

# VARIATIONS IN RESIDUAL DOUBLE BONDS OF A COMMERCIAL SELF ETCHING ADHESIVE

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**Abstract:** The FTIR examination technique allows the identification of the degree of conversion from monomer to polymer of the adhesive systems by determining the infrared absorption spectrum of the studied products. As the current tendency moves towards work simplification, we considered it necessary to make a study on a self etching adhesive. Samples of self etching adhesive Adper Prompt L-Pop (3M ESPE) were prepared, with 5 mm diameter and 1 mm width, polymerized with an Elipar 2500 (3M ESPE) halogen curing light, 600mW/cm<sup>2</sup>, and an Elipar Freelight 2 (3M ESPE) LED light, 1000mW/cm<sup>2</sup>. The samples were examined through the FTIR technique immediately after polymerization, then three months later, after preparing the samples. The results indicate a slightly higher value of the residual double bonds during the LED polymerization than during the halogen one. If the examination of the samples is done later, one can observe a decrease of the double residual bonds compared to the initial values.

**Conclusion:** In this experiment, the value of the double residual bonds of the Adper Prompt L-Pop adhesive has been less influenced by the light curing source used, than by the time the tests have been performed.

**Keywords:** FTIR examination technique, self etching adhesive, double residual bonds

**Rezumat:** Tehnica de examinare FTIR permite stabilirea gradului de conversie din monomer în polimer al sistemelor adezive prin determinarea spectrului de absorbție în domeniul infraroșu al produselor studiate. Deoarece tendința actuală este aceea de a simplifica tehnica de lucru, am considerat util să studiem un adeziv autogravant. S-au realizat mostre de adeziv autogravant Adper Prompt L-Pop (3M ESPE), cu diametrul de 5 mm și grosimea de 1 mm, polimerizate cu o lampă cu bec halogen, Elipar 2500 (3M ESPE), 600mW/cm<sup>2</sup>, și o lampă cu LED, Elipar Freelight 2 (3M ESPE), 1000mW/cm<sup>2</sup>. Probele au fost examinate prin tehnica FTIR imediat după polimerizare și la trei luni după realizarea probelor. Rezultatele arată o valoare ușor mai ridicată a prezenței dublelor legături reziduale la polimerizarea cu LED comparativ cu cea cu bec halogen. Când examinarea probelor se face tardiv, se observă o scădere a dublelor legături reziduale comparativ cu

valorile inițiale. **Concluzie:** în acest experiment, valoarea dublelor legături reziduale ale adezivului Adper Prompt L-Pop a fost influențată mai puțin de sursa de fotopolimerizare utilizată, cât de momentul la care au fost efectuate testele.

**Cuvinte cheie:** tehnica de examinare FTIR, adeziv autogravant, legături reziduale duble

## INTRODUCTION

FTIR technique allows to establish the degree of conversion of the adhesive systems, from monomer to polymer by determining the infrared absorption spectrum of the studied products.(3)

## PURPOSE OF THE PAPER

Based upon the idea that the residual monomers have cytotoxic potential, this study aims at observing the way in which the degree of conversion of the double bonds C=C is modified in the adhesive systems under the influence of two light curing sources and the influence of the storage time for samples.

## MATERIAL AND METHOD

Light curing adhesive samples were accomplished, having a diameter of 5 and 1 mm, which were polymerized with:

- Elipar 2500 (3M ESPE) halogen curing light, Ø 8 mm, 600mW/cm<sup>2</sup>, 20 seconds.
- Elipar Freelight 2 (3M ESPE) LED light, Ø 8 mm, 1000mW/cm<sup>2</sup>, 10 seconds.

The curing time was chosen based on the principle of total energy density. We took into consideration the power density of each lamp and the spectral loss of the halogen lamp.

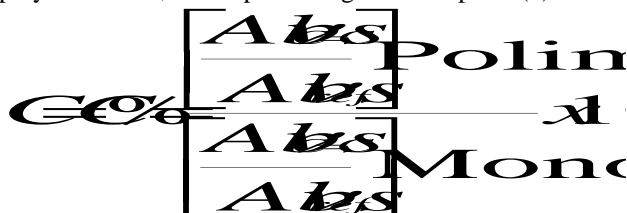
The polymerization was done at 0,5 mm distance over a film of transparent plastic material, so as not to affect the polymerization efficiency through direct bonding to the adhesive system.

The samples were observed by means of FT-IR JASCO 610 under direct collaboration with the Research Institute of Chemistry „Raluca Râpan”, Cluj-Napoca. The samples were taken into study immediately, respectively three months after polymerization.

In order to determine the quantity of the non-reactive methacrylate groups, an absorption band of 1635-

1640 cm<sup>-1</sup> is used because of the valence vibrations of the double bonds C=C of the methacrylate groups. The absorption group C-C from 1610 cm<sup>-1</sup> is used as reference, because of the valence vibrations of the aromatic nucleus from the molecule Bis-GMA.

