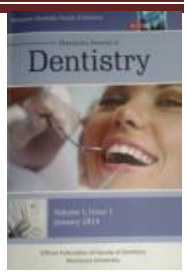

Effect of Etching Method and Time on Surface Roughness and Shear Bond Strength of a Novel Universal Adhesive to Ground Enamel

Amal Mansoor Ali AL-Qadhi[1], Radwa Ibrahim El-Toukhy[2], Salah Hasab Mahmoud[3] BDS, Sana'a University-Yemen
1Post graduate student in Operative Dentistry Department,Faculty of Dentistry,Mansoura University
2 Lecturer, Department of Operative Dentistry Faculty of Dentistry Mansoura University
3 Professor and Chairman of Department of Operative Dentistry Faculty of Dentistry Mansoura University

Abstract:

Purpose: To evaluate the effect of etching method and time on surface roughness and shear bond strength of universal adhesive to ground enamel.

Materials and Methods: Three etching agents, phosphoric acid (PPA), polyalkenoic acid (PLA) and phosphoric acid ester monomer (PPM) with one universal adhesive (G-Premio Bond) were used in the study. Fifty sound human molars were decoronated and sectioned mesiodistally to obtain 100 halves. Each half was fixed in self-cure acrylic resin. Ten specimens were selected as a control group (no etching) whereas, the remaining (n=90) was equally assigned into three tested groups (n=30) according to the etching agents. Every tested group was subdivided into three subgroups (n=10) according to the etching time (5, 10, 15 seconds). Tested groups were treated with acids and surface roughness was measured using a Profilometer. The universal adhesive was applied to all groups. Resin composite block (Tetric N-Ceram) was incrementally built up using a plastic mold. All the specimens were subjected to shear bond strength test.

Results: Surface roughness of enamel treated by PPA was the highest followed by PPM whereas, PLA showed the lowest value.Shear bond strength of universal adhesive to enamel etched with PPM and PPA for 5 to 15 seconds, and PLA for 15 seconds was significantly higher than other groups.

Conclusions: Enamel surface micro-morphology and bonding durability are etching protocol dependent. Enamel treatment withphosphoric acidincreases surface roughness more thanpolyalkenoic acid and phosphoric acid ester monomer. Polyalkenoic acid in 15 seconds enhances the enamel bonding.

.

Introduction

A number of recent studies and literatures showed that success or failure of resin composite restoration in clinical observations and laboratory investigations determines by the quality of the adhesive system at the tooth restoration interface. So, good adhesion to tooth structure is required for clinical success and restoration longevity. [11]

New dental adhesives have been initiated with special focus on their using by decreasing the number of components and clinical steps.Universal adhesives which were introduced to the dental market as a single bottle identified as "multi-mode" or "multi-purpose" that can be used in either etch-and-rinse (E&R), self-etch (SE) or selective etch modes.[10]

Acid etching to enamel causes a selective demineralization, but the best result is revealed with ground enamel rather than unground one because of the high free surface energy, increase porosity, and surface area.[19]

Phosphoric acid dissolves totally the smear layer and increases the wettability of enamel.[1]PPA etching polarizes the surface of enamel which leads to the development of chemical bonding interactions between acidic functional monomers of adhesives and HAp.[17]

PLA is considered one of complex acids that containspolyacrylic, polyitaconic, and polymaleic acids, it is used in etching for cavity cleaning and conditioning in restorations with glass ionomer cement.PLA forms ionic bond between carboxyl groups and calcium of HAp.[4]

Another etching technique is the recently developed PPM.[12] The composition of this acid characterizes by containing a functional monomer 10-MDP. It is a versatile monomer composes oflong carbon chain with two ends of hydroxyl groups. Thus, chemical reaction with dental substrates, resulting in higher bonding strength rather than mechanical adhesion only.[15]

Etching time is an essential step to develop clinical success in adhesive dentistry. However, the increasing in time produced more complex role of irregularities and little projections on surface subsequently high surface roughness.Surface roughness of enamel is important characteristic that is reported to affect adhesion to enamel which is dependent on the resins capacity to infiltrate between rods and crystals leading to micromechanical retention.[16]

Materials and methods:

Three different etching materials: phosphoric acid (Cica, Etching gel, PROMEDICA, Germany), polyalkenoic acid (Ketac Conditioner, 3M ESPE, Germany), phosphoric acid ester monomer (ClEARFIL LINER BOND F PRIMER,Kuraray, Japan). An universal adhesive (G-Primo BOND, GC, Tokyo, Japan) and one type of nanohybrid resin composite restorative material (Tetric N-Ceram, ivoclarVivadent AG, Schaan, Liechtenstein, Batch no. 635780) were used for the restorative procedures.

