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Evaluation of the effect of shelf life of different universal bond systems on dentin bonding

Aybike Bas[1], Periş Çelikel[2]

[1] Atatürk University Faculty of Dentistry, Department of Pediatric Dentistry, Erzurum, Türkiye

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Correspondence:

Dr. Aybike BAS
Atatürk University Faculty of Dentistry, Department of Pediatric Dentistry, Erzurum, Türkiye.
E-mail: aybkbass@gmail.com

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Abstract

Aim: The aim of this study was to investigate changes in dentin bonding caused by the expiry of shelf life of five universal bond systems.

Methodology: According to the manufacturer's instructions, both expired and non-expired samples of five universal bond agents [Prime&Bond (Dentsply), Solare Bond (GC), OptiBond (Kerr), Clearfil S Bond (Kuraray), and G-Premio Bond (GC)] were applied to 10 human teeth. Composite blocks measuring 4-5 mm in thickness were then created on the teeth following the application of the bond. The teeth were cut to obtain samples in the form of square sectioned rods approximately 1.0 × 1.0 mm in width and 8-9 mm in length. These samples were then subjected to microtensile bond strength testing, and the results were statistically analyzed ($p<0.05$).

Results: When evaluating the difference in microtensile bond strength between expired and non-expired adhesive agent groups of the same brand at different shelf life stages, there was a significant decrease between the Kerr, Clearfil, and G-Premio groups ($p<0.05$), while no significant difference was found in the Prime&Bond and Solare groups ($p>0.05$). When microtensile bond strength values were assessed among different groups based on shelf life, no significant difference was observed ($p>0.05$). Among expired adhesive agents, Solare exhibited the highest microtensile bond strength, while among non-expired adhesive agents, G-Premio showed the highest microtensile bond strength.

Conclusion: Universal bond systems should be used in accordance with the manufacturer's instructions under appropriate storage conditions and considering the shelf life. However, it has been shown that there is no difference in bond strength of universal bond systems within the first six months following the expiration date indicated; they can be used during this period if stored under appropriate conditions.

Keywords: Bonding agents, shelf life, universal bond system, microtensile bond strength

Introduction

Adhesive systems are essential agents today, especially in ensuring the connection of restorative materials with the tooth, in both aesthetic applications and in increasing the strength of the tooth structure. From past to present, adhesive agents have been introduced to the market in different forms of use, but currently, universal adhesive systems are preferred because they reduce the multistep applications of previous systems to a single step, their application procedures have been simplified, they require less technical expertise, and they are thus more user-friendly (1)

Despite the many advantages of universal adhesive systems, they may present disadvantages during storage, such as polymerization or hydrolysis of the monomer they contain, evaporation of their contents, or degradation of the additives. Therefore, it is known that the shelf life of the components of these adhesives must be taken into account. Universal adhesive systems containing 2-hydroxyethyl methacrylate (HEMA) or an acidic monomer have been shown to have a shorter shelf life due to a tendency to degrade more rapidly over time (2). In this case, poor bonding can occur, leading to treatment failure. To prevent this, manufacturers recommend ideal storage conditions and shelf life to ensure that adhesive agents are used in their optimal condition (3). Additionally, most manufacturers recommend using single-dose packages to prevent adverse effects during storage (4).

Although manufacturing companies establish the recommended storage conditions and shelf life, many dentists continue to use these agents beyond their expiration dates, assuming that there will be no change in effectiveness (5). Our review of the literature revealed a limited number of studies that have examined whether the bonding of adhesive systems changes after their shelf life has expired, leaving a gap in our knowledge about the optimal use of these products that clinicians continuously apply in treatments. Therefore, this study investigated microtensile bond strength by applying samples of five universal bond systems before and after the expiration of their stated shelf life. Thus, our study will contribute to the literature and shed light on the effective usability of universal bond systems. The null hypothesis of this study was that exceeding the shelf life of bonding agents has no effect on the quality of dentin bonding.

