

Thermophysical properties and bonding with composite resin of premixed mineral trioxide aggregate for use as base material

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Dental bases require low thermal conductivity and good mechanical properties, such as bonding with composite resins. This study aims to elucidate the physicochemical properties of premixed mineral trioxide aggregate (MTA) for its suitability as a dental base and to explore the optimal adhesive strategy with composite resin. The thermal conductivity and compressive strength of this premixed MTA are 0.12 W/(m·K) and 93.76 MPa, respectively, which are deemed adequate for its application as dental base. When bonded to composite resin, the use of 37% phosphoric acid etching before applying the Clearfil SE bond significantly reduced the bonding strength between composite resin and premixed MTA. This was because the compressive strength and Vickers hardness of premixed MTA decreased, and tricalcium silicate was dissolved from the surface during acid etching. Therefore, it is recommended to avoid using 37% phosphoric acid etching when bonding premixed MTA and composite resin as a dental base.

Keywords: Premixed mineral trioxide aggregate, Dental base, Thermophysical property, 37% phosphoric acid etching, Tricalcium silicate

INTRODUCTION

After removal of the infected dentin, tooth cavities may form near the pulp. Two problems can arise if these cavities are not conservatively treated before final restoration. First, patients may experience postoperative hypersensitivity because of temperature changes¹. Second, the long-term prognosis is uncertain because only the thin pulpal wall supports the final restoration^{2,3}. To address this issue, a dental base with a thickness of 1–2 mm is to replace the removed dentin and provide insulation and support for the final restoration⁴. Materials used as dental bases should have low thermal conductivity, good physical properties, and high bonding strengths with the final composite resin⁵. Glass ionomer (GI), zinc phosphate (ZP), and flowable resin (FR) have conventionally been considered high strength dental base materials⁶, with thermal conductivities of approximately 0.4–2.0 Wm⁻¹K⁻¹^{7,8}. The thermal conductivities of enamel and dentin are approximately 0.9 Wm⁻¹K⁻¹ and 0.6 Wm⁻¹K⁻¹, respectively; therefore, materials with appropriate thermal conductivities have traditionally been used as dental base materials⁹.

Mineral trioxide aggregate (MTA) was introduced by Torabinejad in 1993. It is composed of hydraulic calcium silicate and commonly used for direct pulp capping and root canal retrograde filling⁸. MTA has gained immense popularity in the dental field because of its intrinsic properties such as biocompatibility, high microleakage resistance, and potential for reparative dentin formation¹⁰. However, it is manually mixed, making it sensitive to the proficiency of the operator and resulting in difficulties in obtaining uniform thermal

and physical properties¹¹. Moreover, because of its long setting time, MTA is not optimal as a dental base¹². To address these issues, premixed MTA enables a uniform composition and possesses uniform thermal and physical properties¹³. Furthermore, because premixed MTA is based on a pozzolanic reaction, the silica and alumina eluted during the hydration process react with calcium hydroxide to enable rapid setting^{14,15}. To date, no studies have evaluated the thermal and physical properties of premixed MTA as a dental base. The main component of premixed MTA, calcium silicate, has a sufficiently low thermal conductivity for use as an insulator in the building and construction fields¹⁶. Additionally, when premixed MTA was mixed with tertiary distilled water, it exhibited a compressive strength of 100 MPa after 1 day¹⁷. Based on these results, the premixed MTA is expected to be a suitable dental base.

When MTA is used as a dental base, it directly contacts the teeth and the upper restoration. Therefore, many studies have reported methods to increase the bonding strength between MTA and composite resins¹⁹. In general, when comparing the bonding strengths of MTA and composite resin using total-etch and self-etch techniques, some studies have reported that self-etch is more favorable than total-etch¹⁹. Conversely, other studies reported the opposite^{20,21}; this suggests that the bonding technique between MTA and composite resin has not been sufficiently established. Furthermore, there is no information yet on the changes in mechanical strength, surface hardness, surface roughness, chemical composition, and crystal structure when premixed MTA is etched with 37% phosphoric acid. It has been reported that when 37% phosphoric acid etching was

applied to MTA, the surface roughness increased, which increased the ability to seal between MTA and composite resin^{22,23}. However, the increase in surface roughness was predicted only by observing the scanning electron microscopy (SEM) images of the MTA after etching, and a quantitative measurement of the surface roughness increase was not performed. In the case of premixed MTA, it was necessary to observe the effect on bonding strength when 37% phosphoric acid etching is applied prior to bonding.

