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Conventional and High Intensity Halogen Light Effects on Water Sorption and Microhardness of Orthodontic Adhesives

Tancan Uysal;[a] Faruk Ayhan Basciftci;[b] Yagmur Sener;[c] Murat Selim Botsali;[d] Abdullah Demir[b]

ABSTRACT

Objective: To test the null hypothesis that when the equivalent total light energy is irradiated to three orthodontic adhesive resins, there is no difference between the microhardness and water sorption values regardless of the curing light sources.

Materials and Methods: Samples were divided into six groups according to the combination of three orthodontic adhesives (Kurasper F, Light-Bond, Transbond XT) and two light intensities (quartz tungsten halogen [QTH] and high intensity quartz tungsten halogen [HQTH]). One half of each of the 40 samples of three adhesive pastes was polymerized for 20 seconds by a QTH light source, and the other half was polymerized for 10 seconds by a HQTH light source. Water sorption was determined and Vickers hardness was established with three measurements per sample at the top, center, and bottom. Statistical analysis was performed using two-way analysis of variance (ANOVA) with multiple comparisons (Tukey-HSD).

Results: Statistically significant differences were found among all adhesives for water sorption and hardness values cured with QTH and HQTH. The HQTH curing unit resulted in higher values than did the QTH. The highest water sorption values were observed for Kurasper F cured with HQTH and the lowest value was observed for Transbond XT cured with QTH. For microhardness Light-Bond cured with HQTH produced the highest values, and Transbond XT cured with QTH produced the lowest.

Conclusions: When the equivalent total light energy is irradiated to three orthodontic adhesive resins, there are significant differences between the microhardness and water sorption values cured with the QTH and HQTH light source. The null hypothesis is rejected.

KEY WORDS: Water sorption, Vickers hardness, Halogen, Composites.

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INTRODUCTION Return to TOC

A wide variety of photo-activated resin-based composites (PARBC) have become commercially available in the orthodontic field. These are the choice of adhesive for orthodontic bonding because of their ease of use and the extended time they allow for optimal bracket placement. The disadvantage of using light-cured materials is the time required for exposing the adhesive.[1] A reduction in the amount of curing time would be of great advantage to both the orthodontist and patient.[2]

PARBC need adequate light output and irradiation time to obtain an optimal curing level. Incomplete curing of resins results in the increase of water sorption, a decrease in the hardness, and deterioration of the mechanical properties of material through the softening of the polymer matrix by unreacted monomer.[3]

Adhesives used for orthodontic bonding are required to have long-term durability in the oral cavity. The material is in contact with saliva in a complex environment containing bacterial flora composed of a many inorganic and organic species. The materials thus require a certain set of physical properties before the curing process which include microhardness as the most important and water sorption, which were related with the strength.[4]

The use of high intensity units has been recommended almost universally,[5] since they are able to enhance monomer conversion. Hardness, water sorption, and solubility in water are largely related to the conversion of monomers incorporated into composites.[6] Polymerization by a high intensity quartz tungsten halogen (HQTH) curing unit occurs rapidly. Conversely, some authors do not recommend the use of high intensity light units because this type of unit induces higher polymerization shrinkage, lower degree of conversion, and larger residual stress in dental filling composites.[7] In recent years many different methods have been studied aiming to improve the physical properties of PARBC, ie, use of different light activation techniques such as pulse delay,[8] soft-start and pulse cure,[9] development of resins,[10] and

the use of the incremental filling technique.[11]

It is believed that adequate PARBC polymerization and improved mechanical properties may be obtained in a shorter time when using high power light curing units. In recent studies many authors compared the effects of light emitting diodes (LED) and plasma arc curing lights with conventional curing systems. However, there are few reports on the conventional and high intensity halogen curing systems,[12] especially their effect on the microhardness and water sorption properties of PARBC.

The purpose of the present study was to test the null hypothesis that when the equivalent total light energy is irradiated to three orthodontic adhesive resins, there are no differences between the microhardness and water sorption values regardless of the curing light sources (quartz tungsten halogen [QTH] and HQTH).

MATERIALS AND METHODS Return to TOC

One hundred twenty glass ring molds (8.5 mm in inner diameter and 2 mm in height) were prepared (Figure 1) using a low-speed saw (Isomet, Buehler Ltd, Lake Bluff, Ill). The internal surface of the glass rings were roughened and etched for 5 minutes with hydrofluoric acid (Etch-It, American Dental Supply, Easton, Pa). The glass molds were then weighed in air and in water with an electronic balance (Shimadzu AY220, Shimadzu Corp, Kyoto, Japan) to calculate their density and volume.

