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Evaluation of a universal adhesive (Iperbond Ultra)

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The object of this study was to observe the resin-composite/enamel and resin-composite/dentine interfaces achieved with Iperbond Ultra, and to evaluate how well this adhesive system stuck to enamel and dentine by comparison with competing systems.

1) Evaluation of the enamel and dentine interfaces

INTRODUCTION

Iperbond Ultra is a self-etching adhesive system (Self-Etch Mode) requiring no preliminary etching and rinsing stage: practitioners greatly appreciate these systems' simplicity and rapidity in use compared with traditional systems that demand a preliminary etching or "E&R" (Etching and Rinsing) stage.

Nevertheless an application of phosphoric acid, even if not strictly necessary, could strengthen the adhesion of this Self-Etch Mode to dental tissues.

The aim of this first part was to use **a scanning electron microscope (SEM),** at various magnifications, to observe the **resin-composite/enamel** and **resin-composite/dentine** interfaces of composite blocks cemented using Iperbond Ultra, both with and without preliminary etching.

MATERIALS, EQUIPMENT AND METHOD

Preparation of the teeth

Four intact, freshly-extracted **human teeth** were collected, cleaned and kept at 4°C in a 1% chloramine T solution. The roots were eliminated using an irrigation polisher with a coarse (80 grit) abrasive disc. The occlusal faces were polished first with a coarse (80 grit) paper and then with a finer one (800 grits) to obtain a clean, flat surface of enamel (in the case of two of the teeth) or dentine (for the other two). These residual crowns were then covered with cylinders of self-polymerizing acrylic resin (Plexcil®, Escil, Chassieu, France), leaving a flat enamel or dentine surface exposed.

Surface treatment, assembly and conservation

One of the two teeth with a flat surface of **enamel** exposed was **etched** with 37% phosphoric acid for 30 seconds. This tooth was Group 1. It was then carefully rinsed and thoroughly dried. The other of those two teeth (Group 2) **was not treated**.

One of the two teeth with a flat surface of **dentine** exposed was **mordanted** with 37% phosphoric acid for 15 seconds. This tooth was Group 3. It was then carefully rinsed and dried without drying out the dentine. The other of those two teeth (Group 4) **was not treated**.

A Teflon® mould was placed over each sample to create **cylinders of Reflectys composite** 3mm in diameter and 2mm in height, cemented using Iperbond Ultra. These samples were kept in distilled water at 37°C for 24 hours.

Preparation of the samples for observing under the SEM

The samples were sliced in two vertically using a diamond disc cutter under water, the cut being made to pass as close to a diameter of the cylinder as possible. The **interfaces** obtained in this way were polished under water using a metallographic polisher with increasingly fine (800, 1200, 2400 and 4000) SiC abrasive discs, then with one-micron diamond particles on a sheet under lubricant and without solvent, and lastly with a 0.05µm finishing disc under a colloidal suspension of SiO_2. The samples were then cleaned by ultrasound in distilled water for 5 minutes.

They were then progressively dehydrated by dipping in a **series of ethanol** baths of increasing concentration (25% for 15 min, 50% for 15 min, 75% for 15 min, 90% for 15 min and 100% for one hour). Once dried, the samples were put in position and held vertically by a conductive solution on a conductive cylinder and then themselves made conductive by metallization with an approximately 20nm layer of gold deposited using a vacuum metallizer.

They were observed under the SEM at **two magnifications, x800 and x2000**, under a voltage of 15 kV and at a working distance of 9 to 21 microns.

RESULTS

The figures below show the various interfaces obtained with the different modes of assembly and the different dental tissue substrates: Figures 1 and 2 show the interface of the Reflectys/enamel assembly cemented using Iperbond Ultra with preliminary etching; Figures 3 and 4 show the interface of the Reflectys/enamel assembly cemented using Iperbond Ultra without preliminary etching; Figures 5 and 6 show the interface of the Reflectys/dentine assembly cemented using Iperbond Ultra with preliminary etching; lastly, Figures 7 and 8 show the interface of the Reflectys/dentine assembly cemented using Iperbond Ultra without preliminary etching.