Three null hypotheses were tested. The first null hypothesis was that there would be no significant differences in thermal conductivity between MTA and other conventional dental base materials such as GI, ZP, and FR. The second null hypothesis was that there would be no significant difference in the compressive strengths of MTA with GI, ZP, or FR. The third null hypothesis was that there will be no significant difference in bonding strength between MTA and composite resin when 37% phosphoric acid etching is either performed or not performed prior to applying the 6th generation bonding technique. These null hypotheses were tested using various experiments and statistical analyses to determine whether they could be rejected.

MATERIALS AND METHODS

Materials

Four different dental base materials were used: premixed MTA (Endocem MTA premixed regular, Maruchi, Wonju, Korea), GI (Fuji 1, GC, Tokyo, Japan), ZP (Elite cement 100, GC), and FR (Denfil Flow A2 shade, Vericom, Anyang, Korea). The composition of four different dental bases have been summarized in Table 1. To prepare the specimens of premixed MTA, they were immersed in tertiary distilled water for 1 day at 37°C. The GI and ZP specimens were prepared by mixing at a powder-liquid ratio according to the instructions from the manufacturer. FR specimens were fabricated by exposing each surface to curing light (Elipar S10, 3M ESPE, St. Paul, MN, USA) for 30 s.

Evaluation of thermal properties of four materials

The thermal conductivity is given by Equation (1). To measure thermal diffusivity ($\alpha(T)$) a laser flash apparatus (LFA; LFA467, Netzch, Gebrüder, Germany) was used. The specific heat ($C_p(T)$) was measured using differential scanning calorimetry (DSC; DSC204 F1 Phoenix, Netzch) and the density was measured using the Archimedes density measurement method. Premixed MTA, GI, ZP, and FR specimens were fabricated using a metal cylindrical disk-type mold with a diameter of 10 mm and a thickness of 1 mm. The specimens were polished using silicon carbide grit paper (Daesung, Sejong City, Korea) ranging from 400–1200 grit and a polishing machine (EcoMet 30, Buehler, Waukegan, IL, USA) to ensure a smooth and flat surface for accurate thermal diffusivity measurements. The thickness of each specimen was measured using Vernier calipers (Mitutoyo, Kawasaki, Japan).

To protect against reflection of the input thermal energy at the surface, each specimen was coated with carbon. The specimens were then placed on a 4-sample round/10.0 mm holder and the thermal diffusivity was measured at 37°C and 55°C using LFA after setting laser voltage to 230 V and the pulse width to 0.15 ms. The thermal diffusivity was measured 12 times at each temperature condition. After measuring the thermal diffusivity, the specimens were finely ground and the specific heat was measured 12 times using DSC.

$$\text{Thermal conductivity} = \text{Thermal diffusivity} \times \text{Specific heat} \times \text{Density} \quad (1)$$

Evaluation of compressive strengths of four materials

Fifteen specimens were prepared for each material (premixed MTA, GI, ZP, and FR) using a polypropylene cylindrical (PP) mold with a diameter of 4 mm and a thickness of 6 mm. The cross-head speed of the universal testing machine (UTM; 5942 Universal Testing Systems, Instron, Norwood, MA, USA) was set to 1 mm/min, and the compressive strengths of the four materials were measured and compared.

Table 1 Composition of four different base materials

Abbreviation	Product name	Manufacturer	Composition
Premixed MTA	Endocem MTA premixed regular	Maruchi, Wonju, Korea	Tricalcium silicate, zirconium oxide, calcium oxide, silicate oxide, and other metallic oxide
GI	Fuji 1	GC, Tokyo, Japan	Powder: Alumino-fluoro-silicate glass and poly-acrylic acid powder Liquid: Distilled water, polyacrylic acid, and polybasic carboxylic acid
ZP	Elite cement 100	GC	Powder: Zinc oxide and magnesium oxide Liquid: Distilled water and phosphoric acid
FR	Denfil Flow A2 shade	Vericom, Anyang, Korea	Filler: Barium oxide, alumino-silicate, and fumed silica Matrix: Bis-GMA and TEGDMA