Materials and Methods

This study received ethical approval from the Ethics Committee of Atatürk University Faculty of Medicine (29/03/2024 – B.30.2.ATA.0.01.00/239).

Before the experimental phase of our study, 20 teeth extracted from individuals aged 16–40 years were embedded in acrylic blocks. The embedded teeth were sectioned on the occlusal surface using a low-speed diamond saw under water cooling to expose dentin, and the exposed dentin surfaces were prepared by sanding with silicone carbide paper to facilitate the application of adhesive resins.

Applied bonding agents and laboratory procedures

Group 1

Stored under appropriate conditions and never used, samples of bonding agents Prime&Bond [(Dentsply Sirona), (PB)] and Solare Bond [(GC), (SB)] that had exceeded their shelf life of approximately six months; OptiBond [(Kerr), (OP)] that had exceeded its shelf life of approximately eight months; and G-Premio Bond [(GC), (GP)] and Clearfil S Bond [(Kuraray Noritake Dental), (CF)] that had exceeded their shelf life of approximately three years were applied to the extracted teeth.

Group 2

The bonding agents Prime&Bond, Solare Bond, OptiBond, G-Premio Bond, and Clearfil S Bond that had not exceeded their shelf life were applied to the extracted teeth. Information regarding the contents of the bonding agents used in our study is provided in Table 1.

A composite block 4 mm thick was created on the samples of both groups. The teeth with the composite block were cut perpendicular to the bonding interface with a diamond cutting disc rotating at low speed under water cooling. Square-section, rod-shaped samples 1.0 × 1.0 mm wide, ideally cut, and 8–9 mm long were selected from each group.

Microtensile bond strength test

The samples were fixed to the microtensile test apparatus with a cyanoacrylate adhesive and subjected to microtensile bond strength testing by applying a tensile force of 100 N at a speed of 1 mm/min. Samples that broke during preparation or while being placed in the test apparatus were considered prefailures and excluded from the study. Measurements were conducted on 10 samples from each group.

Statistical analysis

Analyses were performed by using SPSS software (IBM SPSS Statistics version 25, IBM Inc., Armonk, NY, USA).

For comparing bonding agent samples exceeding and not exceeding shelf life in dependent groups, the Wilcoxon signed-rank test was applied ($p<0.05$). To compare samples of different bonding agents exceeding and not exceeding shelf life, independent groups were tested using the Kruskal-Wallis test ($p<0.05$).

Table 1. Contents of the universal bonding agents used in the study

Bonding Agent Lot Number (Exceeded shelf life agent / Not exceeded shelf life agent)	Manufacturer	Content
Prime&Bond **Lot:2109000549** **Lot:2307000522**	Dentsplay Sirona Konstanz/Germany	PENTA , 10-MDP,Active Guard Technology cross-linker, CQ tertiary amine, isopropanol, and water
Solare Universal Bond **Lot: 2111221** **Lot:011986**	GC Corporation Tokyo/Japan	4-META, UDMA, TEGDMA, 10-MDP, acetone (25-50%), water (>20%), silanated colloidal silica, initiators.
OptiBond Universal **Lot: 00215375** **Lot:9740868**	Kerr Corporation, Middleton, WI, USA	HEMA, GDMA, GPDM, acetone, water, and ethanol.
G- Premio Bond **Lot: 180501A** **Lot:2207071**	GC Corporation Tokyo/Japan	10-MDP, 4-META, 10-methacryoyloxydecyl dihydrogen thiophosphate, methacrylate acid ester, distilled water, acetone, photo-initiators, silica fine powder
Clearfil Universal Bond **Lot: 000014** **Lot: 000108**	Kuraray, Medical, Sakazu,Okayama, Japan	10-MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, camphorquinone, ethanol, water, silanated colloidal silica

Results

When microtensile bonding strengths of samples of the same bonding agent that had and had not exceeded its shelf life were compared, while there was a statistical difference between Optibond, Clearfil, and G-Premio samples ($p<0.05$), there was no statistically significant difference between Prime&Bond and Solare samples ($p>0.05$). Statistically, there was no significance found when comparing the microtensile bond strengths among samples of different brands that exceeded their shelf life ($p>0.05$). Similarly, there was no statistical significance found when comparing the microtensile bond strengths among samples of different brands that had not exceeded their shelf life ($p>0.05$). A comparison of the microtensile bond strengths of the samples is provided in Table 2.

