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ORIGINAL ARTICLE

The effect of different surface treatments on roughness and bond strength in low fusing ceramics

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Abstract The aim of this study was to evaluate the influence of different surface treatments (air abrasion, acid etching, and laser irradiation) on the surface roughness and bond strength of a low fusing ceramic. Thirty-six discs of low fusing ceramic (Finesse, Ceramco) were prepared (10 mm in diameter and 2 mm in thickness) according to the manufacturer's instructions. Specimens were divided into three groups (n=12), and the following treatments were performed: Air abrasion with alumina particles (50 μm), acid etching with 5% HF and Nd:YAG laser irradiation (distance: 1 mm, 100 mJ, 20 Hz, 2 W, and 141.54 J/cm^2). Following determination of surface roughness (R_a) by profilometry, specimens were examined with scanning electron microscopy (SEM). The luting cement (Clearfil Esthetic Cement) was bonded to the ceramic specimens using Teflon tubes. After 24 h of water storage, shear bond strength test was performed using a universal testing machine at a crosshead speed of 1 mm/min. The data were analyzed with two-way analysis of variance (ANOVA) and Tukey HSD tests (α=.05). Two-way ANOVA indicated that surface roughness was significantly affected by surface treatments (p<.001). Tukey honestly significant difference (HSD) indicated that the air abrasion group had a significantly higher mean value (p<.05) than the other groups. Shear bond strength was significantly affected by surface treatments (p<.001). Tukey HSD indicated that the air abrasion group had a significantly higher mean value (p<.05) than the other groups. No significant difference was found between the acid-etching and laser-irradiation groups (p>.05). The SEM image of the laser irradiation surface appeared to be relatively smooth as compared to the images of other the groups. Air abrasion of low-fusing porcelain surfaces was effective in improving the bond strength as compared to the acid-etching and laser-irradiation methods.

Keywords Bond strength · Luting cements · Surface treatment · Ceramic · Laser

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Introduction

Due to their high esthetic profile, mechanical properties, chemical stability, and biocompatibility, all-ceramic restorations have become the focus of dental practitioners, researchers, and manufacturers alike [1]. Some authors postulate that the advantages of adhesion of all-ceramic crowns and bridges are due to improved retention, better marginal adaptation, and fracture resistance [2]. Current understanding of the adhesion of dental restorative materials is based on two fundamental theories. One theory is based on chemical adhesion, describing inter molecular forces at the interface. The other theory is based on micro-mechanical retention, attributing adhesion to inter penetration of components of the two surfaces [3].

The internal surface of the ceramic restoration must be prepared to optimize micro-mechanical retention of the cement into ceramic micro-roughness. Surface treatments of porcelain increase the surface area and create micro-porosities on the porcelain surface, enhancing the potential for mechanical retention of the cement [4]. Establishing a good bond to all-ceramic restorations improves their retention, reduces microleakages, and enhances the fracture resistance [5].

New self-adhesive cements that rely on a single-step application have been proposed for luting ceramic-based restorations [6, 7]. When using these cements, micro-mechanical retention seems to be come a crucial factor in their bonding performance [8]. Different conditioning systems have been proposed to provide roughness and promote micro-mechanical retention [9, 10]. Airborne particle abrasion with alumina particles and application of a tribochemical silica coating allows for chemical bonding to a silane coupling agent and to resin cement [11]. However, this procedure is not recommended for glass ceramics, since it could lead to micro-cracking and premature failure [12].

Hydrofluoric acid attacks the glass phase producing a retentive surface for micro-mechanical bonding. However, increasing the mechanical strength, by increasing the crystalline content and decreasing the glass content, results in an acid-resistant ceramic whereby any type of acid treatment produces in sufficient surface changes for adequate bonding to resin [13].

Lasers have become widely used in medicine and dentistry. Technological advances during the last decade have resulted in the increased use of lasers in dentistry. Many of these advances have been directed at the use of lasers in clinical applications as an alternative to acid etching of enamel or dentin for bonding dental materials to the tooth surface [14–17]. However, there is a little information on the effects of laser lights on restorative materials. Thus, aiming at further clarifying this issue, the present study evaluated surface roughness and ceramic-composite bond strength, employing several methods for surface treatment associated with an adhesive system. The null hypothesis was that Nd:YAG laser treatment would increase surface roughness and bond strength comparable to air abrasion and acid-etching surface treatment methods.

