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Research Article

Bond Strength of Composite Resins used in the Repair of all Ceramic and Metal Ceramic Crowns - An *In vitro* Study

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Abstract

Purpose of the study: In vivo repair of fractured ceramic restorations with composite resin is a viable alternative to total replacement of the restoration and there is a need to find out the bond strength between the ceramic and composite resin to obtain a clinically acceptable restoration.

Objectives: To find out and compare

- The bond strength obtained with different types of ceramics viz. conventional feldspathic ceramic, heat pressed ceramic and ceramic used in CAD-CAM technology.
- The effectiveness of two etchants viz. acidulated phosphate fluoride (APF) gel (1.23%) and hydrofluoric acid (HF) (9.6%) in providing adequate bond strength of composite resins to fractured ceramic restorations.
- The role of two types of micro hybrid composites viz. Filtek and Fulfil on the bond strength with different ceramics.

Materials and Methods: Twelve Nickel chromium alloy discs of size 12mm x 1.5mm were casted and feldspathic ceramic was built up on them. Twelve discs were made in pressable ceramic and twelve discs were milled using CAD/CAM technology. All the specimens were embedded in acrylic resin blocks. Half the specimens were treated with HF (9.6%) and the remaining half was treated with APF gel (1.23%). The specimens were further treated with silane coupling agent followed by bonding agent and composite resin cylinders with 3mm diameter and 2mm height were built up on them. They were subjected to shear bond strength testing with Universal testing machine following which the specimens were viewed under a scanning electron microscope to ascertain the mode of failure.

Results: The results proved that IPS Empress pressable ceramics provided a higher shear bond strength (SBS) when compared to CAD-CAM ceramics. Metal Ceramics yielded the lowest SBS. Ceramics can be considered as a significant factor influencing the SBS. 9.6% HF etchant was found to give a higher mean SBS when compared to 1.23% APF Gel. Etchant also can be considered as a significant factor that influence the SBS. Even though Filtek Resin recorded a higher mean SBS when compared to Fulfil Universal, the difference between them was not statistically significant. Resins cannot be considered as a significant factor that has an influence on SBS. Higher SBS was found in the combination of CAD-CAM ceramic and etchant 9.6% HF. Either of the resins can be used as there is no significant difference between them. The specimens where HF was used as the etchant, the failure was cohesive in nature irrespective of the resins used. With APF, majority of failures were adhesive in nature.

Conclusions: The type of ceramic and the etchants are significant factors in providing adequate bond strength between the composite resins and fractured ceramic restorations. The two resins provided almost similar bonding characteristics.

Keywords: Dental Ceramics; Ceramic Fracture; Ceramic Repair; Composite Resin; Shear Bond Strength

Introduction

21st century witnessed an overwhelming acceptance of the ceramic based prosthodontic treatment because of its aesthetic superiority and desirable mechanical properties, irrespective of the fact that a paradigm shift has occurred in the treatment policy –from tooth supported to implant supported. Popularity does not avoid the potential hazard of failure which is reported to be between 3 to 8%. and that has happened with the ceramic restorations in increasing proportions [1-4]. Fracture of ceramic dental materials can occur because of the low tensile strength, improper design of framework, poor support, intra-ceramic micro defects caused by mismatch in thermal expansion and multi directional forces generated by repetitive dynamic occlusal contacts and by parafunctional occlusion [5,6].

In the initial phases, fractures occurring in ceramic based fixed dental prostheses were never attempted to be repaired *in situ*. Instead, ceramic restorations were always refabricated in the event of a fracture, and it was considered as an ideal treatment option [7]. Repair with resins were always considered as a plausible solution. But it was put into practice only in the recent past with the improvement in the adhesive properties of the resin. Resin will make a bond to the ceramic only if the ceramic is subjected to surface treatment using an etchant. Hydrofluoric acid (9.6%) and Acidulated phosphate fluoride gel (1.23%) were the commonly used etchants. Etchants selectively dissolve either the leucite or the glassy phase and it is dependent on the concentration of the etchant [8]. Etching creates pores which would eventually increase the surface area. Application of silane to the etched porcelain surface promotes adhesion and the bond strength. These are chemically organo- silane coupling agents which are bifunctional with one end of the molecule capable of reacting with an inorganic surface such as porcelain and the other with an organic surface such as a composite resin [9].