Fig. 1: Group 1: Reflectys/enamel interface cemented using Iperbond Ultra with preliminary etching (x800)

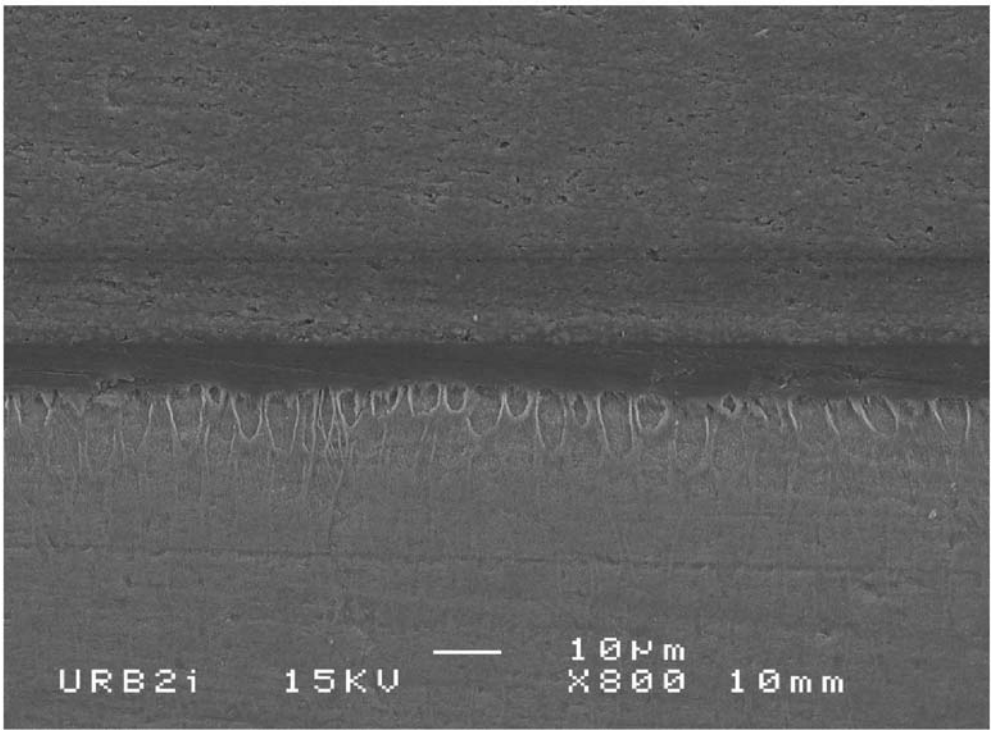

Fig. 2: Group 1: Reflectys/enamel interface cemented using Iperbond Ultra with preliminary etching (x2000)

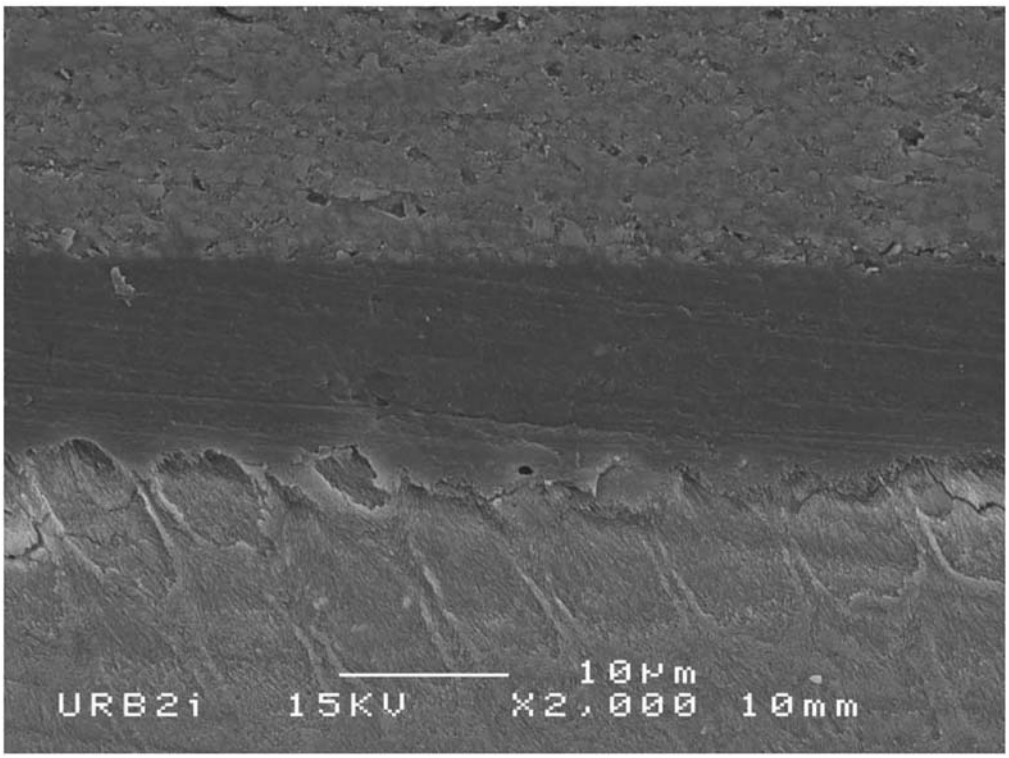

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Fig. 3: Group 2: Reflectys/enamel interface cemented using Iperbond Ultra without preliminary etching (x800)

