

ORIGINAL ARTICLE

Experimental justification of the optimization of modern adhesive protocols ceramic restoration

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ABSTRACT

Aim: The aim of our study is to optimize modern adhesive protocols through experimental investigation.

Materials and Methods: During the study, we used the following methods: laboratory, experimental, scanning electron microscopy, morphological studies and analysis of the elemental composition of structures (samples) were carried out using a scanning electron microscope and statistical.

Results: Scanning electron microscopy of ceramic samples after etching showed changes in the surface structure, which consisted in an increase in micro-spaces, more frequent and deeper relief of irregularities with increasing etching time, acid concentration and activation of the etching gel. On the surface of samples etched for 60 seconds in 9% HF, irregularities of the cellular structure with a size of 5 to 18 μm were detected. The differences between statically and dynamically etched samples are clearly visible.

Conclusions: Thus, our study confirms the safety of using sandblasting to clean ceramics, the importance of dynamic etching at the stage of preparing ceramics for fixation, and the effectiveness of using an alcohol adhesive protocol. The results obtained, based on the principles of evidence-based medicine, can be used by dental professionals to improve the quality of treatment of patients with defects in the hard tissues of the teeth.

KEY WORDS: surface aeroabrasion, ceramic restorations, scanning electron microscopy (SEM), adhesive protocol, acid etching

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INTRODUCTION

A significant development in contemporary aesthetic dentistry is associated with the introduction of adhesive restorative materials into clinical practice. The achievement of reliable fixation through micromechanical retention has enabled minimally invasive tooth preparation and maximum preservation of dental hard tissues [1–4]. The application of adhesive techniques has facilitated the fabrication of ceramic restorations that demonstrate several advantages, which, according to both national and international literature, improve treatment quality and extend clinical longevity [4,5]. At the same time, the increasing use of this technology has highlighted treatment complications related to adhesive techniques, arising from insufficient theoretical knowledge and the unwarranted expansion of their indications [2,3,6]. Errors during adhesive fixation reduce the bond strength between ceramics, zirconia, and dental hard tissues, and may lead to frequent complications such as debonding, fracture of restorations, marginal discrepancies, and secondary caries, ultimately resulting in unfavorable outcomes [2,6].

According to a number of authors, these issues are associated with inadequate understanding of adhesion mechanisms and the influence of enamel, dentin, and ceramic (zirconia) pretreatment procedures on adhesive fixation [1,5–7]. Furthermore, there is no consensus among clinicians regarding the sequence of prosthetic treatment with ceramic restorations and the selection of adhesive systems. This has drawn considerable scientific interest to the subject of adhesive fixation, making it one of the most debated topics in dentistry [7–9].

At present, a unified methodological approach to the protocol and algorithm of adhesive fixation has not been established [3,5,9]. Insufficient knowledge of the factors contributing to the weakening of the adhesive bond and of the underlying mechanisms indicates the need for both theoretical substantiation and experimental research [7,10]. Therefore, this issue represents not only an important scientific direction but also a practical challenge, and thus remains highly relevant.

AIM

The aim of our study is to optimize modern adhesive protocols through experimental investigation.

MATERIALS AND METHODS

To obtain optimal air-abrasion characteristics and to evaluate their effect on the ceramic surface, ceramic specimens were subjected to sandblasting using a Renfert Basic Classic unit (Renfert, Hilzingen, Germany) with a nozzle diameter of 0.8 mm. The sandblasting distance was standardized at 3 cm by means of a custom-made holder fabricated from Triad photopolymer resin (USA). Each specimen was treated for 5 seconds. Surface roughness parameters were determined using a TR 200 profilometer (Time Group Inc., China). Subsequently, the ceramic specimens were etched with either a 4.5% HF solution *Etchant Gel* (Ivoclar Vivadent, Germany) or a 9.5% HF solution *Porcelain Etchant* (BISCO, USA). In part of the specimens, the etching gel was actively distributed with a disposable microbrush, while in others it remained static; following the exposure time, the gel was removed with an air–water spray and the surface dried. Roughness parameters were re-evaluated using the TR 200 profilometer.

