

## HYDROFLUORIC ACID ETCHING OF FLEXOCERAM INLAY PORCELAIN: A MICROSCOPIC STUDY

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مقدمة :-

من الصعوبات التي تواجه فن وعلم التركيبات الثابتة هي كيفية تثبيت الوجه الخزفي المناسب للون الأسنان الطبيعية على الهيكل المعدني وقد تطور طب الأسنان لاستحداث كافة الطرق حتى كانت طريقة تخريش سطح المعدن بالحامض ليكون تثبيت الوجه الخزفي متينا ومن أحدث المعادن المستخدمة هذه الأيام هو الفلوكسوسيرام .  
المواد والطريقة المستخدمة في البحث :

ثلاثون قرصا من معدن الفلوكسوسيرام وقد قسموا إلى مجموعتين ( ١٢ بالمجموعة الأولى وقد استخدم لتخريشها ١٪ حامض الهيدروفلوريك ، ١٨ بالمجموعة الثانية استخدم لتخريشها ١٠٪ من الحامض) وتم دراسة السطح المعدني بعد استخدام الحامض في مدد (١٠ ، ٢٠ ، ٢٥ ، ٣٠ ، ٣٥ ، ٤٠ ، ٥٠ ، ٦٠ ثانية للمجموعة الأولى و ٩٠ ، ٣٠ ، ٢ ، ٣ ، ٥ ، ١٠ دقائق).

التسايح :-

بين الشكل ١ ، ٢ تأثير الحامض في كل المدد على المعدن كان التأثير قويا على ملتقى جزئيات الزجاج وكريستالات الليوسيت .  
تأثير التخريش بالحامض بتركيز ١٠٪ كما يظهر في الشكل من ٣-٦ ومن الجدول رقم ٢ ولم يظهر تحسن بعد مدة دقيقتين .

التخديش الخفيف باستخدام ١٪ من الحامض لمدة وجيزة أشارت على أن مهاجمة الحامض للملتقى جزئيات الزجاج وكريستالات الليوسيت ظهر في ٢٥ ثانية من استخدام الحامض (شكل ٢) ويعزو الشقوق الميكروسكوبية بالمعدن الى عدم تناسب الصفات الطبيعية للزجاج مع كريستالات الليوسيت ولذا فإن تركيز ١٠٪ من الحامض و ٩٠ ثانية هما العاملان الرئيسان في معاملة المعدن للحصول على أفضل النتائج وأحسنها .

This study was carried out to determine the hydrofluoric acid (HF) concentration and time most suitable to use with Flexoceram inlay porcelain which is a new product on the market. This study has shown that 10% hydrofluoric acid is effective in etching Flexoceram inlay porcelain and that this concentration produces optimum etch patterns at 90 seconds of acid etching. It has also shown that hydrofluoric acid preferentially attacks the interface between the glass and the leucite crystals of the material.

### Introduction

It has been suggested that an initial rough surface morphology is appropriate for bonding as it tends to increase the physical area available for reaction and adherence.<sup>12</sup> Etching the fitting surface of the veneer to increase the surface area and to create a

micro-mechanical bond to composite resin, is considered fundamental to the strength of the porcelain-resin interface. Susceptibility of porcelain to etching has been shown by a number of workers.<sup>3,4,5</sup>

It is believed that etching the surface of the veneer is paramount to the achievement of high bond strengths. However, the standard procedure to achieve an adequate etch pattern is yet to emerge as there is no agreement on what etchant to use and for how long to etch. The significance of this etch-

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ing to the bond strength achieved may be responsible for the large number of suggestions, some of which are presented in Table 1. Flexoceram is a new product on the market and, as such, this study was carried out to determine the proper HF concentration and time suitable for etching.<sup>6</sup>