Three different commercially available orthodontic adhesive pastes, Kurasper F (Kuraray, Okayama, Japan), Light-Bond (Reliance, Itasca, Ill), and Transbond XT (3M Unitek, Monrovia, Calif), were used in this study (Table 1). Forty samples of each adhesive paste were placed into the glass molds, which were sandwiched between two glass slides. To ensure that the adhesive paste would be well distributed within the mold, a 5-N force was applied for 30 seconds. The samples were stored in dark and dry conditions at 37°C for 24 hours to standardize the environment prior to the testing procedures after light curing, before they were weighed both in air and in water to calculate their density and volume.

One half of each of the 40 samples of three adhesive pastes was polymerized for 20 seconds by a QTH light source (Hilux 350, Express Dental Products, Toronto, Canada) with a 10-mm diameter light tip. The other half was polymerized for 10 seconds by a HQTH light source (Optilux 501, Kerr, Danbury, Conn) with an 10-mm diameter light tip. Regarding the curing units, the important parameter is the amount of light energy of appropriate wavelength emitted during irradiation. This energy is calculated as the product of the output of the curing unit and the time of irradiation, and it may be termed energy density ($mJcm^{-2}$). The outputs of the light tips were calibrated by a digital curing radiometer (Demetron, Danburry, Conn) as 420 mW/ cm^{-2} for QTH and as 850 mW/cm^{-2} for HQTH. At the start of irradiation, these outputs were measured as 430 mW/cm^{-2} and 865 mW/cm^{-2} for QTH and HQTH, respectively. However, the light intensity decreased 17.2 mW/cm^{-2} for QTH and 24.7 mW/cm^{-2} for HQTH with usage. The total light energy was calculated with the mean output values about:

$$\text{QTH: } 420\ mW/cm^{-2} \times 20\ s = 8400\ mJ\ cm^{-2}$$

$$\text{HQTH: } 850\ mW/cm^{-2} \times 10\ s = 8500\ mJ\ cm^{-2}$$

Water Sorption Measurements

Water sorption was determined according to the method described by Satou et al.[13] The diameter and the thickness of each specimen were measured, and the volume (V_0) was calculated. Each specimen was stored at 37°C in a desiccator for 3 days until a constant weight (W_0) was obtained and subsequently immersed in 10 mL of distilled water maintained at 37°C. Periodically, the specimen was picked up and the surface water was blotted away with paper until free from visual moisture, then it was reweighed (W_1). This procedure was continued until the weight change during 1 week became less than 0.32 μgmm^3. The value of water sorption (W_{sp}) in $\mu g/mm^{-3}$ was calculated using the following equation:

$$W_{sp} = (W_1 - W_0)/V_0$$

Microhardness Measurements

Microhardness measurements were used as indirect evaluation of degree of conversion.[14] Vickers hardness was determined according to the description by Dietschi et al.[15] After water sorption measurements and polishing, the samples were stabilized parallel to the base of the hardness measurement device (Matsuzawa Seiki Co Ltd, MHTZ, Tokyo, Japan, serial number: mh2028) by pressing the sample over a thin layer of a plasticizing material (Plasticine; Beuhlers, Princeton, Ind). Vickers hardness measurements were obtained using a 300-gram load for all specimens. The appropriate load was applied for 30 seconds and the indentation size was recorded 10 seconds later. Vickers hardness was established with three measurements per sample at 10 µm underneath the sample top surface, at the center of the sample and at 10 µm above the sample bottom.

Samples were divided into six groups according to the combination of two light intensities and three orthodontic adhesives. For all groups, the average values and standard deviations (SD) were calculated. Statistical analysis was performed using two-way analysis of variance (ANOVA) (SPSS, Statistical Package for Social Sciences, Version 10.0, Chicago, Ill) and Tukey HSD tests for multiple comparisons (each adhesive and each curing unit). The level of statistical significance was set at $P < .05$.

RESULTS Return to TOC

The water sorption and Vickers hardness mean values and standard deviations of three adhesive resins cured with QTH and HQTH and statistical comparisons are shown in Table 2. Water sorption ($P < .05$) and microhardness ($P < .001$) values irradiated to the three orthodontic adhesive resins varied significantly depending on the different curing units used. Two-way ANOVA revealed significant interaction among the curing unit type and orthodontic adhesives ($P < .05$). The null hypothesis was thus rejected.

For all adhesives, the HQTH light curing unit resulted in more water sorption than did the QTH (Table 2). There are increases in water sorption when the specimens were irradiated using the HQTH, but only statistically significant differences were found for Kurasper F. The highest water sorption values were observed for Kurasper F cured with HQTH (14.03 ± 1.06 μgmm^{-3}) and the lowest value was observed for Transbond XT cured with QTH (5.16 ± 0.84 μgmm^{-3}). All investigated adhesives showed statistically significant differences for water sorption values when irradiated using the QTH and HQTH separately ($P < .001$).