Scanning electron microscopy (SEM) was performed with a ZEISS EVO 50 XVP microscope (Carl Zeiss, Germany) to assess ceramic surface morphology and to confirm the digital micro-roughness parameters after air-abrasion and etching. Elemental composition analysis was conducted by electron probe X-ray microanalysis using an INCA analytical attachment with an X-Max detector. The analysis was performed with a focused electron beam at an accelerating voltage of 15 kV and a probe current of 0.5 nA. Morphological and elemental composition studies of the samples were also carried out using a JEOL JSM-IT300LV scanning electron microscope equipped with energy-dispersive and wavelength-dispersive spectrometers. The spatial resolution in the secondary electron detection mode (high vacuum) was no greater than 3.0 nm at an accelerating voltage of 30 kV and no greater than 15.0 nm at 1 kV.

Experimental testing was performed on ceramic restorations fabricated from Vita Mark II material and on zirconia restorations produced from Dental Zirconia Blank Ø98 mm TT-GT-M Functional (1030–1300 MPa) (Multi-Layered). The restorations were luted with a dual-cure resin cement (*Duolink*, BISCO, USA) and a fourth-generation dental adhesive (*Optibond FL*) using two bonding protocols:

1. the wet-bonding protocol;
2. the ethanol-based bonding protocol.

Statistical analysis of surface roughness parameters was carried out using software for the calculation and

statistical processing of experimental data, as well as the built-in statistical and mathematical functions of Microsoft Excel. Prior to statistical evaluation, the roughness values obtained for each specimen group were pooled into datasets. Each dataset was tested for normal distribution using the Mann–Whitney criterion; mean values, standard deviation (SD), and coefficient of variation (CV) were calculated. Homogeneity of variances and mean values of Ra and Rz parameters across different groups was verified using tests appropriate for small sample sizes, including Fisher's test, Student's *t*-test, and the approximate *t*-test at a significance level of $\alpha = 0.05$, with the number of degrees of freedom determined according to the requirements of each test. Statistical processing of the results was performed using methods of descriptive and inferential statistics; mean values, SD, and CV were computed. The significance of differences between means was determined by Student's *t*-test using Microsoft Excel 2022.

RESULTS

The aim of our study was to identify approaches for improving the effectiveness of prosthetic treatment in patients with defects of dental hard tissues using ceramic restorations, including the refinement of the adhesive fixation protocol and its implementation in clinical practice. Based on this aim, the objectives were directed toward investigating the microstructure of ceramic surfaces prepared for fixation and evaluating their microroughness parameters using different techniques, as well as improving the application technique of adhesive systems on the surface of dental hard tissues.

The assessment of specimen microroughness demonstrated that the Ra and Rz parameters, both without treatment and after sandblasting, showed no statistically significant differences across the groups (Fig. 1).

The next stage of the study was devoted to ceramic etching. Many previous investigations have evaluated different exposure times of etching gels on ceramic surfaces and reported findings similar to ours: an increase in surface microroughness with longer exposure times and the presence of morphological changes expressed as widening of intercrystalline spaces [9,11,12].

A distinctive feature of our study was the evaluation of differences between active and passive ceramic etching. The initial hypothesis assumed that continuous distribution of the etching gel with a microbrush would enhance the etching effect and accelerate its onset. However, the obtained results did not confirm a significant effect of active etching with 4.5% HF, but

