



Original Research Article

Effects of Different Surface Treatments on Ceramo-Metallic Surfaces

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ABSTRACT

The aim of this study was to investigate and compare the roughening effects of different surface treatments on different exposed surfaces of metal and ceramic restorations (porcelain, metal and metal/porcelain surfaces). 135 specimens were prepared for three surface groups; having 45 specimens for each group. Surface treatments were air abrasion alone or in combination with hydrofluoric acid, or phosphoric acid, burs alone or in combination with hydrofluoric acid, or phosphoric acid, hydrofluoric acid alone, and phosphoric acid alone. Statistical analysis showed significant differences between some of the different surface treatments on the same exposed surface of metal ceramic (MC) restoration as well as among the three exposed surfaces of MC restoration.

Key words: Metal, Porcelain, Surface treatments, Phosphoric acid, Hydrofluoric acid.

INTRODUCION

Ceramo-Metallic (MC) restorations have been used in crowns and bridges for aesthetic and functional purposes for several decades due to their excellent biocompatibility and superior aesthetics.^[1,2] However, fracture of porcelain veneered onto a MC restoration is common and is multifactorial.^[3] These include inappropriate coping design, poor abutment preparation, mismatch in modulus between metal and ceramic materials, technical errors, contamination, physical trauma or occlusal prematurity.^[4] Appropriate choice of repair treatment is determined by the cause of failure. Intraoral and extraoral porcelain repair treatments are two possible ways of

repair treatment. Intraoral treatment with direct composite resin is a common and more practical treatment for fractured MC restorations. This is because it demands fewer skills, less time-consuming, is less costly, less destructive, and much more convenient for the patient and dentist.^[5,6] It is indicated for situations when fracture occurs as a result of trauma, fatigue, ceramic of inferior quality, and/or technical errors.^[7]

However, the intra-oral technique is associated with several problems. The most common problem is de-bonding of composite resin material from the fractured MC restoration after short service. The bond of intraoral repair systems depends on mechanical and chemical means. The

chemical means can be achieved by silanization while mechanical retention can be facilitated by surface roughening techniques. The later include macro-mechanical and micromechanical methods.^[4,8,9,5] Macro-mechanical retention can be achieved by sandblasting the fractured surface with aluminium oxide particles, grinding with stone burs or silicon carbide paper.^[4,10,11] These methods help create a microretentive surface and increase the roughened surface area for bonding and thus its wettability.^[8,4] Micromechanical retention can be generated by itching MC surfaces with hydrofluoric, phosphoric or acidulated phosphate fluoride acids.^[12,4,9] These help produce microstructures and large porosities on the itched surfaces, allowing composite resin penetration.^[13,14]

Several studies have evaluated the effect of the mechanical roughening methods or the combination of more than one, on the composite to MC bond strength.^[10,4, 15,11] However, there is still insufficient data on the roughening effects of different surface treatments on MC surfaces and little discussion on their comparison.

The aim of this study was to investigate and compare the effects of several surfaces roughening techniques on three exposed surfaces of MC restoration; porcelain, metal and metal/porcelain. Two null hypotheses were investigated.

1. The roughening effects of eight different surface treatments would not differ within the same exposed surface of MC restorations.
2. The roughening effect of each treatment would not vary among the three exposed surfaces of MC restorations.

MATERIALS AND METHODS

The ceramic material used in this study was feldspathic porcelain color A3 (Vmk 68, VITA, Germany). A nickel-chromium dental casting alloy (Wiron99, Bego, Germany) was used for casting.

Specimen preparation

A total of hundred and thirty five disk-shaped specimens (8mm diameter x 7mm height) were prepared; 45 specimens from feldspathic porcelain (P), 45 specimens from nickel-chromium alloy (M), and 45 specimens from alloy/feldspathic porcelain (M/P).

Preparation of metal specimens (M) and porcelain specimens (P)

90 disk-shaped wax patterns (8 mm diameter x 7 mm height) were prepared using a silicone mold and inlay wax (Figure 1). The wax patterns were then invested in 50/50 % stone and plaster mix and casted using a nickel-chromium dental casting alloy.

