

## Polymerization Efficiency of Glass-Ionomer and Resin Adhesives under Molar Bands

Daniel John Rejman<sup>a</sup>; Theodore Eliades<sup>b</sup>; Thomas G. Bradley<sup>d</sup>; George Eliades<sup>c</sup>

### ABSTRACT

**Objective:** To determine the degree of cure of a light-cured resin-modified glass ionomer (RMGI) under molar bands compared with a light-cured resin and a dual-cured resin.

**Materials and Methods:** The 3 cements used were Fuji Ortho LC, Eagle Spectrum resin, and Variolink II dual-cure. Each sample was indirectly light cured for 20 seconds (10 seconds occlusally, 10 seconds cervically) under sections of molar bands, and the degree of cure was evaluated with micro-MIR FTIR spectroscopy.

**Results:** The RMGI exhibited a significantly higher mean degree of cure (55.31%) than both of the resins (Eagle 19.23%; Variolink II, 25.42%), which did not differ significantly at  $\alpha = .05$  level of significance.

**Conclusion:** Higher degree of conversion can be obtained from RMGIs under molar bands compared with composite resin adhesives provided the proper curing technique is used.

**KEY WORDS:** Bands; Cement; RMGI; Cure

### INTRODUCTION

Despite the continued advances in bonding molar tubes, the use of molar bands continues to be popular.<sup>1</sup> Banded molars not only remain the standard for anchoring many appliances to the dentition, such as headgear and expansion devices, but they are also used in preference to bonded tubes based on the practitioner's discretion and patient variation. Because the retention of bands is achieved mechanically to the tooth by close adaptation and by the cement itself,<sup>2</sup> various properties of cements have been tested and developed to improve retention. Orthodontic applications usually involve thin layers of adhesives and lack areas of bulk material that would seem to favor chem-

ical cure systems. Orthodontic bands, however, are susceptible to areas of variable cement thickness<sup>3</sup> and present a physically larger barrier to irradiation than brackets. Orthodontic cements lend themselves differently to specific applications, and it is important to note the differences in polymerization kinetics and how this may relate to their utilization.

Light-cured resins consist of 2 main components: an organic matrix monomer and a powdered ceramic. Activation of free radicals is used to polymerize the unsaturated methacrylate monomer. Increased irradiation time and light intensity lead to higher strength because of the formation of a structure with a higher density of cross-links (increased degree of cure).<sup>4</sup> In practice, the dominance of surface properties over bulk properties favors the use of light-cured adhesive resins.<sup>4</sup> In dual-cured resin systems, polymerization is initiated by surface exposure to a curing light while the bulk of the material continues to cure by a chemical process. Benzoyl peroxide is used as an initiator, which is activated by a tertiary aromatic amine, and free radicals are formed by a multi-step process. Glass ionomers set as an acid-base reaction that leads to the formation of polycarboxylate salts that make up the cement matrix. Resin-modified glass ionomer (RMGI) cements are dual setting; upon mixing the liquid and powder the acid-base reaction occurs and the light-initiated free-radical polymerization of resin also occurs.

<sup>a</sup> Resident, Department of Developmental Sciences, Orthodontics, Marquette University, Milwaukee, Wis.

<sup>b</sup> Associate Professor, Department of Orthodontics, School of Dentistry, Aristotle University of Thessaloniki, Athens, Greece.

<sup>c</sup> Professor, Department of Biomaterials, School of Dentistry, University of Athens, Athens, Greece.

<sup>d</sup> Associate Professor, Department of Developmental Sciences, Orthodontics, Marquette University, Milwaukee, Wis.

Corresponding author: Dr Daniel John Rejman, Department of Developmental Sciences, Orthodontics, Marquette University, 1801 West Wisconsin Ave, Department of Orthodontics, Milwaukee, WI 53233 (e-mail: thomas.bradley@marquette.edu)

Accepted: June 2007. Submitted: February 2007.

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Particular advancements have been made in the properties of glass-ionomer cements, resin-modified glass ionomers, and compomers. The modified cements combine the favorable properties of glass ionomers: adhesion to enamel and metal, the ability to absorb and release fluoride, and the ability to chemically bond in the presence of moisture. The modified cements also include the favorable properties of resins: light curing for quick set and increased strength.<sup>5</sup> Because of their favorable properties, especially for banding molars, these new cements have replaced the zinc phosphate and polycarboxylate cements that were used in the past.<sup>2</sup>

