Color Characterization of Color Changing Orthodontic Adhesives

Megan Fried
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COLOR CHARACTERIZATION OF COLOR CHANGING ORTHODONTIC ADHESIVES

by

Megan M. Fried, D.D.S

A Thesis submitted to the Faculty of the Graduate School, Marquette University, in Partial Fulfillment of the Requirements for the Degree of Master of Science

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ABSTRACT
COLOR CHARACTERIZATION OF COLOR CHANGING ORTHODONTIC ADHESIVES

Megan M. Fried, D.D.S
Marquette University, 2022

Objective:
A popular product to facilitate removal of excess adhesive during bonding of orthodontic appliances is a color changing adhesive. Before polymerization, these materials are a bright color but upon curing, manufacturers claim they become translucent. After debonding, there is often resin embedded within the enamel, so the appearance of these materials after polymerization can affect the esthetics of a patient’s dentition. The purpose of this study was to analyze the color characteristics of various orthodontic color changing adhesives and determine if there is a significant difference in color changing capabilities and in translucency after polymerization between products.

Methods:
Grengloo and Blugloo by Ormco, BracePaste Color Change by American Orthodontics and Transbond PLUS by 3M Unitek were evaluated. As a non-color changing control, Transbond XT by 3M Unitek was analyzed. A Teflon mold was used to fabricate uniform discs of adhesives (n=10/adhesive). A spectrophotometer was used to analyze the color (L*a*b* values), color change (ΔE_ab*) and translucency at various polymerization stages. The samples were analyzed at 20.6°C and at 37.0°C. Specimens were stored in Fusayama Meyer artificial saliva. Statistical analysis was completed with ANOVA, pairwise comparison analysis, t-test and response profile analysis. P values less than 0.05 were considered significantly different.

Results:
All materials demonstrated an increase in L* upon polymerization, but there was no trend in the changes noted with a* and b* values. There was a significant (p < 0.05) difference in the color change between the materials during all stages of polymerization. Transbond Plus showed the largest color change during initial polymerization, and Grengloo had the smallest change. Upon a decrease in temperature, Transbond XT and BracePaste had minimal color change, and Blugloo and Grengloo had the largest change. Each color changing composite had significantly (p < 0.05) less translucency when compared to Transbond XT.

Conclusion:
All of the color changing composites that were evaluated have significantly different color changing capabilities. Temperature plays a significant role in the appearance of Blugloo and Grengloo. The translucency after polymerization of all the color changing composites is significantly less than Transbond XT, with Blugloo and Grengloo having the largest difference.
I would like to thank Dr. David Berzins and Dr. Shengtong Han for their help in completing this thesis. This project could not have been completed without their knowledge, support, and guidance. I would like to thank the Marquette faculty and the director, Dr. Liu. I would like to thank my co-residents for being such a collaborative group of people to spend residency with. I would like to thank the Graduate School and all the staff in the Orthodontics Department at Marquette University. I would like to thank my husband for his unwavering support throughout my journey to become an orthodontist.
TABLE OF CONTENTS

ACKNOWLEDGEMENTS.................................................................................................i

TABLE OF CONTENTS...............................................................................................ii

LIST OF TABLES..........................................................................................................iii

LIST OF FIGURES......................................................................................................iv

CHAPTER 1...................................................................................................................1

CHAPTER 2..................................................................................................................5

CHAPTER 3..................................................................................................................14

CHAPTER 4..................................................................................................................21

CHAPTER 5..................................................................................................................28

CHAPTER 6..................................................................................................................33

REFERENCES..............................................................................................................34
LIST OF TABLES

Table 1. Study parameters for each orthodontic adhesive sample…………………………18

Table 2. L*a*b* values for each material with cling wrap in place……………………………21

Table 3. L*a*b* values for each material with no cling wrap in place…………………………21

Table 4. L*a*b* values for each material with no cling wrap in place…………………………22

Table 5. Color change ($\Delta E_{ab}^*$) values for each material upon polymerization…………23

Table 6. Color change ($\Delta E_{ab}^*$) values for each material upon change in temperature…..25

Table 7. $p$ values from two sample T test of L*a*b* values for each material at 37.0°C and 20.6°C………………………………………………………………………………………………………..26

Table 8. Translucency parameter values for each material……………………………………27

Table 9. Translucency parameter comparison of each material to Transbond XT………27
LIST OF FIGURES

Figure 1. Teflon molds used to prepare samples..................................................15

Figure 2. Preparation of Grengloo in teflon mold.............................................15

Figure 3. Grengloo sample prior to polymerization on spectrophotometer.............16

Figure 4. Example of L*a*b* reading of a specimen on the spectrophotometer..........17

Figure 5. Transbond Plus post-polymerization with cling wrap in place on spectrophotometer.................................................................17

Figure 6. Transbond Plus post-polymerization without cling wrap in place on spectrophotometer.................................................................18

Figure 7. Samples in artificial saliva at room temperature.................................19

Figure 8. L* values before and after polymerization for each material...............23

Figure 9. a* values before and after polymerization for each material...............24

Figure 10. b* values before and after polymerization for each material...............24

Figure 11. Visual appearance of samples at varying temperatures....................26
CHAPTER 1
INTRODUCTION

The bonding of fixed appliances in orthodontic treatment took many years in the making before it became a routine part of the clinical procedure. The initial appliances utilized in orthodontics involved clamp bands, which were tightened around teeth with a screw attachment (Proffit et al., 2019). Eventually, preformed steel bands that were cemented to the teeth became widespread and used for many years (Proffit et al., 2019). After several decades of the orthodontic profession utilizing bands routinely in orthodontic treatment, Dr. George Newman and Dr. Fujio Miura pioneered the idea of bonding orthodontic brackets to enamel in the mid-1960s (Gange et al., 2015). However, years were spent attempting to develop the proper material and technique for orthodontic bonding. Various bonding materials were examined, along with numerous bracket materials and differing designs of bracket bases (Gange et al., 2015). The formula of the bonding product used today was finally perfected in 1980 by Dr. Byoung Suh (Gange et al., 2015).

