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BIODEGRADATION AND DENTIN BONDING EFFECTIVENESS OF ONE "UNIVERSAL" SELF-ETCH ADHESIVE USED IN MULTI-MODE MANNER

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ABSTRACT

A new type of one-step self-etch adhesives classified as "Universal" or "multi-mode" adhesives appeared in recent years. The idea is that these adhesives can be applied simultaneously with both techniques - etch and rinse and with self-etching technique, without compromising the bonding effectiveness. The aim of this study is to evaluate the micro-tensile adhesive bond strength to dentin of permanent teeth achieved after application of Single Bond Universal (3M ESPE) adhesive system used in multi-mode manner. The results of our study show that the use of this universal adhesive system in multi-mode manner will not lead to the same results regarding the achieved bond strength with dentin. The additional etching with 37% phosphoric acid as well as the application of the adhesive in several layers deteriorates the bond strength right after application and after six months storage in artificial saliva medium (SAGF).

Keywords: adhesion; durability; micro-tensile bond strength; multi-mode manner; permanent teeth; universal all-in-one adhesive.

INTRODUCTION

In recent years, the attention of adhesive dentistry is focused on the development of simplified adhesive systems that reduce the time needed for implementation of the clinical protocol, limit the possibility of technical faults in their administration and at the same time improve adhesion to tooth structure [1]. A consequence of this endeavor are the generations of self-etch adhesives - self-priming and one step self-etching (all-in-one) adhesive. One-step self-etch adhesives provide in one step (etching, priming and bonding) bonding with the enamel and dentin [2-4]. For the moment they are identified as the simplest intermediaries to bind the composite with the tooth structure [1].

The advantages of self-etch adhesives are the easy use and the brief Application Protocol combined with lesser technical sensitivity [2, 5-7]. Due to the lack of a separate etching step there is no risk of over-etching, over-drying or leaving excessively moist dentin, which are effects observed with the use of total etch adhesives [1, 5, 8]. These adhesives are not associated with postoperative sensitivity [9-11] and are accepted as particularly suitable for restoration in primary dentition [12, 13].

Self-etch adhesives also have a number of disadvantages [1, 9, 11, 14]. One of the serious shortcomings stems from their inherent acidic functional monomers [3, 8, 11]. These monomers are highly hydrophilic, favor water sorption and are susceptible to hydrolytic biodegradation, especially all-in-one adhesives that combine self-etch primer and hydrophobic resin in one step [11, 15, 16]. This leads to the creation of highly hydrophilic polymers, which are very permeable to water after polymerization [16]. The adhesive layer formed through their mediation tends to attract water from the inherently humid dentin [17, 18]. As a result of this, instead of providing a reliable sealing of the dentin, polymerized hydrophilic adhesive acts as a permeable membrane [17, 19-21]. This leads to the hydrolysis of the resin polymers and as an end result – to the biodegradation of the adhesive bond over time [22, 23].

Another drawback of the self-etching adhesive approach is the insufficient strength of the bond with the tooth structure and in particular with the enamel [14, 24, 25]. The reason for this is that they are less aggressive in acidity, as compared to the phosphoric acid used in the total etching [25, 26]. On the basis of their acidity (pH) self-etching adhesives are divided into mild (pH > 2), medium (1 < pH < 2) and strong (pH < 1) [4,27]. This acidity affects the interaction with tooth structure and determines the adhesive bond strength [7]. The weaker acidity limits the etching possibility to a greater depth and the creation of micro-retention areas and correlates with enamel bond strength [24, 25]. The depth of demineralization is also determined by the smear layer thickness created during cavity preparation and its buffering effect [28, 29]. A thicker smear layer within the dentin, combined with application of medium aggressiveness self-etch adhesives has been found to limit monomer penetration in the underlying dentin [9, 30-32]. Additional factors affecting the depth of demineralization and monomer infiltration in self-etch adhesives are their composition, viscosity and wetting ability, the way they are mixed and their application on the tooth structure [33, 34].

A new type of one-step self-etch adhesives classified as "Universal" or "multi-mode" adhesives appeared in recent years. The idea is that these adhesives can be applied simultaneously with both techniques - etch and rinse and with self-etching technique, without compromising the bonding effectiveness [35, 36].

