 DOI:10.4047/jkap.2008.46.6.639

DEGREE OF CONVERSION OF BIS-ACRYLIC BASED PROVISIONAL CROWN AND FIXED PARTIAL DENTURE MATERIALS

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INTRODUCTION

Monomers in the polymer-based provisional crown and fixed partial denture materials form polymer network as carbon-carbon double bonds are converted to carbon-carbon single bonds during polymerization. This reaction is not complete. There will be a certain amount of monomer remaining in the resin materials. This is influenced by several factors, such as type of monomer, curing temperature, exposure to oxygen, and the amount of initiator, etc. The degree of conversion is a measure of the carbon double bonds converted into single bonds.[1]

The degree of conversion may influence the ultimate mechanical and physical properties of the materials. The high levels of the residual monomer may cause deleterious effect on properties, such as low hardness, low strength, low resistance to wear, and low color stability.[2,3] In addition, the residual monomer may irritate the surrounding tissues, including pulp, and oral mucosa. A number of reports have appeared in the literature regarding hypersensitivity to the residual monomer, such as pulpitis, burn, glossitis, stomatitis, generalized erythematous rash, although its possibility was low.[4-7] The residual monomer may elute from the polymer-based provisional crown and fixed partial denture materials and diffuse into pulp and saliva resulting in redness and a burning sensation of the oral mucosa.[8]

Polymerization takes place as external energy, such as heat, light and chemicals, forms free radicals from suitable initiator molecules, and a free radical attacks on the double bond (π-bond) of a vinyl group in a monomer, resulting in the new formation of a single bond (σ-bond) to another carbon and an unpaired electron. As polymerization progresses, the amount of aliphatic carbon double bond decreases. Infrared spectroscopy has been used widely for measuring the unreacted carbon double bond. Spectroscopically, the decrease of aliphatic carbon double bond causes a decrease in the absorbance at 1637 cm^{-1}. Bis-GMA has a functional group which does not participate in polymerization. The aromatic double bond of this group (phenyl group) has an absorbance at 1608 cm^{-1}. This absorbance remains constant before and after polymerization. This method is based on comparing the intensities of the absorbance of carbon-carbon double bonds in methacrylate group before and after polymerization. In aromatic moieties, the absorbance of aromatic double bonds can be used as an internal standard.

The purpose of this study was to measure the degree of conversion of three bis-acrylic based provisional crown and fixed partial denture materials by using an infrared spectroscopic method. The null hypothesis to be tested is that there is no significant difference in the degree of conversion among bis-acrylic based provisional crown and fixed partial denture materials.

MATERIALS AND METHODS

Three provisional crown and fixed partial denture materials investigated in this study are presented in Table Ⅰ; three di-methacrylate based materials, LuxaTemp (**LXT**), fast set Temphase (**TMP**) and Protemp 3 Garant (**PT3**). All three materials are chemically activated resins. They were mixed and initiated in accordance with

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Received November 26, 2008 Last Revison December 1, 2008 Accepted December 2, 2008.

Table I. Experimental materials tested

Materials	Code	Lot No.	Shade	Manufacturer
Protemp 3 Garant	**PT3**	FW0063892	A3	3M-ESPE, St Paul, MN, USA
Fast set TemPhase	**TMP**	5572	A3.5	Kerr, Orange, CA, USA
LuxaTemp	**LXT**	120337	A2	DMG, Hamburg, Germany

Fig. 1. Avatar 360 FTIR Spectrometer used to measure the degree of conversion.

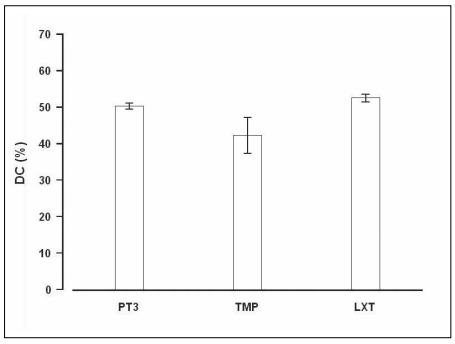

Fig. 2. *Degree of conversion* of the investigated materials.

manufacturers' instructions. All three materials were mixed automatically by the dispenser tip.