Successful bonding of the orthodontic bracket to the tooth is reliant on the attachment base, the tooth surface and its preparation, and the bonding material itself (Proffit et al., 2019). A successful bonding material must have several criteria in order to be clinically acceptable. The material must be dimensionally stable; fluid enough to penetrate into the prepared enamel surface, yet tacky enough to hold its shape; must have excellent inherent strength documented by laboratory testing; and must be easy to use clinically (Proffit et al., 2019). Light-activated composite resin is the most commonly used bonding material today and meets that criterion. In addition, these materials also
provide a controlled polymerization time, more accurate bracket placement and easier removal of excess adhesive prior to curing (Bayani et al., 2015).

Once bonding fixed appliances became routine in orthodontics, two methods of bonding arose to adhere brackets to the dentition: direct bonding and indirect bonding. For direct bonding, the practitioner positions the bracket intraorally, and the ideal position of the bracket is determined during the bonding procedure. The advantages of direct bonding is that it is easier, faster, and less expensive (Proffit et al., 2019). However, the major disadvantage is the accuracy of bracket placement, as the decision of the ideal placement must be made quickly during the bonding procedure (Proffit et al., 2019). Indirect bonding has the bracket positioned in a laboratory on a dental cast (or virtually) and a tray is made to transfer the brackets to the dentition for bonding. The obvious advantage to this is more accurate bracket placement, but the disadvantage is that it can be a laborious process. Both techniques are deemed clinically acceptable and are used routinely in orthodontics (Proffit et al., 2019).

When utilizing the direct bonding technique, as many providers do, an ample amount of bonding material must be applied to cover the base of the bracket. When applied to the tooth, the bracket should be properly positioned and then pressed against the tooth surface to ensure that the bracket is flush against the tooth. Excess material is then present around the margins of the bracket. This material, also known as flash, should be removed prior to final positioning and curing of the bracket. Improper removal of excess adhesive material from the tooth surface when placing fixed appliances can lead to gingival irritation and white spot lesions (Bayani et al., 2015). In addition, excess
adhesive material that is not removed may stain during treatment, leading to an unattractive appearance.

The demand for esthetic orthodontic treatment is continually increasing, so it is important to consider each component of the fixed appliance system and how to optimize esthetics for each patient. The bonding material selected plays a role in the appearance of the fixed appliances. In particular, the removal of the bonding material during the bonding process to minimize flash is critical to the esthetics of the fixed appliance system for the reasons stated above. Although light-cured adhesives allow for easier removal of excess adhesive prior to curing, the concern of improper removal remains (Bayani et al., 2015). In order to address this concern of improper removal of the bonding agent, a popular product that can be selected to facilitate removal during bonding is a color changing adhesive.

Color changing adhesives provide a distinct color prior to polymerization, but upon curing, manufacturers claim that the adhesives become translucent or clear (depending on the product). American Orthodontics (BracePaste Color Change), 3M Unitek (Transbond PLUS) and Ormco (Grengloo and Blugloo) have all developed their own versions of color changing adhesives. The American Orthodontics and 3M Unitek materials both become activated during the light curing process, transforming the material into one that is claimed by the manufacturers to have acceptable esthetics (Ekhlassi et al., 2011). The Ormco products are a dual color changing adhesive. At cooler temperatures they have a distinct color, but at a warmer body temperature, the manufacturer claims that the material becomes translucent (Ekhlassi et al., 2011).
material therefore also has the capability to have its distinctive color returned when cooled with air or water (Ekhlassi et al., 2011).

Although these color changing adhesives aid in removal of excess adhesive material during the bonding of fixed appliances, it has been shown that upon debonding there is often resin adhesive that is left on the tooth surface even after cleanup (Bonetti et al., 2011). Because of the resin that is often left embedded in the enamel after bracket removal, the appearance of the adhesive following polymerization can play an important role for the esthetically minded patient after fixed appliance removal. Although the manufacturers claim that the color changing composites can become clear or translucent, it is uncertain to what degree. Therefore, it is possible that the different color changing adhesives can have varying appearances after they have been cured. The purpose of this study was to analyze the color characteristics of various orthodontic color changing adhesives and determine if there is a significant difference in the color changing capabilities and in the translucency after polymerization between the products utilized by orthodontists.
CHAPTER 2
LITERATURE REVIEW

Shear Bond Strength of Color Changing Orthodontic Adhesives

When deciding what adhesive to use in orthodontics, the shear bond strength of the material is of high importance. With color changing adhesives gaining popularity in orthodontics, questions have arisen on whether the color additives influence bond strength. Therefore, several studies have been done that evaluated the bond strength of orthodontic color changing adhesives. Researchers studied the shear bond strength of color changing adhesives on extracted premolar teeth using a Universal Testing Machine (Turkkahraman et al., 2010). Grengloo, Blugloo, Transbond Plus and Light Bond (control) were the materials studied. The results indicated that all color changing adhesives displayed acceptable bond strengths and can be safely used in orthodontics. There was no significant difference in shear bond strength between the various color changing adhesives. However, there was a significant difference between Light Bond and Transbond PLUS. Although Transbond PLUS showed the lowest shear bond strength value, it still had acceptable bond strength to be used in orthodontics (Turkkahraman et al., 2010).