Materials and methods

Thirty-six feldspathic ceramic disc specimens were prepared (10 mm in diameter and 2 mm in thickness) of a low fusing porcelain (Finesse, Ceramco Inc. Burlington, NJ, USA) according to the manufacturer's instructions. The bonding surface of each disc was polished using silicon carbide paper (grit 300, 400, and 600) on a rotating metallographic polishing device (Isomet 1000, Buehler Ltd, Lake Bluff, Illinois, USA) under water-cooling. The surfaces were cleaned with ethanol and dried carefully in air before surface treatment. After the finishing procedures, the specimens were subjected to ultrasonic treatment (Biosonic UC 50, Coltene Whaledent, Cuyahoga Falls, OH, USA) in distilled water to remove any surface residues and then dried. Thereafter, the ceramic discs were randomly divided into three groups (n=12), according to the surface treatments applied.

Air abrasion (group I): Air abrasion with 50-µm aluminum oxide particles (Korox, Bego, Bremen, Germany) at a pressure of 2.8 bar, a distance of 10 mm, perpendicular to the treated surface for 20 s.
Acid etching (group II): Bonding surfaces of ceramic discs were etched with 5% hydrofluoric acid (IPS Ceramic Etching Gel; Ivoclar Vivadent, Schaan, Liechtenstein) for 60 s as a previous study [18]. The gel was rinsed off with water for 20 s, then dried with oil-free compressed air.
Laser irradiation (group III): Nd:YAG laser (DEKA M.E.L. A., Calenzano, Italy), was used for laser irradiation on the ceramic surfaces. The laser optical fiber (300 mm in diameter) was aligned perpendicular to the ceramic surface at 1-mm distance and scanned the whole ceramic area. The laser parameters used were 100 mJ (pulse energy), 20 Hz (pulse per second), 2 W (power setting), 141.54 J/cm^2 (energy density), 150 µs (pulse duration) [19].

The prepared surface was ultrasonically cleaned in 90% ethyl alcohol for 30 min. The average surface roughness (Ra, µm) of the treated specimens was measured with the Mitutoyo Surftest–402 Surface Roughness Tester (Surftest 402 Analyzer Mitutoyo Corporation, Tokyo, Japan). Three traces were recorded for each specimen at three different locations in each direction (parallel, perpendicular, and oblique) giving nine tracings per sample. The average of these nine mean surface roughness measurements was used as the score for each sample. The ceramic blocks were embedded in acrylic resin (Meliodent; Bayer Dental Ltd, Newbury, UK) ensuring that the ceramic surface remained uncovered.

The luting cement (Clearfil Esthetic Cement, Kuraray Co Ltd, Osaka, Japan) was bonded to ceramic specimens using

Table 1 Results of two-way analysis of variance for surface roughness

Effect	*df*	Sum of squares	Mean square	*F* value	*p* value
Between groups	2	24,777	12,388	205,018	0.000
Within groups	33	1,994	0.060		
Total	35	26,771			

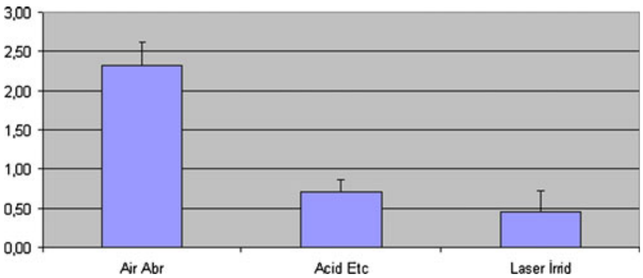

Fig. 1 Surface roughness of each surface-treatment group

Teflon tubes with an internal diameter of 4 mm and 3 mm in height. The bonding procedures were performed according to the manufacturer's recommendations. Curing light was applied to the top of the filled molds for 40 s (Bluephase, Ivoclar Vivadent). After careful removal of the Teflon molds, every side of the cement cylinder was light-cured for a total of 80 s. The bonded specimens were then stored in distilled water at room temperature for 24 h.