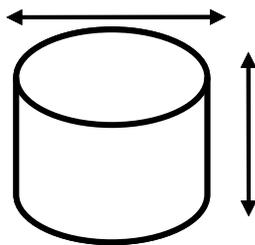


Figure. 1. Diagram represents the metal specimen.

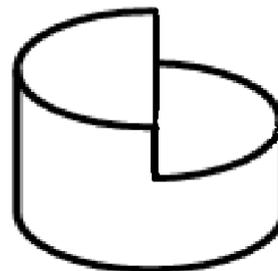


Figure. 2. A semi-circular metal specimen.

All specimens were then sandblasted with 110 μm aluminium oxide abrasive particles and were cleaned in an ultrasonic unit with distilled water to remove any remaining investment. The 90 specimens were then randomly divided into two groups of 45 specimens each. These groups were the metal group (M) and porcelain group (P).

For porcelain group (P), porcelain material (Vmk 68, VITA, Germany) was applied on to the finished surfaces of the 45 specimens selected for this group. Each specimen was held with artery clamps and then inserted into a modified syringe tube (8mm in internal diameter and 9 mm in height) (Discardit II, Spain). The opaque and dentine porcelain layers were mixed according to manufacturer's instructions, and applied to the finished surfaces using the modified syringe tube. Porcelain layers were fired at 9 C° under vacuum in a touch keyboarded computerized porcelain furnace (VACUMAT-30, VITA, Germany) according to the manufacturer's instructions.

For metal/porcelain group (M/P): 45 disk-shaped metal cylinder (8 mm diameter, 7 mm height with depth of 2 mm as semi-circular) were prepared from the same nickel chrome metal. The wax patterns were then invested in 50/50 % stone and plaster mix and casted using a nickel-chromium dental

casting alloy (Figure 2). The 45 specimens were then embedded in a phenolic ring with a cold-polymerised acrylic resin material (De Trey RR. Dentsply, England). The opaque and dentine porcelain layers were mixed according to the manufacturer's instructions, and applied to the finished surfaces using the same modified syringe tube. Porcelain layers were fired at 930 ± 10 C° under vacuum in a touch keyboarded computerized porcelain furnace (VACUMAT-30, VITA, Germany) according to the manufacturer's instructions. A laboratory medium-grit sintered diamond was then used to finish the metal and porcelain surface. All specimens were finished with 110 μm aluminium oxide abrasive particles. The specimens were then cleaned in an ultrasonic unit with distilled water to remove any remaining investment. The one hundred and thirty five specimens were then embedded in a phenolic ring with a cold-polymerised acrylic resin material (De Trey RR. Dentsply, England). The surfaces of the mounted specimens were finished using a wet silicon carbide paper (240grit) to achieve flat surfaces and cleaned in an ultrasonic unit with distilled water to remove any trapped residue. The prepared surface specimens of the three groups embedded in the acrylic resin material are shown in figure 3.

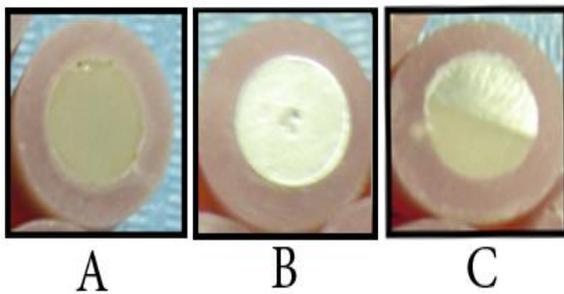


Figure 3. Prepared specimens. A- Porcelain, B- Metal, C- Metal/Porcelain.



Figure 4. Specially designed device for burs treatment.

Five specimens from each surface group were randomly selected and grouped separately as a control sub-group prior to surface treatments. The remaining forty specimens of each surface group were randomly divided into eight subgroups according to surface treatments having five specimens in each group. The nine subgroups are shown in Table 1.

Table: 1. Summary of the treatments of groups and subgroups.

