

Effects of Time Delay on Degree of Conversion of Contemporary Orthodontic Band Adhesives

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ABSTRACT

Objective: For prolonged use in the oral cavity, orthodontic band adhesives should have certain physical properties to ensure the best clinical performance. The degree of conversion (DC) of orthodontic band adhesives has been found to affect their mechanical properties. The aim of this study was to investigate the effects of time delay on the DC of orthodontic band adhesives.

Materials and Methods: Fourier-transform infrared spectroscopy was used to evaluate the DC of 3 adhesives (Bisco Ortho Band Paste LC, Multi Cure Glass Ionomer Band Cement, Transbond Plus Light Cure Band Adhesive) immediately after they had been polymerized and stored in artificial saliva at $37 \pm 1^\circ\text{C}$ for 30 days. Thirty disc-shaped specimens (10 for each adhesive) were fabricated in Teflon molds (6.0×1.0 mm). The samples for each adhesive were divided further into 2 groups ($n=5$).

Results: The DC was significantly influenced by the band adhesive type ($p < 0.05$), and there were significant differences among band adhesive types for the DC after 30 days.

Conclusion: The value of the DC could change with time delay. (*Turkish J Orthod* 2013;26:23–29)

KEY WORDS: Band Adhesive, Degree of Conversion, Time Delay

INTRODUCTION

Hybrid ionomers have been improved by a number of combinations of glass ionomer cements and resin-based composites in recent years. Products with similar composition are also used as pit and fissure sealants and luting composites, and for crown buildups and the bonding of orthodontic brackets and bands. Still, the use of molar bands continues to be popular in orthodontics.¹ The retention of bands to the tooth is achieved mechanically by close adaptation and by the band adhesive itself.² Light-activated band adhesives are very common among orthodontists because of the rapid strength development, shortened setting time (thus, also shorter chair time), increased duration of effectiveness, decreased effects of saliva, metal surface for adherence, and unetched enamel.^{3–6}

Light-activated orthodontic band adhesives are

different from restorative adhesives, as the former are being used in a thinner layer with a greater depth between the teeth and the bands.⁶ For instance, in a tooth with a large metal restoration, the light path through the tooth crown is interrupted, so the adhesive might not polymerize sufficiently because less light can penetrate.⁶ Clinically, therefore, inadequate polymerization would occur because of difficulties both in illuminating the band adhesives evenly from each side of the band/tooth and in reaching the posterior of the oral cavity with the light source.

Although resin-modified glass ionomer cements (RMGIC) or modified composites as orthodontic band adhesive materials are nowadays welcome,

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an inadequate polymerization by means of an inadequate degree of conversion (DC) of the orthodontic band adhesive materials is still a problem for orthodontists. The DC of adhesives is the extent to which the C=C of monomer are converted into C-C to form polymers during setting reactions.⁷ Ideally, the adhesive resin material would have all of its monomer converted to polymer during the polymerization reaction.⁸ However, all dimethacrylate monomers exhibit considerable residual monomer in the final product, with a DC ranging from 36 to 72% under conventional irradiation conditions.⁹ Rejman *et al.*¹⁰ found DCs of 19–55% for Fuji Ortho LC, Eagle Spectrum resin, and Variolink II. Kuappi and Combe¹¹ found DCs of 79–89% for Fuji Ortho LC.

The dissolution or elution of leachable components of adhesive resin may present a deleterious effect in the polymeric network of the material, modifying its structure physically and chemically at short or long periods of time in moisture.¹² If an optimum degree of cure cannot be achieved for band adhesives, it might cause an increase in water sorption, decrease in hardness, and softening of the polymer matrix.¹³ In addition, monomer conversion of resin composites at 72- and 120-hour delays provided higher values than the 5-minute-delay composite group.¹⁴ Therefore, there is a scarcity of data in the literature to date on the DC of orthodontic band adhesives and the length of time of the DC after light polymerization.^{6,10} Due to prolonged use in the oral cavity, the orthodontic band adhesives should have certain physical properties to ensure the best clinical performance.

However, the relative contributions of these variables to the DC are still unknown. In almost all studies concerning the DC, DC values were measured for 24 hours, but there were no comparisons about any differences in DC in moisture after time delay.⁶ The information that would be obtained from such a comparison might be regarded as essential for the development of new strategies to increase DC. Therefore, the objective of the present study was to investigate whether storage in artificial saliva for a definite time period would affect the DC of orthodontic band adhesives. The first null hypothesis of the present study was that there would be differences in DC values of orthodontic band adhesives after illumination, and the second one was that DC values would increase with time in artificial saliva.