Among samples of different brands that had exceeded their shelf life, the highest microtensile bond strength was observed in the Solare group, while among samples that had not exceeded their shelf life, the highest value was measured in the G-Premio group (Fig. 1).

Table 2. Statistical comparison of microtensile bond strengths of samples

	Time to exceed shelf life	Fracture values of bonding agents that have exceeded shelf life		Fracture values of bonding agents that have not exceeded shelf Life		*p*
		Median (Percentile 25-75)	Mean± SS	Median (Percentile 25-75)	Mean± SS	
Prime&Bond	6 M	14.6 (11.4-17.8)	15.3±4	17.3 (14.1-18.7)	17.1±2.8	0.398
Solare	6 M	16.4 (14.1-22.3)	17.5±5.8	16.9 (11.4-20)	17.5±8.5	0.684
OptiBond	8 M	15 (9.6-19.6)	15±4.8	21.9 (13.7-24.1)	19.7±5.4	0.043*
Clearfil	3 Y	10.5 (7.3-13.2)	10.3±3	15.5 (12.8-21.4)	16.6±4.9	0.028*
G- Premio	3 Y	11 (7.3-15.9)	11.8±4.8	27.3 (19.1- 31)	25.7±5.9	0.028*
p		0.073		0.079		

*: p<0.05

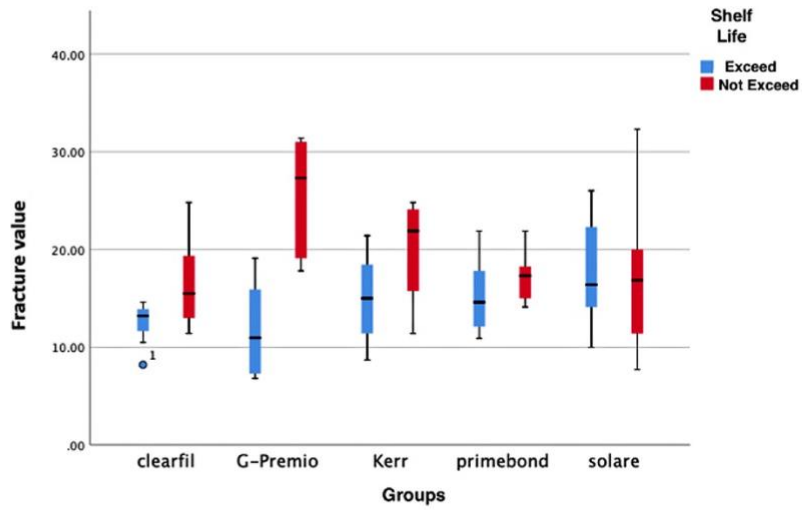

Figure 1. Measured values of all samples that have and have not exceeded their shelf life

Discussion

The microtensile bond strength was compared between samples whose shelf life was exceeded for different periods (six months, eight months, and three years) and those whose shelf life was not exceeded. These are among the universal bond systems that are frequently preferred in today's restorative procedures. The purpose of comparing the dentin bond of universal bond systems that have exceeded their shelf life with those that have not exceeded their shelf life is that although universal bond systems are recommended by manufacturers to be consumed under appropriate storage conditions and within the specified shelf life, this situation can sometimes be ignored by physicians. Our study has shown that universal bond systems that do not contain HEMA monomer can be used during the first six months after exceeding their shelf life. However, it has been observed that if the shelf life of universal bonding agents exceeds six months, the dentin bond strength decreases. Therefore, the null hypothesis of this study was rejected.

