



Effect of Different Modification Techniques on Mechanical and Physical Properties of E-Max CAD Substrate

Marwah Hussein Abdulsattar^{(1)*}
Alaa Hussein Jasim⁽²⁾

⁽¹⁾ Department of Prosthetic Dental techniques, College of Health and Medical Techniques, Middle Technical University, Baghdad, Iraq.

⁽²⁾ Department of Prosthodontic Dental techniques, College of dentistry, Almustansiriyah university, Baghdad, Iraq.

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*Corresponding Author:

Email:

marwahhusseinlazzawi87@gmail.com⁽¹⁾

alaa.hussein88@uomustansiriyah.edu.iq⁽²⁾

(1) Assis. Lec., Department of Prosthetic Dental techniques, College of Health and Medical Techniques, Middle Technical University, Baghdad, Iraq.

Abstract

The world's best-selling glass-ceramic is IPS E.Max CAD. It is known for its versatile applications and is appropriate for the effective manufacturing of comprehensive dental restorations. This experimental study aimed to evaluate the morphological changes, surface roughness, and micro-hardness after modification techniques with KHF_2 and NH_4HF_2 acid etching on e-max cad substrate.

Materials and Methods: Forty disc shape of E.Max CAD specimens (thickness 3mm and diameter 10mm) were fabricated and divided into four groups according modification techniques (control group, sandblasting by $70\mu\text{m}$ of Al_2O_3 , etching with 70 mg of KHF_2 and etching with 70mg of NH_4HF_2), 10 samples for each group. Surface roughness, micro-hardness, and microstructure changes of e-max CAD substrate have been measured.

Results: The higher mean value for the surface roughness (R_a) was in the group (ECNH_4HF_2), followed by (ECKHF_2) whereas the lowest mean value of surface roughness was in the group (ECC) followed by (ECSB). While for the micro-hardness test, the (ECKHF_2) group demonstrated the highest mean value in comparison with other treated groups. Finally, SEM results for both untreated and sandblasted groups revealed there is a smooth and no deep morphological change for their surfaces. While the SEM analysis revealed that the e-max cad surfaces after KHF_2 and NH_4HF_2 showed rough morphology with fine grains result and random porosities.

Conclusions: Etching with KHF_2 and NH_4HF_2 yielded higher microstructure changes and roughness than other techniques. While KHF_2 etchant was more effective on E.Max CAD surface micro-hardness in comparison with other treated groups.

Introduction:

For dental restorations to be successful there are many factors such as anatomical forms, mechanical properties, surface textures, translucency, and colors. ⁽¹⁾ In recent decades, free-metal restorations became appropriate alternatives to metal restorations due to their excellent biocompatible properties and high aesthetic performances. With such benefits, ceramic restorations were suggested for fixed prostheses like ceramic inlay/onlay restoration, partial/full bridges and crowns ⁽²⁾. In 2001, Ivoclar Vivadent introduced the lithium disilicate glass ceramic with superior processing mechanism, IPS e. max Press and the castable lithium disilicated glass ceramics with improved optical & mechanical characteristics. IPS e. max CAD was the latest generation introduced by the manufacturer in 2005, which is the machinable lithium disilicate glass-ceramic introduced for accommodation of the recent advances in technology of CAD/CAM ⁽³⁾.

However, the microstructure and composition of the ceramic restorations are essential constituents for efficient substrate bondings. Thus, different surface modification methods were suggested to improve bonding strength including sandblasting with various different oxides, particle sizes, mechanical grinding, hydrogen fluoride application as well as heat-treatment ⁽⁴⁾.

The irregular surfaces required for micromechanical bonding are produced by both acid etchings and airborne aluminium oxide particle abrasions ⁽⁵⁾. The airborne particle abrasions with alumina were shown to be efficient in the roughening of ceramic surfaces and were commonly utilized in dentistry. Nevertheless, it was difficult to adjust the abrasive blasting directions or regulate the operating air pressures. As a result, excessive airborne particle abrasions lead to micro crack formations and ceramic surface chippings. The chemical treatment with acid etchings is a relatively safer and less aggressive technique to alter ceramic surface microstructure ⁽⁶⁾. Hardness is known as the resistance to permanent surface indentation or penetration; it is a measure of the plastics deformation

resistance and is measured as a force per unit area of indentation, thus, its essential in dentistry ⁽⁷⁾.

The purpose of this study was to investigate how surface modification treatments (control group, sandblasting with 70 μ m of Al₂O₃, etching with 70 mg of KHF₂, and etching with 70mg of NH₄HF₂) impacted the surface roughness, micro-hardness, and microstructure changes of the e-max CAD substrates.