Multiple studies have been done to compare the bond strength of various color changing adhesives at different time intervals after bonding. One study compared the shear bond strength of Transbond PLUS, Grengloo, Blugloo and Trans bond XT (control) on bovine incisors at 15 minutes and 24 hours after bonding with the use of a Universal Testing Machine (Duers et al., 2011). They found that all four orthodontic bonding adhesives demonstrated bond strengths that were clinically acceptable. All adhesives
except for Grengloo had higher shear bond strength at 24 hours than 15 minutes.

Grengloo at 15 minutes displayed the highest shear bond strength, but at 24 hours showed
the lowest shear bond strength of all the adhesives (Duers et al., 2011).

Another study compared the shear bond strength of Transbond PLUS and
Transbond XT (control) on extracted premolars at 15 minutes, 24 hours and 1 week after
bonding with the use of a Universal Testing Machine (Youssefinia and Mortezai 2018).
They found that the lowest shear bond strength tested was Transbond XT at 1 week after
bonding. However, there was no significant difference in the shear bond strengths
between the adhesives for all time points (Youssefinia and Mortezai 2018). Ekhlassi and
colleagues also examined the shear bond strength of Transbond PLUS, Grengloo and
Transbond XT (control) at 15 minutes, 24 hours and 1 week after bonding (Ekhlassi et
al., 2011). They also found that there was no significant difference in shear bond strength
of the adhesives at various time points (Ekhlassi et al., 2011). Others have investigated
the effect of light-curing time on the shear bond strength of various color changing
adhesives. Bayani et al. (2015) collected extracted premolar teeth and bonded a metal
orthodontic bracket on the buccal surface. Greengloo, Transbond PLUS, and Resilience
(conventional light-cured adhesive) were selected as the bonding materials for the various
groups. For each adhesive, the specimens were light-cured for two different times of 20
seconds and 40 seconds. The specimens underwent mechanical testing using a Universal
Testing Machine to analyze shear bond strength. The adhesive remnant index was then
used to assess the amount of adhesive remaining on the tooth surface. The authors found
that decreasing the light-curing time from 40 to 20 seconds decreased the shear bond
strength of the tested adhesives, but it was only significantly decreased in the Transbond
PLUS adhesive. They also found that Grengloo adhesive demonstrated higher shear bond strength than the Transbond PLUS adhesive when light-cured for 40 seconds (Bayani et al., 2015).

**Fixed Appliances and Enamel Color**

With all the elements involved with orthodontic treatment, there is inevitably structural defects of the enamel that occur (Pandian et al., 2017). Enamel can be altered from etching procedures, decalcification and from clean-up procedures at the debonding phase which can lead to microcracks and scratches (Pandian et al., 2017). All of these factors can result in a change in the enamel color (Pandian et al., 2017). In addition to alterations in the enamel itself, it has also been found that resin adhesive is often left on the tooth surface after bracket removal (Fields, 1982). The enamel etching procedure results in the formation of resin tags during bonding because of penetration and polymerization of the bonding adhesive (Pandian et al., 2017). Clean-up procedures cannot completely remove the resin tags without damaging the enamel surface, and therefore resin may remain embedded in the enamel after orthodontic treatment (Pandian et al., 2017 and Bonetti et al., 2011).

An important esthetic aspect of assessing a finished orthodontic case is tooth color (Pandian et al., 2017). It has been shown that part of patient satisfaction after finishing orthodontic treatment is the color of enamel (Pandian et al., 2017). Therefore, more research is being done to evaluate how orthodontic treatment affects enamel color. A systematic review was done to evaluate this, and several results were drawn from the review (Pandian et al., 2017). It was determined that significant color change does occur
following orthodontic treatment, but as alluded to above, this can be for a variety of factors (Pandian et al., 2017). Some of the studies reviewed showed that the etching system used prior to bonding can have an effect on enamel color because this relates to the length of the resin tags in enamel (Pandian et al., 2017). A significant positive correlation exists between enamel color change and resin tag depth (Pandian et al., 2017). A study by Zaher et al. (2012) showed less color change in self-etching systems than the conventional system.

The systematic review also evaluated light-cure systems, showing no significant color difference in enamel between the various systems (Pandian et al., 2017). It reviewed light-cure and chemically cure resins, and two studies showed that the chemical cure resins had significantly increased color change in enamel compared to the light-cure composites (Pandian et al., 2017). The review highlighted that clean-up procedures do influence the color changes in enamel significantly (Pandian et al., 2017). Trakyali et al. (2009) demonstrated that using tungsten carbide burs alone during clean-up can lead to increased surface roughness, which can increase the stain susceptibility of the enamel (Trakyali et al., 2009 and Joo et al., 2011).

Because there are so many components of orthodontic treatment that can affect the color of enamel, more long-term studies are needed to further evaluate surface roughness, corrosion products, stain susceptibility and residual resin tags and what role they play in enamel color (Pandian et al., 2017). More specifically, because the impact that resin tags likely have on the long-term esthetics of the tooth, it is important to be mindful of this when selecting the orthodontic bonding adhesive. Although the actual amount of resin that may remain on the tooth after fixed appliance treatment is minimal,
it is feasible that the color of the adhesive itself can impact the esthetics of the tooth. With color changing orthodontic bonding adhesives becoming more popular, the extent of the color changing capabilities of the adhesives and how clear they actually become after polymerization is a consideration in the overall esthetics of orthodontic treatment. However, there is a lack of analysis of the actual color changing characteristics of the various color changing orthodontic adhesives on the market. This study served to fill this void and provide further insight on the properties of color changing adhesives.

**Alternative Adhesive that can be Utilized in Orthodontics**

In attempt to reduce the quantity of adhesive left on the tooth surface after debonding fixed appliances, researchers have explored the use of alternative materials that may aid in the clean-up process. One material of interest is an orthodontic bonding adhesive that contains fluorescent color that is inducible by visible light, such as from a dental curing unit (Namura et al., 2010). Materials like this have been used in the past for crown restorations but have not been reported in orthodontic bonding adhesives. Namura and colleagues studied the usefulness of orthodontic adhesive containing fluorescent dye and if it would be possible to visualize the adhesive that remains on a tooth more clearly (Namura et al., 2010). They also analyzed what effects the fluorescent dye would have on the esthetics of a clear bracket, on the shear bond strength of the material, and on the adhesive remnant index (Namura et al., 2010).