Treatment Surface	Control(1)	Abr** -HF**(2)	Abr- Ph*** (3)	Abrasion(4)	Bur-HF(5) acid(5)	Bur- Ph. (6)	Bur (7)	Hydrofluoric acid (8)	Phosphoric acid (9)
Metal (M)	M1	M2	M3	M4	M5	M6	M7	M8	M9
Porcelain (P)	P1	P2	P3	P4	P5	P6	P7	P8	P9
Metal Porcelain (M/P)	PM1	PM2	PM3	PM4	PM5	PM6	PM7	PM8	PM9

*Abr = Air abrasion with 50 µm aluminum oxide particles
 ** HF = Hydrofluoric acid *** Ph. = Phosphoric acid

Surface Treatment

Five finished specimens of each surface group were selected randomly and then treated with one of the eight procedures listed below, giving 8 different treated subgroups within each main surface group.

1. Air abrasion.
2. Air abrasion and hydrofluoric acid
3. Air abrasion and phosphoric acid
4. Bur
5. Bur and hydrofluoric acid
6. Bur and phosphoric acid
7. Phosphoric acid (35%) 8)
Hydrofluoric acid (9%)

1. Air abrasion treatment

The finished surface of each specimen was air abraded using 50 µm aluminium oxide particles (Ultradent, USA) with a pressure of 3-4 bars and fixed distance. The specimens were held manually and sandblasted according to the manufacturer's instructions. They were then thoroughly rinsed using an air/water spray and were dried with oil free air using chair-side syringe.

2. Diamond bur treatment

The finished surface of each specimen was roughened using diamond-coated burs of 1.2 µm (Meisinger, Germany). Specimens were held facing a turbine using a specially designed device (Figure 4). The device was fixed away from the test surface. Every three test surfaces were roughened with one diamond bur in a turbine moving in one direction at a speed of 170000 rpm) and static load of 500g. Roughened surfaces were then cleaned using air/water spray and dried with air spray.



Figure 5. The sample during Profilometer testing.

3. Phosphoric acid treatment

The finished surface of each specimen was etched with 35% phosphoric acid (Ultradent, USA) for 60 seconds

according to the manufacturer's instructions. The etched specimens were then thoroughly rinsed using air/water spray for 60 seconds and were dried using free air spray.

4. Hydrofluoric acid treatment

The finished surface of each specimen was etched with 9% hydrofluoric acid (HF) (Ultradent, USA) for 60 seconds according to the manufacturer's instructions. The etched surfaces were then thoroughly rinsed using an air/water spray for 60 seconds and dried using free air spray.

Roughness testing

The 135 specimens of all subgroups were subjected to roughness testing using a

Profilometer (Mitutoyo, Japan). The roughness test was expressed as roughness average "Ra Value" and was calculated in micrometer (μm). Each roughness test specimen was tested by placing it horizontally on the flat fixed surface of the Profilometer with a small amount of glue to avoid unwanted movement Figure 5. Three readings for each specimen were taken. For each reading, the stylus of the Profilometer was moved three times over the test surface of each specimen at least 4 mm away from the center of the surface in three different directions.

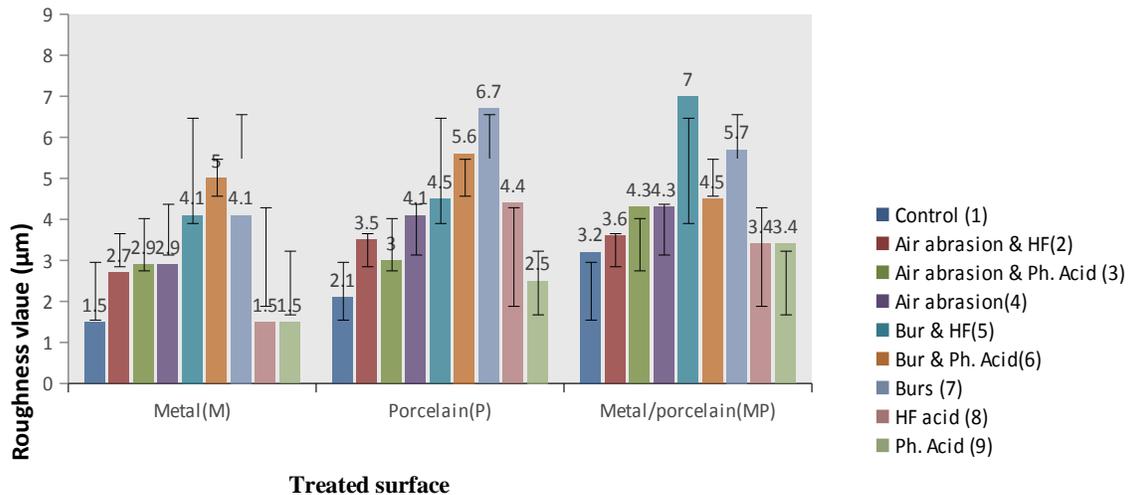


Figure. 6. Roughness mean values (μm) for subgroup and exposed surface ($\pm\text{SD}$)