Increase in surface roughness after 37% phosphoric acid etching

Four specimens of each dental material (premixed MTA, GI, ZP, and FR) were prepared using a metal cylindrical disk-type mold with a diameter of 10 mm and a thickness of 2 mm. The upper surfaces of these specimens were divided into six equal areas by dividing their width into 2 parts and length into 3 parts. Surface roughness was measured using a stylus profiler (DektakXT, Bruker, Middlesex County, MA, US) by profiling a 1 mm section inside each area with a 1 g force. The R_a value of the 200 μm to 800 μm section within the 1 mm length was then calculated. After measuring the surface roughness of all divided areas, the areas were etched with 37% phosphoric acid (Spident, Incheon, Korea) for 30 s, rinsed with tertiary distilled water for 30 s, and air dried for 15 s. Subsequently, the surface roughness of the previously measured area was re-measured to evaluate any increase in surface roughness for each four dental materials.

Bonding strength measurement and fracture mode analysis before and after etching

Forty acrylic blocks (LST-240, Aekyung chemical, Seoul, Korea) were fabricated using a PP cylindrical polypropylene mold with a diameter of 30 mm and a thickness of 20 mm. A cylindrical hole with a diameter of 6 mm and a height of 4 mm was created in the center of surface of each acrylic block, and they were then immersed in tertiary distilled water at 37°C for 2 days. The holes were filled with premixed MTA, and pressure was applied using a glass slide to ensure a flat and parallel surface to the acrylic resin. After being immersed in tertiary distilled water at 37°C for 1 day, premixed MTA was set in the hole. Forty specimens were dried at room temperature for 1 day and divided into two groups. The first group ($n=20$) underwent the steps of etching (30 s of 37% phosphoric acid etching, 30 s washing, and 15 s air drying) and was applied with Clearfil SE bond (Kuraray, Tokyo, Japan) according to the instructions of the manufacturer. The second group ($n=20$) was applied with Clearfil SE bond according to the instruction of the manufacturer without 37% phosphoric acid etching. An acrylic PP mold with a diameter of 5 mm and thickness of 2 mm was placed at the center of the filled premixed MTA, and the mold was filled with FR and light-cured for 30 s. The bonding strength between the premixed MTA and FR in the two groups was compared using the shear bond loading zig of the UTM at a conditions of 1 mm/min cross head speed. All the fractured specimens were collected and examined. An optical microscope (M165FC, Leica, Wetzlar, Germany) was used to evaluate the surfaces of the fractured specimens. The fracture modes were classified into three failure types: adhesive, cohesive, and mixed failures.

Surface micro-hardness and compressive strength change analysis after etching

Using a metal cylindrical-type mold with a diameter of 10 mm and a thickness of 2 mm, four premixed MTA

specimens were prepared and polished with 400–1200 grit paper. Five points on each specimen, at least 1 mm apart, were selected to measure microhardness using a micro-Vickers hardness tester (MMT-X, Matsuzawa, Japan). After the initial measurement, the specimens were etched with 37% phosphoric acid for 30 s, washed for 30 s, and air-dried for 15 s. Subsequently, the microhardnesses at the five points were re-measured to assess the change in microhardness for each specimen.

Using a metal cylindrical-type mold with a diameter of 10 mm and thickness of 2 mm, 20 premixed MTA specimens were prepared and divided into two groups. The first group ($n=10$) underwent etching (30 s of 37% phosphoric acid etching, 30 s, washing for 15 s and air drying) before testing. The second group ($n=10$) was not etched before testing. The compressive strength of each group was evaluated by using UTM testing machine at a crosshead speed of 1 mm/min.

Analysis of alteration of surface morphology and calcium content according to 37% phosphoric acid etching

Observations were made on the surface of a premixed MTA specimen with a diameter of 10 mm and thickness of 2 mm using field-emission scanning electron microscopy (FE-SEM; IT-500HR, JEOL, Tokyo, Japan) at $\times 500$ and $\times 5,000$ magnification. Observations were made under an acceleration voltage of 10 kV and a working distance (WD) of 10 mm. After 37% phosphoric acid etching steps, the surfaces of premixed MTA specimens were observed again. In addition, the calcium content on the surface of the premixed MTA before and after 37% phosphoric acid etching was analyzed using energy dispersive X-ray spectroscopy (EDS) at $\times 1,000$ magnification. Furthermore, EDS analysis was performed to determine the depth of 37% phosphoric acid etching penetration in the premixed MTA specimens. The premixed MTA specimen was sliced in the middle, and the cross-sectional view was analyzed by EDS at $\times 1,000$ magnification. The surface of the premixed MTA specimen was also etched with 37% phosphoric acid, sliced down the middle, and the cross section was analyzed by EDS to observe the penetration depth of acid etching.