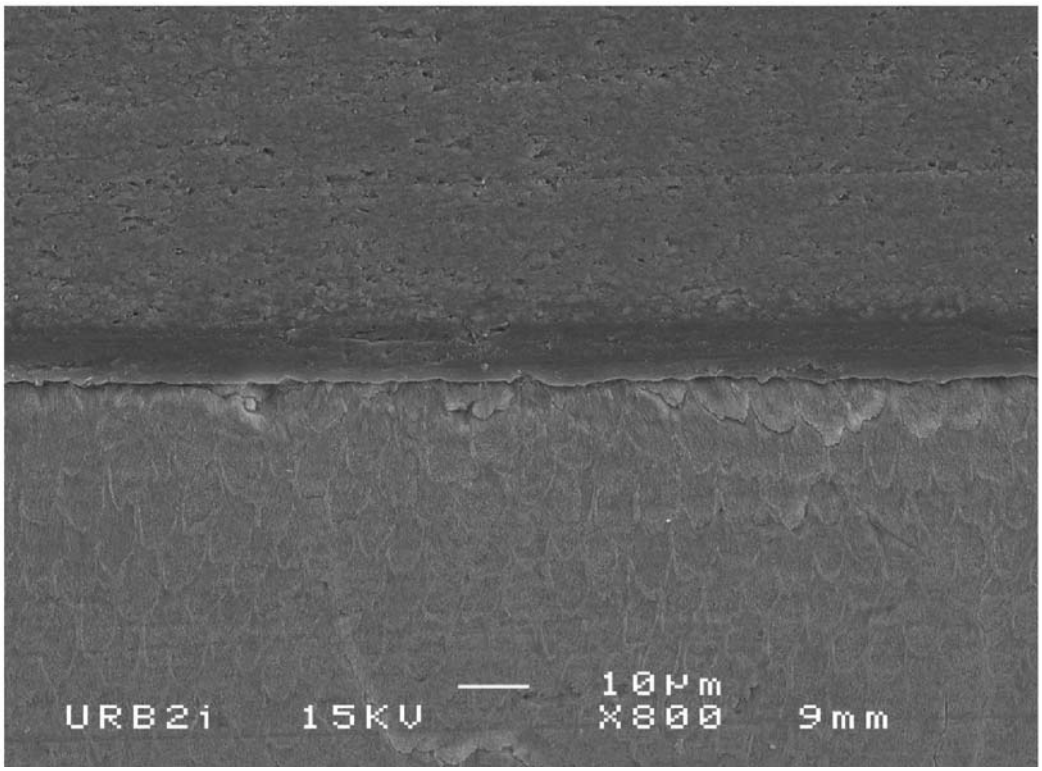


Fig. 4: Group 2: Reflectys/enamel interface cemented using Iperbond Ultra without preliminary etching (x2000)