Comparisons of etchants, resins and ceramics are available in the documented literature [7,10-12]. However, the recent class of machinable ceramics was not included in the studies which evaluate the efficacy of repairing with resins. Hence it was decided to compare the different materials used in the three-component system viz ceramics, etchants and the resins. The three ceramic systems tested were metal ceramic, pressable all ceramic and CAD/CAM ceramic. Two etchants viz Hydrofluoric acid (HF) or Acidulated phosphate fluoride gel (APF) were tried with all the three ceramics. Two composites viz Filtek and Fulfil which could be used for the repair of fractured ceramic restorations were included in the present study.

The objectives of the study were
To find out and compare

- The bond strength obtained with different types of ceramics viz conventional feldspathic ceramic, heat pressed ceramic and ceramic used in CAD-CAM technology.
- The effectiveness of two etchants viz acidulated phosphate fluoride gel (1.23%) and hydrofluoric acid (9.6%) in providing adequate bond strength of composite resins to fractured ceramic restorations.
- The role of two types of micro hybrid composites viz Filtek and Fulfil on the bond strength with different ceramics.

Methodology

The present study was conducted to compare the shear bond strength of composite resin which was bonded to the ceramic.

Fabrication of specimens

Thirty-six-disc shaped specimens of size 12mm diameter and 1.5mm thickness were made. 12 were ceramo-metal specimens, 12 made of pressable ceramics and 12 ceramic specimens fabricated through CAD/CAM process.

Fabrication of ceramo metal specimens using feldspathic porcelain

Twelve discs were casted in Nickel Chromium alloy (Command) through induction casting process. The discs were grit blasted and finished but the sprues were retained to be used as a retentive element in the next stages of the experiment. On the surface of the disc feldspathic porcelain was layered adhering standard processes of adding opaque layer, dentine porcelain and final glaze. The thickness of porcelain was limited to 1.5mm. (Figure 1,2).

Figure 1: Nickel chromium alloy discs.

Figure 2: Finished ceramo metal discs.

Fabrication of specimens in heat pressed leucite IPS Empress ceramic (Figure 7).

Twelve specimens were made in heat pressed ceramic (IPS emax press – Viva dent). Moulds were prepared using lost wax process. Pressing was done in IPS Empress hot press furnace (EP 600 Ivoclar Viva dent). Finishing and polishing was done with green stone and white wheel. Glazing was done with Universal glazing paste. (Figure 3-5).

Preparation of specimens using CAD-CAM technology

Twelve specimens were fabricated using the CAD-CAM technology.

Figure 3: IPS Empress hot press furnace.

Figure 4: eMax press material.

Figure 5: eMax heat pressed discs.

Disc shaped steel die was scanned under InEos digital scanner. Using Cerec 3D software, three-dimensional virtual image of the specimen was made. Sirona InLab CAD/CAM unit was used for milling and the specimens were made in IPS Empress CAD blocks. Wooden sticks with 2 mm diameter and 10mm height were attached to the specimens using cyanoacrylate adhesive to be used for retentive purpose. (Figure 6-9).

Figure 6: IPS Empress CAD block used for milling.

Completed specimens were embedded in acrylic blocks measuring 1.5 x 1.5 x 1.5 cm. A metal mould was used for this purpose (Figure 10). Distribution of specimens are given in table 1.

Application of etchant

Groups C1, E1, F1 were etched using acidulated phosphate gel -1.23% (Flurovil topical fluoride gel) for 7 minutes and groups C2,

Figure 7: Digital scanner used for scanning metal die.

Figure 8: Sirona In Lab CAD-CAM unit.

Figure 9: Milled Ceramic discs

Figure 10: Disc specimens embedded in Acrylic blocks.