Fourier transform infrared spectrometry (FTIR) was used to determined the degree of conversion. The infrared spectrum was recorded by means of a FTIR spectrometer (Avatar 360, Nicolet Analytical Instruments UK) (Fig. 1). The wavelength was 2000 - 550 cm^{-1}. The resolution was 4 cm^{-1}. Scan coaddition was 16. The FTIR spectra of the mixed provisional crown and fixed partial denture materials were immediately obtained after a small amount of each material had been compressed firmly into a thin film between two translucent polyethylene strips. The spectra of the mixed provisional crown and fixed partial denture materials were obtained by using the same method as the above at 24 hours after mixing. The specimens were stored under dry conditions and at 23°C. The approximate thickness of the thin film was between 40 μm and 70 μm.

In this study, the degree of conversion (%) was calculated by determining the proportion of remaining carbon double bonds in the strip at 24 hours in relation to the carbon double bonds in the strip just after mixing by using the internal standard method. The quantity of remaining carbon-carbon double bonds was determined from the spectrum of the absorbance between the aliphatic double bond at 1637 cm^{-1} and the aromatic double bond at 1608 cm^{-1} before and after polymerization using the baseline method. The absorbance at 1637 cm^{-1} originates from aromatic double bonds in the Bis-GMA molecule. Its intensities remain unchanged after polymerization.

The degree of conversion of each material was obtained using Equation 1.

Equation 1)

DC (%) = $\times 100$ - % of remaining double bond

where

% of remaining double bond =

$$\frac{[(_{Abs}C = C)/(_{Abs}C \cdots C)]_{polymer}}{[(_{Abs}C = C)/(_{Abs}C \cdots C)]_{monomer}} \times 100$$

All experiments were carried out in triplicate. The mean values and standard deviations of the results were computed. The data were statistically analyzed using one-way ANOVA (oneway analysis of variance) and the multiple comparison Scheffe test at the 0.05 significance level (SPSS, version 9.0, SPSS Inc., USA).

RESULTS

The results are presented graphically in Fig. 2. The mean values and standard deviations of the degree of conversion were 52.5 $\pm$ 1.1 %, 50.3 $\pm$ 0.8 %, and 42.3 $\pm$ 4.9 % for LuxaTemp, Protemp 3 Garant and *fast set* TemPhase, respectively. There was no significant difference between LuxaTemp and Protemp 3 Garant ($P > .05$). However, there

was a statistically difference between Protemp 3 Garant and fast set TemPhase, and LuxaTemp and *fast set* TemPhase ($P < .05$).

DISCUSSION

Several techniques have been reported to determine the degree of conversion in resins. Gas-liquid chromatography (GLC) has been used successfully to determine residual monomer in resin.[10-12] High-performance liquid chromatography (HPLC) was developed to analyze all components such as eluates from resins.[13-15] Spectroscopic analysis,[16] and wet chemical analysis[17] have been used. Differential scanning calorimetry, which is based on the enthalpy of the exotherm for setting, has been used by many researchers.[18,19] Among them, infrared spectroscopy has been most widely used for measuring the unreacted monomers in resin materials since Ruyter and Györöosi first applied the method to dental materials.[1-3,9,16,20-22]

Fourier transform infrared spectroscopy in this study has advantages, such as sensitivity, specificity, high reliability, and economy.[23] In addition, the thin film technique by using polyethylene strips is easy to apply, very useful, and provides a constant values of degree of conversion. This investigation demonstrated that the degree of conversion for provisional crown and fixed partial denture material can be easily and precisely measured by using FTIR.