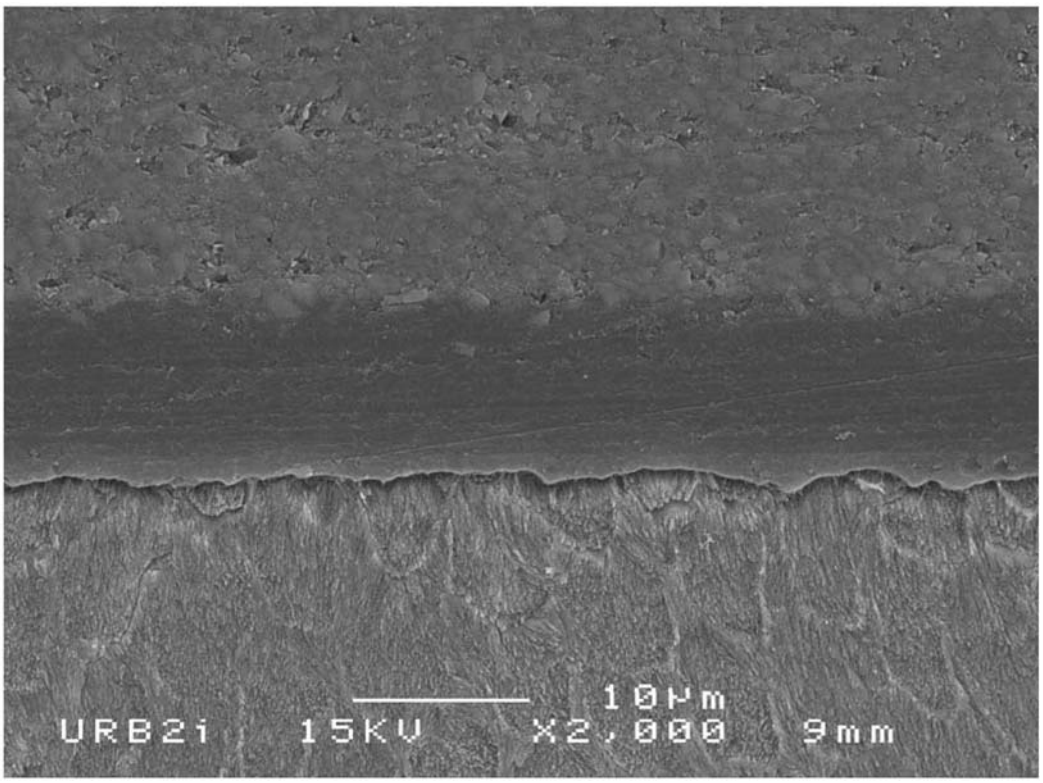


Fig. 5: Group 3: Reflectys/dentine interface cemented using Iperbond Ultra