Namura and others bonded brackets on bovine enamel surfaces and used adhesives with three different concentrations of fluorescent dye: 0.001, 0.002 and 0.003%. To ensure that the dye did not compromise shear bond strength or the adhesive
remnant index score, this was also measured for each material with the various concentrations of fluorescent dye. The color penetrating ability through the clear bracket was also analyzed. Their results showed that at 0.003% fluorescent dye, the shear bond strength was significantly lower than their control of Transbond. This material was also five times more visible behind a clear bracket than the other materials. It was determined that orthodontic adhesives containing 0.002% fluorescent dye can provide sufficient bond strength and esthetics, but also aid in adhesive removal with its fluorescent color (Namura et al., 2010).

**Color Analysis**

The analysis of color, particularly the color of the dentition and of dental materials, is a critical part of dentistry. An important aspect of analyzing color is understanding that color parameters can be recorded in all three planes of space. The Commission Internationale de L’Eclairage (CIE) established the color parameters, recording them in the L*a*b* color space (Gomez-Polo et al., 2016). This system relates human color perception in all three dimensions of color space. L* is the lightness variable, and a* and b* are chromaticity coordinates (Gomez-Polo et al., 2016). Coordinate a* represents where the color falls along the red/purple to green/blue axis and coordinate b* represents where it falls along the yellow to blue/purple axis (Gomez-Polo et al., 2016).

Differences in color can be quantified by using the more traditional CIELab formula ($\Delta E_{ab}^*$) or using the newer CIEDE2000 formula ($\Delta E_{00}$) (Gomez-Polo et al., 2016).
The \( \Delta E_{ab}^* \) formula has been used more historically, and the equation to analyze differences in color is as follows:

\[
\Delta E_{ab}^* = \sqrt{\left(\Delta L^*\right)^2 + \left(\Delta a^*\right)^2 + \left(\Delta b^*\right)^2}
\]

In order to further understand how differences in color can relate clinically, attempts have been made to correlate the color measurement to visual perception. In dentistry, a perceptible color match is a color difference that is at or below the perceptibility threshold, whereas an acceptable color match is a color difference at or below the acceptability threshold (Gomez-Polo et al., 2016). Few published studies have used the CIEDE2000 formula to analyze perceptibility and acceptability thresholds of color (Gomez-Polo et al., 2016). However, multiple studies have reported various thresholds for clinical perceptibility and acceptability utilizing the \( \Delta E_{ab}^* \) formula, although there is a range of values (Gomez-Polo et al., 2016). Typically, in dentistry the acceptability threshold for color difference is greater than the perceptibility threshold (Gomez-Polo et al., 2016).

As stated above, most studies analyzing color have utilized the \( \Delta E_{ab}^* \) formula. For example, in 2006 Sidhu and others studied the differences in color and translucency of resin composites upon light curing. In their study, they analyzed disks of shade A2 resin composite used in restorative dentistry prior to and following polymerization to evaluate how color and translucency changes (Sidhu et al., 2006). They utilized the \( \Delta E_{ab}^* \) formula to study the differences in color. They determined with this formula that the changes during light curing of all the studied materials were statistically different and that for
precise shade matching, clinicians should use the cured material as a reference (Sidhu et al., 2006).

Hyun and Ferracane also utilized the $\Delta E_{ab}^*$ formula in their research in 2016 to study the influence that biofilms have on the optical properties of bioactive glass-containing composites. In their study, bioactive glass-containing composite disks were fabricated and light-cured (Hyun and Ferracane 2016). The discs were divided into four different surface roughness groups and then incubated in a media with and without *Streptococcus Mutans* biofilm for several weeks. The $L^*a^*b^*$ values were measured before and after the incubation period and the $\Delta E_{ab}^*$ formula and translucency parameter were utilized to analyze differences in color and translucency, respectively. Their results showed that highly polished dental composites that contain bioactive glass additives may become slightly more rough and have less translucency when exposed to bacterial biofilms, but they do not discolor more than composites without the bioactive glass additives (Hyun and Ferracane 2016).

Although $\Delta E_{ab}^*$ formula has a long history and has been widely utilized, researchers have raised the question on if there is a better formula that can be used to capture perceived color differences. More specifically, a study was done to compare the $\Delta E_{ab}^*$ formula to the newer CIEDE2000 formula and how they detect perceived color differences (Gomez-Polo et al., 2016). The purpose of this study by Gomez-Polo and others was to evaluate the CIELab and CIEDE2000 formulas and determine which reflects differences in color the best, and also to evaluate whether color perception differs by sex. In the study, forty participants grouped 18 dental resin disks, with the only requirement that each group was formed of discs that were indistinguishable in color. The
authors demonstrated that women detected differences in color better than men. The results of this study also showed that the CIEDE2000 formula captured the color differences perceived by the human eye better than the $\Delta E_{ab}^*$ formula (Gomez-Polo et al., 2016). However, the difference between the two formulas was small (although significant). This difference could also have occurred though due to poorly measured variables or because the correlation was contaminated by other variables that were not taken into account (Gomez-Polo et al., 2016).
CHAPTER 3
MATERIALS AND METHODS

Study Design

All procedures and testing were completed in the Biomaterials Lab at the Wehr Physics Building of Marquette University. For the purpose of this study, the products that were evaluated include: Grengloo by Ormco, Blugloo by Ormco, BracePaste Color Change by American Orthodontics and Transbond PLUS by 3M Unitek. As a non-color changing control adhesive, the commonly used Transbond XT by 3M Unitek, was also analyzed. Ten specimens of each orthodontic adhesive were fabricated (n=10/adhesive). The color and translucency of each specimen was tested using a spectrophotometer (CM-700d; Konica Minolta).