MATERIALS AND METHODS

The orthodontic band adhesives selected were Bisco Ortho Band Paste LC (BO Band; Bisco, Inc, Schamburg, IL, USA), 3M Unitek Multi Cure Glass Ionomer Band Cement (MC GIC; 3M Unitek Orthodontic Products, Monrovia, CA, USA), 3M Unitek Transbond Plus Light Cure Band Adhesive (TB Plus; 3M Unitek). BO Band and TB Plus are light-curing orthodontic band adhesives, and MC GIC is a tri-cured orthodontic band adhesive. Abbreviations, basic ingredients, average filler weights, and lot numbers of adhesives can be seen in Table 1. An Elipar FreeLight 2 (3M ESPE Dental Products, St Paul, MN, USA) was used as a light curing device having a tip of 8 mm with light intensity of 1260 mW/cm².¹⁵

Thirty disc-shaped samples, 6 mm in diameter and 1 mm in depth, were fabricated in Teflon molds between glass slides (2 mm) at room temperature. The manufacturers' instructions were strictly followed for preparing the tested materials. MC GIC was prepared by dispensing 1 level powder scoop (large) and 3 drops of liquid onto the mixing pad with a large cement spatula and then placing the mixture in the moulds. BO Band and TB Plus were directly injected into the moulds. The slides were gently pressed together, and excess material was removed with a spatula. Standardization of the distance between the light source and samples was provided by the thickness of the glass slide; these glass slides also provide a smooth surface for testing. Ten samples for each adhesive were coded according to the brand of adhesive. After that, each adhesive group was divided into 2 subgroups (n=5).^{8,16} These samples were left in artificial saliva at 37 ± 1°C until the appropriate time elapsed, and then were and stored in 6 light-proof boxes coded to the different adhesives to avoid further exposure to light. The time intervals between initial light cure and DC evaluation were 10 minutes and 30 days.¹⁶

Following initial light cure and 30-day storage, each sample was pulverized into fine powder with a percussion mortar and transparent pestle. Fifty micrograms of the ground powder was mixed with 5 mg of potassium bromide powder (Carlo-Erba Reagenti, Milan, Italy) and pressed by a hand press machine to produce a thin potassium bromide pellet. The pressed sample was placed in a microtransmission holder of a beam-condensing unit. A Fourier-transform infrared spectrometer (FTIR; 1600 Series; PerkinElmer, Waltham, MA, USA) was used

Table 1. The orthodontic band adhesives included in the study

| Brand ^a | Basic Ingredient | Filler Weight (average) % | Company | Lot No. |
|--------------------|---|---------------------------|---|-------------|
| Bisco Ortho Band | Bisphenol A diglycidylmethacrylate, dental glass, amorphous silica, sodium fluoride | 81 | Bisco, Inc, Schamburg, IL, USA | 0600004600 |
| Multi Cure GIC | Polycarboxylic acid copolymer, 2-hydroxyethyl methacrylate, water, silane-treated glass, potassium persulfate | 65 | 3M Unitek Ortho Prod, Monrovia, CA, USA | 6CY/6FN0808 |
| Transbond Plus | 2-Hydroxy-1,3-dimethacryloxypropane, citric acid dimethacrylate oligomer, silane-treated glass, silane-treated silica | 77.5 | 3M Unitek Ortho Prod, Monrovia, CA, USA | 6EX0809 |

^a Abbreviations: Bisco Ortho Band, Bisco Ortho Band Paste LC; Multi Cure GIC, 3M Unitek Multi Cure Glass Ionomer Band Cement; Transbond Plus, 3M Unitek Transbond Plus Light Cure Band Adhesive.

to evaluate the DC. Spectral absorption peak heights were recorded then analyzed using the diffuse-reflection mode of the FTIR.