Time to first failure, retentive strength, and shear-peel band strength of glass ionomers have been studied<sup>6,7</sup> but there is little information on the degree of cure of cements under molar bands, which obviously pose a larger physical barrier to light penetration than smaller brackets. A recent study showed that the amount of occlusal metal coverage significantly affects the depth of cure of two resin band adhesives, as determined by Fourier transform infrared (FTIR) spectroscopy.<sup>8</sup>

Previous studies on dental composites have shown that many characteristics of the material, including hardness, tensile and compressive strength, and flexural modulus depend on the degree of resin polymerization.<sup>9</sup> In particular, degree of cure modulates solubility and degradation, which affects the biological performance of the material.<sup>10</sup> Adverse effects could include bacteria reaching the enamel with resulting decalcification, monomer leaching, and the release of plasticizers, polymerization inhibitors, and inhibitors. These have been shown to have detrimental biological effects in cell cultures.<sup>10</sup>

The purpose of this study was to investigate the degree of cure of a light-cured RMGI under molar bands, compared with a light-cured resin and a dual-cured resin. This *in vitro* study sought to add further insight into the complex subject of polymerization and how it may relate to RMGI's use as proper band cement.

## MATERIALS AND METHODS

Fifteen orthodontic band strip samples measuring 4 mm × 8 mm were sectioned from intact first molar bands (Orthos 0.022, Ormco Corp, Orange, Calif) and divided into 3 groups of 5 bands each. A light-cured RMGI (Fuji Ortho LC, GC International, Tokyo, Japan), a light-cured resin (Eagle Spectrum, American Orthodontics, Sheboygan, Wis) and a dual-cure resin composite luting cement (Variolink II Dual Cure, Ivoclar Vivadent, Schaan, Liechtenstein) were selected for this investigation. The adhesives were mixed according to manufacturer's instructions, and approximately



Figure 1. Schematic representation of the specimen preparation.

15 mg of each was applied to the base of each band. The amount of material was weighed. This is important in establishing a reference or starting point in the sense that if a reduced amount is present, application of a standard pressure would result in different adhesive thickness compared with a bulk specimen. Any excess resin was removed from the specimen after application of a uniform pressure by the operator. The band samples were firmly pressed against a glass slide covered by a polystyrene strip (Figure 1). Excess resin was removed before polymerization. An Elipar Trilight curing light (ESPE, GmbH, Seefeld, Germany) was standardized at 950 mw/cm<sup>2</sup>. Each sample was indirectly irradiated for 10 seconds at the occlusal edge and 10 seconds from the cervical edge, with the tip of the curing light held at the edge of the band at a 30° angle. The time frame was chosen to allow for extended irradiation relative to the 10-second period used for irradiation of adhesives under brackets.

The plastic strips were removed, and the flat surface was placed in contact with a KRS-5 microcrystal (Th-ThBr,  $n = 2.4$ , 45° edge angle, 7 internal reflections, 10 × 5 × 1 μm). This was secured in a micro-multiple internal FTIR spectroscopy accessory (Perkin-Elmer Corp, Norwalk, Conn). The accessory was placed on a FTIR spectrometer (Perkin-Elmer 6x, Perkin-Elmer Corp). From each sample micro-multiple internal reflectance (MIR) FTIR spectra were obtained under the conditions: 4000 to 400 cm<sup>-1</sup> wave number range, 4 cm<sup>-1</sup> resolution, 30 scan transients. Additional spectra were collected from a sample of unpolymerized resin for each of the 3 adhesives to serve as a reference.

The spectra of each sample was analyzed, and the degree of cure (DC) was estimated for each using a relative percentage basis with the two frequency method and the tangent baseline technique.<sup>11</sup> For the resins, the aliphatic (C=C) bond stretching vibrations at 1638 cm<sup>-1</sup> were chosen as the analytical frequency, while the aromatic (C···C) bond stretching vibrations at 1605 cm<sup>-1</sup>, which are unaffected by the polymerization reaction were chosen as the reference frequency. For the Fuji RMGI, the ester (C=O) bond stretching vibrations at 1712 cm<sup>-1</sup>, which do not interfere with polymerization, were chosen as the reference frequency. The formula used to determine the % DC is:

$$\% \text{ DC} = 100 \times [1 - (A/B \times C/D)]$$

**Table 1.** Mean Percentage Degree of Cure for a Resin Adhesive, a Dual-Cure Resin, and a Resin-Modified Glass Ionomer<sup>a</sup>

	N	Mean % DC	SD	Tukey Grouping*
Eagle Resin	5	19.23	8.33	A
Variolink II	5	25.42	12.99	A
Fuji Ortho LC	5	55.31	5.71	B

<sup>a</sup> N indicates number of samples; DC, degree of cure; SD, standard deviation.