with preliminary etching (x800)

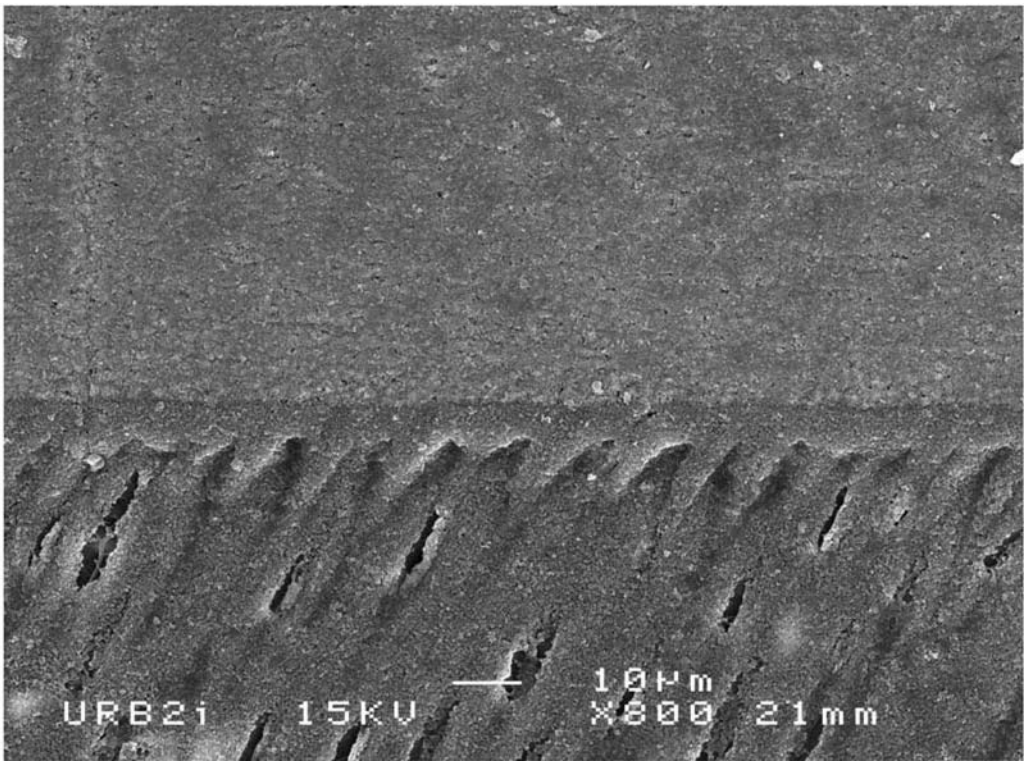
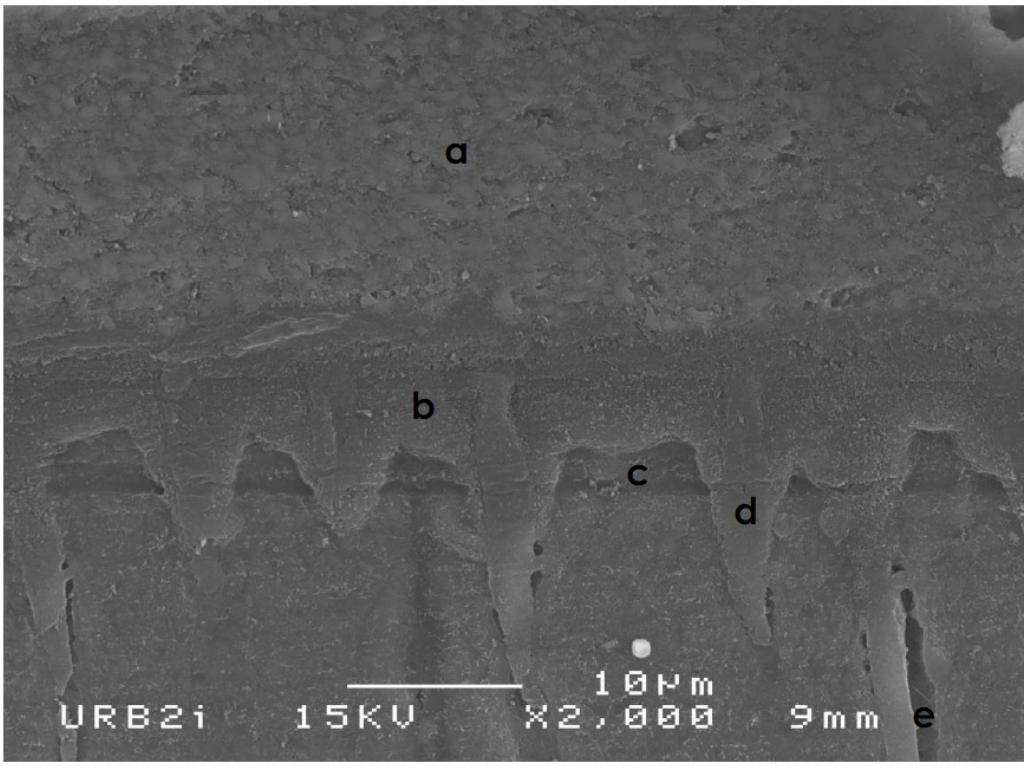


