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Alterations in Microhardness of Clearfil Universal Bond Quick, G-Premio Bond, and Scotchbond Universal after Curing

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Abstract

Background and Aim: This study aimed to assess the alterations in microhardness of Clearfil Universal Bond Quick (CUBQ), G-Premio Bond (GPB), and Scotchbond Universal (SBU) immediately, 24 hours, and 6 months after curing.

Materials and Methods: In this in vitro study, a composite disc was fabricated. A putty/wash impression was made from the disc to serve as a mold. CUBQ, GPB, and SBU adhesives were applied in the mold, and after allowing 3 hours for the solvent to evaporate, they were cured by a LED curing unit for 20 seconds. Ten specimens were fabricated from each adhesive. The microhardness of the specimens was measured by a microhardness tester immediately, 24 hours, and 6 months after curing. Data were analyzed by repeated measures ANOVA (alpha=0.05).

Results: The mean microhardness of the three adhesives was significantly different immediately after curing (P<0.001), and CUBQ showed significantly higher microhardness than GPB (P<0.001) and SBU (P=0.004). The difference in microhardness of the three adhesives was not significant after 24 hours and 6 months (P>0.05).

Conclusion: The present results showed that the microhardness of all three tested universal adhesives increased with time.

Keywords: Composite Resins; Dentin-Bonding Agents; Hardness

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Introduction

Dentin bonding agents are multi-functional organic molecules with functional groups that interact with dentin and restorative resins. They are extensively used for bonding of composite resins to tooth structure [1]. Several types of dentin bonding agents are available in the market, and this diversity often leads to confusion of dental clinicians in selection of an ideal bonding agent. Etch-and-rinse and self-etch bonding agents have long been used for restorative procedures. However, universal adhesives were recently introduced to the market, which can be used in different modes of self-etch and etch-and-rinse on dentin and enamel [2]. These novel bonding agents were introduced aiming to enhance the bond strength to tooth structure, decrease the procedural steps, accelerate the procedure, and simplify the application of bonding agents. Universal adhesives can optimally bond to wet and dry dentin. Also, they can bond to enamel, porcelain, amalgam, and metal [3, 4]. Universal adhesives have unique properties, and are different from other bonding agents in terms of their monomer content [5, 6]. They are also suitable for bonding of indirect restorations such as zirconia, alumina, glass-ceramic, and metal restorations [4]. Clearfil Universal Bond Quick (CUBQ; Kuraray, Noritake, Japan), G-Premio Bond (GPB; GC Corporation, Japan), Scotchbond Universal (SBU; 3M ESPE, St. Paul, MN, USA), Adhese Universal (Ivoclar, Schaan, Liechtenstein), and Clearfil Universal Bond (Kuraray; Noritake, Japan) are among the commonly used universal adhesives available in the market [1, 5].

Since dentin bonding agents are used for bonding of composite resins to tooth structure, their weak performance would result in tooth discoloration, development of secondary caries, or debonding and failure of restorations [7-9].

Hardness is one of the important characteristics of dental materials, which can be affected by a wide variety of mechanical properties such as ductility, elastic stiffness, plasticity, strain, strength, toughness, viscoelasticity, and viscosity [9]. Hardness has been used as a reliable criterion for evaluation of mechanical strength and degree of conversion of bonding agents [10]. Considering the significance of mechanical properties and particularly hardness of dentin bonding agents, particularly universal adhesives, this study aimed to assess the microhardness of CUBQ, GPB, and SBU over time. The null hypothesis was that no significant difference would be found in the microhardness of CUBQ, GPB, and SBU at different time points after curing.

Materials and Methods

This in vitro experimental study was conducted on CUBQ (Kuraray Noritake; Tokyo, Japan), GPB (GC Corporation, Tokyo, Japan), and SBU (3M ESPE, St. Paul, MN, USA) universal adhesives. Table 1 presents the composition of the three adhesives. The study protocol was approved by the ethics committee of the School of Dentistry, Shahid Beheshti University of Medical Sciences (IR.SBMU.DRC.REC.1398.155).

The sample size was calculated to be 30 (10 from each adhesive) assuming alpha=0.05, beta=0.1, and power of 90%.

For the fabrication of specimens, first a composite (Z250; 3M ESPE, St. Paul, MN, USA) disc with 4.4 mm diameter and 0.7 mm thickness was fabricated [11]. Next, a putty/wash impression (Speedex; Coltene/Whaledent, Altstatten, Switzerland) was made from the composite disc to serve as a mold. The bonding agents were applied in the mold, and were allowed 3 hours in order for the solvent to evaporate [12]. Next, they were light-cured using a LED curing unit (Optilux 501; Kerr, Danbury, CT, USA) with a light intensity of 650 mW/cm2 for 20 seconds. Accordingly, 10 specimens were fabricated from each adhesive.