\* Means with same letter do not differ significantly at the  $\alpha = 0.05$  level.

### For Eagle and Variolink

- A: net peak absorbance area of the polymerized material at 1638  $\text{cm}^{-1}$
- B: net peak absorbance area of the unpolymerized material at 1638  $\text{cm}^{-1}$
- C: net peak absorbance area of the unpolymerized material at 1605  $\text{cm}^{-1}$
- D: net peak absorbance area of the polymerized material at 1605  $\text{cm}^{-1}$

### For Fuji Ortho LC

- C: net peak absorbance area of the unpolymerized material at 1712  $\text{cm}^{-1}$
- D: net peak absorbance area of the polymerized material at 1712  $\text{cm}^{-1}$

Analysis of variance (ANOVA) was used to compare the mean % DC of the different adhesives. Pairwise comparisons were made using Tukey's Studentized range test at  $\alpha = .05$  level of significance.

## RESULTS

The mean degree of cure was 19.23% for the Eagle resin, 25.42% for Variolink II, and 55.31% for Fuji Ortho LC (Table 1). Pairwise comparisons indicated (1) no significant difference between the mean % DC between the Eagle and Variolink resins; (2) a significant difference between the % DC of Fuji RMGI and Eagle resin; and (3) a significant difference between the % DC of Fuji RMGI and Variolink resin.

## DISCUSSION

In deciding what material to use for banding molars, the clinician should ask which properties prevail: thin layers of cement or areas with bulk volume. Banded teeth possess both; thin layers dominate when fit well, but areas of relative bulk remain, lending themselves well to dual-cured systems. The thin layers respond well to light curing, where the degree of carbon-carbon double-bond conversion is associated with curing time, light intensity, color shade, and filler loading.<sup>12</sup>

A study on curing patterns demonstrated that below

surfaces exposed to curing lights, there is adequate curing until a certain depth, where both remaining double bonds and Knoop's hardness pattern changed rapidly. This was attributed to the attenuation of the initiating light as a function of distance.<sup>12</sup> Thus, the variability of adhesive thickness, and the influence of the size of a barrier to the light (the molar band) should be expected to influence the degree of cure of a material. The technique used to measure the degree of cure analyzed the adhesives in the portion of the band where presumably the least amount of irradiation penetration occurred, and thus the least amount of polymerization. The spectrometer analyzed the surface of the materials at a mean depth of approximately 3  $\mu\text{m}$ ,<sup>9</sup> and it is this surface that would be in contact with the enamel of the tooth.

The main hypothesis tested in that study related to the extent of carbon double-bond conversion under a significantly larger obstacle (molar bands) than brackets. A previous study, using similar methods and curing parameters for upper incisor steel brackets, showed a 68% DC for a dual-cured resin and a 48% DC for a light-cured resin.<sup>13</sup> Although the resin manufacturers differed, the results of this study showed a 25% DC (dual-cured resin) and 19% DC (light-cured resin), suggesting that the effect of a larger barrier is considerable and leads to a reduced conversion relative to that obtained from brackets. The 55.3% DC obtained for the RMGI sample is significant compared with the resins, and this percentage also compares well to previous findings that 33%–55% of double bonds remain (45%–67% DC) after setting RMGI cements.<sup>14</sup> It could be implied that a favorable degree of cure of RMGIs can be obtained under molar bands. The clinician must keep in mind that the method of curing cements under bands can affect the DC considerably,<sup>8</sup> although the degree of cure affects the mechanical properties of resins; no information is currently available on degree of conversion's effect on RMGI's clinical performance.<sup>14</sup>

The RMGI setting consists of an acid-base reaction accompanied by a free radical polymerization reaction, and exposure of the cement to the curing light may inhibit the acid-base reaction rate (which leads to the formation of the polycarboxylate salt cement matrix).<sup>14</sup> A study examining the efficiency of RMGI's acid-base reaction has shown that the reaction was slowed by light exposure, but Fuji II LC does exhibit an acid-base reaction and resultant salt formation. The study also stated that there was little differences in top-surface and bottom-surface curing efficiency. This was attributed to small specimen thickness and the relatively transparent glasses used in modern RMGIs, which allow higher levels of light transmittance.<sup>15</sup> This may par-

tially explain the favorable DC of the RMGI in this study.

The Variolink II is also a dual-cure material, and final double-bond conversion may differ from those measured initially. A standardized backing was used for all the samples, but it can be presumed that under clinical conditions the enamel backing would differ in the amount of secondary illumination allowed.

## CONCLUSION

- The degree of cure of the resin modified glass ionomer under molar bands was significantly higher than that of the two resins that were tested.

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