Fig. 6: Group 3: Reflectys/dentine interface cemented using Iperbond Ultra with preliminary etching (x2000)



Key: a: Reflectys composite; b: Iperbond Ultra adhesive; c: hybrid layer (penetration of adhesive into the intertubular dentine); d: resinous cord (penetration of adhesive into the tubule); e: dentinal tubule

Fig. 7: Group 4: Reflectys/dentine interface cemented using Iperbond Ultra without preliminary etching (x800)

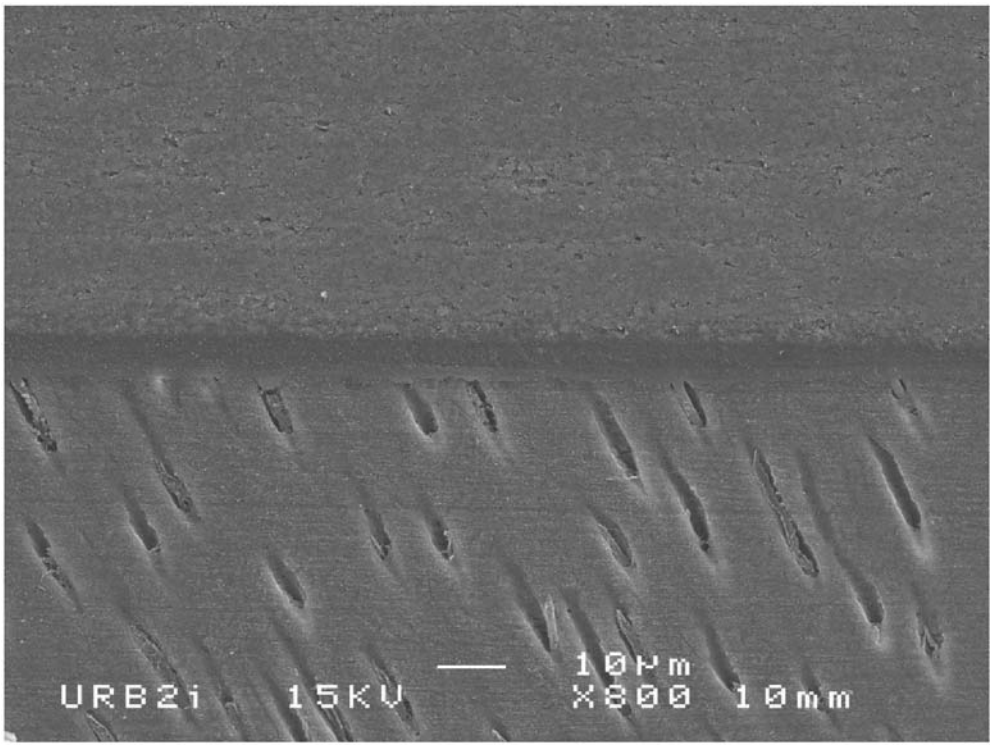
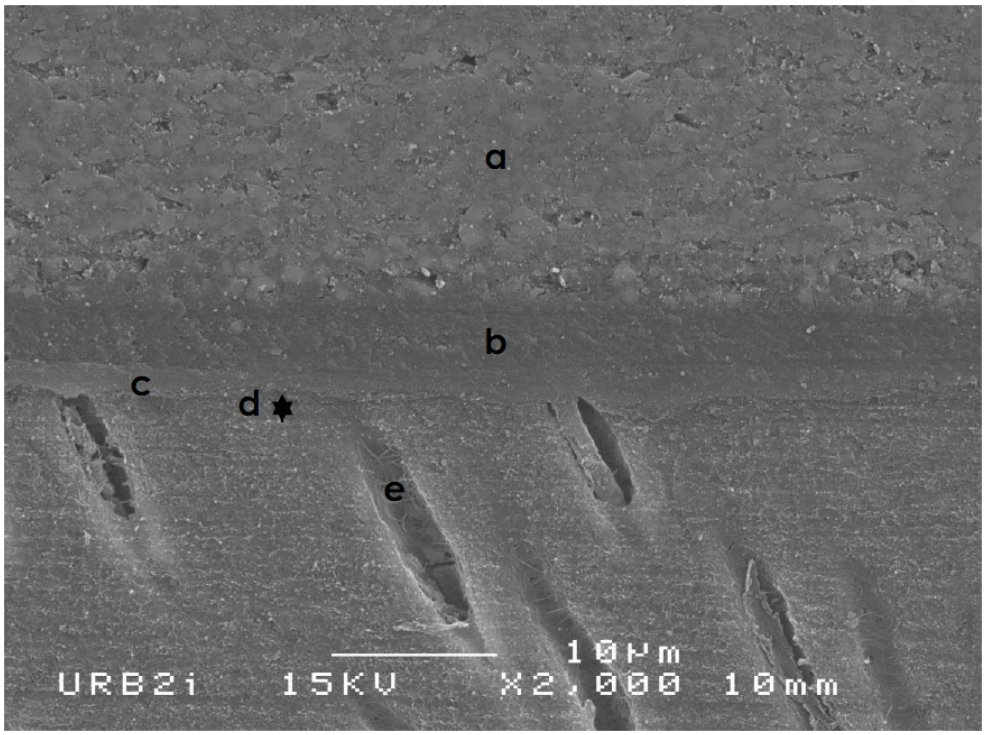


Fig. 8: Group 4: Reflectys/dentine interface cemented using Iperbond Ultra without preliminary etching (x2000)



Key: a: Reflectys composite; b: Iperbond Ultra adhesive; c: dentinal mud; d: hybrid layer (penetration of adhesive into the intertubular dentine); e: dentinal tubule

DISCUSSION

So far as the Iperbond Ultra/enamel interface is concerned:

With preliminary etching, micro-anfractuosities some microns deep were created on the surface of the enamel in both interprismatic and intraprismatic areas; the adhesive seems to have penetrated it well and formed resinous cords and the interface seems very good, with few porosities; the immediate seal also appeared to be good (Figs. 1 and 2).

Without etching, these micro-anfractuosities were not created (Fig. 3); the adhesive seems to have spread uniformly but we found that the interface appeared not to be so good as after etching, with some gaps and porosities the length of the interface (Fig. 3 and 4). This is very probably connected with the pH, which measured around 2.7 in the lab. This pH is usual for universal adhesives.

So far as the Iperbond Ultra/dentine interface is concerned:

With etching, we again found deep penetration of adhesive into the dentinal tubules in the form of regular cords of adhesive, fairly broad and at least ten microns deep (Fig. 6) in the micro-anfractuosities created (Fig. 5).

Without etching, we could observe no really marked resinous cords; we also noted a uniform spread of adhesive on the dentine surface (Fig. 7) and at the higher magnification it was possible to objectivize the adhesive's penetration into the dentinal tubules (Fig. 8).

Whether the dentine had been etched or not, it the adhesive seemed to have spread well over the surface and the immediate seal appeared good.

Moreover, the adhesive layer was thin (under ten microns or so), and its cohesion with the resin-composite used appeared to be good, resulting in excellent copolymerisation.