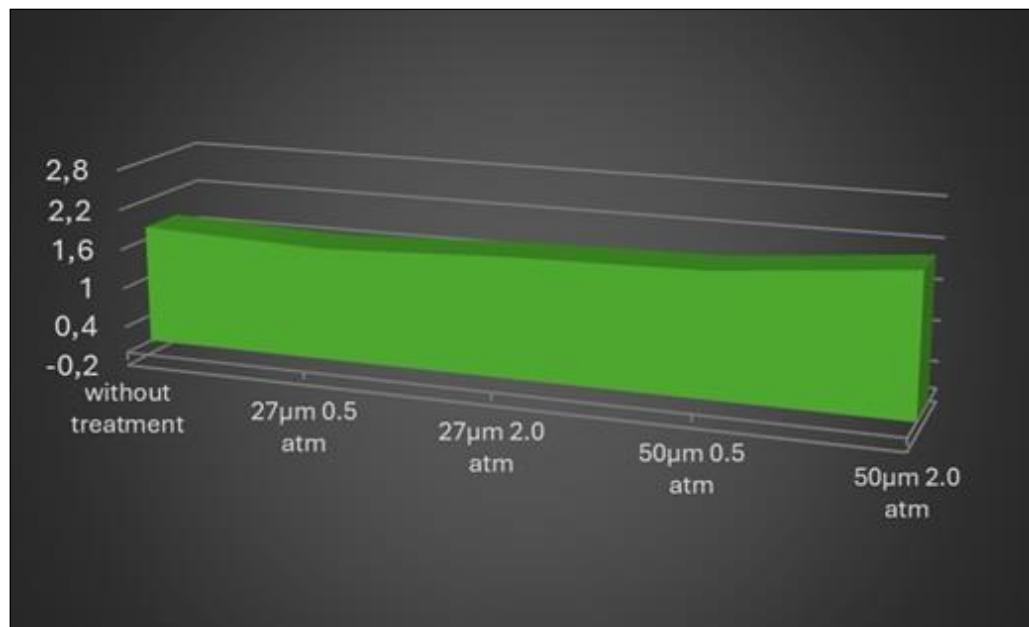


Fig. 1. Microroughness results after air-abrasion treatment
Picture taken by the authors

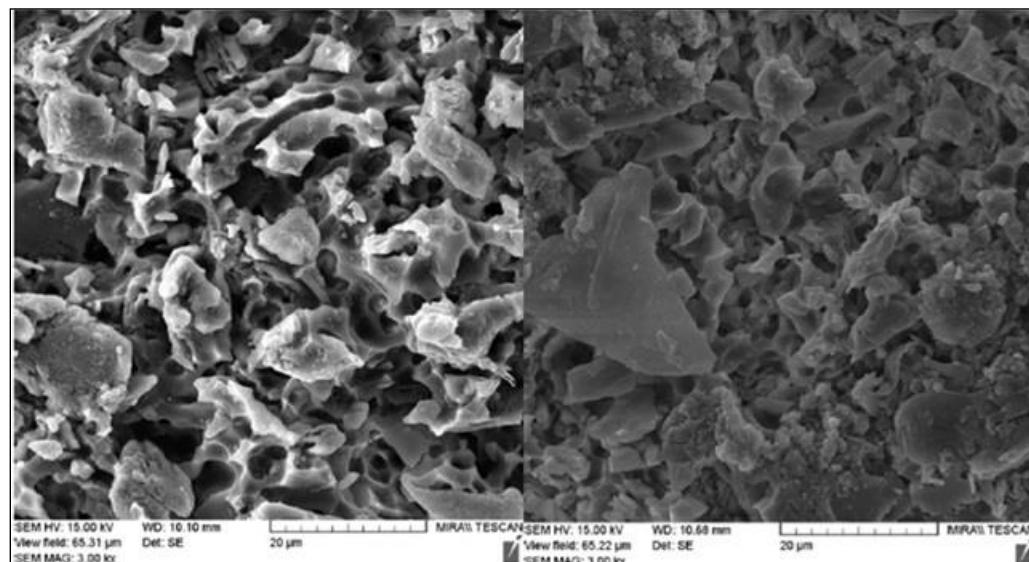


Fig. 2. Left – ceramic specimen etched dynamically for 60 s in 9% HF; right – specimen etched statically for 60 s in 9% HF
Picture taken by the authors

