산부식과정이 mineral trioxide aggregate와 콤포지트 레진과의 결합에 미치는 영향**

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The effect of acid-etch procedure on the bond between composite resin and mineral trioxide aggregate**

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ABSTRACT

Introduction: This study measured the contact angles of mineral trioxide aggregate (MTA) and the shear bond strengths of composite resin to MTA with and without acid-etch procedures. Methods: twenty-seven MTA specimens were prepared. The contact angles were measured using 7 specimens before and after acid-etch procedure. The remaining 20 specimens were divided into 2 groups, each with 10 specimens. Acid-etch procedure was done on etched group and not on non-etched group. Then, Scotchbond Multipurpose (3M ESPE, St Paul, MN, USA) was applied and composite resin was built. Shear bond strengths were measured using universal testing machine, and the data were subjected to Mann-Whitney U test. Results: After acid-etching procedure, the average contact angle was significantly lower than that of unetched MTA surface ($p<0.05$). MTA Specimens with acid-etch procedure showed a significantly higher bond strength than that without acid-etch procedure ($p<0.05$). Conclusions: Acid-etch procedure improved the wettability of MTA surface and the bond strength between MTA and composite resin.

Keyword: mineral trioxide aggregate, shear bond, contact angle, acid etch

서 론

Mineral Trioxide Aggregate (MTA) is well-known as the material of choice for furcation repair, internal resorption treatment, and capping of pulps with reversible pulpitis (Torabinejad & Chivian, 1999). Recently, MTA has been introduced as a potentially alternative dressing material for pulpotomy of primary molars. These applications are possible due to the bioactive, hard-tissue inductive, conductive, and biocompatible nature of MTA (Iwamoto et al., 2006; Maroto et al.,2006). In the presence of moisture, MTA sets into a hard mass by forming calcium hydroxide and silicate hydrate gel. Although MTA is suggested for use in a variety of clinical applications, there is only limited information on the effect of various treatments on the physical properties of MTA. In particular, there is quite a few amount of information on the effect of the conditioning process, including exposure to phosphoric acid, on the physical properties and crystalline structure of MTA, which is a reflection of the hydration process (Lee et al., 2004;

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Nekoofar et al., 2007). The effect of phosphoric acid, which is used to increase the retention and sealability of composite resin restorations, on MTA, could affect its durability and effectiveness (El-Araby et al., 2005). It was evaluated the effect of acid-etch procedures on the compressive strength and surface microhardness of MTA and reported that they had an effect on the compressive strength and surface microhardness of MTA. Acid-etch procedure created surface changes that might have the potential to enhance the bonding of composite resin (Kayahan et al., 2009).

In literature, investigators in some studies have focused on the suitability of placing glass ionomer (GIC), compomer and composite resin on top of MTA (Yesilyurt et al., 2009; Neelakantan et al., 2012). The findings of two previous studies have demonstrated that etch-and-rinse adhesives performed better than one-step self-etch adhesives when used to bond composite or compomer to MTA. The bond strength of etch-and-rinse adhesives in the aforementioned reports averaged between 13 and 23 MPa, whereas the one-step self-etch adhesive demonstrated values ranging between 5 and 10 MPa (Tunc et al., 2008; Bayrak et al., 2009). Authors of another report compared the shear bond strength of GIC to MTA and concluded that GICs might be used on top of MTA after the MTA has set for 45 minutes (Yesilyurt et al., 2009).

Total etching procedure created surface changes that might have the potential to enhance the bonding of resinous materials (Kayahan et al., 2009). The aim of this study was to investigate the effect of acid-etch procedure on the bond strength of composite resin to MTA. Two null hypotheses were tested: 1) no significant difference would be detected between the contact angles on the surface of MTA before and after acid-etch procedure, and 2) the acid-etch procedure would not affect the shear bond strength of composite resin to MTA.

Mateirals and Methods

Specimen Fabrication

Twenty-seven specimens of white ProRoot MTA (Dentsply Tulsa Dental, Tulsa, OK, USA) were prepared using cylindrical acrylic blocks, each with a hole in the middle measuring 4 mm in diameter and 2 mm in height. The acrylic blocks were filled with MTA and covered with a wet cotton pellet. Then, the specimens were stored at 37℃ with 100% relative humidity for 48 hours to encourage setting. The MTA surface was neither rinsed nor polished after removal of the cotton pellet.