Microhardness differences among top, middle, and bottom values in all cured groups irradiated by QTH and HQTH were not statistically significant. For that reason average values were used for the Vickers hardness number for all groups.

All adhesives showed statistically significant hardness differences when irradiated using the QTH and HQTH ($P < .001$). Vickers hardness obtained with HQTH was generally superior when compared to the values obtained with the QTH, but only Kurasper F showed a statistically significant difference ($P < .001$). The highest hardness values were observed for Light-Bond cured with HQTH (95.28 ± 4.51 kg/mm^2) and the lowest value was observed for Transbond XT cured with QTH (55.10 ± 2.46 kg/mm^2).

DISCUSSION Return to TOC

The use of light to polymerize composite resins has increased in the last few years.[2,3,6,8,13] Several light devices have been developed that have greater power density in the curing region of the visible spectrum, 400–500 nm wavelength, which can be used to accelerate the photo-polymerization of composite resin materials, and to improve the physical properties of the set composite.[16] The most widely used light sources for PARBC are QTH lights.[17] Argon lasers, LED, HQTH, and xenon plasma arc lamps have all been shown to achieve rapid polymerization.[18–20] Studies on depth of cure, resin hardness, polymerization contraction, strength properties, water sorption, and water solubility have been performed with some of these systems[20]; however, there is limited published data on newer curing technologies such as HQTH.[12]

The HQTH light is capable of producing light of a greater intensity than that of the QTH light and may be sufficient for the fast curing of adhesives including those used for orthodontic bonding purposes. Nomoto et al[21] found that when the comparable total light energy was irradiated to the resin, the curing depth and the degree of conversion might be similar regardless of the differences in the light intensity or irradiation time. In addition, higher light intensity could result in increased fracture, hardness, and greater flexural strength of resin, which would translate into greater bond strength of brackets bonded to teeth.[3] With these effects in mind, this in vitro study was performed to investigate the effects of QTH and HQTH light sources on mechanical properties such as water sorption and microhardness of three orthodontic adhesives.

Water sorption is a critical property for PARBC because it increases the volume of the material.[22] Moreover, water acts as a plasticizer, increasing the deterioration of the resin matrix. In addition, water sorption usually affects color stability of composite since water-soluble monomers can penetrate the outer border of the brackets and lead to colorization of composite around the bracket base. This phenomenon is esthetically unacceptable.

The degree of cure is one of the critical parameters, which may influence the physical properties of composite materials,[23] and thus the clinical behavior of light curing materials. Knowing the degree of cure of resin composites is also essential in terms of residual monomers and probable allergic susceptibility.[2,24] However, directly measuring the degree of conversion is not easy. Therefore, similar to Usumez et al,[2] the mechanical property of hardness was evaluated in the present study to serve as an indirect indicator of the degree of cure. Microhardness allows for measurements at specific locations within the sample; for this study, evaluations were made at the top, middle, and bottom of the specimens. Though hardness values may not be used for a direct comparison among materials, they are a valuable tool for relative measurements within the same material, and their simplicity facilitates the evaluation of a large number of specimens,[25] making it suitable for comparing different curing techniques.

We used the Vickers hardness values for microhardness measurements. In the literature, both the Vickers and Knoop methods have been used to evaluate the hardness of resin composites. Knoop hardness is said to be more suitable for polymers, because Vickers indentation can distort with relaxation of the materials, whereas the long diagonal of the Knoop indentation is not affected. However, there are neither scientific data supporting this view nor international standards or qualifications favoring either of these methods.[2] Hofmann et al[26] investigated the association between Vickers and Knoop hardness and showed a significant linear correlation, and both may be similarly appropriate for studying resin composites.

The mechanical properties of PARBC are influenced by the type and composition of resin matrix, filler type, filler load, and mode of polymerization.[2] A correlation between volumetric filler content and hardness was demonstrated by Pilo and Cardash.[27] Inorganic fillers are added to reduce polymerization shrinkage and water sorption, to increase hardness and strength, and also to impart color characterization to the material.[28] Li et al[29] reported that changing the level of filler in composite altered the properties of hardness, water sorption, compressive strength, elastic modulus, and wear resistance. In this study, composite Kurasper F showed statistically significant higher water sorption than the other composites. In addition, the highest overall mean hardness value was observed for the Light-Bond specimens. Different results for these composites may be explained by the higher hydrophilicity of organic matrix resins, different composition/filler content, and also by their higher content of organic resins.