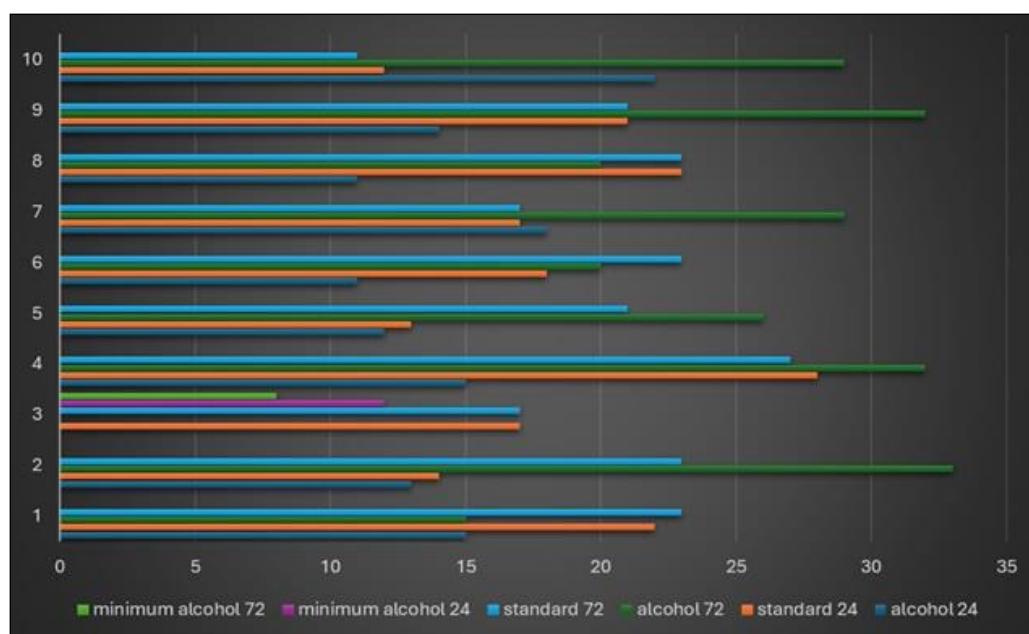


Fig. 3. Comparison of tensile bond strength values among the four groups
Picture taken by the authors

did substantiate the effect of 9% HF on microroughness parameters. The use of 4.5% HF for etching feldspathic ceramic Vita Mark II is therefore not recommended.

Differences in exposure time with 4.5% HF were statistically insignificant with respect to microroughness parameters. In contrast, when using 9% HF gel, a statistically significant difference was observed between the dynamic and static etching methods ($p > 0.05$).

For the analysis of adhesive interface morphology under standard and ethanol-based adhesive protocols, ten second and third molars from both jaws were used, extracted for orthodontic or surgical reasons, without carious lesions, cracks, restorations, or structural defects. The teeth were randomly divided into two groups; each tooth received an adhesively fixed ceramic block made of Vita Mark II with Duolink dual-cure composite cement (Bisco, USA). In the first group ($n = 5$), adhesive protocol No. 1 was applied; in the second group ($n = 5$), adhesive protocol No. 2 was used.

After fixation, the specimens were stored in distilled water for 24 hours. The adhesive interface area was then processed with a turbine handpiece (Dentsply Sirona, Germany) and fine-grit burs (Meisinger, Germany) under water and air cooling. The surface was etched with 37% phosphoric acid for 150 seconds, rinsed with distilled water, and subsequently immersed in sodium hypochlorite solution for 10 minutes, followed by additional rinsing with distilled water. The specimens were stored in distilled water for another 24 hours prior to electron microscopy analysis.

Morphological studies and elemental composition analyses were carried out using a JEOL JSM-IT300LV scanning electron microscope equipped with energy-dispersive and wavelength-dispersive spectrometers. The spatial resolution in secondary electron detection mode (high vacuum) was ≤ 3.0 nm at 30 kV and ≤ 15.0 nm at 1 kV. In low-vacuum mode, the spatial resolution at 30 kV did not exceed 4.0 nm. Under these conditions, the measurement range for linear dimensions extended from 0.03 to 1000 μ m with a maximum permissible relative error of 10%. The magnification range was from 5 \times to 300,000 \times based on a 10 \times 12 cm image size.

Scanning electron microscopy of ceramic specimens after etching revealed surface structural changes characterized by an increase in microporosities, as well as a more frequent and deeper surface relief with longer etching times, higher acid concentrations, and activation of the etching gel. Consistent with the findings of Ravikumar Ramakrishnaiah et al. (2016), specimens etched for 60 seconds with 9% HF exhibited honeycomb-like surface irregularities ranging from 5 to 18 μ m in size. Clear differences were observed between spec-

imens subjected to static and dynamic etching (Fig. 2).