Group	Material	Treatment (Etching)	Number
$C1_f$	CAD - CAM (Filtek Z 250)	APF gel (1.23%)	6
$C1_u$	CAD - CAM (Ultrafil)	APF gel (1.23%)	6
$C2_f$	CAD - CAM (Filtek Z 250)	Hydrofluoric Acid (9.6%)	6
$C2_u$	CAD - CAM (Ultrafil)	Hydrofluoric Acid (9.6%)	6
$E1_f$	IPS Empress (Filtek Z 250)	APF gel (1.23%)	6
$E1_u$	IPS Empress (Ultrafil)	APF gel (1.23%)	6
$E2_f$	IPS Empress (Filtek Z250)	Hydrofluoric acid (9.6%)	6
$E2_u$	IPS Empress (Ultrafil)	Hydrofluoric acid (9.6%)	6
$F1_f$	Feldspathic porcelain (Filtek Z 250)	APF gel (1.23%)	6
$F1_u$	Feldspathic porcelain (Ultrafil)	APF gel (1.23%)	6
$F2_f$	Feldspathic Porcelain (Filtek Z 250)	Hydrofluoric acid (9.6%)	6
$F2_u$	Feldspathic Porcelain (Ultrafil)	Hydrofluoric acid (9.6%)	6

Table 1: Grouping of specimens and different treatments.

E2, F2 were etched using hydrofluoric acid-9.6% (Pulpdent porcelain etch gel) for 4 minutes. The etchants were applied where composite resin cylinders were attached. After etching the specimens were rinsed in running water for 20 seconds and air dried for 30 seconds.

Application of silane coupling agent

Silane coupling agent (3M ESPE) was applied on the etched surface of each specimen for 1 minute using a fresh microtip applicator after which it was air dried for 5 seconds.

Application of bonding agent

Bonding agent was applied to the etched and silanated specimens using a microtip applicator and was light polymerized with visible light of 400-500nm wavelength for 10 seconds.

Preparation of composite resin cylinders

Composite resin cylinders were prepared using a mould fabricated with a teflon sheet of 2 mm thickness having dimensions of 1.5 x 1.5 cm. Two holes were drilled in the teflon sheet corresponding to the composite cylinders to be fabricated on the surface of the ceramic specimen. The cylinders were 3 mm in diameter and 2 mm in height. The distance between the two composite cylinders was 3 mm. The mould was positioned on the acrylic resin block containing the ceramic specimen. The two composite resins - Filtek Z 250 and Ultrafil Universal were placed in the corresponding holes of the mould and light polymerized with visible light at a wavelength of 400-500 nm for 40 seconds. The distance between the light tip and the specimen was fixed as 1mm. The composite resin was light polymerized again for 40 seconds after which the teflon mould was removed. (Figure 11-14).

Figure 11: Teflon mould placed on the specimen to make composite resin cylinders.

Figure 12: Composite resin cylinders fixed on the specimen.

Figure 13: Test specimen - side view.

Figure 14: Schematic diagram of the specimen.

Testing

The shear bond strength of composite resin to ceramic specimens was tested using a single bladed universal testing machine at a crosshead speed of 0.2mm/min. The load at failure was recorded in Newtons and converted to shear bond strength in MPa.

Force(F) per area(πr^2): $F/\pi r^2$, r = radius of the composite resin cylinder in metres. (Figure 15-16).

Figure 15: Shear bond strength tested in Instron.

Figure 16: Close up view of the shear testing.

After shear bond strength testing all the specimens were gold sputtered for 30 minutes after which they were viewed under a scanning electron microscope to find out the effect of etchants and the mode of failure (Figure 17-19). Summary of methodology is given in (Figure 20).

Statistical analysis

The results obtained were subjected to a Factorial -ANOVA test to detect statistically significant differences among the groups.

Results

Three factors influenced the shear bond strength viz. ceramic, etchant and resin. Ceramic was of three types - Feldspathic porcelain of metal ceramic, IPS Empress (pressable ceramic) and CAD-

Figure 17: Specimens after shear testing.

Figure 18: Spcimens after gold sputtering.

Figure 19: Scanning electron microscope.

Figure 20: Flow chart on methodology.

CAM (CAD/CAM milled ceramic). Etchant was of two types - 1.23% APF Gel and 9.6% HF. Resin was of two types – Filtek resin and Fulfil Universal.

The factors and their levels are given table 2.

Factor	Levels
Ceramic	CAD-CAM (IPS Empress CAD), IPS Empress (Emax press), Metal Ceramic
Etchant	1.23% APF Gel, 9.6% HF
Resin	Filtek Resin, Fulfil Universal

Table 2: Factors tested in this study.