Materials and Methods

Specimens design:

Forty disc specimens of all-ceramic (IPS e-max CAD) with a dimension of (3mm in thickness and 10mm in diameter) ⁽⁸⁾ were designed with the aid of CAD software unit to convert the STL file to milling Cam machine Fig. (1). Then, they were sintered at (840 °C in 30 min). Then, it's subdivided into four subgroups according to their modification techniques with 10 specimens in each subgroup (n = 10). a) no treatment or control, b) sandblasted with 70 μ m alumina Al₂O₃ particle at 0.2 MPa for 10 sec. and at 20 mm distance from the nozzle to the specimens, c) coated by 70mg of potassium hydrogen difluoride acid (KHF₂). Then, they were heated at 280°C in a porcelain furnace (ivoclar vivadent), d) coated with 70mg of ammonium hydrogen difluoride acid (NH₄HF₂). Then, they were heated at 170°C in a porcelain furnace (Ivoclar Vivadent) ⁽⁹⁾.

Finally, the treated surfaces were cleaned with the steam cleaner for 15 sec. and then with compressed air for 15 sec. After 30 minutes, all specimens were kept in DW for 24 hours at 37°C and the surface roughness of 10 specimens/group was measured following the surface treatment protocols. Acontact stylus profilometer was used to conduct a quantitative measurement of surface roughness in micrometers (μ m) for all specimens Fig. (2). Three readings parallel with each other were done on the center of each disc specimen, and calculation of mean (Ra) was done to find the general surface properties of the samples.

After surface treatment have been finished,one specimen was selected

randomly from each group for SEM stage, firstly, the specimens were attached to the aluminum holders in the plasma gold-coating device in order to paint their surfaces with pure gold. Then, inserted into the Scanning Electronic Microscope (Inspect –S50, Holland) to determine the morphological changes after surface modification by scanning at different magnifications.

Finally, a digital micro-hardness tester was used to evaluate Vickers micro-hardness, each specimen was subjected to a hardness test in load 9.8N at 15seconds using micro-Vickers hardness tester Fig. (3). Vickers-hardness was used in this analysis due to the small specimen size and multiple compositions. Each specimen was put on the device's stage, the Vickers indenter was then lowered slowly until it came into contact with the treated surface of the specimen. The dots of the indentation remaining on the surface of the specimen after the load has been released automatically are determined with a microscope and the average estimated area of the indentation's sloping surface is calculated.

Results

A Scanning Electronic Microscope (SEM) analysis:

The scanning Electron Microscope (SEM) results for both untreated and sandblasted groups revealed there are smooth and no morphological changes for their surfaces Figures (4) and (5). While the SEM analysis revealed that the e-max CAD surfaces after KHF_2 and NH_4HF_2 showed rough morphology with fine grain result and random porosities Figure (6) and (7).

Surface roughness results:

Tables (1) and Table (2) revealed that the higher mean value for the surface roughness (Ra) was in the group (ECNH_4HF_2), followed by (ECKHF_2) whereas the lowest mean value of surface roughness was in group (ECC) followed by (ECSB).

Surface micro-hardness results:

Tables (3, 4, and 5) as well as Fig. (8), demonstrated that the higher mean value for the surface micro-hardness was in the group (ECKHF_2) followed by (ECNH_4HF_2), whereas there is non-significant difference in the mean values between (ECC) and (ECSB) groups.

DISCUSSION

The long-term success of ceramic restorations depends largely upon the substrate adhesion with either veneering ceramic or resin cement. Thus, methods like roughening by airborne particle abrasions or acids etchings were introduced and suggested for improving the micromechanical interlocking of ceramic's surface^(9, 10).

The surface roughness is essential for obtaining micromechanical retentions for all-ceramic restorations⁽¹¹⁾. According to Tables (1),(2) and SEM results showed there is clear differences in the surface structure of the treated groups when compared to the E-max cad surface without any treatment. however the mechanical treatment with aluminium-oxide (Al_2O_3) is one of an important treatment procedures to remove pollution from the ceramic surfaces and can produce the biggest quantity of monoclinic-phase that is better to bond transformations from tetragonal monoclinic phases coupled with enlarging the local volume, achieving strong adhesion of veneering ceramic^(12,13) But, the findings in the current study of roughness value (Ra) and SEM analysis demonstrated that 70 μm of a sandblasting particle with e-max cad group had non- significant effect in the mean of Ra and microstructure changes when compared with the untreated group that explained by the highly crystalline phase (70%) embedded in the glassy matrix, thus the large grain sizes of alumina oxides and highly pressure concentration post sintering may lead to weakening the surface of e-max cad substrate and accelerated tetragonal to monoclinic phase^(14,15). On the other hand, KVAM,2019 their studies showed that mechanical treatment with 50-70 μm grains' size of sandblast do not cause surface damage and develop the bonding with veneering ceramic.