Elemental composition analysis of the etched ceramic specimens, conducted in addition to SEM evaluation, confirmed that the qualitative elemental composition corresponded to the manufacturer's specifications. One of the modern and most debated approaches to improving dentin adhesion is the use of the ethanol wet-bonding protocol. This shear bond strength test provides quantitative data for the objective evaluation of the effectiveness of conventional and ethanol-based adhesive fixation protocols. In our study, a simplified ethanol bonding technique was applied, consisting of the application of 95% ethanol for 30 seconds in combination with water and Optibond FL primer, which is considerably easier to implement in clinical dentistry. The study included 40 second and third molars extracted from both jaws for orthodontic or surgical reasons, of approximately similar dimensions, without carious lesions, cracks, restorations, or structural defects. Following extraction, the teeth were stored in 2% chlorhexidine solution for no longer than one month; the solution was replaced every 14 days to avoid contamination. The occlusal surface of each molar was sectioned at the equator using diamond disks under water and air cooling, followed by finishing with a turbine handpiece (Dentsply Sirona, Germany) and fine-grit burs (Meisinger, Germany) under water and air cooling. After sectioning, each tooth was mounted in a block of self-curing acrylic resin so that the surface intended for bonding with the test materials remained free and accessible for treatment. The teeth were randomly assigned to four groups. A ceramic fragment with a 3 \times 3 mm base made of Vita Mark II was adhesively bonded to the dentin of each tooth using Duolink dual-cure composite cement (Bisco, USA). Adhesive protocol No. 1 was applied in groups 1 and 3, and adhesive protocol No. 2 in groups 2 and 4. Another distinctive feature of this study was the use of a protocol for indirect ceramic restoration fixation involving co-polymerization, whereas in other studies the adhesive was polymerized separately prior to independent polymerization of the composite block.

A characteristic feature of this method is the considerable stress transmitted to the dental hard tissues and the ceramic block, which often results in cohesive fractures of the specimens. Such outcomes were observed in the 3rd and 4th groups, where 6 and 5 specimens, respectively, fractured cohesively within the ceramic layer. This indicates that the strength of the adhesive joint exceeded that of the ceramic itself and could hypothetically have been even greater than the measured values. The 24-hour results for groups 1 and 2 showed mean bond strength values of 18.40

MPa (standard deviation 4.75) and 14.27 MPa (standard deviation 4.0), respectively. The standard Optibond FL protocol demonstrated higher values, though the difference remained within the margin of error. After 72 hours, specimens from groups 3 and 4 showed mean values of 20.21 MPa and 24.87 MPa, respectively; the ethanol-based Optibond FL protocol demonstrated a statistically significant improvement. Results reported by Shan Shan Duan et al. (2019) in a comparable shear bond strength study also showed a significant increase in adhesion strength in the ethanol bonding groups. The difference in bond strength between the 24- and 72-hour intervals can be explained by the findings of Jang Y. et al. (2021): the degree of conversion of a light-cured resin polymerized for 20 seconds through a 4 mm ceramic block is lower after 24 hours compared with that of a dual-cure material that was not exposed to light polymerization. Therefore, waiting more than 24 hours before testing could have allowed further polymer chain growth and improved adhesive conversion [11,13–15]. The difference between the mean values of the ethanol protocol at 24 and 72 hours was statistically significant with high probability (t -test = 0.0006183). The high variance (53.227035) in the ethanol protocol group tested at 72 hours indicates considerable variability of the parameters. Nevertheless, the lowest bond strength value in this group (12.41 MPa) exceeded the corresponding minimum values in the other groups.

Based on the tensile bond strength results, it was concluded that the ethanol-based adhesive protocol provides superior adhesion strength (Fig. 3).

Analysis of the adhesive interface morphology demonstrated uniform and deep penetration of adhesive tags into dentinal tubules, regardless of the type of adhesive protocol; however, the morphological pattern may vary considerably depending on the sectioning angle relative to the spatial orientation of the tubules [14,16,17].