Scan range was 4400–4450 cm^{-1} , and scanning speed was 60 cm^{-1} per minute for FTIR spectra. The spectral resolution for this work was 16.0 cm^{-1} . Spectra were also acquired from the same number of unpolymerized adhesives. For this, unpolymerized pastes were smeared onto thin potassium bromide pellets. The number of double vinyl bonds remaining in the sample exposed to irradiation is shown by the intensity of the peak at 1637 cm^{-1} , referring to the carbon-carbon double bond (C=C) stretching of the vinyl group, and has been used in the study of polymerization of acrylates and methacrylates.¹⁷ The DC was directly related to the decrease of 1637 cm^{-1} absorption on the FTIR spectra as follows:

$$DC = [(A_0 - A_t)/A_0] \times 100,$$

where A_0 is the absorption of the peak at 1637 cm^{-1} , when time is equal to 0, and A_t is the absorption at time t .¹⁸

The C=C bonds on the methacrylate groups are converted to the carbon-carbon single bonds (C–C) when polymerization occurs. For this reason, when the monomer is cured with light, the peak height of the 1637 cm^{-1} wave number is divided by the uncured value (the peak height is higher), and, thus, the polymerization degree (DC) is calculated. The polymerization degree was calculated by using the $DC = [(A_0 - A_t)/A_0] \times 100$ equation. A_0 is the absorbance of the uncured materials (the polymerization didn't start at this time), and A_t is the

absorbance of the cured materials (the polymerization finished at this time).

The data were analyzed with SPSS 13.0 (SPSS, Inc, Chicago, IL, USA). Shapiro-Wilk and Levene tests were used to test the distribution of normality and homogeneity of variances. As the data distribution was normal in each group, and group variances were homogeneous, 2-way ANOVA was applied. Multiple comparisons were done with the post hoc Tukey honestly significant difference (HSD) test. Statistical significance was set as $p < 0.05$.

RESULTS

According to 2-way ANOVA, the DC was significantly influenced by the band adhesive type ($p < 0.05$), and there were significant differences among band adhesive types for the DC after 30 days. Interaction of the orthodontic band adhesives and the time delay was not found to play a statistically significant role in the DC ($p > 0.05$) for some samples, but time by itself had a statistically significant influence on the DC ($p < 0.05$).

Table 2 and Figure 1 summarize the mean, SD, and Tukey grouping of the DC for 3 commercially available orthodontic band adhesives after initial light cure and 30 days.

Among the materials, the highest mean DC value was obtained from TB Plus, and the lowest mean DC value was observed in MC GIC band adhesive samples. Tukey HSD comparisons indicated that (1) there were no significant differences between DC values of BO Band and TB Plus band adhesive samples, (2) the only difference was in the T1 (initial light cure) of DC values of TB Plus and the T1 and T2 (after 30 days) values of MC GIC, and (3) the

Table 2. Degree of conversion (DC) values with time delay

| Composites ^a | Time | n | DC | | Tukey Grouping ^b |
|-------------------------|------|---|-------|-------|-----------------------------|
| | | | Mean | SD | |
| Transbond Plus | T1 | 5 | 87.13 | 7.34 | A |
| | T2 | 5 | 75.61 | 9.16 | AB |
| Bisco Ortho Band | T1 | 5 | 77.94 | 4.16 | AB |
| | T2 | 5 | 71.12 | 6.71 | AB |
| Multi Cure GIC | T1 | 5 | 69.48 | 11.76 | B |
| | T2 | 5 | 61.07 | 11.52 | B |

^a Abbreviations: Bisco Ortho Band, Bisco Ortho Band Paste LC; Multi Cure GIC, 3M Unitek Multi Cure Glass Ionomer Cement; Transbond Plus, 3M Unitek Transbond Plus Light Cure Band Adhesive; T1, initial light cure; T2, after 30 days.

^b Groups with the same letter indicate no significant difference at $\alpha = 0.05$ level.

mean DC values of all band adhesives were decreased after 30 days.

DISCUSSION

In the present study, the DC of orthodontic band adhesives was measured by using FTIR. The results of this study revealed that there were statistically significant differences between the DCs of orthodontic band adhesives after initial curing. Furthermore, the contact time with the aqueous media had a significant role in the DC of orthodontic band adhesives, and the DC of adhesives decreased with time in the aqueous media. The overall DC values of different adhesives were also statistically different. Therefore, the first null hypothesis was accepted, but the second one was rejected.