The ammonium hydrogen difluoride (NH_4HF_2) and potassium hydrogen difluoride (KHF_2) etchings are chemical treatment methods, they were selected because their melt etching technique lead to fluoridated surfaces that attach to hydroxyl groups following water cleaning causing positive influences on the adhesions ⁽¹⁷⁾. Results of the current research for surface roughness and SEM images showed that etching with 70mg of (KHF_2) and 70mg of (NH_4HF_2) had a significant effect on the e-max cad substrate. SEM images revealed that treatments with KHF_2 and NH_4HF_2 altered the surface characteristics and increased the inter-grain spaces, and the grains of e-max cad are dislodged causing surface porosities ⁽¹⁸⁾. That may be due to aggressive etching agents on silica-based ceramic surfaces, reacting with the crystalline or glassy constituents producing irregular porous surfaces which increase surface areas and increase bonding strength ^(18,19) in agreement with RUYTER, 2017. They indicated that etchings with NH_4HF_2 and KHF_2 lead to the improvement of bonding capacities of zirconia with a tri-*n*-butylborane initiated acrylic resin and participated in the elevation of the surface's free energy of zirconia. The surface micro-hardness of the material denotes how hard a material is so that it resists indentation. The higher the micro-hardness of the material, the higher the mechanical properties must be predictable⁽⁷⁾. According to knowledge

of the authors, there were no previous studies investigating the effect of KHF_2 and NH_4HF_2 on micro-hardness of E.Max cad, so the outcome of the present study could be explained there was a significant effect of KHF_2 on the micro-hardness of e-max cad when compared with control group. The KHF_2 with a melting point of 280°C was more effective on the E.Max CAD surface.

Conclusions

Among our study limitations, it may be concluded that etchings with KHF_2 and NH_4HF_2 surpass alumina blasting in surface roughness and microstructure changes of e-max CAD substrate. While KHF_2 etchant was more effective on e-max cad surface micro-hardness in comparison with other treated groups.

Data availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Conflicts of interests

The authors declare no conflicts of interest.

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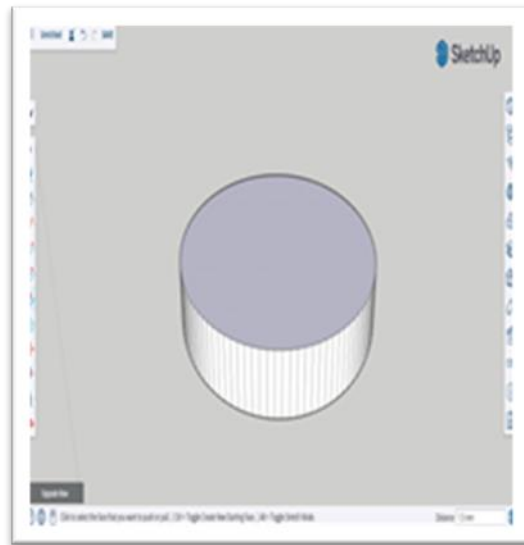


Figure (1): E-max cad specimen



Figure (2): Profilometer device for roughness test.



Figure (3): Digital micro Vickers hardness tester.

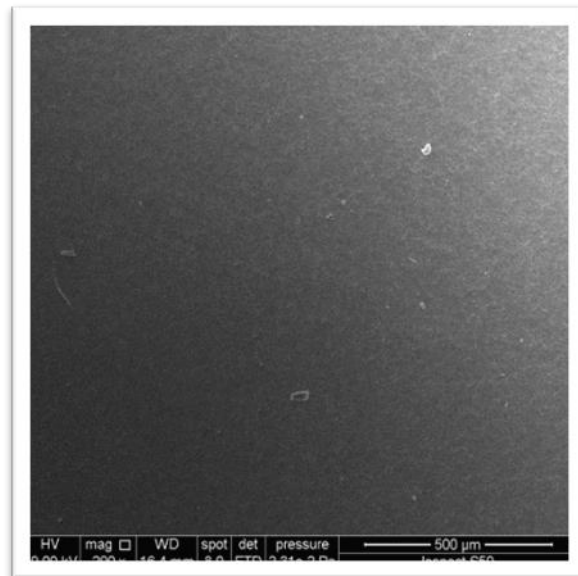


Figure (4): SEM for untreated e-max CAD group (control).