Finally, shear bond strength was tested with a universal testing machine (TSTM 02500, Elista Ltd Sti, Istanbul, Turkey) at a cross head speed of 1 mm/min. A shear load was applied until failure occurred. The results of testing were entered into a spreadsheet (Excel; Microsoft, Seattle, WA) for calculation of descriptive statistics. The data were analyzed by two-way analysis of variance (ANOVA) and Tukey HSD tests (SPSS/PC, Vers. 10.0; SPSS, Chicago) for pairwise comparisons among groups (α=0.05).

In order to perform micro-morphologic examination of ceramic surfaces, one additional specimen from each group was sputter-coated with gold and analyzed using a scanning electron microscope (SEM) (LEO 440, Electron Microscopy Ltd, Cambridge, UK) at 20 kV. Photomicrographs of representative areas for the surface treatments applied on ceramic groups were obtained at 1,000× magnification.

Results

The mean surface roughness values of the three surface treatment groups were as follows: air abrasion (2.33±0.28), acid etching (0.71±0.16), and laser irradiation (0.46±0.27). As seen in Table 1, two-way ANOVA indicated that surface roughness was significantly affected by surface treatments ($p<.001$). Tukey honestly significant difference (HSD) indicated that the air -abrasion group had a significantly higher mean value ($p<.05$) than the other groups. No significant difference was found between the acid-etching and laser-irradiation groups ($p>.05$) (Fig. 1).

The mean shear bond strength values of the three surface-treatment groups were as follows: air abrasion (84.72±2.46), acid etching (59.33±3), and laser irradiation (58.99±5.77). As seen in Table 2, two-way ANOVA indicated that shear bond strength was significantly affected by surface treatments ($p<.001$). Tukey HSD indicated that the air -abrasion group had a significantly higher mean value ($p<.05$) than the other groups. No significant difference was found between the acid -etching and laser-irradiation groups ($p>.05$) (Fig. 2).

SEM photographs of the porcelain surfaces treated by the different surface-treatment techniques are presented in Figs. 3, 4, and 5. The surface of the air-abrasion group appeared rough compared to that of the acid-etching group. The SEM image of the laser irradiation surface appeared to be relatively smooth compared to the images of other groups.

Discussion

Creating an effective micro-roughness on ceramic surfaces is crucial for an adequate retention of ceramic restorations [19, 20]. This study demonstrated an alternative ceramic etching pattern by laser treatment in comparison to conventional HF conditioning [21]. Although many studies have investigated the effects of laser on enamel surfaces [3, 22–24], its effect on porcelain surfaces still remains unknown. SEM analysis showed conchoidal irregularities on the irradiated porcelain surface. It was hypothesized that these irregularities enhance mechanical retention between resin composite and porcelain [25]. The indication of the study is that laser irradiation and acid etching showed similar surface roughness and bond strength values between porcelain and composite resin. The air-abrasion method created rougher surfaces than the other surface-treatment methods (laser irradiation, acid etching) and showed the highest shear bond strength values. Wood et al. [13] recommended that sandblasting be the most effective surface treatment for In Ceram Alumina ceramic. Ersu et al. [18] demonstrated that the air-abrasion method provides rougher surfaces than laser irradiation and acid etching, which is in agreement with our study.

Table 2 Results of two-way analysis of variance for shear bond strength

Effect	*df*	Sum of squares	Mean square	*F* value	*p* value
Between groups	2	5228,87	2614,43	161,93	0.000
Within groups	33	532,799	16,14		
Total	35	5761,67			

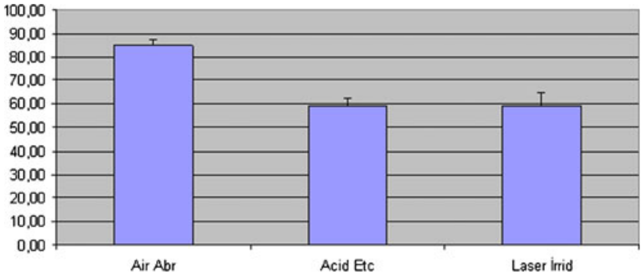

Fig. 2 Shear bond strength of each surface-treatment group

In this study, shear bond strength test was used, a commonly used bond strength test which is fast and easy to perform and also reflects the clinical situation. Shear bond strength tests are more appropriate for evaluating the adhesive capabilities of resin agents to ceramics [26].