The short forms used in the description are.

- SBS: Shear bond strength
- CAD/CAM: Computer aided designing/Computer aided machining.
- APF gel: Acidulated phosphate gel
- HF: Hydrofluoric acid

Test Procedure

Null Hypotheses

- **$H_{0(a)}$:** There was no significant difference between the different types of ceramics.
- **$H_{0(b)}$:** There was no significant difference between the different etchants.
- **$H_{0(c)}$:** There was no significant difference between the different resin materials.
- **$H_{0(d)}$:** The interaction (joint effect) of various factors was not significant.

Alternate hypotheses

- **$H_{1(a)}$:** There was a significant difference between the different types of ceramics.
- **$H_{1(b)}$:** There was a significant difference between the different etchants.
- **$H_{1(c)}$:** There was a significant difference between the different resin materials.
- **$H_{1(d)}$:** The interaction (joint effect) of various factors was significant.

Level of significance: $\alpha = 0.05$.

- **Decision Criterion:** p-values were compared with the level of significance. If $P < 0.05$, the null hypothesis was rejected and accepted the alternate hypothesis. If $P > 0.05$, the null hypothesis was accepted. If there was a significant difference, multiple comparisons were carried out (post hoc-test) using Bonferroni method to find out among which pair or groups there existed a significant difference.

- **Statistical technique used:** Factorial ANOVA

- **Computations:** Various computations and P-values are presented in the tables.

The difference in mean SBS between the different ceramics was found to be statistically significant ($P < 0.001$). Ceramic was found to be a significant factor influencing SBS. The difference in mean SBS between the different etchants was also found to be statistically significant ($P < 0.001$). Etchant was found to be a significant factor influencing SBS. No statistically significant difference was observed between the different resins ($P > 0.05$) with respect to mean SBS. Resin was not a significant factor influencing SBS. The interaction (joint effect) of Ceramic and Etchant on SBS (in MPa) was not statistically significant ($P > 0.05$). The interaction (joint effect) of Ceramic and Resin on SBS (in MPa) was also not statistically significant ($P > 0.05$). The interaction (joint effect) of Etchant and Resin on SBS (in MPa) was also not statistically significant ($P > 0.05$). The interaction of three factors viz. Ceramic, Etchant and Resin on SBS (in MPa) was also not statistically significant ($P > 0.05$). (Table 3,4). There was a significant difference between Metal Ceramic (Feldspathic) and IPS Empress ($P < 0.001$) as well as between Metal Ceramic and CAD-CAM ($P < 0.01$) with respect to the mean SBS (in MPa). However, no statistically significant difference was observed between IPS Empress and CAD-CAM Ceramic ($P > 0.05$) with respect to the mean SBS (in MPa). (Table 5).

In short, the results prove that IPS Empress pressable ceramics provide a higher SBS when compared to CAD-CAM ceramics. Metal Ceramics yielded the lowest SBS. Ceramics can be considered as a significant factor influencing the SBS. 9.6% HF etchant was found to give a higher mean SBS when compared to 1.23% APF Gel. Etchant also can be considered as a significant factor that influence the SBS. Even though Filtek Resin recorded a higher mean SBS when compared to Fulfil Universal, the difference between them was not statistically significant. However, resins cannot be considered as a significant factor that has an influence on SBS (Figure 21). It was found that higher SBS was found in the combination of CAD-CAM ceramic when etched with 9.6% HF. Either of the resins can be used as there is no significant difference between them (Figure 22).

The mode of failure after the shear bond testing was evaluated through scanning electron microscopy. The specimens where HF was used as the etchant, the failure was cohesive in nature irrespective of the resins used. With APF, majority of failures were adhesive in nature. (Table 6, Figure 23-27).