Analysis of X-ray diffraction (XRD) graph before and after 37% phosphoric acid etching

Premixed MTA was loaded into an X-ray holder within the XRD (Ultima IV, Rigaku, Tokyo, Japan) with $\text{CuK}\alpha$ radiation ($\lambda=0.154$ nm) at 40 kV and 30 mA. The scan range (2θ) was set at 10° – 60° with a step size of 0.02° and $2^\circ/\text{min}$ scan speed of $2^\circ/\text{min}$. XRD graphs of the premixed MTA before and after etching were obtained and compared to identify the changes in the structure of the material.

Statistical analysis

Statistical analysis was performed at $\alpha=0.05$. Level of significance and error bars represent 95% confidence intervals in Figures. The mean values and standard deviations of all data are presented in the Table. Statistical analyses were performed using SPSS

Statistics 23 (IBM, Armonk, NY, USA). After equal variance for all statistical data was determined by the Shapiro-Wilk test, these values were subjected to the *t*-test and one-way analyses of variance (ANOVA), as well as *post-hoc* analyses using Tukey's test.

RESULTS

Thermal properties of four different dental base materials

The $\alpha(T)$ and $C_p(T)$ were functions dependent on temperature. According to Equation (1), thermal conductivity, which is defined as the product of thermal diffusivity, specific heat, and density, is also dependent on temperature. From Tables 2 and 3, the thermal diffusivity was in the order ZP>premixed MTA>GI>FR, and the specific heat was in the order GI>FR>premixed MTA>ZP. When the temperature increased from 37°C to 55°C, the thermal conductivity also increased. The order of the increase in thermal conductivity from 37°C to 55°C was GI (+23%)>ZP (+9%)>premixed MTA (+4%)>FR (+2%).

Compressive strengths of four different dental base materials

The mean compressive strength values for each dental material were measured and presented in Table 4, premixed MTA=92.28 MPa, GI=99.97 MPa, ZP=95.35

MPa and FR=368.48 MPa. There were no statistically significant differences in the compressive strengths of premixed MTA, GI, and ZP. However, the compressive strength of FR was significantly different from those of premixed MTA, GI, and ZP.

Increase in surface roughness after 37% phosphoric acid etching

The initial surface roughnesses of the dental bases may differ; therefore, the average increase in surface roughness after 37% phosphoric acid etching was measured for each dental base group. The mean increases in surface roughness for the four dental bases were as follows: ZP=1.285 μm >premixed MTA=0.457 μm >GI=0.228 μm >FR=0.008 μm (Table 4). All four groups showed statistically significant differences. The increase in surface roughness of FR was almost negligible, even after 37% phosphoric acid etching. This indicates that resistance to acid was in the order FR>GI>premixed MTA>ZP.

Bonding strength measurement and fracture mode analysis before and after etching

In the bonding strength comparison between premixed MTA and the composite resin, the bonding strength of the before-etching group (2.95 MPa) was statistically higher than that of the after-etching group (2.15 MPa), as shown in Table 5. This resulted in a decrease in

Table 2 Comparison of thermal properties of four different base materials at 37°C

Material (37°C)	Thermal diffusivity [mm ² /s] (Mean±Standard deviations)	Specific heat [(W·s)/(Kg·K)] (Mean±Standard deviations)	Density [Kg/mm ³] (Mean±Standard deviations)	Thermal conductivity [W/(m·K)] (Mean±Standard deviations)
Premixed MTA	0.533±0.001	0.895±0.001	2.487±0.000	1.187±0.002 ^B
GI	0.248±0.001	1.141±0.001	1.816±0.000	0.513±0.002 ^C
ZP	0.634±0.001	0.832±0.000	2.996±0.001	1.581±0.003 ^A
FR	0.208±0.000	1.130±0.000	1.723±0.000	0.404±0.000 ^D

Different capital letters denote statistically significant differences of thermal conductivity between the four dental materials at 37°C ($p<0.05$) according to ANOVA.