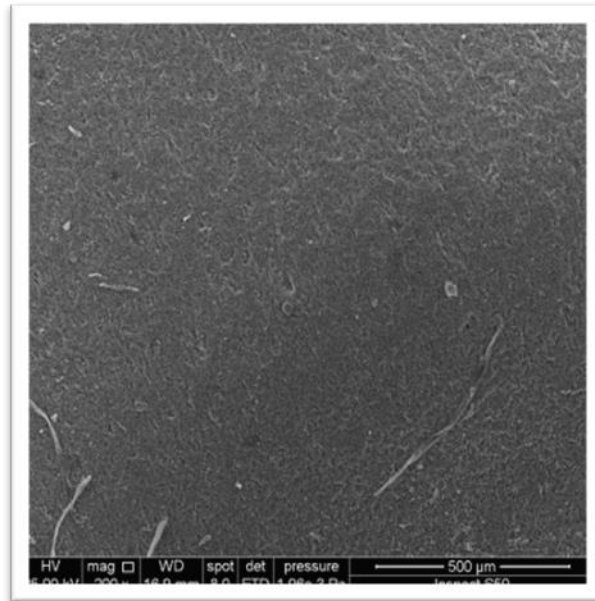


Figure (5): SEM for sandblasted e-max CAD group.

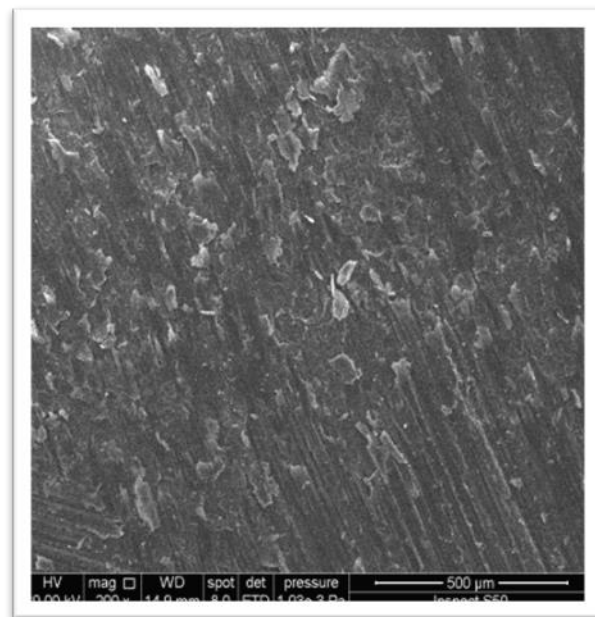


Figure (6): SEM for etched with KHF₂ e-max CAD group.

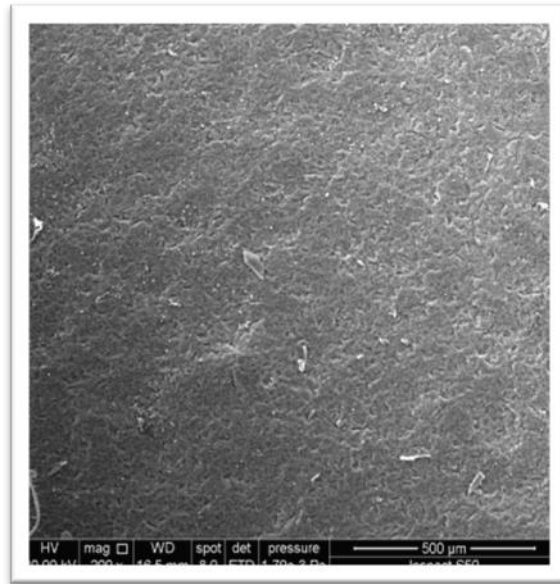


Figure (7): SEM for etched with NH_4HF_2 e-max CAD group.