In the evaluation of bonding to the tooth structure, in vitro test methods that provide reliable data are preferred for determining the effects of material development and/or experimental variables. Microtensile bond tests are frequently preferred in vitro tests for evaluating dentin bonding strength. The advantages of this method are that it provides reliable measurements even on small samples, more samples can be taken from a single tooth, SEM examination can be performed on more than one sample at the same time, and it provides more reliable results in the evaluation of bonding. Therefore, the microtensile bond strength test was used to measure our study samples (6).

HEMA monomer is an ingredient currently used in universal bond systems because of its hydrophilic nature, which creates a more wettable area on the tooth surface and increases adhesive-tooth bonding (3, 7). However, rapid hydrolysis and degradation pose a threat to the shelf life. The results of this study showed that the bond strength of the OP containing HEMA monomer, which exceeded the shelf life of approximately 8 months, was significantly reduced compared to the sample that did not exceed the shelf life. It was found that the dentin bonding values of P&B and SB, which did not contain HEMA and exceeded the shelf life by a similar period of time (6 months), did not differ from the samples that did not exceed the shelf life. One reason for this situation may be the negative effect of the HEMA monomer on the shelf life, as stated in the literature (3, 4, 7).

Similar to the results of our study, Cuevas-Suarez et al. reported no significant difference between the dentin bond strengths of P&B that did not exceed the shelf life and P&B that had reached the end of the shelf life. It was stated that this might be due to the fact that the PENTA-P monomer in the P&B content makes the material more resistant to hydrolytic degradation and, thus more stable during the shelf life (3).

When the literature is examined, it is observed that the change in dentin bonding depending on the shelf life of SB and OB has not been evaluated. Considering the content of SB, 4-MET and 10-MDP monomers are important ingredients that provide bonding to enamel and dentin (2). Since there is no HEMA in the content of SB, it is thought that these ingredients do not degrade and can maintain bond strength even if the shelf life is exceeded.

Universal bonds are self-etch systems, and hydrolysis events occur frequently because of their acidic nature (8). The hydrolysis of the ester portion of the HEMA molecule is closely related to the acid concentration in the solution, and hydrolysis begins before the bonding agent is used (9). It has been shown that a pH value of less than two for the bonding agent containing HEMA leads to rapid hydrolysis of the molecule (10). Since OB has a pH value of 1.4, it is thought that the HEMA structure in its content is rapidly hydrolyzed, and its content changes faster. Therefore, OB may have shown a lower microtensile bond strength than P&B and SB, although they exceeded the shelf life in similar periods.

Among our study groups, GP and CF were bonding agents that exceeded the shelf life of approximately three years. Since restorative treatments could not be performed at our university during the pandemic, the shelf life of these agents expired, and they were not used in patients. However, these bonds in the form of disposable packages were stored in accordance with the manufacturer's recommendations for preclinical training and various studies. In the literature, there are limited studies evaluating the microtensile bond strength of bonding agents that have exceeded their shelf life; however, in these studies, the maximum shelf life was three months (3, 5, 11). Before starting our study, it was thought that bonding agents (GP and CF) with a shelf life of more than 3 years would not bond to dentin. The results of this study showed that although these two groups showed a bond below the desired dentin bonding level (12), this bond was not completely lost. This indicates that the functional monomers of the universal bond systems stored under appropriate conditions are not completely degraded, although they are well beyond their shelf life.

It is known that the ideal bond strength of bonding agents to dentin should be 15-28 MPa (12). In our study, the average microtensile bond strength of all the bond groups that did not exceed the shelf life was measured in the range of 15-28 MPa and was measured in the GP, OB, SB, P&B, and CF groups in order from the highest to the lowest value. There were no statistically significant differences between the groups in terms of bond strength. In a study by Jafarnia et al., similar to our findings, the microtensile bond strength of GP to dentin was found to be the highest, whereas that of the CF group was the lowest. In addition, the mean bond strength values were lower than those in the present study. It was stated that the high bond strength of the GP group was related to the three strong bonding compounds 10-MDP, 4-META, and 10-methacryoyloxydecyl dihydrogen thiophosphate in the bonding agent. It was stated that the reason for the lowest bond strength of the CF group may be the lack of functional monomers that provide adhesion (13). In addition, high concentrations of HEMA may need to be added to facilitate the solubilization of Bis-GMA in CF13. High concentrations of HEMA may adversely affect the binding of MDP with hydroxyapatite (14, 15).