Over the years, there has been a continual development of the provisional crown and fixed partial denture materials. One of the modern classes of developed provisional crown and fixed partial denture materials is di-methacrylate based composites, which were introduced to overcome the negative properties of mono-methacrylates. During polymerization, the di-methacrylate monomer undergoes cross-linking. In case of restorative composite resins, unsaturated residual monomer ranges from 25 % - 45 %. The materials exhibit lower degrees of conversion ranging from 55 % to 75 % than acrylic resins.[21,24-26] One reason is the limitation of movement of the monomers because of the earlier onset of vitrification of the polymer network. The other is that the methacrylate groups of polymer chain which has not-reacted side can not diffuse through the matrix because they are already attached to the polymer.[27]

The degrees of conversion of provisional crown and fixed partial denture materials tested in this study were lower than those of restorative composite resins investigated in the previous studies.[24-26] This is because the provisional crown and fixed partial denture materials have higher proportion of di-methacrylate monomers than the restorative composite resins. The greater the proportion of di-methacrylates in the composites, the lower the degree of conversion. As noted in the introduction, the degree of conversion is influenced by several factors such as the type of monomer, curing temperature, exposure to oxygen, the amount of initiators, filler particles, and the ability of monomer to diffuse to the reactive radical ends of polymer through the matrix, etc.

The provisional crown and fixed partial denture materials tested varied in the extent of the polymerization. LuxaTemp and Protemp 3 Garant polymerize to a relatively higher degree than fast set TemPhase which cured the least of the three materials. The results of this study suggest that the degree of conversion may be correlated with the rate of polymerization. Thus, the faster the setting, the lower the degree of conversion. After polymerization, a certain amount of the unpolymerized monomer may affect the mechanical and physical properties, and biocompatibility. It may reduce the dimensional stability, the strength, and wear resistance, and tends to soften the resin material.[2,3] Besides the effects on mechanical and physical properties, the residual monomer can leach out from the resin to the oral environment, causing cytotoxic effects on pulp and oral mucosa, and inhibiting the protein synthesis of the oral epithelial cells.[28] Therefore, a higher degree of conversion can mean the improved biocompatibility of the polymer-based provisional crown and fixed partial denture materials.

CONCLUSIONS

1. *Fourier Transform Infrared Spectroscopy* is a useful method for measuring the degree of conversion of bis-acrylic based provisional crown and fixed partial denture materials.
2. The degree of conversion of *fast set* TemPhase was lower than those of the others, LuxaTemp and Protemp 3 Garant. This difference was significant ($P < .05$).

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STATEMENT OF PROBLEM: The degree of conversion may influence the ultimate mechanical and physical properties of provisional crown and fixed partial denture materials. The high levels of the unreacted residual monomer may cause deleterious effect on the properties. **PURPOSE:** The purpose of this study was to measure the degree of conversion of bis-acrylic based provisional crown and fixed partial denture materials by using an infrared spectroscopic method. **MATERIAL AND METHODS:** Chemically activated three bis-acrylic based provisional crown and fixed partial denture materials, LuxaTemp [DMG, Hamburg, Germany], *fast set* TemPhase [Kerr, Orange, CA, USA] and Protemp 3 Garant [3M-ESPE, St Paul, MN, USA], were investigated by *Fourier transform infrared spectrometry* (FTIR). The FTIR spectra of the materials tested were immediately obtained after mixing. The specimens were stored under dry conditions and at 23°C for 24 hours, and then the spectra of the materials were also obtained. The degree of conversion (%) was calculated from the spectrum of the absorbance between the aliphatic double bond at 1637 cm^{-1} and the aromatic double bond at 1608 cm^{-1} using the baseline method. The data were statistically analyzed using one-way ANOVA and the multiple comparison Scheffe test at the significance level of 0.05. **RESULTS:** The mean value and standard deviation of the degree of conversion were 52.5 % ± 1.1 %, 50.3 % ± 0.8 %, and 42.3 % ± 4.9 % for LuxaTemp, Protemp 3 Garant and *fast set* TemPhase, respectively. There was no significant difference between LuxaTemp and Protemp 3 Garant, whereas there was a statistically difference between Protemp 3 Garant and *fast set* TemPhase, and LuxaTemp and *fast set* TemPhase ($P < .05$). **CONCLUSION:** The degree of conversion of fast set TemPhase was significantly lower than those of the others. The degree of conversion may be correlated with the rate of polymerization.

KEY WORDS: Degree of conversion, provisional crown and fixed partial denture materials

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Article history
Received November 26, 2008 Last Revison December 1, 2008 Accepted December 2, 2008.