Among some methods, FTIR has been selected as an appropriately analytical and powerful technique and has been widely used as a reliable method, as it detects the C=C stretching vibrations directly before and after curing of materials.^{8,16,17,19,20} However, the FTIR technique has

some limitations, such as the time-consuming nature of the protocol of evaluating the DC of a bulk composite by FTIR and also the need to pulverize prepared samples.¹⁹ Furthermore, the depth of cure could not be estimated when using this method, so differences in the level of polymerization between the top and bottom layers of the sample were imperceptible. Therefore, the average values for the DCs of the band adhesive materials observed in this study are from the bottom layers of the band adhesive materials. The relatively small sample size of specimens per test group ($n=5$) might be considered another limitation in the present study; however, this sample size has been recommended as optimal for studies of this nature, and some investigations with a smaller sample size could be found in the orthodontic literature.^{8,10,16}

In the current investigation, 3 orthodontic band adhesives were selected because they were the most widely used, and all were light-activated adhesives, so the DC could easily be investigated. Two of the band adhesives were modified composite, and the last was a resin-modified glass ionomer cement (RMGIC). All of them were flowable, opaque, and manufactured for orthodontic band cementing. In addition, factors influencing the transmission of light, such as thickness of the band adhesive material and distance of light tip to sample surface, were standardized, and the light source for curing and storage conditions were the same. Hence, in the present investigation, any differences in the DC could be attributed to the differences in compositions and types of materials.

Our results showed that the DC values were much higher for TB Plus than for MC GIC. The DC is dependent upon the light permeability of the filler, as well as the monomer composition, type, concentration, inhibitor, curing time, and accelerator in the

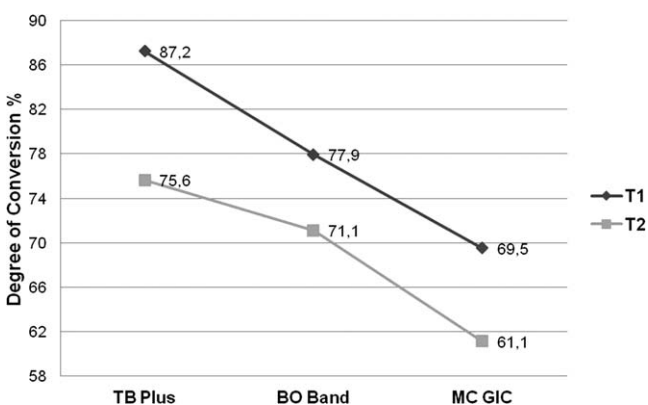


Figure 1. Degree of conversion of orthodontic band adhesives.

resin materials, and also the curing way of adhesives.^{6,8,10,11,14,19} Materials used in this study include different inorganic filler types and monomer compositions (molecular structure and shade; Table 1). Moreover, MC GIC, an RMGIC, consists of two main components: an organic matrix monomer and a powdered ceramic. It could be prepared via dispensing of powder and liquid by a clinician, but the others are modified composites and no mix or one paste. The clinician's skills can be an explanation as to what might affect quality of band adhesive or filler composition.

Additionally, MC GIC is a tri-cured RMGIC for curing mechanisms, which are acid-base reaction, light curing, and self-curing. TB plus and BO band are light-activated, modified composites for curing mechanisms. A previous study¹⁰ using different adhesives affirmed that a favorable DC of RMGICs (55% for Fuji Ortho LC) can be obtained under molar bands. This was in contrast with the DC of the light-cured resins (81% DC and 78% DC) of the present study, but closer to that of the RMGIC (69% DC). Direct irritation of samples in the present study might be a reason for the DC of the adhesives. Another investigation using similar sample preparation technique showed a 79–89% DC for a tri-cured resin.¹¹ That was in contrast with the current study. The characteristic of continued polymerization via chemical curing of RMGICs might have caused this differentiation after initial light exposure. Also, with the MC GIC samples, the differentiation may have been a result of the overlap of the acid-base reactants on the spectrum and the diminished resolution of the carbon-carbon analytical peak as polymerization occurred. Therefore, determination of chemical conversion could be difficult, and the DC values of the MC GIC samples would be lower than the others.

