



## Introduction:

The group of all-in-one self-etching adhesives are in today's focus of interest in dental material science and gaining increasingly acceptance by the practitioners. All manufacturers claim high bonding efficiencies of their products. Usually, the bonding efficiency is given as shear bond strength (SBS) or micro tensile bond strength after 24 h water storage or after thermocycling for several days. However, only few is known concerning the the bonding efficacy within the first 24 h.

The disadvantage of the bond strength test is that it has to be done by an experienced operator and is very time consuming. In contrast to that the determination of the degree of conversion (DC) by FTIR-spectroscopy is a fast method to evaluate the material properties of dental composite.

## Objective:

This study was conducted to determine the degree of conversion of self-etching adhesives by FTIR-spectroscopy using two different evaluation methods at different times after light activation.

## Materials:

*Absolute*, Lot: 388-007, Dentsply-Sankin; *Clearfil S<sup>3</sup> Bond*, Lot: 011111, Kuraray; *G-Bond*, Lot: 0405111, GC-Corporation; *Hybrid-Bond*, Lot: KK2, J. Morita Europe; *iBond*, Lot: 010061, Heraeus Kulzer, *experimental iBond*, Lot: VP 070904AK3, Heraeus Kulzer; *Xeno IV*, Lot: 050119, Dentsply-Caulk; *Venus A2*, Lot: 010111, Heraeus Kulzer. The adhesives were applied according to manufacturers instructions. Light activation of adhesives and composite (Venus, Heraeus Kulzer) was done with a standard halogen curing unit (Translux Energy, Heraeus Kulzer).

## Method:

DC was determined using FTIR-ATR spectroscopy (Spectrum One, Universal ATR Sample Accessory, Perkin-Elmer). FTIR-spectra were recorded immediately, 1 h, 2 h, and 24 h after irradiation from five adhesive specimens covered with Venus, each. DC values were calculated from the ratio of absorbance intensities of aliphatic carbon double bonds (1636 cm<sup>-1</sup>) before and after polymerization using baseline corrected original spectra or second derivative spectra.

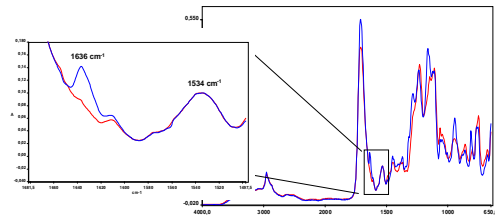


Figure 1: Example of FTIR spectra of iBond before (blue line) and after (red line) light activation. Small box: Magnification of relevant region.

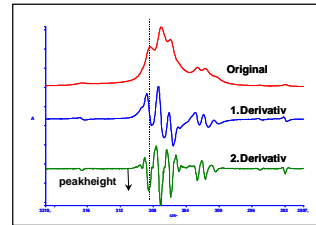


Figure 2: Example of FTIR second derivative spectrum.



Figure 3: FTIR-ATR spectroscope, Spectrum One, Universal ATR Sample Accessory, Perkin-Elmer (left photograph) and prepared adhesive film on sample holder before measurement (right photograph).

## Results:

### 1. Degree of Conversion – Baseline Method:

	Absolute	Clearfil S <sup>3</sup>	G-Bond	Hybrid Bond	iBond	exp. iBond	Xeno IV
Immediate	58.7 <sup>a</sup>	82.5 <sup>b</sup>	83.0 <sup>b</sup>	56.2 <sup>a</sup>	78.6 <sup>b</sup>	72.7 <sup>a</sup>	63.7 <sup>a</sup>
1 h	65.7 <sup>b</sup>	91.3 <sup>c</sup>	87.6 <sup>b</sup>	66.6 <sup>b</sup>	89.3 <sup>b</sup>	81.6 <sup>b</sup>	70.6 <sup>b</sup>
2 h	67.0 <sup>b</sup>	91.4 <sup>b</sup>	88.6 <sup>b</sup>	68.1 <sup>b</sup>	90.5 <sup>b</sup>	84.1 <sup>b</sup>	72.3 <sup>b</sup>
24 h	71.1 <sup>c</sup>	94.4 <sup>b</sup>	90.5 <sup>b</sup>	74.0 <sup>b</sup>	92.9 <sup>b</sup>	89.7 <sup>b</sup>	78.0 <sup>b</sup>

Figure 4: Degree of Conversion at different times after light activation. Same small letters denote groups that are not significantly different by columns. Same capital letters denote groups that are not significantly different by rows.