Experimental studies of adhesion were conducted according to the criteria modified by Nathaniel C. Lawson et al. (2020). These and most other clinical studies on adhesion were carried out for direct composite restorations, whose rate of change and degradation is several times higher than that of ceramic restorations. Nathaniel C. Lawson et al. (2020) reported 19 cases of marginal defects, 6 cases of marginal discoloration, 3 cases of secondary caries, and 3 cases of debonding among 126 treated teeth one year after restoration. Most of the unsatisfactory results were observed in groups using self-etch adhesive systems [18].

In the case of more reliable ceramic restorations combined with a fourth-generation adhesive system, the results included 2 cases of marginal defects and 8

cases of superficial marginal discoloration. No statistically significant difference was observed between groups 1 and 2.

The analysis of these findings indicates that the clinical evaluation method demonstrates low sensitivity for investigating specific aspects of adhesive system performance. Several authors have confirmed this conclusion in their studies. For more precise clinical assessment of the effect of the ethanol bonding protocol, new methods of clinical evaluation are required, whereas laboratory methods consistently confirm higher bond strength when it is used.

DISCUSSION

Both domestic and international research groups have conducted a considerable number of studies on the use of air-abrasion and hydrofluoric acid etching. Authors such as Goro Nishigawa et al. (2019), Alireza Keshvad et al. (2019), Michele Carrabba et al. (2021), and İlknur Caglar et al. (2018) reported positive effects of sandblasting and etching on adhesive bond strength [14,16,18,19]. However, in most of these studies, one type of air-abrasion protocol was applied that is rarely used in actual clinical practice—sandblasting for 10–30 seconds (Goro Nishigawa et al., 2019; Michele Carrabba et al., 2020).

Moreover, in the majority of cases, the studies focused on evaluating the direct effect of sandblasting on bond strength without analyzing the morphology and microroughness of the surface (Goro Nishigawa et al., 2021) [18,20].

SEM analysis in our study confirmed earlier findings regarding the numerical values of microroughness by showing no morphological differences between specimens subjected to different sandblasting protocols. This contrasts with the results of Uwalaka C.O. et al. (2022) and other investigations that assessed the effect of air-abrasion [18,20,21]. It should be noted, however, that the sandblasting duration in those studies (10–30 s) differed substantially from that applied in our study (5 s).

The obtained results may be applied in clinical dentistry to justify the use of short-term air-abrasion for cleaning contaminated ceramic surfaces without causing surface damage or compromising surface quality.

In the field of adhesive systems, fourth-generation adhesives remain the “gold standard.” Publications by David Pashley et al. (2020), Shan Shan Duan et al. (2021), and Muhammet Kerim Ayar et al. (2023) support the use of the ethanol bonding protocol to improve the quality of dentin adhesion.

The original ethanol bonding technique involved sequential dentin saturation with 50%, 70%, 80%, and

95% ethanol for 30 seconds each, lasting a total of 3–4 minutes, as applied in the studies by C. Yesilyurt et al. (2018). Li F. et al. (2019) reported enhanced bonding of commercial adhesive systems to dentin when dentin was saturated with 100% ethanol for 1 minute, similar to the method proposed by F.T. Sadek et al. (2018), which, however, represents a clinically challenging protocol [2,8,16,22]. The effectiveness of the ethanol protocol remains inconclusive: most authors studying this issue suggest that it should be considered primarily as an *in vitro* method and emphasize the lack of sufficient evidence supporting its *in vivo* effectiveness (Muhammet Kerim Ayar et al., 2020). In our study, adhesive shear bond strength was evaluated using the Macro Shear Bond Strength Test (SBS), comparable to the methodologies applied by Shan Duan et al. (2021), Yesilyurt et al. (2019), and Li F. et al. (2019).

For more accurate data, the Macro Tensile Bond Strength Test (TBS) may be employed, in which the force is applied perpendicularly to the adhesive interface and cohesive specimen fractures occur less frequently, allowing for more reliable results [18,21–23].