**Table 1. Etching agents and time suggested in previous studies.**

Study	Etchant	Time
Hobo & Iwata, 1985 <sup>10</sup>	2N hydrochloric acid	15 min
Sorensenetal, 1991 <sup>11</sup>	Stripit(20%HF)	3 min
Simonsen&Calamia, 1984 <sup>12</sup>	7.5% HF	20 min
Hsuetal, 1985 <sup>13</sup>	Strip it	2.5 min
Horn, 1985 <sup>14</sup>	10% HF	15 min
	Strip it	20 min
Stangeletal, 1987 <sup>2</sup>	20% HF	2.5 min
Lacyetal, 1988 <sup>15</sup>	9.5% HF	4 min
	1.23%APF	10min
Bailey & Bennett, 1988 <sup>1</sup>	10% acid, ammomium bifluorideStripit	1 min
		1 min

HF = Hydrofluoric acid; APF = Acidulated phosphate fluoride.

### Materials and Methods

Flexoceram inlay porcelain powder and liquid\* and hydrofluoric acid\*\* were used in this study. Thirty porcelain discs, measuring 8mm x 4mm x 2mm, were fabricated according to a method described in 1992.<sup>7</sup> Twelve discs were used with 1 % HF and eighteen with the 10% HF.

The phase(s) being etched within the Flexoceram porcelain were studied under the optical and the scanning electron microscopes following short etching times with 1 % hydrofluoric acid for the following periods: 10 seconds, 20 seconds, 25 seconds, 30 seconds, 35 seconds, 40 seconds, 50 seconds and 60 seconds.

The degree of etching within the ceramic, following treatment with 10% HF during long etching times, was also studied to determine a suitable etch time for the ceramic. The samples were etched for 30 seconds, 90 seconds, 2 minutes, 3 minutes, 5 minutes and 10 minutes.

Three specimens for each period of etching were weighed using a microbalance\* before and after etching following ultrasonic cleaning and drying in the oven at 200°C for at least six hours. The loss in weight was calculated from the difference between the two measurements.

All etching was done in the fume hood and the specimens were washed under a running tap of laboratory grade distilled water before being ultrasonically cleaned for at least 30 minutes.

### Results

Scanning electron micrographs (SEM) representing the results of etching Flexoceram discs for the short etch times with 1% hydrofluoric acid are shown in Figures 1 and 2. The figures show that while the acid attacked all the phases in the material, there is a preferential attack at the interface of the leucite crystals with the glassy phase. Etching of the leucite crystals exposed the lenticular plates and the intertwined internal substructure of the leucite crystals. This became most prominent between 25 and 35 seconds etch times. No other phases were evident.

The average loss in weight following etching of the samples with 10% hydrofluoric acid for various lengths of time are summarized in Table 2. The resulting etch patterns following the treatments are also shown in Figures 3-6. These results show that for the 10% concentration of hydrofluoric acid, there was no significant change or improvement in the etch pattern despite increase in loss of the material following etching for more than 2 minutes. The etched surface progressively became uniformly non-porous with distinguishable micro-undercuts that presents a complex honeycomb appearance. The higher magnification in Figure 6 shows that the etched surface was both intricate, complicated and probably delicate.

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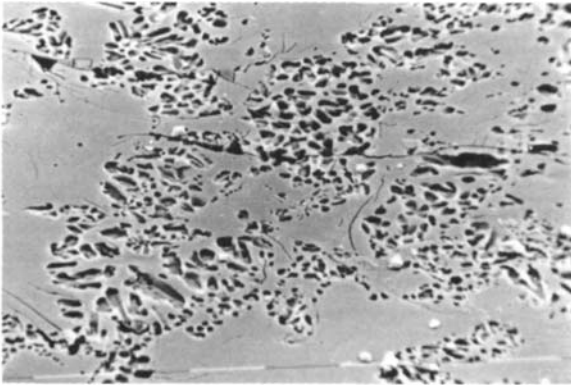


Figure 1. SEM of polished Flexoceram etched with 1% HF acid for 10 seconds. (Magnification X 1250)