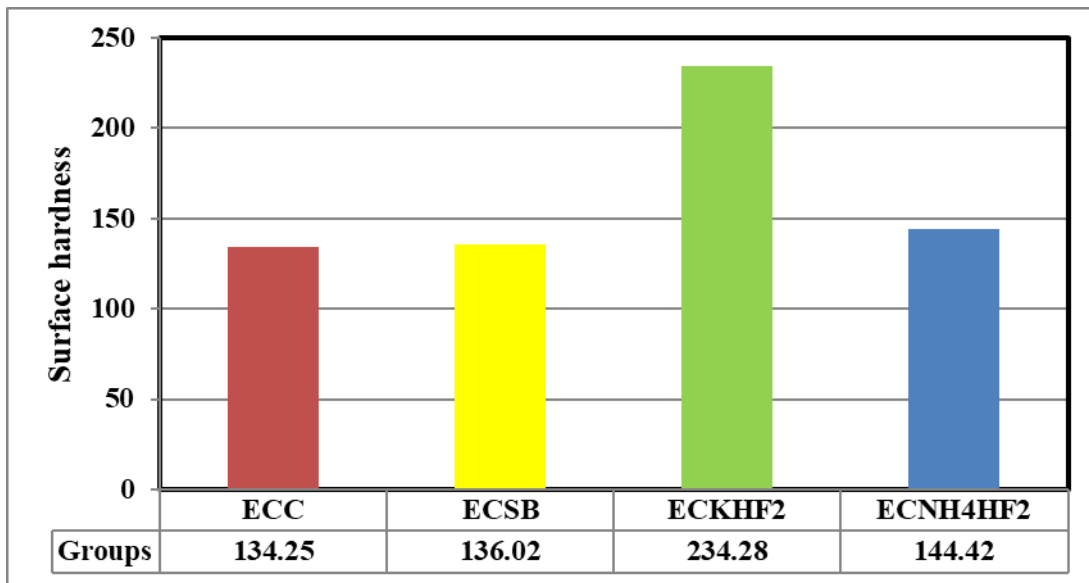


Figure (8): Diagram showed the micro-hardness values for all groups.

Table (1): Descriptive Statistics for surface roughness value (Ra) for all groups.

| Groups | Mean | SD | Min. | Max. |
|-----------------------------------|--------|--------|------|------|
| ECC | .2330 | .05164 | .39 | .45 |
| ECSB | .3325 | .01097 | .90 | .93 |
| ECKHF ₂ | 1.6520 | .02702 | 1.49 | 1.57 |
| ECNH ₄ HF ₂ | 1.9630 | .02500 | 1.92 | 1.97 |

Table (2): One-way ANOVA test for surface roughness values (Ra) for all groups.

| | Sum of Squares | Df | Mean Square | F | Sig. |
|----------------|----------------|-----------|--------------|-----------------|--------------|
| Between Groups | 11.669 | 3 | 3.226 | 2535.121 | 0.000 |
| Within Groups | .048 | 33 | .002 | | |
| Total | 11.717 | 36 | | | |

Table (3): Descriptive Statistics for surface micro-hardness value for all groups.

| Group | N | Mean | SD | Minimum | Maximum | SE | *P-value |
|-----------------------------------|----|--------|-------|---------|---------|------|--------------|
| ECC | 10 | 134.25 | 2.58 | 130.10 | 137.30 | 0.81 | H.S 0.001 |
| ECSB | 10 | 136.02 | 1.34 | 133.20 | 137.50 | 0.42 | |
| ECKHF ₂ | 10 | 234.28 | 2.22 | 230.70 | 237.00 | 0.70 | |
| ECNH ₄ HF ₂ | 10 | 144.42 | 2.19 | 140.30 | 146.40 | 0.69 | |
| Total | 40 | 162.24 | 42.34 | 130.10 | 237.00 | 6.69 | |

Table (4): One-way ANOVA test for surface micro-hardness value of all groups.

| Hardness test | F | Sig. | Sig |
|---------------|--------|------|-----|
| Among group | 5089.1 | .000 | H.S |

Table (5): LSD test for surface micro-hardness value of all groups.

| Group | Groups | Mean Difference | Std. Error | *Sig. |
|-----------------------------------|-----------------------------------|-----------------|------------|-------|
| ECC | ECSB | -1.77000 | .95526 | .072 |
| | ECKHF ₂ | -100.03000* | .95526 | .000 |
| | ECNH ₄ HF ₂ | -10.17000* | .95526 | .000 |
| ECSB | ECKHF ₂ | -98.26000* | .95526 | .000 |
| | ECNH ₄ HF ₂ | -8.40000* | .95526 | .000 |
| ECKHF ₂ | ECNH ₄ HF ₂ | 89.86000* | .95526 | .000 |
| ECNH ₄ HF ₂ | ECNH ₄ HF ₂ | -89.86000* | .95526 | .000 |

*. The mean difference is significant at the 0.05 level.

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