CONCLUSION

The Iperbond Ultra adhesive system seemed to have spread well over the enamel and dentine surfaces; nevertheless, preliminary etching did seem to improve its spreading over enamel. In the case of dentine we found an interface which also seemed very good, even without etchting. From this point of view it seems that Iperbond Ultra behaves both as a "Self –Etch Mode" and as a "E&R" "Etching & Rinsing" adhesive. It should be pointed out that this is the current tendency of universal adhesives.

2) Evaluation of the adhesion to enamel and dentine achieved by Iperbond Ultra in its two modes of application. Comparison with four competitors.

INTRODUCTION

As we have already pointed out, practitioners value "Self-Etch Mode" for their simplicity of use. Nevertheless a preliminary etching is likely to allow deeper and stronger infiltration of adhesive into the micro-anfractuosities created (especially on enamel), and to improve adhesion values.

The aim of this in vitro study was to test **Iperbond Ultra's adhesion to enamel and to dentine**, in each of its two recommended modes of application (**with and without etching with phosphoric acid**), and to compare its adhesive performance with those of competing systems in the market: one "E&R" adhesive (Excite F, *Ivoclar Vivadent*) and three "Self-Etch Mode" (Xeno III, *Dentspy*; Adhese One F, *Ivoclar-Vicadent* and iBond, *Heraeus*).

MATERIALS, EQUIPMENT AND METHOD

Preparation of the teeth

150 intact, freshly-extracted **human teeth** were collected, cleaned and kept at 4°C in a 1% chloramine T solution. The roots were eliminated using an irrigation polisher with a coarse (80 grit) abrasive disc. Their occlusal faces were first polished with a coarse (80 grit) paper and then with a finer one (800 grits) so as to obtain a clean, flat surface of enamel (in the case of 90 teeth) or dentine (60 teeth). These residual crowns were then covered with cylinders of self-polymerizing acrylic resin (Plexcil®, Escil, Chassieu, France), leaving a flat enamel or dentine surface exposed.

Each of the samples obtained was randomly assigned to one of **fifteen groups**: Nine groups of 10 samples each for the teeth with a flat enamel surface exposed, and six groups of 10 samples each with a flat dentine surface exposed.

Surface treatment, assembly and conservation

The adhesives Iperbond Ultra, Xeno III, iBond and Adhese One F (all three Self-Etch Mode) were tested in both modes of use, **with and without etching with 37% phosphoric acid**. Excite F (E&R) was tested using its sole mode of application, i.e. with phosphoric acid etching.

In the groups where mordant was used on enamel it was left on for 30 seconds, then carefully rinsed off and thoroughly dried. In the groups where it was used on dentine it was left on for 15 seconds, then carefully rinsed off and dried without drying out the dentine.

A Teflon® mould was placed over each sample to create **cylinders of Reflectys composite** 3mm in diameter and 2mm in height, cemented using Iperbond Ultra. The samples were kept in distilled water at 37°C for 24 hours, and then shear-tested.

Adhesion tests

The strength of adhesion was determined using a universal testing machine (LRX, *Lloyd Instruments*, England). The samples were immobilised on a device which allowed a shearing blade to be applied to the junction between the composite cylinder and the enamel or dentine, at a speed of 0.5mm/min.

Statistical analysis

The series of tests accordingly covered nine groups of 10 samples for enamel and six groups of 10 samples for dentine. ANOVAs followed by Tukey's tests were used to compare adhesion values. The significance level was set at $p<0.05$. The statistical calculations were done using the Stata 12 software.

RESULTS

Table 1 summarises the enamel and dentine adhesion values obtained for the various groups tested.

		Adhesion to enamel (in MPa)		Adhesion to dentine (in MPa)	
Adhesive	**Number**	Without etching	With etching	Without etching	With etching
Iperbond	n=40	27.6 (±2.3)[1]	30.9 (±2.7)[1]	27.1 (±3.2)[A]	29.1 (±5.2)[A]
Xeno III	n=30	28.2 (±2.7)[1]	29.5 (±4.0)[1]	15.6 (±1.7)[B]	
iBond	n=30	21.4 (±3.9)[2,3]	26.0 (±3.2)[1,3]	18.6 (±3.2) [B]	
Excite F	n=20		29.4 (±3.9)[1]		27.0 (±4.3)[A]
Adhese One F	n=30	16.4 (±5.5)[2]	20.8 (±7.5)[2,3]	6.9 (±5.2)[C]	
Means shown with the same superscript number or letter do not differ significantly at the 5% threshold.					