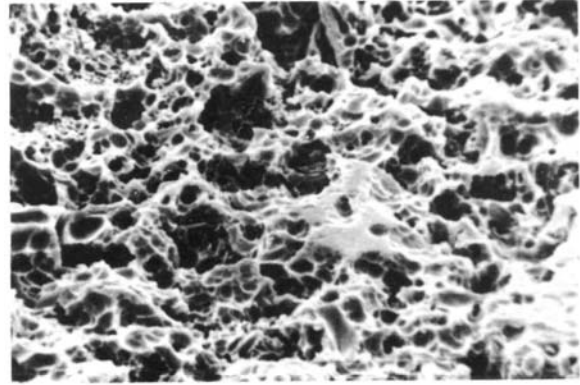


Figure 4. SEM of polished Flexoceram etched with 10% HF acid for 90 seconds. (Magnification X 1250)

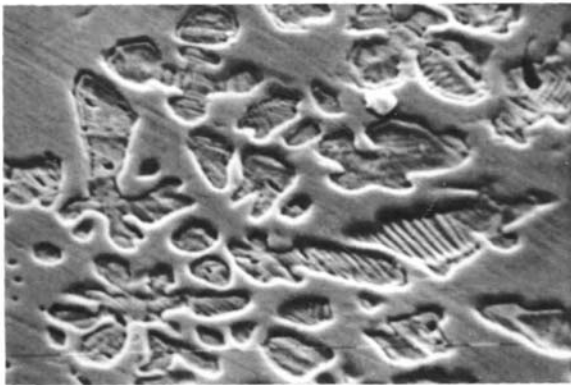


Figure 2. SEM of polished Flexoceram etched with 1% HF acid for 25 seconds. (Magnification X 5000)

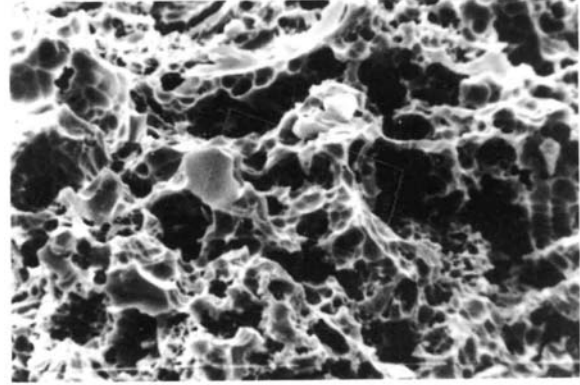


Figure 5. SEM of polished Flexoceram etched with 10% HF acid for 10 minutes. (Magnification X 1250)

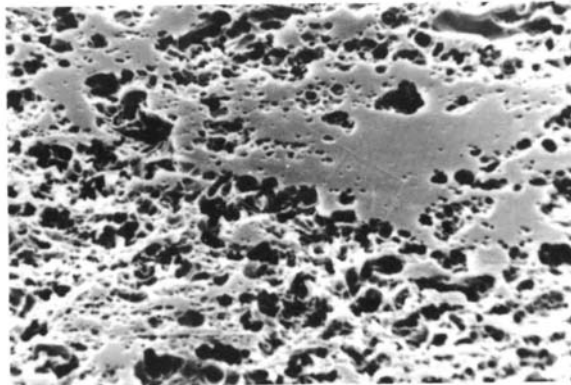


Figure 3. SEM of polished Flexoceram etched with 10% HF acid for 30 seconds. (Magnification X 1250)

