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Effect of Dentin Bonding Agent Acidity on Surface Microhardness of Mineral Trioxide Aggregate

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Purpose: This study investigated the effect of dentin bonding agent acidity on surface microhardness of MTA. **Materials and Methods:** Forty cylindrical molds (3 mm×5 mm) were prepared, and three dentin bonding agents with different acidities: Adper Single Bond 2 (ASB), Single Bond Universal (SBU), and Clearfil SE bond 2 (CSE) were applied to the inner surface of the molds (n=10). No bonding agent was applied in the control group. MTA was mixed and inserted into the molds and sealed with a wet cotton pellet for 4 days. After setting, the Vickers microhardness (HV) test was done at 200, 400, 600 μm from the inner surface of the mold. One-way ANOVA was conducted for all samples. A *P*-value of less than .05 was considered significant. Tukey HSD test was performed for post-hoc analysis. **Results:** The mean HV values and standard deviations were 67.02±11.38 (Con), 48.76±11.33 (ASB), 43.78±11.19 (CSE), 37.84±9.36 (SBU), respectively. The difference between the control group and the experimental groups was statistically significant ($P<0.001$). The difference between ASB and SBU was statistically significant ($P<0.001$), while the difference between SBU and CSE was not. There were no statistically significant differences between the various points from the inner surface of the mold within each group ($P>0.05$). **Conclusion:** Results of the current study indicate that use of dentin bonding agents with MTA can reduce the surface microhardness of MTA. Moreover, there is a direct relationship between the acidity of dentin bonding agents and the surface microhardness of MTA. [J Korean Dent Sci. 2024;17(1):36-44]

Key Words: Acidity; Dentin adhesive; Vickers microhardness; Mineral trioxide aggregate

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Introduction

Mineral trioxide aggregate (MTA) is a biocompatible dental material which has been used in vital pulp therapy and root perforation repair since its introduction as a retrograde filling material in 1993[1-3]. MTA shows superior sealing and antibacterial properties compared to conventional retrograde materials. However, it has disadvantages such as a long setting time, difficulty of manipulation, and potential of discoloration of teeth. White MTA with a reduced ratio of metal oxides was introduced to facilitate the use of MTA in aesthetically important areas such as the anterior teeth[4]. The color of the MTA was improved, but bismuth oxide contained in the product to make the material radiopaque maintains its oxidizing properties which induce discoloration[5-9]. Despite the use of White MTA, discoloration of teeth was still observed. Studies report that the use of dentin bonding agents reduces or prevents discoloration caused by MTA[7,8,10]. Results of these studies indicate that the use of dentin bonding agents may be clinically beneficial to prevent discoloration caused by MTA. However, no research has been conducted to determine whether dentin bonding agents affect MTA and dental pulp in clinical situations such as vital pulp therapy.

MTA is an aggregate which changes as it is hydrated. The hydration process hardens the cement. That is, the hydration rate represents the hardening rate. The porosity, which is an indicator of the degree of hydration, can be confirmed through SEM images[11,12]. Vickers microhardness (HV) is defined as the resistance to plastic deformation after indenting the surface of a material. Vickers test was originally used to measure metals, but it is also used for various other materials such as ceramics and cement pastes. Hardness tests display the bulk properties of materials and are used to evaluate mechanical properties sensitive to microstructural traits such as changes in the ratio of water to cement[13]. Although surface microhardness does not represent all the material properties of MTA, linear relationships between microhardness and compressive strength, porosity, and modulus of elasticity have been demonstrated[14]. Surface microhardness is a non-destructive test that can be used as an indicator of the degree of hydration during hardening[13]. The surface hardness of MTA is affected by various factors such as the pH value of the environment, the thickness of the material, the condensation pressure, the amount of entrapped air in the mixture, humidity, acid etching treatment, and temperature[15].

Studies on bonding to MTA indicate that bonding process has several effects on MTA. Total-etch system shows higher bonding strength to MTA than the self-etch system[16,17]. In the presence of bonding agent, gap was not observed between MTA and composite resin, but gap was observed in the absence of bonding agent[15]. In addition, acid-etching affects the micromorphology and adhesion of MTA, compressive strength, and surface microhardness[12,18-20]. However, has been no research published on the effect of light-cured bonding agents on MTA.