Acid etching with solutions of hydrofluoric acid (HF) or ammonium bifluoride can achieve proper surface texture and roughness [9, 27–30]. In the procedure, the glassy matrix is selectively removed, and crystalline structures are exposed. Crystals influence the formation of micro-porosities. In a study performed by Szep et al. [31], it was found that HF acid leaves an amorphous precipitate of fluoride on tooth structures, which may influence caries resistance and bonding interaction that acid etching creates. Some low-fusing ceramics and glass ceramics contain minimal amounts of leucite crystals, which may inhibit the formation of highly retentive micro-porosities with HF acid etching [32].

Nd:YAG laser irradiation has been proposed for surface modification, so a glazed surface layer on ceramics will be formed [33]. Osorio et al. [19] evaluated the effect of different surface treatments (Sandblasting, Nd:YAG laser irradiation, Rocatec system) on In-Ceram Alumina roughness. They selected 2 W (100 mJ=pulse at 15 Hz) output power for laser irradiation in their AFM (Atomic Force Microscope) study and found similar topographic AFM images for all surface treatments. Li et al. [21] reported that there were no significant differences in the shear bond strength of a composite resin to ceramic after Nd:YAG laser irradiation at power outputs of either 0.9 W (60 mJ=pulse at 15 Hz) or 1.2 W (80 mJ=pulse at 15 Hz). In the present study, 2-W output power (100 mJ=pulse at 20 Hz) was used for laser irradiation. Laser irradiation promoted a favorable retention pattern for bonding with resin cement on tested ceramic surfaces, but roughness values were similar to the acid-etched surfaces. Li et al. [21] found that the pulsed Nd:YAG laser could replace the etching method to pretreat the fracture porcelain surfaces for bonding with composite resin at appropriate energy parameters. Akyıl et al. [34] reported that shear bond strength after HF acid etching is more effective than Nd:YAG laser etching. SEM analysis revealed that when