Table 3 Comparison of thermal properties of four different base materials at 55°C

Material (55°C)	Thermal diffusivity [mm ² /s] (Mean±Standard deviations)	Specific heat [(W·s)/(Kg·K)] (Mean±Standard deviations)	Density [Kg/mm ³] (Mean±Standard deviations)	Thermal conductivity [W/(m·K)] (Mean±Standard deviations)
Premixed MTA	0.518±0.001	0.949±0.000	2.487±0.001	1.224±0.002 ^B
GI	0.273±0.001	1.272±0.000	1.816±0.000	0.632±0.002 ^C
ZP	0.640±0.001	0.900±0.001	2.996±0.000	1.728±0.003 ^A
FR	0.197±0.000	1.224±0.000	1.723±0.000	0.416±0.000 ^D

Different capital letters denote statistically significant differences of thermal conductivity between the four dental materials at 55°C ($p<0.05$) according to ANOVA.

Table 4 Comparison of compressive strengths and the increase in surface roughness of four different dental bases

Groups	Compressive strength (MPa) (Mean±Standard deviations)	Surface roughness increments (µm) (Mean±Standard deviations)
Premixed MTA	92.28± 9.98 ^A	(0.457±0.163) ^A
GI	99.97±13.53 ^A	(0.228±0.084) ^B
ZP	95.35± 13.45 ^A	(1.285±0.161) ^C
FR	368.48±39.74 ^A	(0.008±0.004) ^D

Different capital letters denote statistically significant differences of compressive strength and surface roughness increments between the dental materials ($p<0.05$) according to ANOVA. The surface roughness increments represent the differences between the surface roughness values before and after 37% phosphoric acid etching.

Table 5 Comparison of bonding strength before and after etching conditions

Groups	Bonding strength (MPa) (Mean±Standard deviations)
Before etching	2.95±0.63 ^A
After etching	2.15±0.42 ^B

According to *t*-test; different capital letters denote statistical differences between the bonding strengths of premixed MTA and composite resin before and after etching ($p<0.05$).

Table 6 The changes in surface micro-hardness before and after etching

Groups	Surface micro-hardness (HV) (Mean±Standard deviations)	Compressive strength (MPa) (Mean±Standard deviations)
Before etching	39.82±4.73 ^A	92.28±9.98 ^A
After etching	33.18±4.91 ^B	70.12±8.74 ^B

According to *t*-test; capital alphabets indicate statistical difference of surface micro-hardness and compressive strength of premixed MTA before and after etching ($p<0.05$).

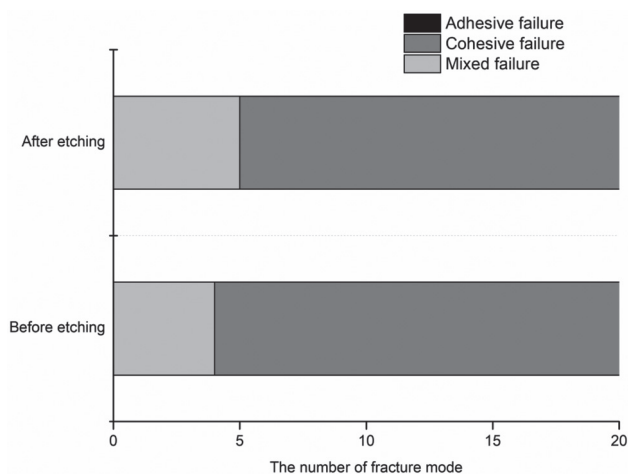


Fig. 1 Observation of fractured surfaces of specimens after completion of bonding strength test for comparison of number of fracture mode distributions before and after etching.

bonding strength of approximately 27%. The fracture mode distribution data presented in Fig. 1 show that in

the before-etching group, there were four mixed failures and 16 cohesive failures in the premixed MTA. Similarly, in the after-etching group, there were five mixed failures and 15 cohesive failures in the premixed MTA. Adhesive failure was not observed between premixed MTA and the composite resin. Thus, the third null hypothesis was accepted. To determine the reason why 37% phosphoric acid etching might have caused a deterioration in bonding strength between premixed MTA and composite resin, additional experiments were conducted, such as surface hardness tests, compressive strength tests, SEM/EDS, and XRD before and after etching.