Ceramic	Etchant	Resin	Mean	Std dev	Min	Median	Max
Metal Ceramic	1.23% APF Gel	Filtek Resin	11.52	4.91	7.44	10.21	19.79
		Fulfil Universal	16.23	6.61	8.26	16.75	26.99
	9.6% HF	Filtek Resin	22.78	5.70	14.96	23.66	30.42
		Fulfil Universal	19.19	5.30	13.56	17.05	26.77
IPS Empress (Emax press)	1.23% APF Gel	Filtek Resin	25.56	2.39	22.02	25.94	28.14
		Fulfil Universal	20.40	4.68	13.02	22.00	24.74
	9.6% HF	Filtek Resin	29.87	8.89	16.86	33.15	39.77
		Fulfil Universal	24.45	6.41	16.38	22.88	32.22
CAD-CAM (IPS Empress CAD)	1.23% APF Gel	Filtek Resin	20.26	4.52	14.30	19.16	27.54
		Fulfil Universal	14.87	2.83	11.20	14.69	18.57
	9.6% HF	Filtek Resin	29.43	1.82	26.71	30.15	31.20
		Fulfil Universal	29.57	7.32	23.11	25.94	39.18

Table 3: Shear bond strength (SBS) recorded in different factors and their levels are given below (MPa).

Source	df	Sum of squares (SS)	Mean SS	F	P-Value
Ceramic	2	783.46	391.73	13.00	<0.001*
Etchant	1	1079.34	1079.34	35.82	<0.001*
Resin	1	108.22	108.22	3.59	0.063
Ceramic*Etchant	2	183.68	91.84	3.05	0.055
Ceramic*Resin	2	103.00	51.50	1.71	0.190
Etchant*Resin	1	4.60	4.60	0.15	0.698
Ceramic*Etchant*Resin	2	144.77	72.38	2.40	0.099
Error	60	1807.82	30.13	---	---
Total	71	4214.88	---	---	---

Table 4: ANOVA Values.

Dependent variable: Shear Bond Strength (MPa) Bonferroni						
(I) Ceramic	(J) Ceramic	Mean Difference (I-J)	Std. Error	Sig	95% Confidence Interval	
					Lower Bound	Upper Bound
Metal Ceramic	IPS Empress	-7.6375*	1.58457	.000	-11.5402	-3.7348
	CAD-CAM	-6.1029*	1.58457	.001	-10.0056	-2.2002
IPS Empress	Metal Ceramic	7.6375*	1.58457	.000	3.7348	11.5402
	CAD-CAM	1.5346	1.58457	1.000	-2.3681	5.4373
CAD-CAM	Metal Ceramic	6.1029*	1.58457	.001	2.2002	10.0056
	IPS Empress	-1.5346	1.58457	1.000	-5.4373	2.3681

Table 5: Multiple Comparisons (Post-Hoc test) to find significant difference between different Ceramics using Bonferroni method.

Based on observed means.

*. The mean difference is significant at the .05 level.

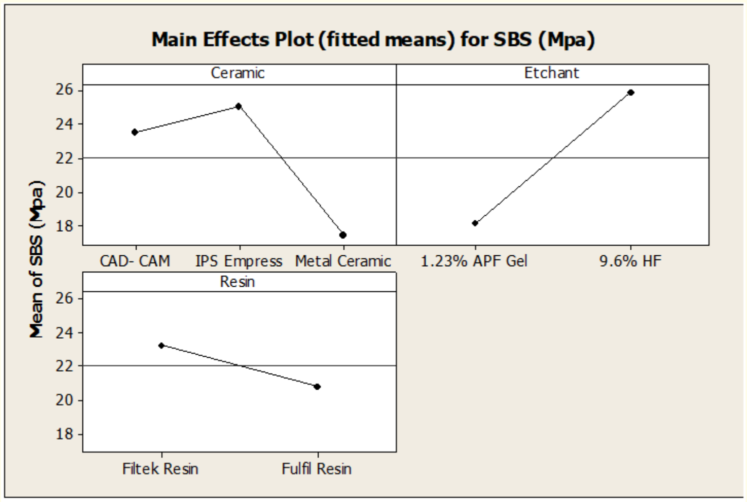

Figure 21: Main effects plot.

Figure 22: Interactions plot.

Etchant	Filtek resin		Fulfil resin	
	Cohesive failure	**Adhesive failure**	**Cohesive failure**	**Adhesive failure**
HF	100%	0%	100%	0%
APF	22.2%	77.8%	38.8%	61.2%

Table 6: Comparison of mode of failure between ceramic and composite resin as seen under scanning electron microscope.

Cohesive failure: Failure is cohesive in nature within ceramic.