Today, the literature describes a large number of different methods that increase the adhesion of zirconia to luting cements, which indicates the relevance of this problem. Based on the analysis of available sources, the following methods were identified as enhancing zirconia–cement adhesion: grinding with diamond burs, sandblasting, acid etching, laser surface treatment, electrical discharge machining (EDM), tribochemical silica coating (TBS method), selective infiltration etching (SIE), nano-alumina coating, zirconia powder fusion sputtering (FS), MDP-containing primers, MDP-free primers, and universal adhesive systems [7,8,14,20–23].

Sandblasting involves treating the material surface with particles (most commonly aluminum oxide) propelled by a high-speed source. Particles of various diameters, ranging from 25 to 150 μm , may be used. The recommended particle size depends on the type of material, particularly its translucency, in order to achieve greater surface relief while minimizing damage. Other parameters—including air pressure, particle shape, distance and angle of impact, and treatment duration—also influence the final outcome. Sandblasting removes surface contaminants, increases bonding surface area and roughness, and improves wettability [16,18,20–23]. However, this method also has drawbacks, including the potential formation of defects and microcracks, as well as excessive tetragonal-to-monoclinic phase transformation, which reduces the mechanical properties of the final restoration [23–25].

Zirconium dioxide is generally considered a

polycrystalline ceramic resistant to acid exposure at room temperature for short, clinically relevant periods. Nevertheless, several attempts have been made to modify etching conditions, and recent studies have demonstrated improved effectiveness of this method [8,12,18,22]. Various highly concentrated acids—such as nitric, hydrofluoric, and hydrochloric acid, as well as their mixtures—can be used for etching. Additional components, including alcohol, hydrogen peroxide, or ferric chloride, may be added to the solution. Different temperature regimes and etching times may also be employed. Under properly selected conditions, this method enables the creation of a uniform, homogeneous, and rough surface on the fitting surface of the restoration. Furthermore, the absence of direct physical impact helps avoid microdamage to the material and unwanted phase transformations [20,22].

Despite the numerous proposed methods and their combinations, no universally accepted adhesion protocol has yet been established that ensures consistent and stable long-term results. None of the reviewed methods can be unequivocally recommended as the most effective, accessible, and non-destructive for clinical practice. However, based on comparative evaluation, certain methods appear more favorable and may be considered suitable for broader clinical application.

Thus, additional research is required to evaluate the long-term outcomes of adhesion and to develop standardized protocols for zirconia surface pretreatment.

CONCLUSIONS

Comparative analysis of the results of aeroabrasive treatment of the ceramic surface in different modes showed the absence of significant differences in its microroughness. Such treatment can be used for surface decontamination. Dynamic or static etching for 30–60 s does not have a significant effect on the microroughness of Vita Mark II ceramics when etched with 4.5% hydrofluoric acid and does not differ from the results obtained after aeroabrasive treatment. At the same time, the effect of dynamic etching with 9% hydrofluoric acid on the ceramic surface leads to an increase in the average microroughness parameters Ra and Rz by 1.58 μm compared to static etching, increasing the area of the adhesive surface and improving the quality of adhesive fixation. SEM study of the ceramic surface showed an increase in the microroughness relief with increasing exposure time and concentration of the etching gel. Dynamic etching causes visualized morphological changes in the microstructure. *In vitro* adhesion strength analysis demonstrated maximum adhesion strength in the

alcohol adhesive protocol group after 72 hours and no increase in adhesive bond strength in the standard protocol group at the same time. SEM morphology analysis of the adhesive interface showed high-quality and deep dentin hybridization, no visible differences between the standard and alcohol protocol groups.

Thus, our study allows us to answer questions in clinical dentistry that are currently under active discussion

in the dental community, namely: the possibility and safety of using sandblasting to clean ceramics, the role of dynamic etching at the stage of preparing ceramics for fixation, and the effectiveness of using the alcohol adhesive protocol. The results obtained, based on the principles of evidence-based medicine, can be used by dental professionals to improve the quality of treatment of patients with hard dental tissue defects.

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CONFLICT OF INTEREST

The Authors declare no conflict of interest

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