The changes in surface micro-hardness and compressive strength of premixed MTA after 37% phosphoric acid etching

In Table 6, the surface microhardness values for before and after etching were 39.82 HV and 33.18 HV, respectively, which were statistically significantly different ($p<0.05$). The decrease in surface microhardness was approximately 17%. Similarly, in Table 6, the compressive strength values for before and after etching were 93.76 MPa and 70.12 MPa, respectively, which were statistically significantly different ($p<0.05$). The

decrease in compressive strength was approximately 25%.

Analysis of alteration of surface morphology and calcium content according to 37% phosphoric acid etching

Premixed MTA samples were observed under $\times 500$ and $\times 5,000$ magnification using the SEM images shown in Fig. 2. Before etching, as shown in Figs. 2(a) and (c), the surface appeared relatively flat and smooth with the existence of micro channels approximately $1\ \mu\text{m}$ in diameter. However, after etching, the matrix was dissolved and globular particles in the premixed MTA

were exposed, resulting in newly formed grooves, as shown in Figs. 2(b) and (d). Therefore, it can be concluded from SEM images that when 37% phosphoric acid etching was applied, the matrix of premixed MTA dissolved, causing changes in the surface morphology.

The EDS analysis provided information on the chemical composition of the SEM images of the premixed MTA. Before etching, the calcium content of the premixed MTA was 30%. However, after etching, the calcium content of the premixed MTA decreased to 0.3% (Fig. 3). Almost all of the calcium on the surface of premixed MTA disappeared after 37% phosphoric acid

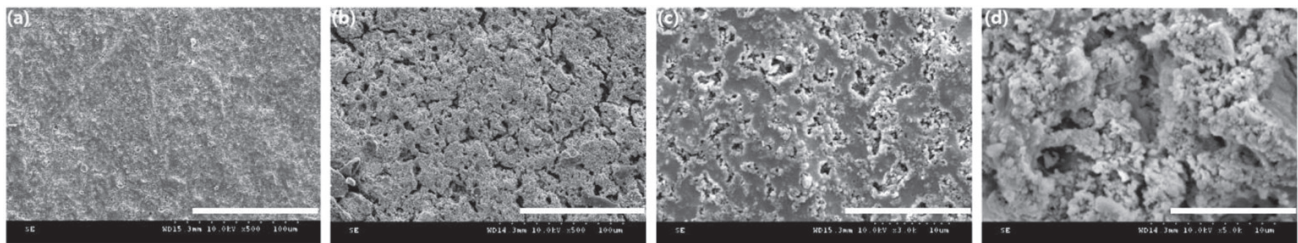


Fig. 2 SEM images of premixed MTA at (a) $\times 500$ for before etching, (b) $\times 500$ for after etching, (c) $\times 5,000$ for before etching, and (d) $\times 5,000$ for after etching (scale bar= $100\ \mu\text{m}$).

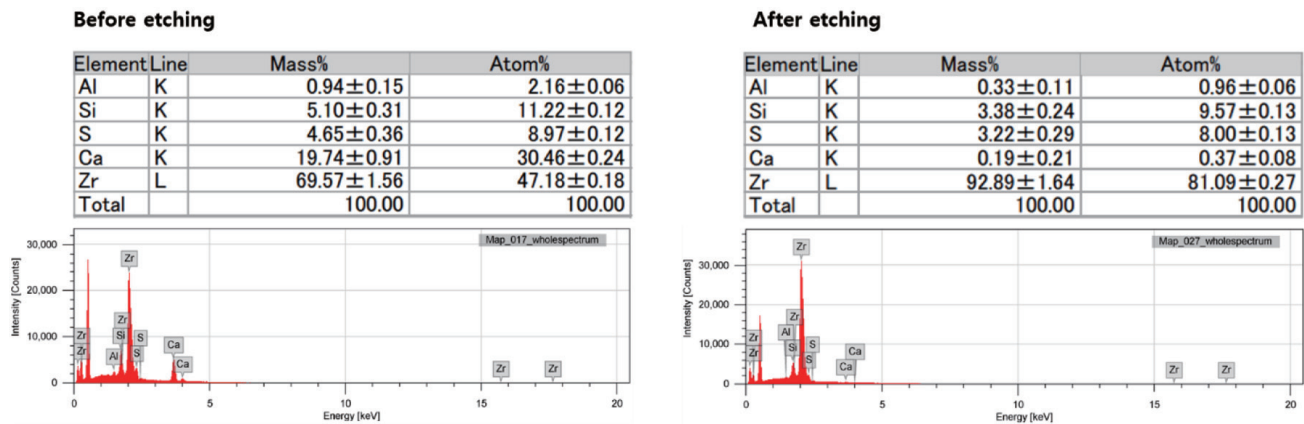


Fig. 3 EDS analysis of surface of premixed MTA before (left) and after (right) etching.