Tab. 1: Adhesion (in MPa) to enamel and to dentine of the various adhesives tested, with and without preliminary etching

So far as adhesion to enamel is concerned:

First of all, it should be noted that the values for adhesion to enamel were significantly higher when etched with phosphoric acid (Student's test: p=0.0025); this was expected in view of the data in the literature.

Iperbond Ultra's adhesion values with etching (30.9 MPa) and without (27.6 MPa), **were not significantly different**, though adhesion was better with etching (a greater number of samples would probably show that it is better, in terms of immediate adhesion, to mordant the enamel before applying Iperbond).

We may note that Iperbond Ultra's adhesion values **with etching** were **the highest** of the five adhesive systems under evaluation (even though the number of samples did not make it possible to establish a significant superiority).

With or without etching, Iperbond Ultra's adhesion to enamel was not significantly different from that of Excite F (29.4 MPa), which requires etching, nor from that of Xeno III, with (29.5 MPa) or without (28.2 MPa) etching. By contrast, although the adhesion values of Iperbond Ultra were comparable to those of iBond with preliminary etching (26.0 MPa), the latter had significantly lower values for adhesion to enamel than those of Iperbond Ultra without etching (21.4 MPa). Lastly, while Adhese One F's adhesion values were not significantly different with (20.8 MPa) and without (16.4 MPa) etching, both were significantly lower than those obtained with Iperbond Ultra.

So far as adhesion to dentine is concerned:

Iperbond Ultra's adhesion values, with (29.1 MPa) and without (27.1 MPa) etching, were the highest of the five adhesive systems tested (even though statistical significance was not demonstrated). Adhesion **was not significantly different** with and without etching the dentine with phosphoric acid, but it did seem that etching gave the higher values.

The adhesion values of Iperbond Ultra were not significantly different from those of Excite F (27.0 MPa), which requires etching.

They were, on the other hand, significantly higher than those of Xeno III (15.6 MPa), iBond (18.6 MPa) and Adhese One F (6.9 MPa) without mordanting.

DISCUSSION

On enamel

Iperbond Ultra is the adhesive system which achieved the best values for immediate adhesion to enamel against shearing in this evaluation which covered five adhesive systems. The number of samples tested did not allow proof of the improvement in adhesion after mordanting with orthophosphoric acid, but adhesion does at least appear to be better if that stage is included. Iperbond Ultra's adhesion without mordanting the enamel seems good enough for this adhesive system to be used in this way when required by the clinical situation.

On dentine:

Iperbond Ultra is likewise the adhesive system which achieved the best values for immediate adhesion to dentine against shearing in this evaluation covering five adhesive systems. As with enamel, the number of samples tested did not allow proof of the improvement in adhesion after etching with orthophosphoric acid, but adhesion does at least appear to be better if that stage is included. Iperbond Ultra's adhesion without etching the dentine also seems good enough for the adhesive system to be used in this way when required by the clinical situation (pre-operational sensitivity, deep loss of substance, &c.).

CONCLUSION

This structural evaluation of the interface and the mechanics of the Iperbond Ultra system's adhesion to dental tissue seems to indicate that the system is very good in terms of immediate adhesion. Iperbond Ultra's adhesion values as measured in this study were indeed higher (though not always with statistical significance) than those of the four other adhesive systems tested, in the case of enamel or dentine alike. Iperbond Ultra would therefore appear to be a **highly worthwhile adhesive system**.

This system is offered as a universal adhesive system: it can accordingly be used with or without preliminary etching of the enamel or dentine.

So far as the adhesion of this adhesive to enamel is concerned, this evaluation seems to confirm previous findings in the literature: etching of the enamel with phosphoric acid seems to improve the spreading of the adhesive (as observed using a scanning electron microscope on the interface) and to improve adhesion (as measured by gross shearing, though this study was unable to show a statistically significant difference). However, the adhesive power of Iperbond Ultra without preliminary etching appeared to be sufficient for this option to be considered when the clinical situation requires.

So far as the adhesion of this adhesive to dentine is concerned, it does indeed seem that it can be used with or without etching. The interface appeared to be good in either case. Adhesion seemed (though not to a statistically significant extent) to be slightly better after etching the dentine; but the drawbacks of etching dentine (post-operational sensitivity, risk of incomplete infiltration of the demineralized dentine and aging, &c.) might steer the practitioner's choice towards the use of this

adhesive system as a self-etching one, especially in certain particular clinical situations (deep cavities, &c.).

This universal adhesive could well be an option enabling the practitioner to respond to virtually all clinical situations, particularly if certain other aspects (biocompatibility, aging of the interface over time, compatibility with two-factor and chemically polymerized adhesives, &c.) are validated in high-quality scientific studies.