If bonding agents are to be used in the clinical context, such as vital pulp therapy, the effect of the agent's acidity on MTA microhardness should be explained. Based on the lack of data investigating the effect of dentin bonding agents on MTA, the purpose of the current study is to investigate the effect of dentin bonding agent acidity on the surface microhardness of MTA. The null hypothesis was that there are significant differences in surface microhardness of MTA depending on the acidity of the dentin bonding agents, and there are significant differences in surface microhardness of MTA between distances from bonding surface.

Materials and Methods

1. Experimental groups

The experiments were conducted using the following dentin bonding agents: Adper Single Bond 2 (ASB; 3M ESPE, St. Paul, MN, USA) which is a two-step

total-etch bonding system with a pH of 4.7, Single Bond Universal (SBU; 3M Deutschland GmbH, Neuss, Germany) which is a universal adhesive with a pH of 2.7, and Clearfil SE bond 2 (CSE; Kuraray Noritake Dental Inc., Okayama, Japan) which is a two-step self-etch bonding system with a pH of 2.0 (primer) (Table 1). The MTA used was ProRoot® MTA (White) (Dentsply Tulsa Dental, Tulsa, OK, USA)

Experiments were conducted in the following four groups (n=10);

Con: No bonding agent (control)
ASB: Adper Single Bond 2
SBU: Single Bond Universal
CSE: Clearfil SE bond 2

2. Specimen preparation

For the control group, ProRoot MTA was mixed according to the manufacturer's instructions; 0.34 g of liquid and 1 g of MTA was placed in a clean, empty amalgam capsule. The capsule was mixed for 30 seconds using amalgamator (D-650, TPC Advanced Technology Inc., City of Industry, CA, USA). A cylindrical mold (length 5 mm×diameter 3 mm) made of polymethyl methacrylate was placed on a glass plate and mixed MTA was inserted into the cylindrical mold using hand instruments such as amalgam carrier and condensers. The extra moisture was removed using dry gauzes.

For the experimental groups, three dentin bonding agents (ASB, SBU, CSE) were applied to inner surface of the cylindrical molds (Fig. 1). After bonding agent treatment, MTA was mixed and inserted into the molds. For ASB and SBU groups, one layer of dentin bonding agent was applied in mold according to the manufacturer's instructions; ASB and SBU were applied and gently air thinned for 5 seconds to evaporate solvents, then light cured for 10 seconds. For the CSE group, two layers of dentin bonding agent were applied in mold according to the manufacturer's instructions; a CSE primer was applied for 20 seconds, and the volatile ingredients were evaporated with a mild oil-free air stream. Then, the CSE bonding agent was applied and the excess was scattered with a gentle air stream, followed by light curing for 10 seconds.

The specimens were then covered by a wet cotton pellet and a damp paper towel in a sealed plastic container and maintained for 4 days at room temperature with 95% humidity[21]. The specimens were wet polished using a Buehler Ecomet 250 Grinder Polisher (Buehler, Lake Bluff, IL, USA) with minimal pressure. Polishing was performed with 400-grit, 800-grit, 1200-grit, and 2000-grit silicon carbide grinding papers.

Table 1. Materials used in this study

Material	Abbreviation	pH	Composition	Lot number
ProRoot® MTA (white) (Dentsply Tulsa Dental, Tulsa, OK, USA)	MTA	-		0000249677
Adper Single Bond 2 (3M ESPE, St. Paul, MN, USA)	ASB	4.7	Dimethacrylate Resins, HEMA, Vitrebond™ Copolymer, Filler, Ethanol, Water, Initiators	NC24034
Single Bond Universal (3M Deutschland GmbH, Neuss, Germany)	SBU	2.7	MDP, Dimethacrylate Resins, HEMA, Vitrebond™ Copolymer, Filler, Ethanol, Water, Initiators, Silane	00221A
Clearfil SE bond 2 (Kuraray Noritake Dental Inc, Okayama, Japan)	CSE	2.0	Primer: MDP, HEMA, Hydrophilic aliphatic dimethacrylate, dl-Camphorquinone, Water Bond: MDP, HEMA, Bis-GMA, Hydrophobic aliphatic dimethacrylate, dl-Camphorquinone Initiators, Accelerators, Silanated colloidal silica	1T0617

HEMA: 2-hydroxyethymethacrylate; MDP: methacryloyloxydecyl dihydrogen phosphate; Bis-GMA: Bisphenol A diglycidylmethacrylate