The specimens then underwent the microhardness test in a hardness tester (Z020; Zwick Roell, Ulm, Germany). For this purpose, each specimen was subjected to 50 g load for 10 seconds. The microhardness of each specimen was measured at 3 points, and the mean of the three values was calculated and reported as the microhardness of the respective specimen. The microhardness of the specimens was measured immediately after curing. Next, they were incubated at 37°C for 24 hours, and their microhardness was measured again. The specimens were then incubated at 37°C for 6 months and subsequently underwent the measurement of microhardness again [13, 14].

Normal distribution of data was evaluated by the Shapiro-Wilk test. Accordingly, repeated measures two-way ANOVA was applied to assess the effect of type of adhesive and time of measurement on microhardness. Since the interaction effect of the two factors was found to be significant, microhardness of each adhesive was evaluated over time using repeated measures one-way ANOVA. Pairwise comparisons were performed by the Bonferroni test. Also, the microhardness of adhesives was compared at each time point using one-way ANOVA, followed by pairwise comparisons with the Tukey's HSD test. Data were analyzed by SPSS version 20 (SPSS Inc., IL, USA) at 0.05 level of significance.

Results

Table 2 presents the measures of central dispersion for the microhardness of the three groups at different time points. The Shapiro-Wilk test showed normal distribution of microhardness data in all three groups at all time points (P>0.05).

Repeated measures ANOVA revealed a significant increase in the mean microhardness over time (P<0.001). Also, the interaction effect of type of adhesive and time of measurement on microhardness was significant (P<0.001). In other words, the gradient of increase in microhardness was different among the three adhesives. Thus, subgroup analysis was performed using repeated measures one-way ANOVA to assess the change in microhardness within each group over time. The results showed a significant increase in microhardness over time in all three adhesives (P<0.001). Pairwise comparisons of the time points within each group with the Bonferroni test showed significant differences between all time points, showing a significantly ascending trend over time (Table 3).

Comparison of the microhardness of the three adhesives at each time point by one-way ANOVA also revealed a significant difference (P<0.001). Thus, pairwise comparisons were performed by the Tukey's test. The results showed that GPB had minimum, and CUBQ had maximum microhardness immediately after curing. All pairwise comparisons revealed significant differences immediately after curing (P=0.00 for all) as follows: GPB< SBU<CUBQ.

The magnitude of increase in microhardness in the first 24 hours was significantly different among the three adhesives (P<0.001). Pairwise comparisons by Tukey's test showed minimum increase in CUBQ, and maximum increase in GPB with significant differences among all three (P<0.01 for all, Figure 1). The same results were obtained at 6 months, compared with baseline.

Table 1. Composition of the three adhesives evaluated in this study

Manufacturer	Adhesive	Composition
Kuraray Noritake (Japan)	Clearfil universal bond quick	bis-GMA, MDP, HEMA, hydrophilic amide monomer, filler, ethanol, water, NaF, photo-initiators, chemical polymerization, accelerator, silane coupling agent, others
GC Corporation (Japan)	G-Premio bond	MDP, 4-MET, MEPS, BHT, acetone, dimethacrylate resins, initiators, filler, water
3M (USA)	Scotchbond Universal	MDP, HEMA, dimethacrylate resins, vitrebond copolymer, filler, ethanol, water, initiators, silane

Table 2. Measures of central dispersion for the microhardness of the three groups at different time points

Material		Immediately after curing	24 hours	6 months
Clearfil Universal Quick	Mean	9.8000	11.3660	19.8330
	Std. Error of Mean	0.45915	0.42290	0.31909
	Minimum	7.00	10.00	18.33
	Maximum	12.67	14.00	21.33
G-Premio	Mean	1.9000	12.1350	20.2320
	Std. Error of Mean	0.21123	0.36267	0.63627
	Minimum	1.00	10.67	18.00
	Maximum	3.00	14.67	23.33
Scotchbond	Mean	7.8330	11.5000	21.3000
	Std. Error of Mean	0.45342	0.57823	0.32375
	Minimum	6.00	8.33	20.00
	Maximum	10.67	14.67	22.67

Table 3. Pairwise comparisons of the time points regarding microhardness within each group

Material	(I) Time		(J) Time		Mean Difference (I-J)	Std. Error	Sig.[a]
Clearfil Universal Quick	Dimension 2	1	dimension3	2	-1.566*	0.522	0.045
				3	-10.033*	0.527	<0.001
			dimension3	3	-8.467*	0.605	<0.001
G-Premio	Dimension 2	1	dimension3	2	-10.235*	0.442	<0.001
				3	-18.332*	0.573	<0.001
				3	-8.097*	0.803	<0.001
Scotchbond	Dimension 2	1	dimension3	2	-3.667*	0.519	<0.001
				3	-13.467*	0.534	<0.001
				3	-9.800*	0.524	<0.001