At present, there is little available information about the performance of these adhesives, due to their recent introduction into clinical practice. As regards the problem area - adhesion to dentin, it is not yet firmly established if we can expect equivalent results regarding the quality and adhesive bond strength, regardless of the route of administration, and whether these new adhesives can overcome some of the above-mentioned critical points of the self-etch approach.

The aim of this study is to evaluate the adhesive bond strength to dentin of permanent teeth achieved after application of Single Bond Universal (3M ESPE) adhesive system used in multi-mode manner. The null hypotheses tested were that: (1) there is no difference in the immediately registered micro-tensile bond strength, irrespective of the application of the adhesive system; (2) six-month artificial saliva storage does not affect the adhesive bond strength in different groups.

MATERIALS AND METHODS:

The study included 60 intact permanent molars. They were collected from healthy individuals with signed informed consent. After extraction teeth were placed for 10 min in a 10% formalin solution and cleaned. Until the testing they were stored in saline solution for no longer than 1 month. The teeth were divided randomly into three groups (20 teeth per group). All teeth underwent a thermal stress test. The micro-tensile bond strength of half of the teeth (N=10) from each group were tested immediately after their preparation and of the other half of the teeth after 6 months in artificial saliva medium (SAGF) (37p).

Preparation of the Tooth Surface:

The occlusal enamel of each tooth was removed with turbine fissure diamond bur (ISO 806204108524835010) under water and air cooling and controlled depth, pre-marked with round turbine bur (806 314 001 534 014). The resulting surface is polished with an abrasive disk. This yields a flat dentin surface of comparable distance from the central fissure. The samples were observed with an optical microscope OLYMPUS VANOX-T and under magnification 25x to 100x to ascertain whether the enamel is completely removed from the occlusal surface. The teeth were divided into three groups according to the method of application of the adhesive system (Table 1). The tested adhesive system is Single Bond Universal (3M ESPE), pH=2.7, which is implemented in a multi-mode manner. In group 1, the adhesive system was applied following the manufacturer‘s instructions - by rubbing on the exposed dentinal surface for 20 seconds. In the samples from Group 2 adhesive was applied in three layers by rubbing for 20 seconds and blowing between the rubs. There are studies that show that the application of the self-etching adhesives in several layers on dentin leads to a greater bond strength [28, 37]. That was our reason to implement such a protocol in the samples from group 2. In group 3 we etched dentin for 15 seconds, washed, dried and subsequent rubbed adhesive for 20 seconds.

Table 1. Distribution of samples by groups according to the method of application of the adhesive system.

Group	N	Method of adhesive application
Group 1	20	Rubbing on dentin for 20 sec., drying for 5 sec, 10 sec polymerization (following manufacturer‘s instructions)
Group 2	20	Application of adhesive in three layers - by rubbing for 20 seconds and blowing between rubs for 5 sec., 10 sec polymerization after the last application of the adhesive.
Group 3	20	Etching dentin for 15 sec. with 37% phosphoric acid, washing for 5 sec, drying with mild air stream, rubbing of the adhesive for 20 sec ., drying for 5 sec ., 10 sec polymerization

**n=number of teeth per group*

Restorations with composite Filtek Ultimate (3M), color A3 body, applied in 3 layers, each with a thickness of 2.0 mm were made on the dentin surface of all samples. Each of these layers is light polymerized for 40 s with a photopolymer lamp (Elipar S10 3M ESPE). After 24 hours of storage in saline solution (37°C) samples were subjected to thermal loads in a temperature range from 5°C to 55°C for 1 500 cycles (Thermocycler SD Mechatronik).

Sample preparation for μTBS - testing:

Samples from each group were cut perpendicular to the restored surface (with microtome Leica SP 1600) to achieve rods with square cross-section (width of 2 mm by 2 mm, height of 20 mm 2x2x20). The research covered rods from central areas of the prepared samples. Half of the samples from each group were prepared in this way immediately after thermal loads. The other half of the teeth of each group were kept in artificial saliva medium (SAGF) at 37°C for 6 months, and then subjected to the same micro-tensile test (μTBS).