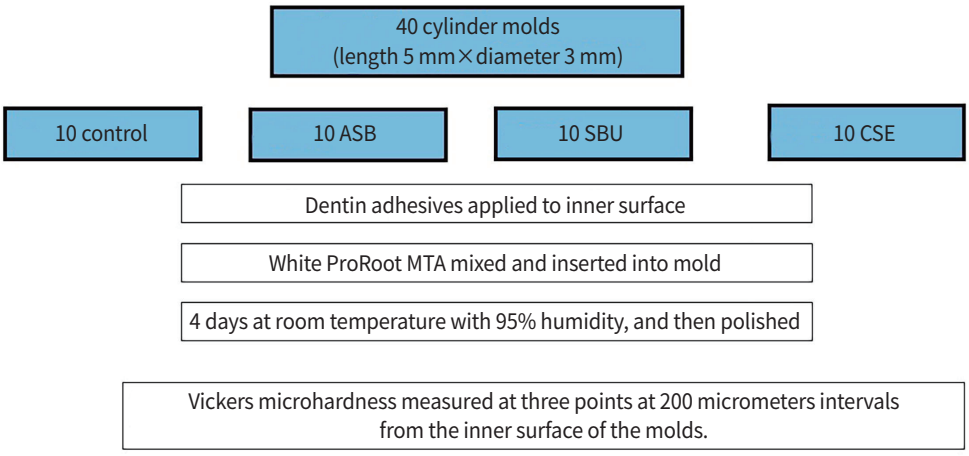

Fig. 1. A diagram showing the experimental design of this study.
ASB: Adper Single Bond 2; SBU: Single Bond Universal; CSE: Clearfil SE bond 2; MTA: Mineral trioxide aggregate.

3. Surface microhardness measurements

The investigated parameter was Vickers microhardness. For the measurement of the microhardness, polished surface of each sample was loaded with a diamond indenter point (MMT-X, Matsuzawa, Akita, Japan) with a weight of 50 g for 5 seconds dwell time[12,15]. The Vickers microhardness of each sample was calculated by measuring the diagonal diameter of the resulting indentation. Microhardness was calculated using the following equation[22]:

$$HV = \frac{2F\sin\frac{136}{2}}{d^2} \approx 1.854 * \frac{F}{d^2}$$

where F=load, 1.854=Vickers constant, d=the mean of the two diagonals.

Each specimen sample was measured at three points at 200 micrometers intervals from the inner surface of the molds.

4. Statistical analysis

The mean microhardness value was calculated for each specimen. Data were analyzed using a R studio statistical software (ver0.99.903). One-way ANOVA was conducted, where a *P*-value less than .05 was considered statistically significant. The Tukey HSD test was then performed to assess differences between the groups.

Results

1. Surface microhardness

In order of highest to lowest, the mean Vickers surface microhardness (HV) values of the control group, ASB, CSE, and SBU groups were 67.02±11.38, 48.76±11.33, 43.78±11.19, 37.84±9.36, respectively (Table 2). There was a statistically significant difference between the groups under the 95% confidence level. Tukey's post hoc test revealed that the difference between the control group and the other experimental groups was statistically significant ($P<0.001$). The difference between the ASB and SBU groups was statistically significant ($P<0.001$), but the difference between the SBU and CSE groups was not. Moreover, there was no statistically significant difference in surface microhardness between the 200 μm, 400 μm, and 600 μm points within each group ($P>0.05$) (Fig. 2). However, there was a statistically significant difference in surface microhardness between groups at each point ($P<0.05$). At each point, the control group showed higher values than all experimental groups, except for the ASB group at the 400 μm point ($P<0.05$) (Fig. 3).

Table 2. Mean Vickers microhardness (HV) and standard deviations (SD)

Group	Point (μm)	HV	SD
Control	mean	67.02	11.38
(n=10)	200	74.27	8.93
	400	63.54	14.04
	600	63.24	7.91
ASB	mean	48.76	11.33
(n=10)	200	45.26	9.77
	400	53.27	12.87
	600	47.65	10.65
SBU	mean	37.84	9.36
(n=10)	200	37.94	6.59
	400	38.18	12.25
	600	37.42	9.25
CSE	mean	43.78	11.19
(n=10)	200	42.03	9.98
	400	44.79	8.79
	600	44.51	15

HV: Vickers microhardness; SD: Standard deviation; ASB: Adper Single Bond 2; SBU: Single Bond Universal; CSE: Clearfil SE bond 2.