Statistical analysis

Data obtained from the roughness testing were statistically analyzed using SPSS 16 (2007) and was found to be parametrically distributed. The One Way ANOVA test was used to compare the effects of the nine subgroups across the main groups and within-subject. Chi-Square test was also used to compare results within-subject contrasts at 95 % level of confidence. A significant difference between values was considered when $p < 0.001$ due to multiple testing.

RESULTS

The mean roughness values (Ra) and the standard deviations of twenty seven subgroups are given in Figure 6. The results of the roughness testing were given in μm . The roughness values of all treatments within all subgroups ranged from 1.5 ± 0.14 to 7.0 ± 0.6 . The results also showed some significant differences in the effect of the nine treatments across the three groups.

Metal-porcelain combined surfaces (MP) treated with bur-hydrofluoric acid (MP5) had the greatest roughness value ($7.0 \mu\text{m}$) compared to the overall treatment sub-

groups tested in this study. The specimens of the control treatment of all surfaces (MP1, P1 and M1) had the lowest roughness values (3.2 μm , 2.1 μm , 1.5 μm respectively).

The mechanical treatment with bur alone was the most effective technique for roughening porcelain surfaces (P). The most effective surface treatment for roughening metal-porcelain combined surfaces (MP) and metal surfaces (M) was the bur-chemical combined treatment; bur-hydrofluoric acid and bur-phosphoric acid respectively.

The comparisons of the nine treatments with the control subgroups in each group are given in Table 2. It shows that the control treatment sub-groups of all surfaces (MP1, P1 and M1) had the lowest roughness values (3.2 μm , 2.1 μm , 1.5 μm respectively) compared to the overall treatment sub-groups tested in this study. It also shows that the comparisons between the roughness values of the nine treatments are affected by the material type.

Table 2. Comparison of the roughness values of the nine surface treatments against the control sub-group.

Source	Treatment	Degrees of freedom (Df)	F	p-value
Treatment	2 vs. 1	1	227.640	< 0.001
	3 vs. 1	1	179.031	< 0.001
	4 vs. 1	1	239.939	< 0.001
	5 vs. 1	1	1545.890	< 0.001
	6 vs. 1	1	520.208	< 0.001
	7 vs. 1	1	989.120	< 0.001
	8 vs. 1	1	237.832	< 0.001
	9 vs. 1	1	15.169	0.001
	Treatment Group	2 vs. 1	2	18.592
3 vs. 1		2	3.049	0.064
4 vs. 1		2	7.610	0.002
5 vs. 1		2	34.564	< 0.001
6 vs. 1		2	36.706	< 0.001
7 vs. 1		2	43.044	< 0.001
8 vs. 1		2	181.785	< 0.001
9 vs. 1		2	6.725	0.004

For the (MP) group, the roughness values of bur-hydrofluoric acid treatment (MP5) and bur treatment (MP7) were the greatest (7.0 μm and 5.7 μm respectively). (MP8) and (MP9) had the lowest roughness

values (3.4 μm). The roughness of (MP2), (MP3), (MP4) and (MP6) was intermediate in value (3.6 μm , 4.2 μm , 4.2 μm and 4.5 μm respectively).

There were some significant differences between the roughness values of the (MP) treated surfaces. The roughness value of bur-hydrofluoric acid treatment (MP5) was significantly greater than those of the other MP treatments. (MP7) had a significantly lower roughness value than (MP5) but a significantly greater roughness value than the other MP treatments.

The roughness value of (MP6) was significantly different from those of the other metal/porcelain treatments except those of (MP4) and (MP3).