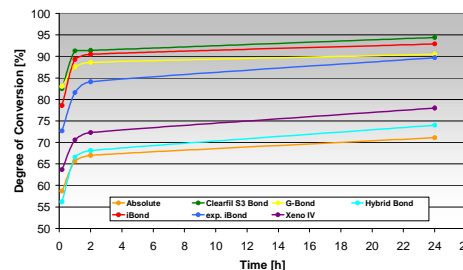


Figure 5: Degree of Conversion [%] versus Time [h]

### 2. Degree of Conversion – Derivative Method:

	Absolute	Clearfil S <sup>3</sup>	G-Bond	Hybrid Bond	iBond	exp. iBond	Xeno IV
Immediate	51.3 <sup>a</sup>	83.0 <sup>b</sup>	75.7 <sup>a</sup>	49.6 <sup>a</sup>	72.6 <sup>a</sup>	64.9 <sup>a</sup>	61.6 <sup>a</sup>
1 h	59.3 <sup>b</sup>	87.9 <sup>b</sup>	81.4 <sup>b</sup>	59.4 <sup>b</sup>	87.0 <sup>b</sup>	78.0 <sup>b</sup>	68.5 <sup>b</sup>
2 h	61.0 <sup>b</sup>	88.7 <sup>b</sup>	82.7 <sup>b</sup>	61.2 <sup>b</sup>	88.9 <sup>b</sup>	81.1 <sup>b</sup>	70.6 <sup>b</sup>
24 h	66.2 <sup>c</sup>	90.8 <sup>b</sup>	87.7 <sup>b</sup>	67.2 <sup>b</sup>	93.7 <sup>b</sup>	89.1 <sup>b</sup>	74.1 <sup>b</sup>

Figure 6: Degree of Conversion at different times after light activation. Same small letters denote groups that are not significantly different by columns. Same capital letters denote groups that are not significantly different by rows.

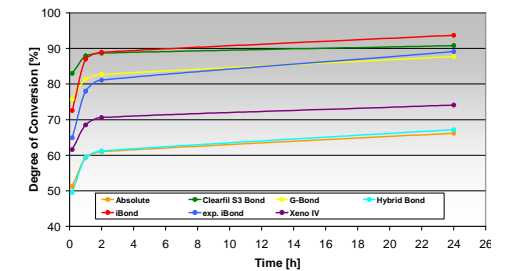


Figure 7: Degree of Conversion [%] versus Time [h]

## Conclusion:

Derivative method produced significant lower values concerning degree of conversion. The values resulted from the baseline method are approx. 5 % higher when comparing both evaluation methods.

Both FTIR evaluation methods are able to calculate the degree of conversion. For future evaluations the derivative method is recommended due to it's ease of use.

Degree of conversion of adhesives depends on time of testing.

Apart from G-Bond and Clearfil S<sup>3</sup> Bond the degree of conversion of the adhesives increases by about 15 % within 24 h. DC of G-Bond and Clearfil S<sup>3</sup> Bond increases by 10 %.

Clearfil S<sup>3</sup> Bond shows the highest initial DC, Absolute and Hybrid Bond the lowest.

Only for Clearfil S<sup>3</sup> Bond nearly all double bonds have been reacted after 1 h. All other adhesives post-cured up to 24 h.

After 24 h Clearfil S<sup>3</sup> Bond, G-Bond, iBond and the exp. iBond show higher DC than Xeno IV, Absolute and Hybrid Bond.