OB contains a GPDM monomer instead of a 10-MDP monomer. Studies have shown that bonding agents containing GPDM monomers have a significantly lower bond strength to dentin than MDP (16, 17). This may be explained by the fact that although GPDM adsorbs to hydroxyapatite, it does not form a stable calcium salt. There is no strong bonding between GPDM and hydroxyapatite, unlike the strong binding formed by MDP with hydroxyapatite (16). As a result of our study, OB without 10- MDP content showed lower bonding strength than GP containing 10- MDP. However, in the other groups (P&B, SB, CF), although 10- MDP was present, the bonding strength was lower than that of OB.

Although functional monomers play a major role in the bond strength of universal bond systems, another important factor is the pH of materials. Today, bonding agents are classified according to their acidity as ultramild (pH>2.5), mild (pH=2), intermediately strong (pH=1-2), and strong (pH<1) (18). By increasing the pH of bonding agents, more effective demineralization can be achieved, and the micromechanical bond surface can be increased. When we look at our study groups, it is known that the pH value of the GP, OB, and SB groups is intermediately strong (1.3-1.5), while the P&B and CF groups are ultramild (pH>2.5). Therefore, although the functional monomers of OB cannot show as effective bonding as the 10-MDP monomer, their acidity close to strong may provide more demineralization in dentin (15). Thus, the hydrophilic monomers HEMA and GPDM may have increased dentin bonding by providing a greater diffusion area (17).

Although SB does not contain HEMA, it has a pH close to strong and contains a strong functional monomer, such as 10-MDP. It showed a lower bond strength than OB and similar bond strength to P&B with mild acidity containing 10-MDP. In the study by Cochinski et al., SB showed lower dentin bond strength than another universal bond without HEMA and with mild acidity, and it was stated that this may be due to other ingredients in the SB content worsening the bond (15).

The shelf life of the universal bonding agents was determined to be 2 years on average. In this process, many factors, such as whether they are kept under appropriate storage conditions, whether they are disposable, their acidity, and the stability of the monomers in their content, are very important for their effectiveness (4). For this reason, bonding agents that had never been used before and were kept under appropriate storage conditions were used in our study. The main limitation of our study was that the measurements were performed on 10 samples. In addition, the fact that the tooth samples were obtained from people of different age groups and that the dentin structure varied from person to person may have affected the amount of smear layer formed and changed the bonding.

In accordance with the literature, the results of our study showed that HEMA content and acidity may decrease the shelf life (3, 4), as well as the monomers in the content and pH value (15, 17, 19, 20) are important in the connection of universal bond systems. The effect of the content of bonding agents on the shelf life should be revealed by conducting new studies with more samples, different contents, and universal bonding agents that have exceeded the shelf life at different times.

Conclusion

Universal bonding systems should be stored and used according to the manufacturer's recommendations. The bonding efficacy of universal bond systems that have exceeded their shelf life depends on the content of the materials and the elapsed time. It was shown that there is no difference in the bond strength of HEMA-free universal bond systems within the first six months following shelf life expiration; they can be used throughout this period if stored under appropriate conditions. The presence of functional monomers and acidity are important factors in increasing the strength of bonding to dentin in universal bond systems that have not exceeded their shelf life.

Disclosures

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Ethical Approval: Ethics committee approval was received for this study from the Atatürk University Faculty of Medicine Ethics Committee, in accordance the World Medical Association Declaration of Helsinki, with the approval number: 2024 - B.30.2.ATA.0.01.00/239.

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