Specimen Preparation

Teflon plates (2 mm thick) with holes 9 mm in diameter were used as molds to fabricate standardized discs of orthodontic adhesive samples (Figure 1). The samples remained in the Teflon molds for the duration of the study. Each mold was placed on Glad Cling Wrap (polyethylene, approximately 13 microns thick) on a glass slide, filled with the orthodontic adhesive material and then smoothed with another glass slide to achieve uniform thickness (Figure 2).
**Experimental Procedure**

The spectrophotometer was white calibrated using a White Calibration Cap (Konica Minolta). Each sample was transferred on to the spectrophotometer with the Cling Wrap in place (Figure 3). The spectrophotometer was used to analyze the color of samples prior to polymerization (Figure 4). Each sample was light-cured with a Kerr
Demi Plus curing unit for 20 seconds on each side. The samples remained at room temperature (20.6°C ± 0.2°C) for 30 minutes to allow for further polymerization, and the color was then analyzed. The samples were tested with and without Cling Wrap to analyze any possible effect it may have on the L*a*b* values (Figure 5 and Figure 6). The samples were measured at various time points and temperatures, according to the parameters listed in Table 1.

Figure 3. Grengloo sample prior to polymerization on spectrophotometer.
Figure 4. Example of L*a*b* reading of a specimen on the spectrophotometer.

Figure 5. Transbond Plus post-polymerization with cling wrap in place on spectrophotometer.
Figure 6. Transbond Plus post-polymerization without cling wrap in place on spectrophotometer.

<table>
<thead>
<tr>
<th>Polymerization Status</th>
<th>Temperature</th>
<th>Background</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pre-Polymerization</td>
<td>20.6°C</td>
<td>None</td>
</tr>
<tr>
<td>Post-Polymerization</td>
<td>20.6°C</td>
<td>None</td>
</tr>
<tr>
<td>30 minutes</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Post-Polymerization</td>
<td>37.0°C</td>
<td>None</td>
</tr>
<tr>
<td>7 days</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Post-Polymerization</td>
<td>37.0°C</td>
<td>White</td>
</tr>
<tr>
<td>7 days</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Post-Polymerization</td>
<td>37.0°C</td>
<td>Black</td>
</tr>
<tr>
<td>7 days</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Post-Polymerization</td>
<td>20.6°C</td>
<td>None</td>
</tr>
<tr>
<td>8 days</td>
<td></td>
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</tbody>
</table>

Table 1. Study parameters for each orthodontic adhesive sample.

The samples were stored in Fusayama Meyer artificial saliva (without the organic and sulfur components) (Schiff et al., 2005 and Ghanbarzadeh et al., 2015) and placed in an incubator set at 37.0°C ± 0.2°C (to mimic intraoral temperature). The composition of the artificial saliva, which closely resembles natural saliva, was: KCl (0.4 g/l), NaCl (0.4
g/l), CaCl₂ (0.6 g/l) and NaH₂PO₄ (0.690 g/l). Samples were removed from the incubator after 7 days, patted dry and the color was analyzed with the spectrophotometer. A heater was used to maintain an elevated temperature during data collection. Samples sat in the artificial saliva at room temperature for an additional day and the color was analyzed again (Figure 7). The color difference between the various samples was measured using the $\Delta E_{ab}^*$ formula.

Figure 7. Samples in artificial saliva at room temperature.

In addition to analyzing color, the spectrophotometer was also used to analyze the translucency of each adhesive sample at the elevated temperature of 37.0°C, 7 days post-polymerization. A standard white tile (Lucideon) was placed over the sample and the L*a*b* values were recorded. Without moving the sample, the white tile was removed, a
standard black tile (Lucideon) was placed, and another reading was made. From the L*a*b* values for both tile backgrounds, the translucency was calculated by using:

\[
TP = \sqrt{((L_b - L_w)^2 + (a_b - a_w)^2 + (b_b - b_w)^2)}
\]

Where TP is the translucency parameter and the “b” and “w” after L, a and b values refer to black and white, respectively.

**Statistical Analysis**

After the data was collected, the average values of each variable were calculated for each material evaluated. An analysis of variance (ANOVA) test was performed to evaluate the equality among the five study groups for each parameter. A response profile analysis was done to analyze the L*a*b* values of the materials at different polymerization stages. A pairwise comparison was used to compare the color changing composite translucency parameter values to the control, Transbond XT. A two sample T test was used to compare the L*a*b* values of each material at 37.0°C and 20.6°C. The significance was set to \( p < 0.05 \). The calculations and analyses were performed with statistical analysis software – R version 4.1.2 (The R Foundation).
CHAPTER 4
RESULTS

Color Change

The L*a*b* values for each material was determined for each parameter shown in Table 1. An ANOVA test was performed for each L*a*b* value for every study parameter. There were significant differences \((p < 0.05)\) with respect to all L*a*b* between the materials for each study parameter. The average L*a*b* values for each parameter are shown in Table 2, Table 3 and Table 4.