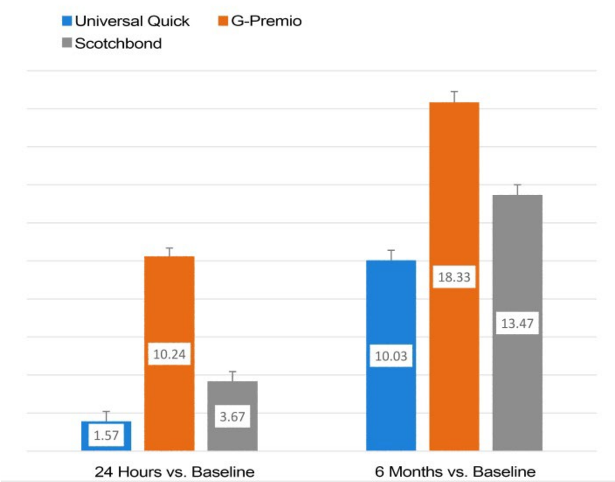

Figure 1. Mean increase in microhardness of different adhesives at 24 hours and 6 months compared with baseline

Discussion

Low bond strength and difficult procedural steps of the application of dentin bonding agents have adverse consequences such as increased frequency of procedural errors due to high technical sensitivity, high number of application steps, time-consuming nature, and tooth discoloration due to caries recurrence and subsequent treatment failure. Considering the significance of mechanical properties of dentin bonding agents, particularly universal adhesives, this study assessed the microhardness of CUBQ, GPB, and SBU over time.

The null hypothesis was that no significant difference would be found in the microhardness of CUBQ, GPB, and SBU at different time points after curing. The results showed that the mean microhardness of the three adhesives at 24 hours and 6 months was close, with no significant difference between the two-time points. Studies evaluating the mechanical properties of dental materials over time typically choose 6 months to simulate long-term clinical use in the oral environment, and also 24 hours to assess the changes shortly after polymerization [13, 14]. The order of the mean microhardness was as follows immediately after curing: CUBQ>SBU>GPB with a significant difference among the three adhesives. Low immediate hardness of GPB and its low degree of conversion, compared with other bonding agents, have been previously reported in the literature [15, 16]. The safety data sheet of the bonding agents used in the present study showed the highest solvent content in GPB. Moreover, water is another constituent of this bonding agent. Both the solvent and water serve as plasticizers and decrease the degree of polymerization. This statement explains low immediate hardness of GPB, at least partly. Additionally, the manufacturer of GPB recommends drying with high-pressure air spray, which was not performed in the present study, increasing the possibility of phase separation in the cured bonding agent, which can also decrease hardness. Lower microhardness of GPB at 24 hours may be due to the higher solvent content and its insufficient evaporation. In other words, insufficient solvent evaporation in GPB and water insolubility of camphorquinone as the main initiator in this bonding agent, which causes incomplete polymerization of its hydrophilic phase may be other reasons for its low immediate hardness.

Statistical analyses also showed a significant difference in microhardness over time within each group, such that the mean microhardness increased over time, similar to some previous studies [17, 18]. CUBQ and SBU experienced a greater increase in microhardness than GPB over time. Thus, the null hypothesis of the study was rejected. In GPB, passage of time resulted in evaporation of the hydrophilic phase. Thus, at 6 months, absence of water and minimal amount of the hydrophilic phase improved the long-term hardness. Evidence shows that dark polymerization can increase the degree of conversion and subsequently the hardness over time [19].

SBU contains methacrylate modified carboxylic acid copolymers that enhance the bond to dentin. Also, SBU and CUBQ contain methacryloxypropyltrimethoxysilane, which enhances their bond strength to other substrates. CUBQ has a high success rate attributed to the presence of 10-methacryloyloxydecyl dihydrogen phosphate in its composition [20, 21]. A meta-analysis by De Munck et al. [22] reported that CUBQ is the best self-etch adhesive in terms of performance, and has excellent long-term clinical service both in vitro and in vivo.

Papadogiannis et al. [16] evaluated the microhardness of several universal adhesives. They showed low microhardness of universal adhesives. They explained that adhesives that contain active methacrylates cannot preserve the water molecules and thus, their low microhardness can be attributed to the plasticizing effects and low cross-linking capacity of their molecules. They indicated that SBU had a higher microhardness than others, probably due to the presence of carboxylic acid polymers in its chemical composition. This polymer absorbs water through hydrogen bonds, and significantly decreases the rate of unstable failures. These results confirm the present findings regarding higher microhardness of SBU at 6 months compared with other groups. In justifying this finding, it should be noted that application of bonding agent (irrespective of its type) and its penetration into dentinal tubules and formation of hybrid layer in composite restorations reinforce the tubular structure and enhance the microhardness [16].

This study had an in vitro design. Thus, the results cannot be reliably generalized to the clinical setting due to inherent limitations of in vitro studies. Controlling solubility and water sorption is essential for maintaining optimal microhardness of dental adhesives. Future studies are recommended to assess the microhardness of adhesives over longer periods.

Conclusion

The present results showed that the microhardness of all three tested universal adhesives increased with time. CUBQ showed significantly higher microhardness immediately after curing. However, the microhardness of the three adhesives was not significantly different at 24 hours and 6 months.

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