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## International Journal of Adhesion &amp; Adhesives

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# Influence of radiant exposure on degree of conversion, water sorption and solubility of self-etch adhesives



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## ARTICLE INFO

## Article history:

Accepted 7 May 2013

Available online 31 May 2013

## Keywords:

Dental adhesives

Cure/hardening

Infrared spectra

Mechanical properties of adhesives

## ABSTRACT

The purpose of this study was to investigate the influence of the radiant exposure on the degree of C=C conversion (DC), water sorption (WS), and solubility (S) of the Clearfil SE Bond (CSE) and Filtek LS (LS) self-etch adhesive systems. The primer of the LS (LSP), and bond agents of the CSE (CSEB) and LS (LSB) were tested. Specimens were light-cured using a light-emitting diode (LED) at different radiant exposures (6.1, 12.2, 13.9, and 27.8 J/cm<sup>2</sup>). DC ( $n=10$ ) was measured using Fourier-transform infrared spectroscopy (FT-IR). WS and S were determined according to ISO 4049. Data were subjected to two-way ANOVA and Tukey's test at pre-set alpha of 0.05. The highest DC was exhibited by LSP, followed by CSEB and LSB, all with statistical difference ( $p < 0.001$ ). The DC was increased with higher radiant exposure and extended light-curing time ( $p < 0.001$ ). LSB and CSEB showed the lower WS followed by LSP, all with statistical difference ( $p < 0.001$ ). CSEB and LSB presented no significance difference on the S values ( $p > 0.05$ ) and were lower than LSP ( $p \leq 0.05$ ). The WS and S were not influenced by the different radiant exposures evaluated ( $p=0.9548$  and  $p > 0.05$ , respectively). The monomer conversion is related to improvement on the mechanical properties of resinous material, but these properties also depend on the polymer network structure formed.

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## 1. Introduction

Currently, conservative restorative procedures can be performed with use of dental adhesives, through the bonding between the restorative material and hard tissues of the tooth [1]. An adequate adhesion to dental tissues is necessary for a long-term durability of the integrity of tooth-restorative material interface. Its failure results in the loss of the retention and marginal adaptation, and is one of the main reasons for adhesive restorations replacement [2,3].

Contemporary adhesives are classified in etch-and-rinse and self-etch [3]. These adhesives are light-cured and the radiant exposure that reaches the material surface is an important factor that provides better mechanical properties, a low monomer conversion decreases the bonded interface strength and degradation resistance [4,5]. Water sorption (WS) and solubility (S) can be associated to degree of C=C conversion (DC) of resin-based materials [6].

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Aqueous environment may cause softening of the resin-based materials by swelling of the polymer network and reduction of the frictional forces between polymer chains [7], affecting the WS and S of resinous materials. Water uptake causes chemical degradation by filler-polymer de-bond and residual monomer release [6]. Moreover, the insufficient cross-linked polymer is more susceptible to plasticization effect by chemical substances that enter during eating and drinking [8].

The increase of the distance between the light guide tip and resinous material surface in deep cavity restoration decreases the irradiance that reaches the restorative material, reducing the DC [9] and/or leading to the formation of more linear polymers [10]. The increase of the radiant exposure available for monomer conversion can improve the physical properties [11–13] and increase adhesive restoration longevity [14]. High conversion of monomer into polymer is related to improve mechanical behavior [1,6,7], but DC is not enough to characterize the 3-dimensional network structure, whereas different C=C bond concentrations coexist in the same polymer [7]. Similar DC value may result in different linear and cross-linked polymer contents [10,15].

Daily deep cavities are found in dental, clinical and the photo-activation away from the surface material is commonly performed; thus, the evaluation of different radiant exposure, time and

distance light-curing are important for the knowledge of light-cured resins behavior, especially of the specific adhesive system used on the bonding procedures of the low-shrink silorane composite (Filtek LS). Clearfil SE Bond consists of a hydrophilic self-etch primer and hydrophobic bond agent, this viscous hydrophobic resin coating layer improves mechanical properties and increases longevity of the bonding interface [4], and is a reference standard of self-etch adhesive for comparison.

LS System Adhesive is classified as two-step self-etch adhesive; firstly the hydrophilic LS primer is applied and light-cured forming the hybrid layer [16,17]. Thus the bi-functional hydrophobic monomer (phosphorylated methacrylate) of the LS bond applied after the primer cured acts as a low viscosity resin liner of connection between methacrylate monomers (by reaction with acrylate group) and silorane monomer (by reaction of the phosphate group with oxirane) [18]. So LS primer acts as one-step self-etch adhesive [16,17].

The objectives of this investigation were to evaluate the influence of the different radiant exposures on the DC, WS, and S of the primer of the Filtek LS, and bond agents of the Filtek LS and Clearfil SE Bond. The hypotheses tested were: (1) there would be no difference between the materials and that (2) the highest radiant exposure would improve the physical properties evaluated.

## 2. Material and methods

The commercial self-etch adhesive systems investigated were Clearfil SE Bond (CSE) and Filtek LS System Adhesive (LS). The primer of the LS (LSP) and bond agents of the CSE (CSEB) and LS (LSB) were tested. Details of the materials are shown in Table 1.