(MP8) and (MP9) had significantly lower roughness values than the other metal/porcelain surface treatments except (MP2). (MP9) had significantly lower roughness values than the other porcelain treated surfaces but significantly greater than the porcelain control specimens (P1).

The difference between the roughness values of (MP9) and (MP8) and that of the metal/porcelain control specimens (MP1) was not significant.

For the porcelain surfaces (P), bur treatment (P7) and bur-phosphoric treatment (P6) had the greatest roughness values (6.7 μm and 5.6 μm). Porcelain surfaces itched with the phosphoric acid (P9) and those sandblasted with phosphoric acid (P3) had the lowest roughness values (2.5 μm and 3.0 μm respectively). The roughness of (P2), (P4), and (P5) and (P8) were intermediate in value (3.5 μm , 4.1 μm , 4.5 μm and 4.4 μm respectively).

There were some significant differences between the roughness values of the porcelain treated surfaces (P). Porcelain surface treated with bur treatment (P7) had a significantly greater roughness value than all the other porcelain surface treatments. (P6) had a significantly lower roughness value

than (P7) but a significantly greater than the other metal/porcelain treatments ($p < 0.001$). The differences in roughness values between the sandblasted porcelain surfaces (P4), hydrofluoric itched surfaces (P8) and bur-hydrofluoric treated surfaces (P5) were not significant. (P4) had a significantly greater roughness value than (P2) and (P3). (P3) and (P9) had significantly lower roughness values than the other surface treatments. The difference between their roughness values was significant. The roughness value of (P3) was significantly greater than that of (P9). (P9) showed significantly greater roughness value than the porcelain control specimens (P1).

For the metal alloy surfaces (M), bur-phosphoric acid treatment (M6), bur-hydrofluoric acid (M5) and bur treatment (M7) had the greatest roughness value (5.0 μm , 4.1 μm and 4.1 μm). Metal alloy surfaces treated with hydrofluoric acid (M8) and phosphoric acid (M9) had the lowest roughness values (1.5 μm). The sandblasted metal alloy surfaces (M4), sandblasted with phosphoric acid (M3) and sandblasted with hydrofluoric (M2) had intermediate roughness values (2.9 μm , 2.9 μm and 2.7 μm respectively).

There were some significant differences between the roughness values of the metal alloy treated surfaces (M). There was a significant difference in roughness values between the metal alloy surfaces treated with bur-phosphoric acid (M6) and those of the metal alloy surfaces treated with bur-hydrofluoric acid (M5) and bur alone (M7). The difference in roughness value between (M5) and (M7) was not significant. The difference in roughness value between the specimens of (M5), (M6) and (M7) and those of the other metal alloy surface treatments was significant. The difference in roughness value between the metal alloy surface treated with sandblast-hydrofluoric (M2), sandblast-phosphoric (M3) and

sandblast alone (M4) was not significant. The difference in roughness value between (M2), (M3) and (M4) and those of the other metal surface treatments was significant. The roughness values of the metal surfaces treated with hydrofluoric acid (M8) was not significantly different from those treated with phosphoric acid (M9). The roughness values of (M8) and (M9) were significantly different from all those of the other metal surface treatments except the control subgroup of porcelain surfaces (M1).

DISCUSSION

This *in vitro* study has demonstrated that there were significant differences between the effects of different surface treatments on different exposed surfaces of the MC restorations.

Intra-oral repair of fractured metal-ceramic (MC) restorations with composite resin is a practically advantageous alternative to the indirect repair method. The application of composite to a fractured (MC) restoration in the clinic can be cheap, straightforward and time saving. However, the weak bond of resin to porcelain and/or metal is one disadvantage of the use of direct intraoral repair with composite. Therefore, such bond strength is of fundamental importance for long-term serviceability of composite resin bonded to metal-ceramic restorations. It is well-documented that roughening of the fractured surface of the metaloceramic restoration is one of the important factors that contribute to an effective and durable bonding between resin composite and silica-based ceramics. [13,14,16-19] This is because the increased roughness of ceramic surface improves the mechanical interaction of the luting cement to the ceramic surfaces and also increases the total surface energy of the ceramic surface, thus, its wettability. [4,20]

Similarly, the roughening of metal alloy surface can also contribute to the bond

strength of composites to metal substrate. Several studies found that air roughening of a metal alloy surface resulted in a significant increase in bond strength of composite-to-base alloy. [21,4]

Previous studies have investigated the effect of different ceramic surface treatments. A small number of roughening techniques were used and a limited number of combined treatments were compared. [16,22,23] This *in vitro* study has included a larger number of surface treatments and compared many surface-roughening combined methods.