<table>
<thead>
<tr>
<th>Material</th>
<th>With Cling Wrap</th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Pre-Polymerization</td>
<td>30 Minutes Post-Polymerization</td>
<td></td>
</tr>
<tr>
<td></td>
<td>L*</td>
<td>a*</td>
<td>b*</td>
</tr>
<tr>
<td>Brace Paste</td>
<td>40.2 ± 1.2</td>
<td>7.3 ± 0.5</td>
<td>-6.8 ± 0.9</td>
</tr>
<tr>
<td>Blugloo</td>
<td>44.5 ± 0.6</td>
<td>-4.4 ± 0.1</td>
<td>-17.4 ± 0.3</td>
</tr>
<tr>
<td>Grengloo</td>
<td>39.7 ± 0.9</td>
<td>-15.4 ± 0.6</td>
<td>7.4 ± 0.5</td>
</tr>
<tr>
<td>Transbond Plus</td>
<td>59.2 ± 0.5</td>
<td>37.9 ± 0.6</td>
<td>2.5 ± 0.2</td>
</tr>
<tr>
<td>Transbond XT</td>
<td>56.6 ± 2.8</td>
<td>0.2 ± 0.1</td>
<td>4.5 ± 1.0</td>
</tr>
</tbody>
</table>

Table 2. L*a*b* values for each material with cling wrap in place. Measurements taken at 20.6°C.

<table>
<thead>
<tr>
<th>Material</th>
<th>No Cling Wrap</th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>30 Minutes Post-Polymerization</td>
<td>8 Days Post-Polymerization</td>
<td></td>
</tr>
<tr>
<td></td>
<td>L*</td>
<td>a*</td>
<td>b*</td>
</tr>
<tr>
<td>Brace Paste</td>
<td>64.7 ± 0.6</td>
<td>-3.5 ± 0.1</td>
<td>-1.9 ± 0.3</td>
</tr>
<tr>
<td>Blugloo</td>
<td>67.8 ± 1.6</td>
<td>-5.4 ± 0.2</td>
<td>-7 ± 2.1</td>
</tr>
<tr>
<td>Grengloo</td>
<td>53.8 ± 0.7</td>
<td>-15.2 ± 0.2</td>
<td>10.6 ± 0.2</td>
</tr>
<tr>
<td>Transbond Plus</td>
<td>59.1 ± 0.3</td>
<td>0.5 ± 0.1</td>
<td>5.6 ± 0.1</td>
</tr>
<tr>
<td>Transbond XT</td>
<td>62.8 ± 2.2</td>
<td>-0.3 ± 0.2</td>
<td>-2.8 ± 1.2</td>
</tr>
</tbody>
</table>

Table 3. L*a*b* values for each material with no cling wrap in place. Measurements taken at 20.6°C.
Table 4. L*a*b* values for each material with no cling wrap in place. Measurements taken at 37.0°C.

The color change between various time points was also analyzed. The ΔE*ab value between pre-polymerization and 30 minutes after polymerization was analyzed for each material and the average value is shown in Table 5. An ANOVA test was performed and there was a significant difference (p < 0.05) in ΔE*ab between the various materials upon polymerization. The ΔE*ab value was also analyzed from 30 minutes after polymerization to 8 days following polymerization and the average value for each material is shown in Table 5. An ANOVA test was also performed and demonstrated that there was a significant difference (p < 0.05) in ΔE*ab between the various materials over this time period. Figures 8, 9 and 10 demonstrate the average change in L*a*b* values after polymerization.

<table>
<thead>
<tr>
<th>Material</th>
<th>7 Days Post-Polymerization</th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>L*</td>
<td>a*</td>
<td>b*</td>
<td>L*</td>
</tr>
<tr>
<td>No Background</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Brace Paste</td>
<td>61.6 ± 1.2</td>
<td>-1.0 ± 0.7</td>
<td>3.7 ± 0.4</td>
<td>67.1 ± 1.0</td>
</tr>
<tr>
<td>Blugloo</td>
<td>72.3 ± 0.4</td>
<td>-2.7 ± 0.0</td>
<td>3.0 ± 0.2</td>
<td>72.8 ± 0.4</td>
</tr>
<tr>
<td>Grengloo</td>
<td>66.5 ± 0.5</td>
<td>-5.0 ± 0.2</td>
<td>10.9 ± 0.4</td>
<td>68.2 ± 0.6</td>
</tr>
<tr>
<td>Transbond Plus</td>
<td>62.7 ± 0.8</td>
<td>3.8 ± 0.8</td>
<td>8.5 ± 0.7</td>
<td>69.3 ± 0.7</td>
</tr>
<tr>
<td>Transbond XT</td>
<td>62.3 ± 2.3</td>
<td>-0.3 ± 0.2</td>
<td>-1.2 ± 1.1</td>
<td>70.3 ± 1.9</td>
</tr>
</tbody>
</table>

Table 5. An ANOVA test was also performed and demonstrated that there was a significant difference (p < 0.05) in ΔE*ab between the various materials over this time period.
<table>
<thead>
<tr>
<th>Material</th>
<th>$\Delta E_{ab}^*$ of Pre-Polymerization to 30 Minutes Post-Polymerization</th>
<th>$\Delta E_{ab}^*$ of 30 Minutes Post-Polymerization to 8 Days Post-Polymerization</th>
</tr>
</thead>
<tbody>
<tr>
<td>Brace Paste</td>
<td>28.4 ± 0.8</td>
<td>6.4 ± 0.4</td>
</tr>
<tr>
<td>Blugloo</td>
<td>26.9 ± 2.3</td>
<td>7.6 ± 2.2</td>
</tr>
<tr>
<td>Grengloo</td>
<td>18.9 ± 1.1</td>
<td>3.5 ± 0.5</td>
</tr>
<tr>
<td>Transbond Plus</td>
<td>37.5 ± 0.6</td>
<td>4.7 ± 0.4</td>
</tr>
<tr>
<td>Transbond XT</td>
<td>10.9 ± 1.1</td>
<td>1.5 ± 0.3</td>
</tr>
</tbody>
</table>

Table 5. Color change ($\Delta E_{ab}^*$) values for each material upon polymerization. Measurements taken at 20.6°C.

