubility (SL) were quantified utilizing a modified version of the ISO-4049. The degree of conversion (DC) was analyzed by Raman spectroscopy and attenuated total reflection Fourier transform infrared (ATR-FTIR) spectroscopy. DC data were examined by one-way ANOVA test (adhesives) and independent sample T-test (resin composites). WS and SL data were analyzed by Kruskal-Wallis (adhesives) and Mann–Whitney U tests (resin composites). Three-way ANOVA statistically examined the μTBS data. Post hoc multiple comparisons were performed using the Bonferroni and Sidak test when needed, and the significance level was set at 5%.

EBX2 displayed the lowest DC value, significantly lower than that of BBX. There was no difference in the DC of the two RCs. No significant difference in WS was detected between BBX, EBX1, EBX2, and FBII ($p > 0.05$). The WS of EFB1 was the lowest ($p < 0.05$). The SL of FBII and EFB1 were significantly lower than BBX and EBX1 ($p < 0.05$). No significant difference in SL was detected among BBX, EBX1, and EBX2 ($p > 0.05$). The WS and SL of BFP were significantly higher than EBP2 ($p < 0.05$). Pairwise comparisons showed that after 24h, EBX1 and EBX2 restored with BFP showed higher bond strengths than restoring with EBP2 ($p < 0.001$, $p = 0.006$, respectively). After 6m of water storage, EBX1, EBX2, and FBII restored with BFP showed significantly lower bond strength than 24h ($p = 0.002$, $p = 0.008$, $p < 0.001$, respectively). Fracture surface SEM revealed that the predominant failure mode for all combinations of adhesives and resin composites was 'adhesive failure.' Aging appeared not to have an evident impact on the failure-mode distribution.

Within the design of this study, it was possible to conclude that adhesives presented distinct degrees of conversion, while resin composites showed similar conversion percentages. The adhesives did not comply with the ISO 4049-2019 requirements of water sorption; only resin composites did. The 2-step self-etch adhesives followed the prerequisites of ISO's water solubility, but the other materials did not. The effect of adhesive systems on the performance at 24 hours depended on the restorative resin composite. The commercial Beautifil Flow Plus resin composite (with S-PRG filler content) positively affected the 24-hour bond strength of the experimental universal adhesives, regardless of their HEMA concentration. The long-term bonding performance depended on the adhesive-resin combinations.