The present study demonstrated that while mechanical treatment with bur alone was the most effective technique for roughening porcelain surfaces, the most effective surface treatment for roughening metal-porcelain combined surfaces (MP) and metal surfaces (M) was the bur-chemical combined treatment. Bur-HF acid (MP5) was found to be the most effective treatment for roughening the metal/porcelain surfaces (MP) whereas bur-phosphoric acid treatment was the most effective method for roughening the metal surface.

The findings of this study have also suggested that the roughening of MC surfaces using chemical treatments alone was ineffective compared to the mechanical roughening techniques alone or mechanical-chemical combined treatments. However, the only exceptional treatment was HF acid on porcelain surfaces. In common with the findings of previous studies, the findings of this study indicated that HF acid itching of porcelain surface was effective in producing irregular, retentive, porous structures on ceramic surfaces. [23,24] This can facilitate better micromechanical interaction between composite and the roughened surface of fractured MC restoration, increasing the bond strength of composite resin.

Treatment with bur alone was the most effective technique for roughening

porcelain surfaces compared to the other surface treatments. The roughness values of porcelain surfaces ranged between 6.7 μm and 2.5 μm . Bur treatment had the greatest roughening value of (6.7 μm) and phosphoric acid had the lowest value of (2.5 μm).

The findings of this study indicated that the porcelain surfaces treated with HF acid (P8) had significantly greater roughness value than those after itching with phosphoric acid (P9). This may indicate that itching ceramic substrate with HF can produce a greater bond strength value than that of phosphoric acid. This is in agreement with the findings of previous studies who demonstrated that roughening ceramic surfaces with HF acid produced a greater shear bond strength value compared with phosphoric acid etching. [22] The findings of this study suggested that the roughness value of the porcelain surfaces after HF acid itching was greater than that of sandblasted porcelain surfaces. Unlike the findings of another study, [4] the findings of this study suggested that the difference in roughness value between the porcelain surfaces itched with HF and those sandblasted was not significant.

The results of this study also showed that the porcelain surfaces treated with mechanical treatments alone (sandblast (P4) or bur (P7)) had significantly greater roughness values than when those mechanical techniques were combined with chemical treatments. This means that chemical treatments may have a negative effect in roughening porcelain surfaces

Generally, the nine roughening methods had a significantly different roughness value among the three main surface groups. Grinding the three fractured surfaces with bur alone, bur-HF acid or bur-Ph acid had significantly greater roughness values than the other materials.

However, the findings of a few previous studies claimed that the roughening of MC surfaces can have no significant difference in the bond strength of the composite-to-base alloy. It was reported that roughening of the bonding surface may decrease the bond strength between the composite and the fractured surface of metaloceramic restorations. Other studies' findings also demonstrated that there were no significant differences in bond strength between diamond roughening, air abrasion, and hydrofluoric acid treatment.^[4,21] Moreover, the findings of other studies suggested that the roughness value of the treated porcelain surfaces is not dependent solely on the techniques used but also on other factors. These include application time of these techniques and the nature of the produced roughness.^[24] Bond strength values between composite resin and the roughened MC surfaces can also be related to post-roughening conditions rather than roughness itself. These include storage conditions, cleaning, heat treatments, ceramic composition and silane treatment.^[3,8,25,18,19]

CONCLUSIONS

Within the limitations of this *in vitro* study it can be concluded that: There were significant differences in the effects of eight surface treatments on the same exposed surface of MC restoration. There were also significant differences in the effects of the each treatment among the three exposed surfaces of MC restoration. Burs treatment is the technique of choice for roughening porcelain exposed surfaces, bur-phosphoric acid treatment is the technique of choice for roughening metal exposed surfaces (M) and bur- hydrofluoric treatment is the technique of choice for roughening metal-porcelain combined surfaces (MP).

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