μTBS - testing

The rods received (from tooth samples) were still fixed by cyanoacrylate adhesive on plates bearing apparatus LMT- 100 (LAM Technologies electronic equipment - Italy) and subjected to micro-tensile test conducted at a sliding speed of 1 mm/min. The test was terminated after the sample was destroyed, marking the value of strength in MPa, causing destruction. The values obtained from the test were subjected to statistical analysis using SPSS 19. We used a t-test to check the difference in the average values of the respective groups, adopting a 95% confidence interval, and 5% significance or risk of error from the first type.

RESULTS AND DISCUSSION

In this study we evaluated whether multi-mode application manner of universal adhesive system Single Bond Universal (3M ESPE) affects the adhesive bond strength to the dentin of permanent teeth immediately and after a 6-month artificial saliva storage. Values and standard deviation for the resulting bond strength of the three groups are presented in Table 2.

Table 2. Mean values and standard deviations of micro-tensile bond strength (MPa) to dentine at the beginning and after 6 months.

Group	N		Mean values		Std. Deviation	
	Start	After 6 m	Start	After 6 m	Start	After 6 m
Group 1	40	40	11.2325	11.6450	3.0507	2.4847
Group 2	40	40	9.4125	8.0275	2.5149	2.4375
Group 3	40	40	8.8725	7.3425	3.7139	2.5322

The highest average immediate bond strength to dentin was registered in samples of Group 1 (m=11.23±3.05 MPa, Table 2). A statistically significant difference was found between the average value of the immediate bond strength after a test conducted between samples of group 1 and those values for samples from group 2 (m=9.41 ± 2.51MPa, Table 2) and 3 (m=8.87 ± 3.71 MPa, Table 2) ($p < 0.05$, Table 3). The difference in averages between Group 2 and Group 3 was not confirmed as statistically significant (Table 3).

Table 3. Mean values of micro-tensile bond strength (MPa) to dentine at the beginning.

Group	Compare with	Mean difference	95% Confidence Interval of the Difference		T	P
			Lower	Upper		
Group 1	Group 2	1.8200	.5579	3.0821	2.917	0.006
Group 1	Group 3	2.3600	.8647	3.8553	3.192	0.003
Group 2	Group 3	.5400	-.7841	1.8641	.825	0.414

Application of adhesive in 3 layers and the additional etching with 37% phosphoric acid did not lead to better results regarding immediate adhesive bond strength, and a weaker dentin bond strength was registered. These results reject the first hypothesis tested by us. The application method of the adhesive system affects the dentin bond strength. Our results for micro-tensile dentin bond strength after additional dentin etching coincide with reports by other authors in the study of self-etch adhesives with this acidity (pH=2.4-2.7) [3, 9, 11, 38-42]. Recorded average values for bond strength in group 2 do not coincide with the results reported by the authors, that the application of the self-etching adhesives in several layers leads to greater strength of bonding to the dentin [28, 37]. Possible reason for this could be the difference in experimental design and the brand of adhesive system.

After six months saliva storage it was again the samples from group 1 that showed the highest measured average bond strength (11.65 ± 2.48 MPa, Table 2). In samples of Group 2, and 3 registered average is respectively 8.02 ±7.34±2.43 MPa and 2.53 MPa (Table 2). After this period a statistically significant difference was found between the averages of samples from group 1, compared with the average of the registered bond strength in samples of Group 2 and 3 (Table 4). The difference in mean values between the groups 2 and 3 cannot be confirmed as a statistically significant (Table 4).

Table 4. Mean values of micro-tensile bond strength (MPa) to dentine after 6 months.

Group	Compare with	Mean difference	95% Confidence Interval of the Difference		T	P
			Lower	Upper		
Group 1	Group 2	3.6175	2.2996	4.9354	5.552	0.000
Group 1	Group 3	4.3025	3.1164	5.4886	7.337	0.000
Group 2	Group 3	.6850	-.2531	1.6231	1.447	0.148

Artificial saliva storage in order to age the specimens [43] demonstrated a repeated trend in recorded averages of immediate bond strength to dentin (Table 3), and again the highest values were recorded in samples from group 1 (Table 4).