![L* Values Before and After Polymerization](image)

Figure 8. $L^*$ values before and after polymerization for each material. Measurements taken at 20.6°C with no cling wrap in place for the post-polymerization samples. Since the pre-polymerization group had cling wrap on the surface of the specimen, the values in this figure for 0 (pre-poly) group was adjusted statistically to account for this.
Figure 9. $a^*$ values before and after polymerization for each material. Measurements taken at 20.6°C with no cling wrap in place for the post-polymerization samples. Since the pre-polymerization group had cling wrap on the surface of the specimen, the values in this figure for 0 (pre-poly) group was adjusted statistically to account for this.

Figure 10. $b^*$ values before and after polymerization for each material. Measurements taken at 20.6°C with no cling wrap in place for the post-polymerization samples. Since the pre-polymerization group had cling wrap on the surface of the specimen, the values in this figure for 0 (pre-poly) group was adjusted statistically to account for this.
Temperature Effect

Each sample had its L*a*b* values analyzed at room temperature and at 37.0°C (near intraoral temperature). The average of these values are shown in Table 2, Table 3, and Table 4. The color change of the materials from an elevated temperature to room temperature was also evaluated and the average $\Delta E_{ab}^*$ value for each material is shown in Table 6. An ANOVA test was done and there was a significant difference ($p < 0.05$) in the $\Delta E_{ab}^*$ values of the different materials. Figure 11 shows the visual appearance of the samples at the varying temperatures. A two sample T test was performed comparing the L*a*b* values for each material at both temperatures and the $p$ values are shown in Table 7. There was a significant difference ($p < 0.05$) in all L*a*b* values for Blugloo and Grengloo, and also for the Transbond Plus a* value from room temperature to an elevated temperature. There was not a significant difference ($p > 0.05$) between all other L*a*b* values for the other materials when the temperature was changed.

<table>
<thead>
<tr>
<th>Material</th>
<th>$\Delta E_{ab}^*$ of 37.0°C to 20.6°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Brace Paste</td>
<td>0.8 ± 0.5</td>
</tr>
<tr>
<td>Blugloo</td>
<td>18.5 ± 1.3</td>
</tr>
<tr>
<td>Grengloo</td>
<td>16.5 ± 0.9</td>
</tr>
<tr>
<td>Transbond Plus</td>
<td>4.3 ± 0.8</td>
</tr>
<tr>
<td>Transbond XT</td>
<td>0.7 ± 0.3</td>
</tr>
</tbody>
</table>

Table 6. Color change ($\Delta E_{ab}^*$) values for each material upon change in temperature.
Translucency Analysis

The translucency of each material was analyzed at 37.0°C, and the average translucency parameter for all of the materials is shown in Table 8. An ANOVA test was performed and there was a significant difference ($p < 0.05$) in the translucency parameter between all the materials. A pairwise comparison was also performed to compare each
color changing composite to the control, Transbond XT, and the $p$ values are shown in Table 9. All the color changing composites had a significantly different translucency parameter value when compared to Transbond XT.

<table>
<thead>
<tr>
<th>Material</th>
<th>Translucency Parameter at 37.0°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Brace Paste</td>
<td>9.7 ± 0.7</td>
</tr>
<tr>
<td>Blugloo</td>
<td>1.1 ± 0.2</td>
</tr>
<tr>
<td>Grengloo</td>
<td>2.5 ± 0.3</td>
</tr>
<tr>
<td>Transbond Plus</td>
<td>9.7 ± 0.2</td>
</tr>
<tr>
<td>Transbond XT</td>
<td>11.7 ± 1.3</td>
</tr>
</tbody>
</table>

Table 8. Translucency parameter values for each material.

<table>
<thead>
<tr>
<th>Translucency Parameter Comparison to Transbond XT</th>
<th>$p$ values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Brace Paste</td>
<td>0.001</td>
</tr>
<tr>
<td>Blugloo</td>
<td>&lt;0.001</td>
</tr>
<tr>
<td>Grengloo</td>
<td>&lt;0.001</td>
</tr>
<tr>
<td>Transbond Plus</td>
<td>0.001</td>
</tr>
</tbody>
</table>

Table 9. Translucency parameter comparison of each material to Transbond XT.
CHAPTER 5
DISCUSSION

There are several different color changing composites available on the market for orthodontists to utilize during bonding. Many studies have been previously done analyzing the shear bond strength of these materials. This study focuses on the actual color changing capabilities of the materials and analyzes how translucent these materials become following polymerization. In this study, the color changing properties (ΔE_{ab}^*) and the translucency parameter were analyzed of four popular color changing composites: BracePaste, Blugloo, Grengloo and Transbond Plus. A non-color changing composite that is widely used, Transbond XT, was also analyzed as a control. The various materials were studied at different time points after polymerization and at varying temperatures. The elevated temperature of 37.0°C was selected to relate to intraoral temperature.

Patients are increasingly more critical of esthetics, and there is no exception when it comes to orthodontic treatment. That is why it is important to maximize esthetics for every product used during treatment. Although the amount or resin that can remain on the teeth after the debonding process is minimal, the appearance of the bonding materials used should be of strong consideration to achieve a maximally esthetic result for the patient. In this study, the L*a*b* values that correspond to color was analyzed at room temperature prior to polymerization, 30 minutes after polymerization and 8 days after polymerization. These values were also analyzed 7 days after polymerization at 37.0°C.

For all the specimens, polymerization led to an increase in L*, meaning the lightness of the materials increased. There was a significant difference between the L* values of the materials for every time point evaluated. BracePaste, Blugloo and Grengloo
appeared to have the largest increase in the L* value upon initial polymerization (Table 2). There was also a significant difference between the a* and b* values of the materials for all time points. Unlike L*, there was not a general trend in the direction that a* and b* shifted during polymerization for all the materials. Transbond Plus displayed one of the larger shifts in a* value upon polymerization where it transitioned from a large amount of red (large a* value) to a value close to zero. Its b* value however, only had minimal change (Table 2). Blugloo on the other hand displayed minimal change in a* and a larger change in b* during polymerization (Table 2).