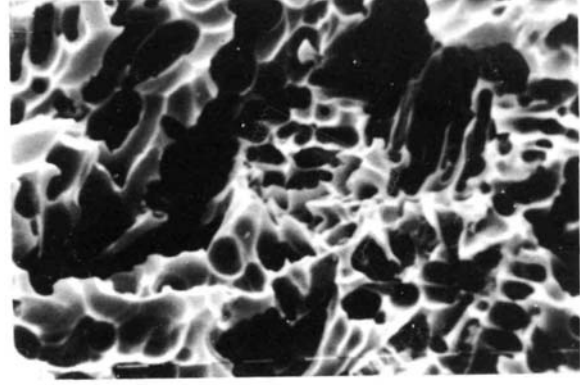


Figure 6. SEM of polished Flexoceram etched with 10% HF acid for 2 minutes. (Magnification X 5000)

Table 2. Average weight loss of Flexoceram following etching with 10% hydrofluoric acid.

Time(min.)	No.	Weight loss (gm.)	% Weight loss
0.5	2	0.00105	0.57
1.5	3	0.00260	1.59
2.0	3	0.00310	1.61
3.0	3	0.00437	2.65
5.0	3	0.00777	4.29
10.0	3	0.01407	8.50

### Discussion

Using 1 % hydrofluoric acid for short periods of time demonstrated the initial stages of acid etching of Flexoceram inlay porcelain and showed that there is a preferential attack at the leucite/glass interface in addition to the dissolution of the glassy phase. This becomes clearly evident at about 25 seconds of etching as shown in Figure 2. This figure also reveals that the leucite crystals are attacked as their internal substructure is exposed showing the lenticular plates and intertwined structure. No other phases were evident in Flexoceram inlay porcelain.

The presence of microcracks in this porcelain is evident in Figures 1 and 2. These cracks also served as areas of penetration for the etching solution. Other studies have shown that the microstructure of Flexoceram inlay porcelain is heterogenous, consisting of leucite crystals in a glassy matrix. Further, the large polygonal crystals of leucite were shown to be surrounded by microcracks and an intertwined dendritic substructure of the leucite crystals.<sup>8</sup> The microcracks are thought to be due to either the incompatibility in the coefficients of thermal expansion of the leucite crystals and the glass and/or due to volumetric changes that occur within the leucite crystals during cooling.<sup>8,9</sup>

Investigation of the loss in weight following etching for various lengths of time with 10% HF was done to determine the ceramic's resistance to dissolution. Calamia<sup>6</sup> suggested that each porcelain should be tested to determine the appropriate etch time and concentration that would produce an acceptable etch pattern. In this study, etching for longer than two minutes did not produce any remarkably different surface pattern despite the increase in weight loss.

The increase in weight loss of the material, Table 2, suggests a decrease in strength of the laminate veneer when etched beyond 2 minutes. Using a Flexoceram inlay porcelain, there was no evidence of reduction in the etching ability of hydrofluoric acid with time as reported in some previous studies.<sup>1,2,5</sup>

The results of bond strength tests from other studies showed that surface treatment of dental porcelain significantly improved the bond between the porcelain and composite resin.<sup>2,7,10,13</sup> The reported improvement in bond strength after etching the porcelain surface may be explained by the micro-mechanical interlocking between the etched porcelain surface and composite resin cement. Hydrofluoric acid dissolves the glassy phase of dental porcelain. It also attacks the interface between the leucite crystals in Flexoceram inlay porcelain preferentially. It is possible that dislodgement of those leucite crystals that are surrounded by microcrack occurs during etching and rinsing the surface. The resultant effect creates micropores and micro-undercuts within the porcelain resembling an intricate honey-comb structure into which the resin can flow to produce a micro-mechanical interlocking and the reported higher bond strengths.

### Conclusion

This study has shown that 10% hydrofluoric acid is effective in etching Flexoceram inlay porcelain. This concentration produces optimum etch patterns at 90 seconds acid etching time. It has also been shown that etching produces a micro-mechanical retentive effect on the porcelain surface that is likely to improve its bond strength to composite resin. This study has also showed that hydrofluoric acid attacks all the phases in Flexoceram porcelain but preferentially invades the interface between the glass and the leucite crystals.

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