Freshly 50 extracted sound human permanent molars without any defects or restoration werepreserved in distilled water (4°c) and refrigerated until were used.All the collected molars were sectioned mesiodistally. Roots were removed andspecimens were fixed in blocks of self-cure acrylic resin (Acrostone, Cairo, Egypt.
Only 10 specimens were selected as a control group, whereas the remaining halves (n=90) were divided into three tested groups (n=30) according to the type of etching materials (phosphoric acid, Polyalkenoic acid and Phosphoric acid ester monomer). Each tested group was subdivided into three subgroups (n=10) according to etching time into (5, 10, 15-seconds).

Surface Roughness

Seven specimens from each subgroup were chosen to evaluate the surface roughness. Before the etching material was applied, each enamel surface was measured and recorded in three different directions.Enamel surfaces were photographed by (USB) with a built-in camera attached to a personal computer (PC) from IBM and utilizing 120X set zoom .Images were documented at 1280 × 1024 pixels of resolution per image.The contour of surface specimens was transformed into 3D images.

Etching Protocol

Eachof etching materials (PPA, PLA and PPM) was applied to enamel surface in (5, 10, 15 seconds). Both Phosphoric acid and polyalkenoic acid were put by micro brush, then washed with water for 15 seconds and dried with air syringe. While, phosphoric ester monomer was washed using acetone alternating with distilled water.After the etching protocol was placed, the final Ra was measured.

Restorative Technique

G-Premio Bond adhesive was applied by a micro brush according to the manufacturer's instructions to all etched subgroups and control group that had not been treated with any acidic agent before. The universal adhesive was agitated for 10 seconds. After curing of the adhesive in all specimens, a plastic mold with an inner diameter of 2.3 mm, and a height of 3mm was used to build up a resin composite block incrementally by gold plated hand instrument and polymerized for 20 seconds.

Shear Bond Strength Test

All specimens were separately and horizontally fixed on a computer controlled materials testing machine (Model 3345; Instron Industrial Products, Norwood, USA) with a cell of 5 kN in weight and data were documented using computer software (Bluehill Lite; Instron Instruments).

Results:

All the collected data were tabulated and statistically analyzed using a statistical package (SPSS ™ Software, V.21, IBM, NY, USA). Data were checked for normal distribution according to Shapiro-Wilk Test then, evaluated using two way analysis of variance (two-way ANOVA)

Surface Roughness (Ra)

There was no significance difference between the etching times (5, 10,15seconds) in each materials used. At the same time, etching materials (PPA, PLA, and PPM) showed no significance difference between each other when used in the same time of etching.On the other hand, results exposed a significant difference when compared different groups of etching agents through different times.**(Table 1)**

Table 1 The results of one-way ANOVA and post-hoc Tukey tests

Group	**Mean ±SD**
PPA_5s	.00620 ±.001742 [a,b,c]
PPA_10s	.00714 ±.001209[a,b,c]
PPA_15s	.00840 ±.001426 [a]
PLA_5s	.00529 ±.001687 [c]
PLA_10s	.00639 ±.001496[a,b,c]
PLA_15s	.00716 ±.000772[a,b,c]
PPM_5s	.00551 ±.001243[b,c]
PPM_10s	.00674 ±.001101[a,b,c]
PPM_15s	.00756 ±.000759 [a,b]
Total	.00671 ±.001544

Shear Bond Strength (MPa)
Regarding to the comparison between etching materials, results indicated that no significant difference in PPA group when used in different times.During the comparison between etching time in PLA group, results showed a significant difference between 10 and 15 seconds (p=0.000).). However, no significant difference was detected within PPM groups between different times.
All these data were presented as mean values and standard deviation in **(Table 2)**