Contact Angle Measurement

The contact angles were measured on the MTA surface by using seven specimens, which were dried in an incubator. The measurements were conducted before and after acid-etch procedure.

Non-etched group: An optical tensiometer (KSV Cam100, KSV Instruments, Helsinki, Finland) performed five measurements for each specimen. Droplets of distilled water were placed on the MTA surface of specimen using a microsyringe. The profiles of the droplets were recorded for 5 s and analyzed to calculate the contact angles with a frequency of one reading per second. Etched group: Before contact angle measurement, MTA surface was etched. Acid-etching procedures consist of 37% phosphoric acid etching gel (Scotchbond Etchant, 3M ESPE, St. Paul, MN, USA) for 15 s and water rinsing for 20 s.

Shear bond strength test

Etched group: Twenty specimens were divided into two groups with 10 specimens each. The MTA surface of 10 specimens was etched for 15 s with 37% phosphoric acid etching gel (Scotchbond Etchant), rinsed with water for 20 s. Excess water was removed by blotting with absorbent paper, leaving the surface visibly moist dried. 3-step, etch-and-rinse adhesive (Scotchbond Multi-purpose, 3M ESPE) was applied over MTA according to the manufacturer's instructions. By using layering technique in 1 mm thickness, a composite resin (Filtek Z100, 3M ESPE) was applied into a cylindrical-shaped plastic matrix with an internal diameter of 2 mm and a height of 2 mm. Light curing was administered with a light-emitting diode light-curing unit (EliparFreeLight 2; 3M ESPE) with an intensity of 1,200 mW/cm^2 for 20

Table 1. The average contact angle (degree)

Material	min.	max.	mean	standard deviation
Non-etched group	33.6	54.7	41.8	7.57
Etched group	11.8	28.1	19.4	4.91

Non-etched group: The same procedures were performed as in group one, but for 10 specimens, the MTA surface was not etched with phosphoric acid etching gel.

The polymerized specimens were stored in 100% relative humidity at 37℃ for 24 hours. For shear bond strength testing, the specimens were secured in a holder placed on the platen of the testing machine and then sheared with a knife-edge blade on a universal testing machine (Z020, Zwickl, Ulm, Germany) at a crosshead speed of 1.0 mm/min. Shear bond strength in MPa was calculated by dividing the peak load at failure with the specimen surface area.

Statistical Analysis

The relationship between the bond strength and etching was statistically assessed using the nonparametric Mann-Whitney U test. The association between the contact angle and etching was tested by the nonparametric Wilcoxon signed rank test. The significance level of 0.05 was considered significant. The IBM SPSS statistics 20.0 (IBM Co., Armonk, NY, USA) was used for all analytical procedures.

Results

Mean and standard deviation of the measured contact angles on treated MTA surfaces are listed in Table 1. The contact angle measurements showed that the average contact angle of non-etched group was 41.8 ± 7.57 degree. The average contact angle of etched group was 19.4 ± 4.91 degrees. By acid-etching pro- cedure, better wetting of MTA surface was achieved (P = 0.02) (Fig 1a, 1b, and Table 1).

Figure 2 shows the statistics of shear bond strength for each group. The mean shear bond strength of non-etched group is 24.5 MPa and the mean shear bond strength of etched group is 37.8 MPa. Mann-Whitney U test indicated that Scotchbond multi-purpose with acid-etch procedure showed significantly higher bond strength to MTA than that without acid-etch procedure (P = 0.003).

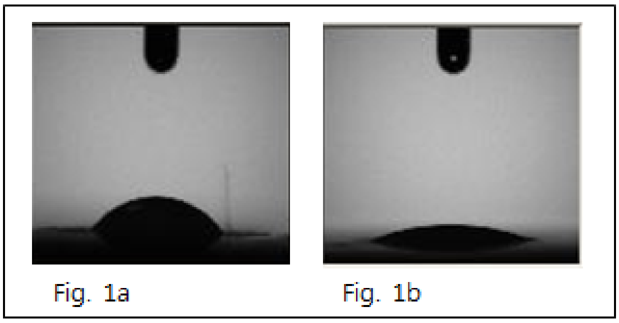

Figure 1. *Contact angle measurement of MTA surface. Representative image of groups (1a. non-etched group, 1b. etched group)*