Adhesive failure: Failure is adhesive in nature at the ceramic- composite resin Interface.

Figure 23: Untreated ceramic surface.

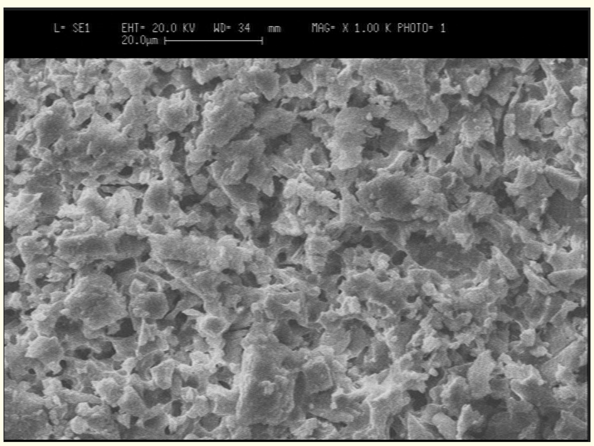

Figure 24: Ceramic etched with HF (9.6%).

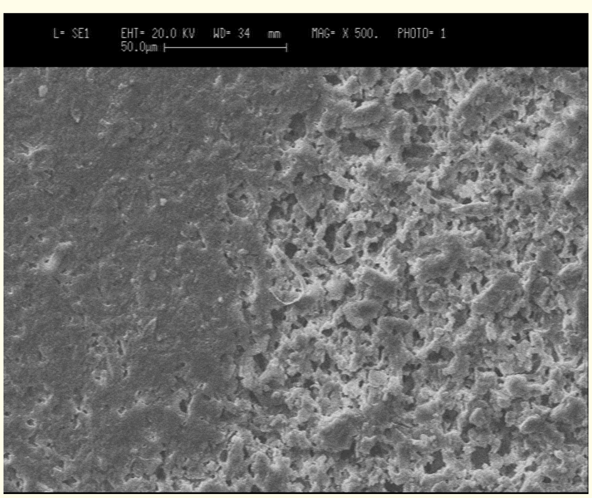

Figure 25: Ceramic etched with APF gel (1.23%).

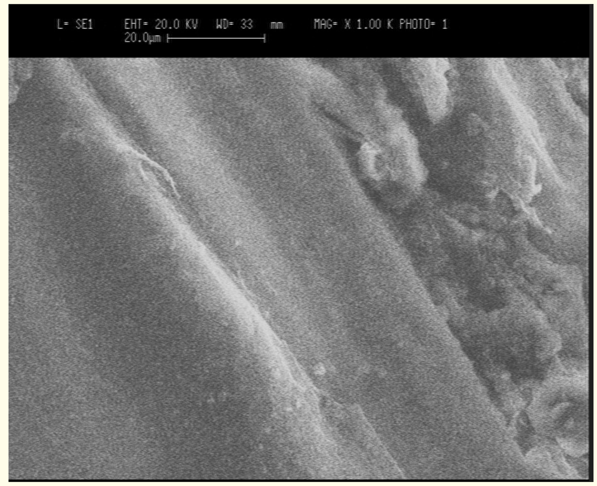

Figure 26: Cohesive failure of ceramic.

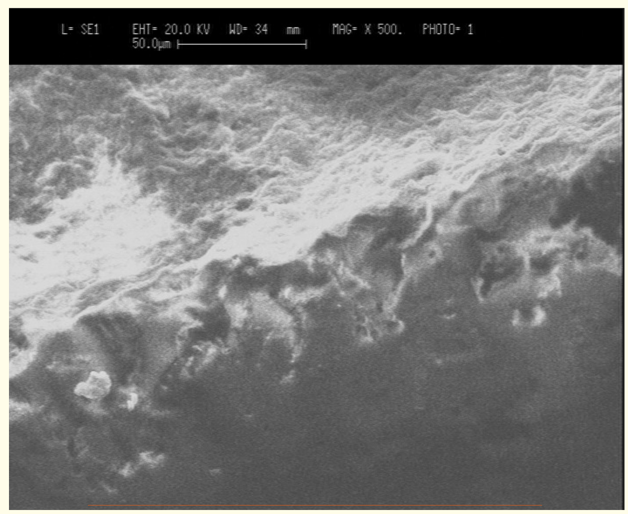

Figure 27: Adhesive failure through the interface of ceramic and composite resin.

