Verifying mechanism of bonding materials led to high adhesive strength to dentin by blended calcium salts of functional monomer

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[Introduction]

The adhesive restorative material is essential for dental treatment. However, there are some points to be improved in the material such as long-term durability. Recently, the development of adherent monomer, 4-MET linked with calcium (4-MET-Ca) was reported that possibilities of long-term durability improvement. 4-MET-Ca blended the sealing coat material was also expressed remineralization ability (Okuma et al., 2009; Motai et al., 2011, 2015). On the other hand, another adhesive monomer, MDP, higher chemical bonding strength to dentine than 4-MET was reported (Yoshioka et al., 2002; Yoshida et al., 2001, 2004). The effect of blend of the MDP with calcium salt (MDP-Ca) has not been studied.

This study aims to develop MDP-Ca newly, and to investigate of new bonding material that MDP-Ca was blended into the all-in-one self-adhesive material (HC) comprising 4-META.

[Material and method]

Experiment 1: Synthesis of MDP-Ca

MDP and ethanol were mixed under cooling and aqueous solution of calcium hydroxide was dropped into the mixed solution. The obtained reactant with aqueous solution of calcium hydroxide was dried to obtain the product. The product was analyzed by FT-IR and XRF to identify the MDP-Ca.

Experiment 2: Determination of proper concentration of MDP-Ca

HC was used as the control. Each synthesized MDP-Ca of which concentration was 0.75%, 1.50%, 3.00% and 4.50% was blended into the respective HC. These mixtures were regarded as the experimental group. Flat dentin surfaces of extracted molar tooth were created in mid-coronal dentin perpendicular to the tooth's longitudinal axis using a slow speed diamond saw. HC and each MDP-Ca blended HC bonding

resin were applied on the dentin surface. Following adhesive treatment, the composite resin was built up and light-cured on the treated dentin surface to obtain the specimens. The specimens were cut perpendicularly to the dentin-resin interface in order to these specimens be the 1 mm^2 adhesive area and stored in in distilled water for 24 hours. The micro tensile test was carried out to the obtained specimens. After the measurement, the mix fracture surface and dentin-resin interface of the specimens were observed using SEM.

Experiment 3: Observation of morphological characterization of bonding layer

HC, 1.5%MDP-Ca blended HC（MDCP）, and 1.5%4-MET-Ca blended HC（CMET）were used as the experimental group. As described above, following adhesive treatment with each bonding material, the specimens were stored in the artificial saliva. The specimens before penetration and after penetration for 1 day, 7 days and 1 month, were taken out and cut vertically to the dentin-resin interface, and then ion etched and observed the morphological characterization of bonding layer on the dentin-resin interface by SEM.

Experiment 4: Examination of microhardness

HC, MDCP and CMET were used as the experimental group. Bonding material was dropped into embedding plate and light-cured. The cured bodies of bonding material were embedded in epoxy resin and mirror polished, microhardness of the specimens were measured by nano-indentation tester.

Experiment 5: Examination of the long-term durability

HC, MDCP and CMET were used as the experimental group. As described above, the specimens were prepared. The specimens were stored in distilled water for 3, 6 and 12 months. The micro tensile test was carried out to the obtained specimens. After the measurement, the mix fracture surface and dentin-resin interface of the specimens were

observed using SEM.

Experiment 6: Examination of maximum water absorption rate and maximum dissolution rate.

HC, MDCP, CMET and SE-Bond were used as the experimental group. As described above, the cured bodies of bonding material were prepared. After the obtained specimens were measured the initial weight and stored in distilled water, maximum water absorption rate was calculated from the weight of the specimens with the maximum amount of water absorbed. Then the water absorption, the specimens were dried and maximum dissolution rate was calculated from the decrease in weight.

Experiment 7: Examination of conversion rate

HC, MDCP and CMET were used as the experimental group. Each bonding material before light-cured and after light-cured was measured an absorption spectrum by FT-IR analyses. The conversion rate was calculated from the absorption peak of carbon double bonds.

Experiment 8: Examination of calcium release

MDP-Ca and 4-MET-Ca were immersed respectively into the ultrapure water and stored for 1 week 37℃. MDP-Ca and 4-MET-Ca were immersed respectively into the artificial saliva, stored in in distilled water for 1 week at 37℃. After insoluble substance was removed, calcium amount in the solution was measured by an atomic absorption spectrophotometer.

[Results]

Experiment 1: It was revealed that the product was compound containing phosphate salt and carbon double bonds by analyze of FT-IR, and that the product was compound containing phosphorus and calcium by analyze of XRF.

Experiment 2: In the measurement of the micro tensile strength of the specimens with different amounts of MDP-Ca blending, 1.5%MDP-Ca blended HC showed the highest value significantly compared to the HC.

Experiment 3: In the morphological characterization of bonding material, high tolerance of ion etching was observed in the bonding layer on the dentin-resin interface with MDCP after 24 hours.

Experiment 4: In the examination of microhardness, compared to the HC, MDCP showed significantly high values. On the other hand, no significant difference was recognized between MDCP and CMET

Experiment 5: In the examination of the long-term durability, MDCP maintained high value compared to the HC after 1 year and the significant difference was recognized. On the other hand, no significant difference was recognized between MDCP and CMET in the whole period. In SEM observation, compared to the HC, degradation of the bonding interface was hardly observed in MDCP and CMET. The mix fracture images were often observed in MDCP and CMET even after 1 year storage.

Experiment 6: In the examination of maximum water absorption rate, MDCP and SE-Bond showed significantly lower values of maximum water absorption rate, compared to HC. On the other hand, in the examination of maximum dissolution rate, MDCP, CMET and SE-Bond showed significantly lower values of maximum water dissolution rate, compared to HC.

Experiment 7: HC, MDCP and CMET no difference was recognized.

Experiment 8: In the specimens that MDP-Ca was immersed into the ultrapure water, calcium was hardly extracted. On the other hand, in the specimens that 4-MET-Ca was immersed into the ultrapure water, calcium was extracted. In the specimens that MDP-Ca was immersed into the artificial saliva, calcium decresed compared to only artificial

saliva. On the other hand, in the specimens that 4-MET-Ca was immersed into the ultrapure water, calcium increased compared to only artificial saliva.

[Discussion]

It was suggested that the obtained the product in this experiment was MDP-Ca synthesized by chemical reaction with MDP and calcium hydroxide. MDCP showed the high adhesive strength. MDP-Ca was reported that hardly released calcium (Yoshida et al., 2004; Takahashi, 2014). MDP-Ca was considered difficult to adhere to dentin. However, blend of MDP-Ca brought about improvement of mechanical characterization and chemical stability, which led to high adhesion strength. MDP-Ca blocked expansion of the nano-space between bonding layer and dentin, inducing long-term durability. On the other hand, as 4-MET-Ca which has the remineralization substance blocked the nano-space by release of calcium (Motai et al., 2015). We suggested that MDCP improved the adhesion strength in a different manner from CMET.

[Conclusion]

MDCP formed excellent bonding layer, showed high adhesive ability compared to the HC. MDCP was improved dissolution resistance and long-term durability of cured bonding material. Compared to the CMET, MDCP was no significant but showed superiority. It was suggested that MDP-Ca improved the adhesion strength in a different manner from 4-MET-Ca.

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