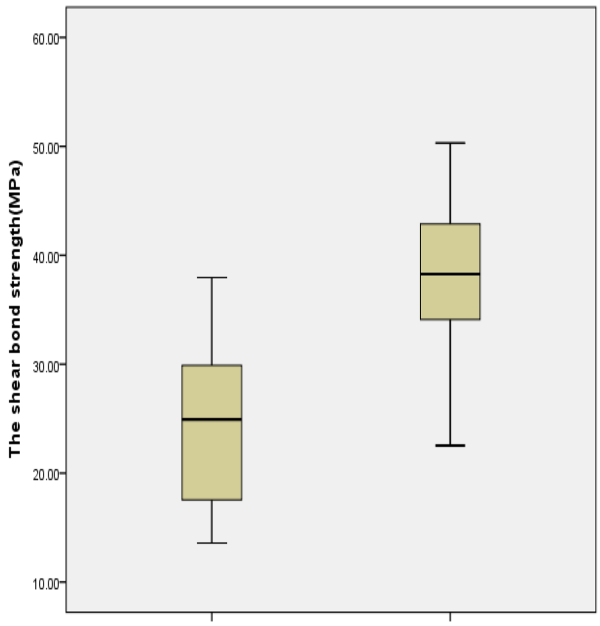

Figure 2. *Shear bond strength values of groups (MPa) to MTA.*

Discussion

Due to its many potential advantages, MTA is increasingly being used in various clinical modalities such as repair of root perforations and as a pulp capping agent in vital pulp therapy, when the final coronal restoration will be in intimate contact with MTA (Uyanik et al., 2009; Accorinte et al., 2009). However, the effects of various restoration procedures on the chemical and mechanical characteristics of MTA have not been evaluated adequately despite their significance.

It was recommended that composite resins used with etch-and-rinse adhesive system was an appropriate final restoration in contact with MTA (Tunc et al., 2008). Recently, it was also reported that MTA showed better shear bond strength than self-etch adhesive systems when it is used with etch-and-rinse adhesive systems (Atabek et al., 2012). Therefore, it can be supposed that separate acid-etch procedures are beneficial to the shear bond between resin and MTA. However, there has been no direct investigation about the effect of acid-etch procedures on it.

In the present study, the effects of an acid-etch procedure on the surface contact angle of MTA and shear bond strength of MTA to composite resin were investigated. It was observed that the MTA surface after acid-etch procedures produced lower contact angle in comparison with the MTA surface before acid-etch procedure, which led to rejection of the first null hypothesis.

Contact angle measurement is a useful indicator of the wettability of a liquid. Good wetting promotes capillary penetration and adhesion, and indicates strong attraction between the liquid and solid surface molecules (O'Brien WJ, 1997). Surface tension is the result of intermolecular attraction of a liquid in contact with a solid surface. When this intermolecular attraction is weakened, the surface tension decreases. Surface tension may be reduced by using a surfactant or heat (Abou-Rass & Patonai, 1982; Cameron, 1986). To achieve optimal wettability, the surface tension of a liquid contacting a substrate should be as low as possible (Attal et al., 1994).

The foregoing discussion makes the acid-etch procedure essential to improving the bond between MTA and composite resin, as observed in the present investigation. This leads to rejection of the second null hypothesis, since etched group achieved higher bond strength results than non-etched group.

It was showed that acid-etch procedure affects the compressive strength and surface microhardness of MTA, and the surface morphology of MTA after acid-etch procedures created a selective loss of matrix from around the crystalline structures that resulted in a relatively uniform 'honeycomb' etched pattern without penetrating deeply or removing a substantial amount of the cement (Kayahan et al., 2009). Authors also suggested that etching created surface changes that might have the potential to enhance bonding of composite resin.

On the other hand, it was reported that the shear bond strengths of self-etch adhesives were lower than that of etch-rinse adhesive and no correlation was observed between the pH of self-etch adhesives and their shear bond strength values (Bayrak et al., 2009). What is important for a better bond between MTA and composite resin, we can suppose, is having a separate step for acid-etch procedure rather than the pH of acid. For more insights and a better understanding of the adhesion mechanism of adhesive systems to MTA, further investigations, which is focused on the mechanical interlocking between them, are certainly needed.

Conclusion

It was concluded that acid-etch procedure improved the wettability of MTA surface and the bond strength between MTA and composite resin. Under the conditions of this study, it appears that acid-etch procedure is essential for a better bond between MTA and composite resin.

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