The degree of C=C conversion assessment was obtained using a Fourier Transform Infrared (FT-IR) spectrometer (Spectrum 100 Optica, Perkin Elmer, MA, USA), equipped with an attenuated total reflectance (ATR). A constant volume of the resinous material (5  $\mu$ L) was placed on the horizontal face of the ATR cell. Absorption spectra ( $n=10$ ) were obtained by the baseline technique in the region between 1800 and 1400  $\text{cm}^{-1}$  with 16 scans at 4  $\text{cm}^{-1}$  of resolution. To calculate the DC, the ratio ( $R$ ) between the peak heights of the C=C aliphatic (1638  $\text{cm}^{-1}$ ) and aromatic group (1608  $\text{cm}^{-1}$ , used as internal standard) band absorptions for cured and uncured composite was used, according to the formula:  $\text{DC} (\%) = [1 - (R_{\text{polymer}}/R_{\text{monomer}})] \times 100$ .

Simulating clinical restorative procedure in a cavity of 6 mm of depth, light-curing of the samples were performed at 0 (control)

**Table 1**  
Materials used.

Material	Composition <sup>*</sup>
Clearfil SE Bond (Kuraray Medical Inc. Okayama, Japan)	Lot. 01416 A bond (CSEB) MDP, Bis-GMA, HEMA, CQ, hydrophobic dimethacrylate, N,N-diethanol p-toluidine, colloidal silica.
Filtek LS (3M ESPE AG, Seefeld, Germany)	Lot. 9BN primer (LSP) Bis-GMA, HEMA, water, ethanol, silica treated silica filler, CQ, phosphoric acid-methacryloxy-hexylesters mixture, phosphorylated methacrylates, copolymer of acryl and itaconic acid, phosphine oxide. Lot. 9BK bond (LSB) Hydrophobic dimethacrylate, phosphorylated methacrylates, TEGDMA, silica treated silica, CQ, stabilizers.

Abbreviations—MDP: 10-methacryloyloxydecyl dihydrogen phosphate; HEMA: 2-hydroxyethylmethacrylate; CQ: camphorquinone; Bis-GMA: bisphenol-A glycidyl dimethacrylate; TEGDMA: triethylene glycol dimethacrylate; and Bis-EMA: ethoxy-1,2-bisphenol-A dimethacrylate.

\* As informed by manufacturers.

**Table 2**  
Experimental groups.

Material	Curing time (s), distance of photo-activation (mm), and irradiance ( $\text{mW}/\text{cm}^2$ )	Radiant exposure ( $\text{J}/\text{cm}^2$ )
CSEB/LSP/LSB	10, 6, 610	6.1
CSEB/LSP/LSB	20, 6, 610	12.2
CSEB/LSP/LSB	10, 0, 1390	13.9
CSEB/LSP/LSB	20, 0, 1390	27.8

Clearfil SE Bond "bond" (CSEB); LS System Adhesive "primer" (LSP); e LS System Adhesive "bond" (LSB).

and 6 mm of distance with a second-generation light-emitting diode (LED) Bluephase 16i (Vivadent, Bürs, Austria) device at 1390  $\text{mW}/\text{cm}^2$  of irradiance according to experimental groups (Table 2). A low-pressure air stream was gently applied during 10 s for solvent evaporation of the solvated LSP. A holder coupled to the light source was used to standardize the distance between the light guide tip and material, controlled by digital caliper. The optical power (mW) delivered by the device was measured with a power meter (Ophir Optronics, Har-Hotzvim, Jerusalem, Israel). The tip diameter was measured with digital caliper to determine tip area ( $\text{cm}^2$ ). Irradiance ( $\text{mW}/\text{cm}^2$ ) was calculated dividing optical power by tip area, and radiant exposure ( $\text{J}/\text{cm}^2$ ) the irradiance multiplied curing time divided by 1000.

The water sorption and solubility were performed in compliance with ISO 4049:2009 standard specifications, except for the specimen dimensions and curing protocol. Silicon mold was used to prepare circular specimens (5 mm diameter and 0.5 mm thickness) of the materials. Solvated LSP was stored in dark room at 37 °C until constant mass is obtained, indicating the solvent evaporation. The LSP, LSB, and CSEB materials were placed into mold, covered by Mylar strip, and irradiated according to experimental groups (Table 2).

The specimens ( $n=5$ ) were stored in desiccator at 37 °C containing silica gel. The specimens were weighted daily in an analytical balance (Tel Marke, Bel Quimis, São Paulo, SP, Brazil) accurate to 0.001 mg, constituting a weighing cycle every 24 h. The complete cycle was repeated until a constant mass ( $m_1$ ) was obtained until that the mass loss of each specimen was not more than 0.1 mg per 24 h cycle. Thickness and diameter of each specimen were measured using a digital electronic caliper. Mean values were used to calculate the volume ( $v$ ) of each specimen (in  $\text{mm}^3$ ).

Then, the specimens were stored in water at 37 °C for 7 days, the volume of water for immersion being at least 6 mL per specimen. After this period, the specimens were carefully wiped with an absorbent paper and weighted again; this value was recorded as  $m_2$ . After this weighing, the specimens were returned to the first desiccators and the entire mass reconditioning cycle was repeated and the constant mass was recorded as  $m_3$ . The values for WS and S, in micrograms per cubic millimeters, were calculated using the following equations:  $\text{WS} = (m_2 - m_3)/v$  and  $\text{S} = (m_1 - m_3)/v$ .

The DC and WS data were analyzed by two-way analysis of variance (ANOVA) and Tukey's test ( $\alpha=0.05$ ). The S data did not assume presuppositions of normality, so non-parametric Kruskal–Wallis test was used to determine if there was significant difference, and Dunn's test to multiple comparisons at the 5% significance level.

## 3. Results

ANOVA showed statistical difference for factors interaction of the DC test ( $p < 0.001$ ). The highest DC was exhibited by LSP, followed by CSEB and LSB, all with statistical difference ( $p < 0.0001$ ). The DC

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