The change in the values of the mean bond strength within each group at the beginning and after 6 months is also examined. The results are presented in Table 5.

Table 5. Comparison between mean values of micro-tensile bond strength (MPa) to dentine immediately after preparation and after 6 months water storage in each group.

Comparison	Mean difference	95% Confidence Interval of the Difference		T	P
		Lower	Upper		
Gr. 1 immediate **Gr. 1 after 6m**	-.4125	-1.3177	.4927	-.922	0.362
Gr. 2 immediate **Gr. 2 after 6m**	1.3850	.3852	2.3848	2.802	0.008
Gr. 3 immediate **Gr. 3 after 6m**	1.5300	.2502	2.8098	2.48	0.020

Tracking the change in values of measured average bond strength to dentin immediately and after 6 months brings important information (samples from the same group). Moreover, at present there is no data on the change in the bond strength after long-term saliva storage and thermal aging of samples prepared with universal adhesives in multi-mode manner [44, 45]. The difference in the average bond strength cannot be confirmed as statistically significant regarding the samples of the first group (Table 5). After 6 months in groups 2 and 3 we measured lower average value of the bond strength. The registered changes were statistically significant (Table 5). These results reject the second tested hypothesis regarding samples from groups 2 and 3. With respect to the samples of group 1 it remains valid. Subject to the manufacturer's recommended application protocol of adhesive system after six months there was no change in the dentin bond strength within our research. The possible clinical interpretation of this result is that over time no failure of the filling should be registered. In samples from group 2 and 3 the 6 months storage in artificial saliva medium (SAGF) leads to a decrease in the dentin bond strength. This is probably due to its biodegradation caused by the adverse impact of adhesive application (group 2) and the additional etching dentin (group 3). Our results coincide with the results of other authors [36, 45].

In literature the effect of the additional demineralization with phosphoric acid is associated with the composition of the functional monomer and the generation of adhesive [11, 45]. Due to the fact that there are many product dependent aspects that determine the interaction between these functional monomers and tooth structure, final conclusions about the effect of additional etching with phosphoric acid on the dentin bond strength of various adhesive systems cannot be made [46-48]. In general, all-in-one adhesives are characterized by much more hydrophilic nature [16]. Their inherent hydrophilic nature is discussed as a possible reason for the significant negative impact on the border area, compared to self-priming adhesives [15]. This naturally leads to the creation of a weak and unstable hybrid layer that is more susceptible to hydrolytic biodegradation and proteolysis [17, 23].

Limiting the acid etching only in enamel during selective etching in a clinical setting is extremely difficult and depends on many factors - the size of the cavity, location, experience of the clinician, viscosity of the etching agent. For these reasons, acid may often fall on the dentin [45, 49]. The negative effect of additional etching with phosphoric acid can be combined consequence of excessive etching, incomplete removal of the acid, incomplete infiltration of the adhesive due to deep demineralization and/or denaturation of the collagen network, extracting the residual hydroxyapatite on the collagen fibers, which affects the ability to chemically binding [11, 42, 50-52].

Additional reason to conduct our research is that the possibility of applying universal adhesive in multi-mode manner will enable the dentist to choose how to use the same adhesive system according to the specific situation determined by the characteristics of the cavity and the conditions for making of the restoration. The results of our study indicate that additional dentin etching and application of this adhesive system does not result in better adhesive strength to dentin. This strength deteriorates over time.

CONCLUSION

The in vitro tests are useful and important for the prediction of the effect of new adhesive systems and their possible correlation with the clinical practice, even though in vitro studies have not been able to fully predict the clinical success.

The results of our study show that the use of universal adhesive system Single Bond Universal (3M ESPE) in multi-mode manner will not lead to the same results regarding the achieved bond strength with dentin. The additional etching with 37% phosphoric acid as well as the application of the adhesive in several layers deteriorates the bond strength right after application and after six months saliva storage.

Further studies are needed to evaluate the effect over the border area adhesive - dentin after long artificial saliva storage as well as clinical studies on the performance of these "universal" adhesive systems.

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