The report of the intensity of the absorbents C=C/C-C is measured before and after the polymerization, Abs representing the absorption.(3)



The composition of the Adper Prompt LP adhesive is:

Liquid 1

- Methacrylate Phosphoric esters
- Bis-GMA
- Initiators based on camphorquinone
- Stabilizers

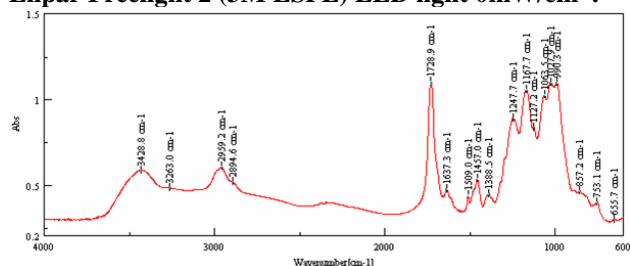
Liquid 2

- HEMA
- Polyalkenoic liquid
- Water
- Stabilizers

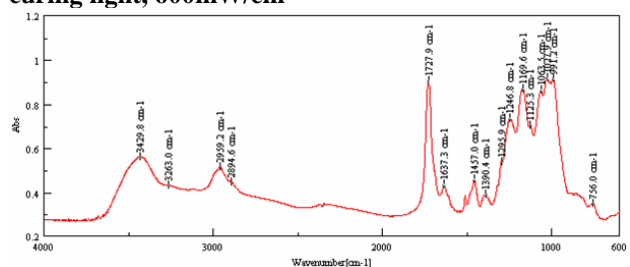
## RESULTS

The characteristics of the absorption spectrum of the polymerized adhesive systems are represented in pictures 1 and 2 for immediate measurements and in pictures 3 and 4 for delayed measurements.

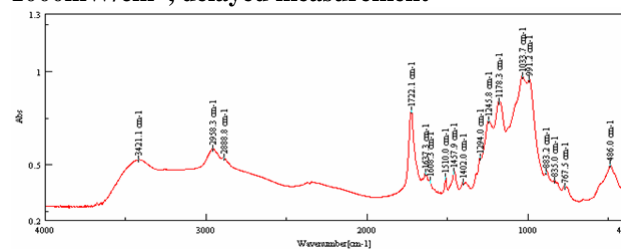
Picture no. 1. Absorption spectrum of the Adper Prompt L-Pop (3M ESPE) self etch adhesive, with Elipar Freelight 2 (3M ESPE) LED light 0mW/cm<sup>2</sup>.



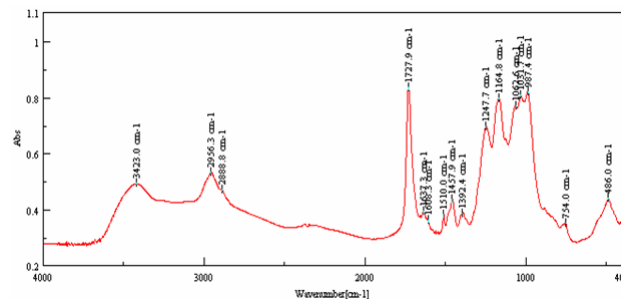
Picture no. 2 Absorption spectrum of the Adper Prompt L-Pop (3M ESPE) self etch adhesive, polymerized with an Elipar 2500 (3M ESPE) halogen curing light, 600mW/cm<sup>2</sup>



Picture no. 3. Absorption spectrum of the Adper Prompt L-Pop (3M ESPE) self etch adhesive, with Elipar Freelight 2 (3M ESPE) LED light, 1000mW/cm<sup>2</sup>, delayed measurement



Picture no. 4. Absorption spectrum of the Adper Prompt L-Pop (3M ESPE) self etch adhesive, polymerized with an Elipar 2500 (3M ESPE) halogen curing light, 600mW/cm<sup>2</sup> 3 months after.



The measured values of the double residual bonds are illustrated in table 1:

Table 1. Values of the double residual bonds.

%C=C Source v	Immediate measurement t %	Delayed measurement nt %	Time variation s %
Halogen	92,995	87,921	5,074
LED	93,168	89,908	3,260

When performing the same tests three months after the preparation of samples and being stored at room temperature and in normal humidity conditions, a decrease in the residual double bond content was observed.

## DISCUSSIONS

An important factor which influences the biocompatibility is the degree of conversion of the monomer polymerization, which penetrates the collagen fibre network. The polymerization degree inside the hybrid layer depends on the following:

- Polymerization type: photo-, auto-, or dual cure;
- Location where the reaction begins (in the thickness of the hybrid layer in the light curable systems or at the surface for the self- or dual cure adhesives);
- The number of C-C bonds available;
- An improper curing within the hybrid layer, makes it act like a "reservoir", releasing monomers with

## CLINICAL ASPECTS

cytotoxic potential.(5)

This experiment is based on the concept of total energy density, which refers to the energy supplied to the material and not to the power of the curing lamp.(8, 2)

Adper Prompt LP is a self-etch adhesive, which uses camphorquinone as a photoinitiator. The absorption spectrum of camphorquinone matches with the one of the halogen lamp emission spectrum (7) and also with the one of the LED lamps. (8)

The LED emission spectrum is very narrow, very tight and may not comply with the maximum absorption of the photoinitiators. This may lead to a low energy internal conversion and to deficient physical chemical properties.(5)

The modification of the residual double bond values in time, can be explained by the fact that the polymerization reaction is not complete immediately after the photopolymerization. There still remains a mobility of the reactive species, which changes the degree of polymerization of the adhesive. This mobility can be influenced by the amount of the solvent remaining in the adhesive, by the exposure to ambient light, by the initial degree of polymerization.

Advanced studies performed on the same commercial adhesive showed similar results.(6,7)

The light source does not influence the degree of conversion from monomer to polymer in the hybrid resin composites.(1, 2,4)

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## CONCLUSIONS

- This experiment showed that the amount of residual double bonds of the Adper Prompt L-Pop adhesive has been less influenced by the photopolymerization source used, than by the moment the tests have been performed.
- The use of the total energy concept of polymerization leads to predictable results regarding the degree of conversion of the photopolymerisable materials monomers.

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