Table 2 The results of one-way ANOVA and post-hoc Tukey tests

Group	Mean ±SD
PPA_5s	9.7752 ±.89837 [b,c,d]
PPA_10s	10.1007 ± 1.76209[a,b,c]
PPA_15s	11.3569 ±.82544 [a,b]
PLA_5s	9.6330 ± 1.00905 [b,c,d]
PLA_10s	8.1080 ± 1.20263 [d]
PLA_15s	10.7855 ± 1.32344[a,b,c]
PPM_5s	10.5867 ± 1.30765 [a,b,c]
PPM_10s	11.6284 ± 1.53495 [a]
PPM_15s	11.7817 ± 1.24121 [a]
control	9.5463 ± 0.9679 [c,d]
Total	10.3002 ± 1.61277

Discussion

In the recent years, adhesive dentistry has undergone great improvement to achieve conservative dental methodology as in minimal-invasive dentistry. The clinical success of a filling depends on quality and durability of the adhesion at restoration/dental tissues interface.[13]Enamel etching and time of etchingare major factor to create surface roughness and then good adhesion.[3]Results of PPA surface roughness (Ra) evaluation revealed the highest surface roughness than PPM and PLA.This can be attributed to the ability of PPA to converting the intact enamel surface into an irregular one.However, Ra results of PLA and PPM showed no different statistically between them,but revealed lower values in comparison to PPA, this can be due to the less aggressive enamel demineralization of these acids.[14]
Polyalkenoic acid cannot infiltrate the surface very deeply, it just make superficially and moderately demineralizes because of its moderately large molecular size and organic nature of it.Whereas, PPM contains functional monomer which considered as "ultra-mild "and less acidic (pH≈2.7).[21]
On the other hand, the result of effect the time on surface roughness showed increasing the roughness with increased time regardless of the type of acid. PLA in 5 seconds produced the lowest demineralization comparing to other etching protocols, this could be related to the higher pH value (pH=2).[20]
Results of SBS showed the highest strength in PPM followed by PPA and then PLA. This could be related to the different composition, concentration or PH of each one. The bond durability of universal adhesive to PPM etched enamel was high, this can refer to 10-MDP which create acid- base resistance zone (ABRZ).[8]However, the use of PLA with universal adhesives has not been investigated enough until now. Regarding to effect of time within etching agents, the finding showed improved the bond strength by increasing time. However, SBS in 15 seconds exhibited a statistically better bonding strength in all acid agents.[18]These results agreed with Wong J, et al[19]and Shimatani Y, et al[14]who reported that enamel bonding with universal adhesives may be developed with etching protocols of PPA for reduced etching times from 15 to 5 seconds or with PLA etching for 15 seconds.

References

1. Barkmeier WW, Erickson RL, Kimmes NS, Latta MA, Wilwerding TM. Effect of Enamel Etching Time on Roughness and Bond Strength. Operative Dentistry. 2009;34:217-222.

2. Cerci BB, Roman LS, Guariza-Filho O, Camargo ES, Tanaka OM. Dental Enamel Roughness with Different Acid Etching Times: Atomic Force Microscopy Study. European Journal of General Dentistry. 2012;1:187-194.

3. Ersahan S, Sabuncuoglu FA. Effect of Surface Treatment on Enamel Surface Roughness. Journal of Istanbul University Faculty of Dentistry. 2016;50:1.

4. Fukuda R, Yoshida Y, Nakayama Y, Okazaki M, Inoue S, Sano H et al. Bonding Efficacy of Polyalkenoic Acids to Hydroxyapatite, Enamel and Dentin. Biomaterials. 2003;24:1861-1867.

5. Hass V, Abuna G, Pinheiro Feitosa V, Martini EC, Sinhoreti MA, Furtado Carvalho R et al. Self-Etching Enamel Bonding Using Acidic Functional Monomers with Different-Length Carbon Chains and Hydrophilicity. J. Adhes. Dent. 2017;19:497-505.