There are many studies in restorative dentistry investigating the relationships between curing light type and mechanical properties of PARBC. The shrinkage of the resin caused by the rapid curing with high intensity lights was considered a disadvantage for restorative applications, and fast curing can generate excess shrinkage and gap formation along the resin-preparation interface.[2] Despite this fact, these types of reports are rare in the orthodontic field.[12,30] Bang et al[30] irradiated equivalent total light energy with QTH and plasma arc units on orthodontic adhesives and found statistically significant differences in polymerization characteristics. Present findings indicate that when the equivalent total light energy was irradiated all three adhesive resins that cured with the HQTH light showed more water sorption and higher microhardness values than did the QTH. However, these differences were statistically significant only in Kurasper F for investigated properties. When looking at the QTH and HQTH lamp results, there was almost no decrease in hardness in the depth (top, middle, and bottom) of the samples, and no statistically significant differences were found. The deviation of the results from top to bottom was generally small, with both curing devices, which indicates a good reproducibility and reliability of the curing protocol.

To take osmotic pressure into consideration, acrylic polymers in the hydrogel matrix immersed in distilled water should absorb more water than those in saliva. Nicholson[31] showed greater equilibrium water uptake in pure water than in a salt solution, results which were consistent with the above theory. Because of these reasons, clinical conditions may significantly differ from an in vitro setting and our findings must be interpreted carefully. For clinical significance, the lower water sorption was observed for Transbond XT cured with QTH, and the higher microhardness was observed for Light-Bond cured with HQTH.

CONCLUSIONS Return to TOC

- Statistical analysis revealed significant interaction among the curing unit type and orthodontic adhesives.

- There are increases in water sorption and microhardness when the specimens were irradiated using the HQTH, but only statistically significant differences were found for Kurasper F.

- Curing units with higher intensity improved the hardness values, but differences among top, middle, and bottom in all cured groups irradiated by QTH and HQTH were not statistically significant.

- Multiple comparison results indicated that water sorption and hardness characteristics of orthodontic composites showed statistically significant differences when irradiated with the QTH and HQTH separately.

- Recommendations as to which adhesive or light source should be chosen for a specific circumstance cannot be done from this study.

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TABLES Return to TOC

Table 1. Orthodontic Adhesive Resins Used in the Present Study

Adhesive Code	Brand Name	Composition	Batch Number	Manufacturer
KF	Kurasper F	Bis-GMA, TEGDMA, HEMA, NaF and MF-MMA copolymer containing fluorine, silica filler.	41123	Kuraray, Japan
LB	Light-Bond	UDMA, TEGDMA, fused silica, sodium fluoride	104160	Reliance, III
TX	Transbond XT	Bis-GMA, Bis-EMA, TEGDMA, silanated quartz, submicron silica	200401	3M Unitek, Calif

Table 2. The Water Sorption and Vickers Hardness Mean Values and Standard Deviations of Three Orthodontic Adhesive Resins Cured With QTH and HQTH and Multiple Statistical Comparison Results[a]

			QTH (n = 20)		HQTH (n = 20)		Light Source Comparisons	Adhesive Resin Comparisons Cured With QTH			Adhesive Resin Comparisons Cured With HQTH		
	Groups	(n = 40)	Mean	SD	Mean	SD	QTH vs HQTH	Group A-B	Group A-C	Group B-C	Group A-B	Group A-C	Group B-C
Water uptake (μg mm^{-3})	A	Kurasper F	13.35	0.99	14.03	1.06	*						
	B	Light-Bond	7.69	0.92	7.84	1.33	NS	***	***	***	***	***	***
	C	Transbond XT	5.16	0.84	5.44	1.39	NS						
Microhardness (Vickers hardness)	A	Kurasper F	67.63	2.24	71.91	2.11	***						
	B	Light-Bond	92.96	2.49	95.28	4.51	NS	***	***	***	***	***	***
	C	Transbond XT	55.10	2.46	55.42	2.70	NS						

[a] NS indicates not significant; n, sample size; QTH, quartz tungsten halogen light; HQTH, high intensity quartz tungsten halogen light; and SD, standard deviation.
* $P < .05$; *** $P < .001$.

FIGURES Return to TOC

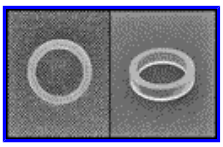

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Figure 1. Glass ring molds

[a] Associate Professor and Department Chair, Department of Orthodontics, Faculty of Dentistry, Erciyes University, Kayseri, Turkey

[b] Associate Professor, Department of Orthodontics, Selcuk University, Konya, Turkey

[c] Assistant Professor, Department of Pediatric Dentistry, Selcuk University, Konya, Turkey

[d] Research Fellow, Department of Pediatric Dentistry, Selcuk University, Konya, Turkey

Corresponding author: Dr Tancan Uysal, Erciyes Üniversitesi Dişhekimliği Fakültesi, Ortodonti A.D. Melikgazi, Kampüs Kayseri, 38039 Turkey (E-mail: tancanuysal@yahoo.com)