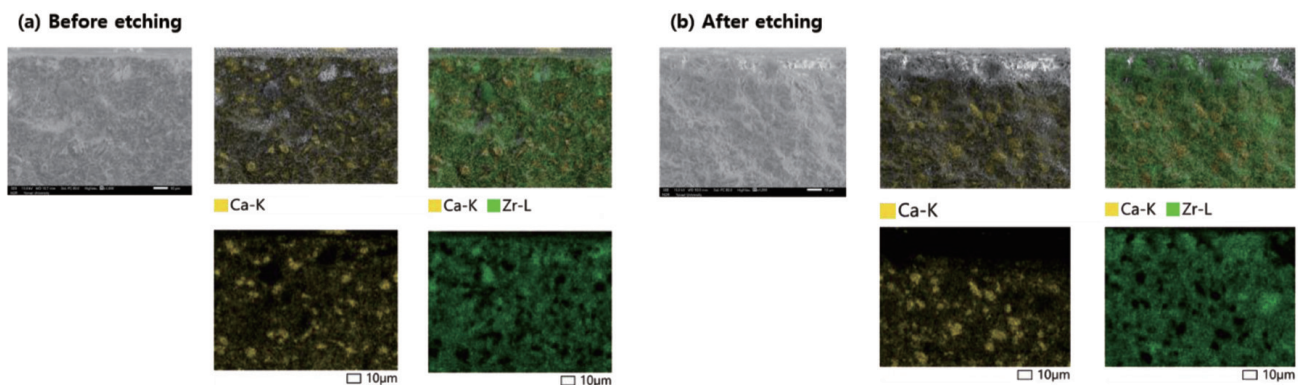


Fig. 4 The SEM/EDS images of cross section view of premixed MTA (a) before and (b) after etching. Yellow color indicates calcium ions and green indicates zirconium ions.

etching. Therefore, the disappearing matrix in Fig. 2 is likely composed of calcium.

Based on the disappearance of calcium ions from the surface of the premixed MTA after etching, the depth of the etching effect can be determined. In the cross-section image shown in Fig. 4, both calcium and zirconium ions were still present in the upper surface before etching; however, calcium disappeared from the upper surface up to a depth of 20 μm after etching. Therefore, it can be concluded that the depth of penetration of the 37% phosphoric acid etching was approximately 20 μm , based on the results shown in Fig. 4.

XRD data analysis before and after etching

XRD analysis of the premixed MTA before and after etching revealed two differences. First, the peaks at 27.0°, 29.4°, 32.2°, and 32.6°, which represented the tricalcium silicate phases, disappeared after etching. Second, the intensity of the peaks corresponding to zirconia increased after etching owing to dissolution of the matrix.

DISCUSSION

The temperature in the oral cavity was reproduced by 37°C, and 55°C reproduced the temperature that causes necrosis within the pulp²⁴). GI showed the highest increase in thermal conductivity compared to other materials when the temperature was raised from 37°C to 55°C, as presented in Tables 2 and 3. This is because the C_p of GI has a slope that rapidly increases with temperature. Although GI exhibited a high C_p slope, the thermal conductivity and diffusivity of the premixed MTA were still higher than those of GI. The thermal diffusivity depends on the structure of the materials. The premixed MTA had a large number of micropores, as shown in Figs. 2(a) and (c). Because of these micropores, the actual distance through which the input heat passes was shortened, likely leading to high thermal diffusivity and conductivity in the premixed MTA group²⁵). The thermal conductivity of premixed MTA, which ranges from 1.187–1.224 $\text{Wm}^{-1}\text{K}^{-1}$, is higher than that of enamel (0.9 $\text{Wm}^{-1}\text{K}^{-1}$) and dentin (0.6 $\text{Wm}^{-1}\text{K}^{-1}$)⁹). Although premixed MTA has a relatively high thermal conductivity, considering its potential for reparative dentin formation, antibacterial properties, microleakage resistance, and high compressive strength (Table 4), it could be a suitable dental base material^{26–28}). However, for premixed MTA, there was a significant difference in the compressive strength between the initial setting (5 min) and final setting (1 day). Therefore, patients should be advised to refrain from chewing teeth restored with premixed MTA on the first day of placement.