Discussion

The purpose of this study was to investigate the effect of dentin bonding agent according to its acidity on MTA surface microhardness. Materials based on calcium silicate are used in vital pulp therapy and perforation repair. After treatment, a cavity is filled with a final restoration. At this time, acid etching and dentin bonding agents are used. MTA properties such as solubility, compressive strength, hardness can be affected by the surrounding environment including acid etching and dentin bonding agents. The pH of dental adhesives used during treatment affect the microstructure and surface hardness of MTA, even after hardening.

Dry particles of MTA are hydrated and crystallized with interlocking mass. Crystallization occurs by the formation of cubic and needle-like crystals[11]. When it is hydrated by a solution with a pH 7, the cubic, needle-like structure is maintained. However, normal crystal structure is not maintained when MTA is hydrated with a pH 5 acidic solution. Moreover, when MTA is stored in a low pH solution, it has low micro-

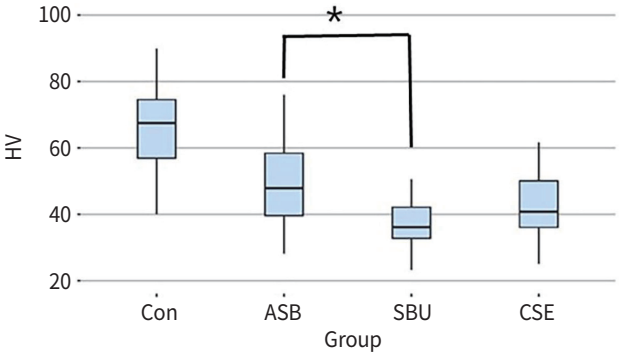

Fig. 2. Mean Vickers microhardness (HV) of each group.
*Indicated statistically significant difference between group at P<0.001.
HV: Vickers microhardness; Con: No bonding agent (control); ASB: Adper Single Bond 2; SBU: Single Bond Universal; CSE: Clearfil SE bond 2.

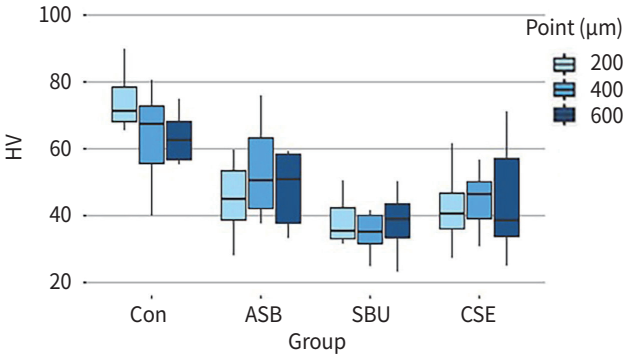

Fig. 3. Mean Vickers microhardness (HV) of each point.
HV: Vickers microhardness; Con: No bonding agent (control); ASB: Adper Single Bond 2; SBU: Single Bond Universal; CSE: Clearfil SE bond 2.

hardness[12,23]. Studies have also reported that etching above hardened MTA increases microcracks on the surface and reduces surface hardness[18]. MTA treated with phosphoric acid shows surface cracks and interior holes, and the lamellar structure disappears. Even treated with a self-etching primer, MTA shows no lamellar structure, and larger pores[19]. However, no research has been carried out on the effect of light-cured bonding agents before MTA hardening.

In this experiment, the surface microhardness of the control group was higher than that of the other groups. The mean HV value of the control group was 67.02±11.38 and the mean HV values of the experimental groups was were 48.76±11.33 (ASB), 37.84±9.36 (SBU), and 43.78±11.19 (CSE). The pH of each group was 4.7 (ASB), 2.7 (SBU), and 2.0 (CSE), respectively. The experimental groups were more acidic than the control group. ASB and SBU contain a Vitrebond™ Copolymer which is a polyalkenoic acid copolymer that chemically binds to calcium of tooth[24]. SBU and CSE contain MDP which imparts acidic characteristics[25]. Even if bonding agents are cured, an acidic environment can be created because unreacted monomers remain in the oxygen inhibition layer[26]. Previous studies have shown that exposure of MTA to acidic environments reduces its microhardness[11,12,20,23]. It has been demonstrated that MTA exposed to acidic environments shows no needle-like structures and incomplete cubic structures[11]. An acidic environment dissolves the needle-like crystal structure quickly and weakens the hardness of MTA.