When analyzing the samples prior to polymerization, cling wrap needed to be placed over the spectrophotometer to prevent the material from sticking to the spectrophotometer surface. To allow for an accurate comparison of the pre-polymerization values to the post-polymerization values, the L*a*b* values at 30 minutes after polymerization were measured with and without cling wrap in place. Thus, the degree that cling wrap affects the L*a*b* measurements was determined and the pre-polymerization values were then adjusted accordingly when compared to other measurements post-polymerization. Overall, removing the cling wrap changed the color minimally. Nearly all the samples became more of their given color without the cling wrap in place (i.e. if the material had a positive a* or b* value, it would become more positive and if it had a negative a* or b* value, it would become more negative). The cling wrap likely reflected off some light from the spectrophotometer, allowing for a greater color of the materials when it was removed.

The color changing capabilities of the materials during polymerization was analyzed at room temperature. All materials displayed color change upon polymerization
when compared to 30 minutes after polymerization. Of the color changing composites, Transbond Plus showed the largest change in color during this time period, and Grengloo had the smallest change in color (Table 5). All of the materials also exhibited a color change from 30 minutes after polymerization to 8 days after polymerization, although not as drastic of a change from initial polymerization. Of the color changing composites, Blugloo showed the largest change in color over this time period and Grengloo showed the smallest change (Table 5). There was a significant difference in the ΔE*_{ab} values of the materials during both of these time periods.

The effect that changing temperature has on the color of the various materials was also evaluated. There was a significant difference in how much the various materials changed colors. Transbond XT and BracePaste had minimal change due to a difference in temperature, and Blugloo and Grengloo had the largest change in color (Table 6). Both Blugloo and Grengloo had a large decrease in L* when transitioning to a cooler temperature, indicating that the materials got darker. Blugloo also became more blue (b* became more negative) and Grengloo became more green (a* became more negative) when the materials transitioned from 37.0°C to room temperature.

In a study by Gomez-Polo and colleagues, the clinical perceptibility level was set at ΔE*_{ab} = 1.0 to 3.7 (Gomez-Polo et al., 2016). Following these parameters, all materials displayed a perceptible difference in color upon initial polymerization (Table 5). Grengloo and Transbond XT did not have a perceptible difference in color upon greater polymerization over time (Table 5). Blugloo, Grengloo and Transbond Plus all had a clinically perceptible change in color when transitioning from 37.0°C to room temperature.
The translucency parameter of the materials was only measured at the elevated temperature because the Blugloo and Grengloo specimens were obviously opaque at room temperature. In addition, the elevated temperature is similar to intraoral temperature, making the results about translucency more clinically relevant. There was a significant difference amongst the translucency parameters of the five materials (Table 8). Furthermore, each color changing composite had a significantly different translucency parameter value when compared directly to the control, Transbond XT (Table 9). However, BracePaste and Transbond Plus translucency parameter values were much more similar to Transbond XT than Blugloo and Grengloo compared to the control.

There were some limitations in this study. First, this study was done all extraorally in a laboratory. Therefore, a direct comparison to what can be seen clinically cannot be made. The materials were also evaluated within Teflon molds and not on the tooth surface. The appearance of the materials likely are different when it is bonded to a tooth, rather than within a mold. In addition, the thickness of the samples studied was 2 mm, which is larger than the amount of material used in orthodontics. However, this thickness is commonly used when evaluating the color characteristics of esthetic dental materials (Gomez-Polo et al., 2016, Hyun et al., 2016, Paravina et al., 2002, and Sidhu et al., 2006). More specimens or repeat measurements also could have been analyzed to increase the validity of the data.

More research is needed in the future to better understand not only the esthetics of these color changing composites, but also how they can potentially aid in the debonding process. American Orthodontics claims that under UV light, BracePaste can fluoresce to help remove the resin during the debonding process (American Orthodontics 2020).
Ormco also claims that the color of Blugloo and Grengloo returns when they are exposed to a blast of air (Ormco). This is believable considering in this study the Blugloo and Grengloo specimens had a considerable amount of color at room temperature. Therefore, in the future it could be beneficial to analyze if these materials aid in the debonding process of fixed appliances and if one material is superior to another. That way, providers would be able to make a decision on which material has adequate bond strength, acceptable esthetics and ease of removal during the debonding process.
CHAPTER 6
CONCLUSION

All of the color changing composites that were evaluated have significantly different color changing capabilities. All materials demonstrated an increase in L* upon polymerization, but there was no trend in the changes noted with a* and b* values. There was a significant difference in the color change between the materials during all stages of polymerization. Transbond Plus showed the largest color change during initial polymerization and Grengloo had the smallest change. Upon further polymerization, Blugloo demonstrated the largest change in color and Grengloo showed the smallest change. However, all the color changing composites changed color more than Transbond XT upon polymerization. Temperature plays a significant role in the appearance of Blugloo and Grengloo. When transitioning from an elevated temperature to room temperature, Transbond XT and BracePaste had minimal color change, and Blugloo and Grengloo had the largest change.

The translucency after polymerization was significantly different between all the materials evaluated. More specifically, all of the color changing composites had significantly different translucency parameter values when directly compared to Transbond XT. Blugloo and Grengloo displayed the largest difference and were most opaque, and BracePaste and Transbond Plus had similar translucency. Overall, the color-changing composites were not as translucent as Transbond XT. Further study is required in order to make a clinical evaluation on the role that these color changing composites have on the esthetics of the dentition after debonding orthodontic appliances.
REFERENCES