Another result derived from this study was that the mean DC values of all tested adhesives decreased after 30 days, which may be a proper aging time.¹⁶ This result was in disagreement with the finding of Loza-Herrero *et al.*,¹⁴ as we determined a definite time. They investigated both an unheated control group and a postcure heating delay group to determine conversion of resin composites and summarized that the DC values of unheated cured-resin materials increased after 76 and 120 hours and decreased remarkably after postcured heated-resin materials with time elapsed. Corekci *et al.*¹⁶ used a similar study design for orthodontic composites, and their results were contrary to ours: there was no

statistically significant influence for time on the DC. At this point, it should be clarified that on one hand, the type of band adhesive used could affect the DC considerably; on the other hand, the DC has been found to affect the mechanical properties of orthodontic band adhesives. Nonetheless, no information is currently available on the effect of DC on the clinical performance of orthodontic band adhesives.²¹ Therefore, for prolonged use in the oral cavity, the orthodontic band adhesives should have certain physical properties to ensure the best clinical performance.

Mouth-simulating conditions of the present study might be a reason for declining the DC values of the band adhesives. Firstly, the artificial saliva in etuve temperature could penetrate as water into the resin matrixes of band adhesives. Secondly, this action may result in chain depolymerization by means of hydrolytic degradation.²² Because there is a definite link between water sorption, solubility, and polymerization, the increased monomer concentration and carbon double-bond unsaturation might predispose the material to potent degradation reactions.²² Therefore, the polymer chain splits, and some derivatives may become free radicals or an inactive species. Degradation reactions could be a reason for the reduction in the DC values of the adhesives. On the other hand, 30 days may be enough time for chain depolymerization of tested adhesives in aqueous media at $37 \pm 1^\circ\text{C}$.

The ratio of filler to resin is important because the lower the proportion of filler, the easier the light penetrates the composite.¹⁹ The percentage of filler in the tested resins of the current investigation ranged between 65 and 81% by weight (Table 1). Although it is claimed that type, size, and percentage of filler in resin material may influence polymerization and DC, the results of the present study indicated that the DC may not be dependent upon the percentage of filler for the orthodontic band adhesives.^{3,17} The BO Band revealed approximately the same DC values as the MC GIC; however, they have different ratios of filler to resin than does the TB Plus. On the other hand, TB Plus, which has a relatively high percentage of filler content, revealed the highest DC values, and this result is in contrast with Yoon *et al.*¹⁹ As a result of filler content, oxygen may penetrate into the adhesive from the surface; therefore, oxygen diffusion occurs via new pathways absorbed by the filler particles. Of our tested adhesives, MC GIC must be mixed by a clinician, and that might be a problem for the internal

homogeneity of the adhesive. Reduction of the DC may occur at the composite/atmosphere interface because of the increased oxygen solubility of the uncured resin.²³ For these conditions, our results on band adhesives should be interpreted carefully. Even so, the clinical significance of the findings in this study is that the TB Plus adhesive exhibited higher DC values than the MC GIC adhesive. Since orthodontic bands will be in the mouth for a long time period, these properties should be considered in order to ensure good clinical performance.

Oestrogenity is an essential factor for adhesive resin materials because of bisphenol A (BPA). BPA exhibits oestrogenity²⁴ and could be detected in serum,²⁵ breast milk,^{25,26} and dental cement.²⁷ BPA is a small molecule that is used as a Bis-GMA monomer precursor in polymerization reactions to produce adhesive resin materials. Eliades *et al.*²⁸ studied the DC of resin materials and reported a statistically significant linear correlation between the DC of directly irradiated specimens and the Bis-GMA concentrations eluted by these groups of specimens. They concluded that very little or no Bis-GMA leach was present when the DC reached 55–60%.²⁸ In the present study, only BO Band contains Bis-GMA, and its DC values reached up to 71–78% (Table 1), which might be adequately high to prevent monomer leach from the orthodontic band adhesive tested. Rejman *et al.*¹⁰ reported overall DC values of 19–55%, and Namura *et al.*⁶ determined overall DC values 41–86% in their studies. However, this was a hypothesis and should be supported with further research.

CONCLUSIONS

Within the limitations of this study, the following conclusions can be drawn:

- DC was found to be dependent on the band adhesive type. BO Band and TB Plus showed high levels of DC, and the DC values of TB Plus were significantly higher than those of MC GIC.
- The value of DC could change within 1 month, and all adhesives were similarly affected under time-delay conditions.

In future *in vivo* or *in vitro* studies, the contemporary orthodontic band adhesives should be investigated alongside the level of the DC for a long period of time (approximately 2 years).

Time delay affects the degree of cure of orthodontic band adhesives. Since orthodontic bands will be in the mouth for a long time period, these properties should be considered in order to ensure good clinical performance.

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