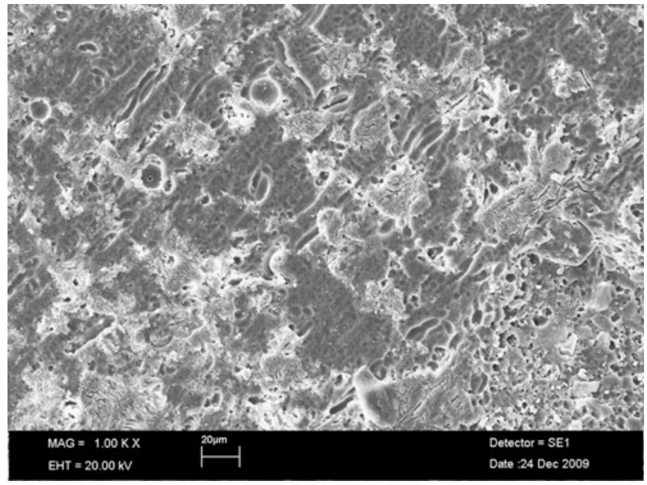

Fig. 4 SEM photomicrograph of the ceramic-applied acid etching

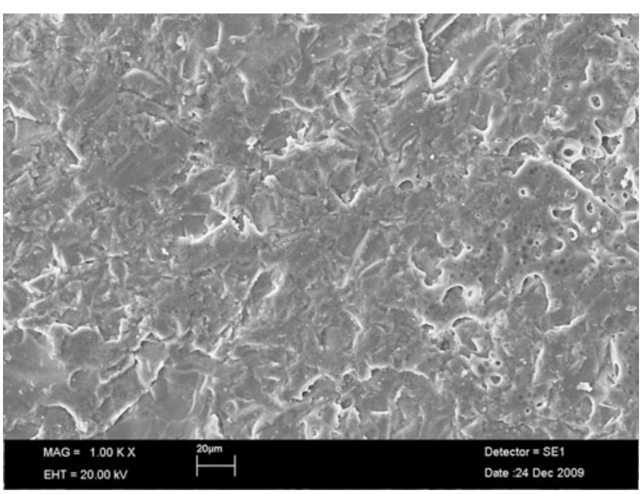

Fig. 3 SEM photomicrograph of ceramic irradiated with Nd:YAG laser

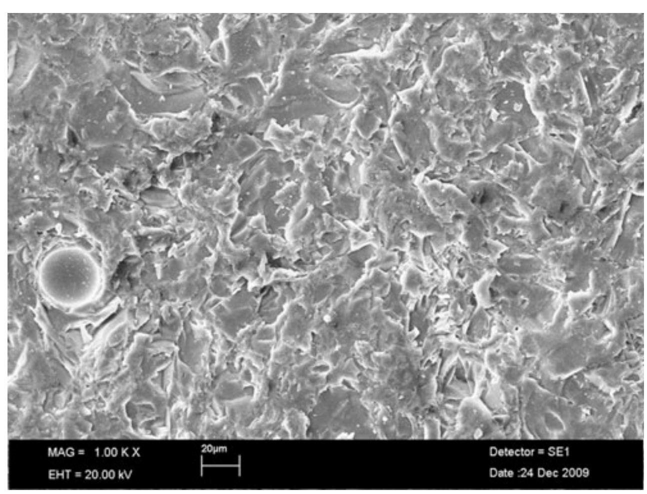

Fig. 5 SEM photomicrograph of the ceramic applied air abraded

HF acid was applied after laser irradiation, the fissures and cracks were larger than seen on the only laser-irradiated surfaces, and the shear bond strength after laser irradiation could be increased by HF acid etching. In the present study, there were no significant differences between laser irradiation and HF acid etching.

Excessive air abrasion induces chipping or a high loss of ceramic material and is therefore not recommended for cementation of silica-based and feldspathic all ceramic restorations [35, 36]. Clinicians should consider the relatively weak bond when preparing ceramic restorations for bonding, although the surface may be more retentive than polished or glazed surfaces. In a study by al Edris et al. [37], a noticeably different chemical etchant efficacy was observed between the glazed and air-abraded dental ceramics. Lacy et al. [38] showed that air abrasion provides sufficient bond strengths. Also in the current study, air-abrasion specimens showed the highest surface roughness and shear bond strength values. SEM analysis showed that air-abraded specimens showed an irregular or hollowed appearance compared to the morphology of the laser-irradiated and acid-etched surfaces. Ersu et al. [18] found that air abrasion created a rougher surface, but it did not enhance bond strength. This may be due to the fact that air abrasion creates surface irregularities without micro-mechanical retention. Wood et al. [13] reported that air abrasion created surface irregularities without undercuts; therefore, the bond strength may be reduced after prolonged periods.

The differences in composition and microstructure of all ceramic restorations might be an important factor in obtaining effective bond strengths between the ceramic and teeth. Self-adhesive cement systems have been proposed for luting ceramic-based restorations [6]. The matrix of these systems consists of multifunctional acid methacrylates that should react with the substrate and contribute to the adhesion mechanism [39], but these porcelain surfaces require a pretreatment [18, 19, 40]. To attain a strong resin bond, micro-mechanical interlocking to ceramic surface is desired, and it requires surface roughening and cleaning for adequate surface interaction [18, 19, 41]. Even though, Hummel et al. and Won-suck et al. [42, 43] postulated that shear bond strength improves with abraded surfaces, which is in agreement with our study. The current study revealed that there was a relationship between surface roughness and shear bond strength for feldspathic ceramics. A limitation of the present study is that no combined effects of higher acid concentrations and different power settings for laser irradiations were observed. Future research is required to evaluate the effect different power settings and durations for laser applications have on ceramic surfaces for obtaining optimum bond strength and roughness values.

Conclusions

This study demonstrated an alternative ceramic etching pattern by laser treatment in comparison to conventional HF conditioning. The results of this study showed that;

1. The air-abrasion method created rougher surfaces than the other surface-treatment methods (laser irradiation, acid etching) (0.05). There were no significant differences between Nd:YAG laser irradiation and HF acid etching on surface roughness (0.05).
2. The air-abrasion method showed the highest shear bond strength values (0.05). There were no significant differences between Nd:YAG laser irradiation and HF acid etching on shear bond strength (0.05).
3. Shear bond strength was significantly affected by surface treatments.

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