In the current study, surface microhardness was significantly lower in the SBU group compared to the ASB group probably because SBU has lower pH value (pH 2.7) than that of ASB (pH 4.7). Thus, it can be inferred that the lower the pH, the lower the microhardness. Nevertheless, in the current experiment, CSE (pH 2.0), which has lower pH than SBU (pH 2.7), showed higher microhardness than SBU, although it was not statistically significant. We speculate that these results may be due to the pH of CSE bonding agent (unknown), and not the CSE primer (pH 2.0) itself. Previous research has demonstrated that MTA shows higher Vickers microhardness when stored in moist conditions compared with when it is stored in dry conditions[27]. In addition, solvated adhesive show higher water sorption than solvent-free adhesives[28]. Therefore, SBU, which is a solvated adhesive, absorbs more water, thus creating a dry environment which may result in a low MTA microhardness. The null hypothesis that there are significant differences in surface microhardness of MTA depending on the acidity of the dentin bonding agents was partially accepted.

In the current study, there were no differences in surface microhardness of MTA between the 200, 400, and 600 μm points from the inner surface of the mold. Thus, the null hypothesis that there are significant differences in surface microhardness of MTA between distances from the bonding surface was rejected. During specimen preparation, MTA was inserted into a mold and a wet cotton pellet and paper towel were placed on the top, then the specimens were maintained in a sealed plastic container for four days. It is speculated that the cotton pellet itself creates an overall acidic environment, and this may be the reason that there were no significant differences in surface microhardness of MTA between distances.

The application of dentin bonding agents is limited in the clinical conditions; bonding agents are applied to the dentin inside the access cavity with a canal opening. ASB is a fifth generation adhesive that uses the two-step total etch technique which requires the use of etchant and rinses before a bonding agent is applied. During this process, etchant may flow into the canal. SBU is a seventh-generation universal adhesive with two application modes: the self-etch mode and the total etch mode. According to data provided by the manufacturer, a higher dentin bonding strength can be obtained when the product is used in the self-etch mode. However, according to other publications, there is no significant difference in bond strength between application modes, both of which are reliable.

It is useful to apply it several times using the self-etch mode for a long time[29-32]. Both SBU and CSE bonding agents have low pHs. It is believed that dental adhesive systems of low pH may weaken the structure of MTA to a higher degree than those with a higher pH. Thus, further research on the clinical use of dentin bonding agents along with MTA is needed.

The pH of MTA is about 12.5 which is attributed to its antimicrobial properties and hard tissue formation capacity[33]. MTA stored in acidic pH solutions increases silicon and calcium ion release[34]. Silicon and calcium ion imply osteogenic and odontogenic potential. However, this increase in ion emission means greater solubility which may affect the sealing properties and surface hardness of MTA. Calcium silicated bioceramics such as MTA form calcium-phosphate salts when they meet phosphoric acid solutions. Hydroxyapatite formation capacity is an important factor in determining bioactivity. Acidic environments not only inhibit the formation of hydroxyapatite but also reduce cell viability. Further research is needed to determine whether dentin bonding agents used with MTA have an effect on another characteristic of MTA when an acidic environment is produced.

In this study, resin cylinder molds were used. If the experiment was conducted using dentin substrate, as in clinical situations, the results may have been different. Self-etching primers of low pH values increase in pH when in contact with dentin. In particular, functional monomers in a primer form insoluble particles when in contact with dentin[35,36]. Thus, if the experiment had used dentin substrate, the pH of the primer would have been offset and increased, which would have minimally affected the MTA. While the authors attempted to reproduce the clinical situation in designing the experiment, in this experiment freshly mixed MTA was placed on unset dentin adhesive while in clinical situations, dentin adhesive is placed on unset MTA. While the authors presumed the difference to be negligible, this is another limitation of this study.

Conclusion

Within the limitations of this study, three dentin bonding agents applied before MTA setting reduce microhardness of MTA. The higher acidity of dentin bonding agents may lead to the lower MTA microhardness. In clinical situation, it is recommended that MTA before setting is kept away from dentin bonding agent.

Conflict of Interest

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

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