The changes in surface roughness because of 37% phosphoric acid etching indicated acid resistance. The results in Table 4 show that the premixed MTA and ZP groups had low resistance to 37% phosphoric acid etching. The surface roughness of the premixed MTA group increased as the calcium matrix dissolved, as shown in Figs. 2–4. Similarly, the surface roughness of

the ZP group increases as zinc oxide dissolves²⁹). When enamel was etched with 37% phosphoric acid for 30 s, the increase in surface roughness was approximately 0.15 μm ^{30,31}). Among the premixed MTA, GI, ZP, and FR groups, GI showed the most similar acid resistance to enamel.

To prevent water absorption by the premixed MTA, the acrylic block was presaturated with water before the bonding strength test¹⁹). Clearfil SE bond is considered the gold standard bonding agent for composite resin–tooth bonding in many previous studies^{32–35}). Because the premixed MTA has hydrophilic properties, the Clearfil SE bond was selected for this study. As shown in Fig. 1, most failure modes were cohesive failures of the premixed MTA, indicating that the bonding strength between the Clearfil SE bond and premixed MTA was greater than the strength of the premixed MTA. Although the compressive strength of the FR was four times that of the premixed MTA, cohesive fracture in the composite resin did not occur because of the high bonding strength between the Clearfil SE bond and premixed MTA. Furthermore, it was difficult for adhesive failure to occur because of the high adhesion strength of the Clearfil SE bond and the premixed MTA.

In Figs. 2–4, it can be seen that the tricalcium silicate matrix dissolved by etching to a depth of 20 μm , which is expected to weaken the bonding strength. The dissolution of the premixed MTA matrix exposed more zirconium oxide particles and the intensity of the zirconium oxide peak increased, as shown in Fig. 5. In clinical practice, enamel roughness is not easily increased using a self-etching primer. To overcome this difficulty, a selective etching technique was developed to increase enamel roughness³⁶). Generally, an increase in surface roughness is proportional to an increase in bonding strength. However, premixed MTA may be physically weakened despite the increase in surface roughness after 37% phosphoric etching. Therefore, it is recommended to avoid contact between premixed MTA and 37% phosphoric acid.

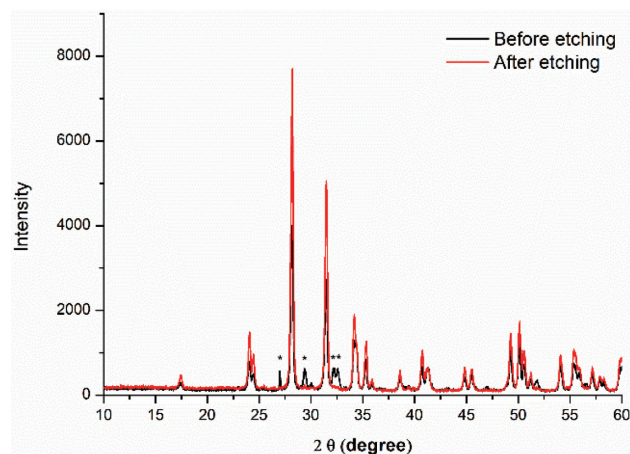


Fig. 5 XRD data before and after etching. * indicates disappeared tricalcium silicate peaks.

There are some important limitations to consider when interpreting the results of this study. The conclusions drawn from this study are specific to premixed MTA and may not necessarily apply to different types of MTA or bonding agents. Further research using a broad range of MTA types and bonding agents is required to fully understand the implications of these findings. Finally, it is important to investigate whether the observed fractures in the 20 μm section were because of decalcification from the etching process or other factors, as this could have implications for the long-term durability of the restoration.

CONCLUSION

The findings of this study suggest that premixed MTA has potential for use as a dental base, despite not having the lowest thermal conductivity and the best physical properties compared to other dental bases. If premixed MTA is used as a dental base with a composite resin as the upper restoration, it is recommended to use a 6th generation bonding technique without using 37% phosphoric acid etching for bonding strength. Overall, further research is required to explore the potential of different types of MTA and bonding techniques for use as dental bases.

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