Discussion

The three ceramics which were used in the present invitro study when compared, IPS Emax press showed the maximum bond strength of 25.07 MPa, followed by CAD/CAM ceramic (IPS Empress CAD) where the bond strength was 23.53 MPa and the lowest bond strength of 17.43 MPa was seen with the metal ceramic specimens. The difference between metal ceramic and IPS Empress was statistically significant. Similarly metal ceramic and CAD/CAM ceramic was also statistically different whereas the difference between IPS Empress and CAD/CAM ceramic was not statistically significant.

Amongst the two etchants HF, showed a higher bond strength of 25.88MPa whereas with APF gel, 18.14 MPa was obtained and the difference between the two was statistically significant. Filtek resin showed a higher bond strength of 23.24 MPa while Fulfil showed a bond strength of 20.79 MPa and the values were not statistically significant. (Table 3).

Composite resin will not bind mechanically to the fractured porcelain surface unless it is subjected to surface treatment. Etching creates surface irregularities and that fosters mechanical bonding. Fractured porcelain denture teeth were quickly repaired using composite resin by Jochen D.G [14]. It has been observed that minimum bond strength of 17-20 MPa would provide a successful bond between tooth material and composites [13]. Shirani., *et al*. after reviewing seventeen studies have found that the bond strength between ceramic and composite resin have gone only up to 29.7 MPa [15]. In the present study, the two brands of micro hybrid composite resins viz Filtek and Fulfil showed values of 23.24 MPa and 20.79 MPa respectively which fall within the clinically acceptable limits.

When samples were etched with hydrofluoric acid, most of the failures were found to be cohesive in nature and which happened in the ceramic while in samples that were etched with APF gel, most of the failures were adhesive in nature and which happened at ceramic-composite interface. The acid that creates more irregularities on the surface will promote good adhesion of composite resin to the porcelain surface. HF acid etched patterns appear more pronounced and aggressive. Voids and channels appeared larger, deeper and more numerous in samples treated with HF for 4 minutes as compared to samples etched with APF gel. APF etched the porcelain surfaces but created a relatively smooth homogeneous surface which probably might be insufficient to create a micro mechanical bonding when compared to that of HF treated specimens. (Figure 23-27). The findings of Canay., *et al*. [16] and Kukiattrakoon., *et al.* [17] are comparable to the results of the present study in which etching with HF showed greater shear bond strengths as compared to APF gel. The higher efficiency of HF is attributed to the greater surface roughness produced by it when compared to the etching efficiency of APF gel.

In this context, the observations made about the preferential dissolution characteristics by Stangeli., *et al.* [8] become relevant. When the etchant concentration is as high as 52%, the glassy phase would be dissolved preferentially where as a 20% concentration would seem to dissolve the crystalline phase preferentially. The main crystalline component of dental porcelain is leucite which dissolves more rapidly than the surrounding glass on etching with HF and APF gel because lower concentrations are used. Energy dispersive spectroscopy analysis revealed a decreased concentration of Si, Al, Ca and Na in the etched zone. The concentration of leucite in feldspathic porcelain is 15-20% by volume whereas it is 35 - 45% by volume in IPS Emax press which was the ceramic that was used for heat pressed specimens. Since etching dissolves leucite faster than the glass, the increased presence of leucite in IPS Empress would have allowed enhanced etching and thereby increased the surface area. This explains the higher bond strength seen with pressable ceramics.

Application of silane to the etched surface may cause the fluorosilicate salts to dissociate by hydrolysis and the silane gets adsorbed to the ceramic surface. Silane also promotes wetting of the porcelain surface and enhances the flow of the resin thereby improving the bond strength of composite to porcelain by approximately 25% [16,18].

Conclusions

The type of ceramic has a role in determining the bond strength with the composite resin that is used for repairing ceramic fractures. Heat pressed and CAD/CAM ceramics can provide better bond strength than feldspathic porcelain. It was observed that Hydrofluoric acid (HF) provided better etching and better bonding than APF gel. However, both the brands of micro hybrid composite resins used for repairing of ceramic fractures provided bonding without significant difference.

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