6. Kakaboura A, Fragouli M, Rahiotis C, Silikas N. Evaluation of Surface Characteristics of Dental Composites Using Profilometry, Scanning Electron, Atomic Force Microscopy and Gloss-Meter. Journal of Materials Science: Materials in Medicine. 2007;18:155-163.

7. Li N, Cui W, Cong P, Tang J, Guan Y, Huang C et al. Biomimetic Inorganic-Organic Hybrid Nanoparticles from Magnesium-Substituted Amorphous Calcium Phosphate Clusters and Polyacrylic Acid Molecules. Bioactive materials. 2021;6:2303-2314.

8. Nikaido T, Takagaki T, Sato T, Burrow MF, Tagami J. The Concept of Super Enamel Formation—Relationship between Chemical Interaction and Enamel Acid-Base Resistant Zone at the Self-Etch Adhesive/Enamel Interface. Dental Materials Journal. 2020.

9. Osorio R, Toledano M, Garcia-Godoy F. Bracket Bonding with 15-or 60-Second Etching and Adhesive Remaining on Enamel after Debonding. The Angle Orthodontist. 1999;69:45-48.

10. Perdigão J, Araujo E, Ramos RQ, Gomes G, Pizzolotto L. Adhesive Dentistry: Current Concepts and Clinical Considerations. Journal of Esthetic and Restorative Dentistry. 2021;33:51-68.

11. Samorodnitzky-Naveh GR, Geiger SB, Levin L. Patients' Satisfaction with Dental Esthetics. The Journal of the American Dental Association. 2007;138:805-808.

12. Sato T, Takagaki T, Baba Y, Vicheva M, Matsui N, Hiraishi N et al. Effects of Different Tooth Conditioners on the Bonding of Universal Self-Etching Adhesive to Dentin. J Adhes Dent. 2019;21:77-85.

13. Scotti N, Cavalli G, Gagliani M, Breschi L. New Adhesives and Bonding Techniques. Why and When? The international journal of esthetic dentistry. 2017.

14. Shimatani Y, Tsujimoto A, Nojiri K, Shiratsuchi K, Takamizawa T, Barkmeier WW et al. Reconsideration of Enamel Etching Protocols for Universal Adhesives: Effect of Etching Method and Etching Time. J Adhes Dent. 2019;21:345-354.

15. Sofan E, Sofan A, Palaia G, Tenore G, Romeo U, Migliau G. Classification Review of Dental Adhesive Systems: From the Iv Generation to the Universal Type. Annali di Stomatologia. 2017;8:1-17.

16. Takeda M, Takamizawa T, Imai A, Suzuki T, Tsujimoto A, Barkmeier WW et al. Immediate Enamel Bond Strength of Universal Adhesives to Unground and Ground Surfaces in Different Etching Modes. European journal of oral sciences. 2019;127:351-360.

17. Tsujimoto A, Barkmeier W, Takamizawa T, Latta M, Miyazaki M. The Effect of Phosphoric Acid Pre-Etching Times on Bonding Performance and Surface Free Energy with Single-Step Self-Etch Adhesives. Operative dentistry. 2016;41:441-449.

18. Tsujimoto A, Barkmeier WW, Takamizawa T, Watanabe H, Johnson WW, Latta MA et al. Influence of Duration of Phosphoric Acid Pre-Etching on Bond Durability of Universal Adhesives and Surface Free-Energy Characteristics of Enamel. European Journal of Oral Sciences. 2016;124:377-386.

19. Wong J, Tsujimoto A, Fischer N, Baruth A, Barkmeier W, Johnson E et al. Enamel Etching for Universal Adhesives: Examination of Enamel Etching Protocols for Optimization of Bonding Effectiveness. Operative dentistry. 2020;45:80-91.

20. Yao C, Ahmed MH, Yoshihara K, Mercelis B, Gré CP, Van Landuyt KL et al. Bonding to Enamel Using Alternative Enamel Conditioner/Etchants. Dental Materials. 2019;35:1415-1429.

21. Yoshihara K, Hayakawa S, Nagaoka N, Okihara T, Yoshida Y, Van Meerbeek B. Etching Efficacy of Self-Etching Functional Monomers. Journal of dental research. 2018;97:1010-1016.