An Investigation on Dentin Surface Characterization Changes and Nano-Leakage of Two Different Universal Adhesives

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By:

Nayera Hassanien Mohamed

B.D.S. MSA University 2012

Prof. Dr. Farid Mohammed Sabry El-Askary

Professor, Operative Department and Vice Dean of Education and Students Affair

Faculty of Dentistry, Ain Shams University

Dr. Mohamed Salah Abdelaziz Nassif

Assistant Professor, Dental Biomaterials Department

Faculty of Dentistry, Ain Shams University

Dr. Nermeen Kamal Hamza

Lecturer, Operative Department

Faculty of Dentistry, MSA University

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DEDICATION

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Introduction

Resin-dentin interface remains the most critical part of adhesive restorations; however significant improvements of adhesive systems have been introduced to the market [1]. Since the quality of resin-dentin interface is primarily affected by the infiltration of resin into the exposed collagen[2]; new multi-mode/ universal adhesives have been introduced for use as either self-etch or etch-and-rinse adhesives[3] .

It is believed that the capability of dentin adhesives to attain a stable and long-term bond depends mainly on the complete infiltration of resin monomer through the exposed collagen network, in such a way that the partially demineralized dentin surface is entirely reached, therefore failure to appropriately penetrate the collagen network into the partially demineralized dentin may create a weak porous layer of exposed collagen, not encapsulated by resin or protected by hydroxyapatite[4]; resulting in nanaoleakage formation which is a pathway formed in hybrid layer-adhesive interfaces, without any formation of gap. The term nanoleakage was expressed by **Sano *et al*** [5]after using silver nitrate as a tracer to observe the leakage pattern of hybrid layers.

All universal adhesives contain water in their composition due to the importance of water in the ionization of the acidic monomers to enable them to interact with enamel and dentin. Moreover, due to the intrinsic wetness of the underlying dentin substrate, hydrophilic monomers were added into the composition of dentin bonding systems since years[6,7] , but the presence of residual water provokes hydrolytic degradation of polymers and collagen which is potentiated by the acidic pH of the monomer[8,9]. Therefore; adequate solvent evaporation is essential during the application of any water-based adhesives. In addition to the presence of 10-MDP which is one of the few monomers used in adhesive dentistry shows chemical bonding to the tooth tissues via ionic bonding to calcium found in hydroxyapatite forming stable MDP-Ca salts which is deposited in self-assembled nanolayers[10,11] .

Atomic Force Microscope (AFM) was used previously to evaluate the dentin surface demineralization and correlate it with the performance of the different adhesives after each treatment. In this

previous study[12]; increasing the bond strength of the two-step self-etching adhesive were positively correlated to the change in the different parameters assessed using the AFM. According to the knowledge retrieved from this previous study using AFM as a tool to evaluate surface characterization; nanoleakage was not the parameter of choice to be evaluated.

Since specific factors of several bonding systems can influence the development of nanoleakage, such as; the type of the solvent (water, acetone or ethanol), the chemical consistent of the adhesive (HEMA, Bis-GMA) and their molecular weight [5,13]. In addition to the modifications to standard clinical protocols can increase bonding stability[14],[1]; thus, it is necessary to identify which factors, especially in the etching mode and thereby affect bonding durability. Accordingly, the correlation between the changes in surface characterization using the AFM assessment and nano-leakage of two universal adhesives applied on ground dentin surface with either etch-and-rinse mode or self-etching mode might be of value and this is what our study will be conducted on.

Review of Literature

I- Universal Adhesives:

Universal adhesive was one of the most recent novelties in adhesive dentistry that have been used since 2011 in clinical practice showing broader applications than 7th generation system of adhesive bonds (self-etching single-bottle or "all-in-one" systems)[15] . They are known as "multi-purpose" or "multi-mode" adhesives due to its ability to be used as etch-and-rinse (ER) adhesives, self-etch (SE) adhesives, or as ER adhesives on enamel (a technique commonly referred to as "selective enamel etching) or as SE adhesives on dentin [16,14], depending on the personal preferences of the operator and on the specific clinical situation[17,18].

Multi-functional monomers which are synergistic, very specific and cross-linking are required to develop truly universal adhesives. These functional monomers should be able to co-polymerize with chemically compatible resin-based restoratives and cements in addition to the need of being hydrophilic/ hydrophobic in character; hydrophilic in order to wet the dentin which have a significant amount of water, while hydrophobic once polymerized in order to avoid water sorption and hydrolysis over time.

Moreover, ideally universal adhesives should be acidic enough to be effective when used in self-etching mode but yet not so acidic in order not to breakdown the initiators added for the polymerization in dual-cure or self-cure resin cements[19]. The presence of water in the composition of any universal adhesives is essential for the dissociation of the acidic functional monomer, inherent in all these systems that makes self-etching possible, but although its presence is very important, too much amount of water can cause several problems such as;1- difficulty in evaporation during air-drying step, 2-degradation of the chemistry of these systems, 3- decrease the shelf-life of the universal adhesive and finally one of the most problems that can be faced is 4- phase separation of monomers which is considered a fatal problem[20,21].

The addition of acetone or ethanol into the universal adhesive formulations improves the wetting of resin and its infiltration into tooth tissues which also helps in water removal and its evaporation during air-drying step.

10-MDP monomer was firstly introduced by Panavia™ adhesive resin cement which showed many positive credits to be used as an effective monomer in universal adhesive composition such as; 1- being a versatile amphiphilic functional monomer with a hydrophilic polar phosphate group

on one end (capable of chemical bonding to tooth tissues, zirconia and metals) while the other end carries the hydrophobic methacrylate group (capable of chemical bonding to methacrylate-based cements and restoratives). In addition to the presence of long carbon chain backbone which increases the hydrophobicity of the monomer which shows a partition coefficient of 4.1 (partition coefficient is essentially a measure of how hydrophobic or hydrophilic a chemical substance is), therefore 10-MDP is considered to be the most hydrophobic among all functional monomers used in dental adhesives[22].

This hydrophobicity of monomer is considered to be of a great importance for the durability of bond in terms of hindering hydrolysis breakdown and water sorption at the adhesive interface over time which is considered as one of the main reasons for bond failure [23, 24] moreover, this hydrophobicity prolongs the shelf-life of the adhesive as it relatively stabilizes the solution. Additionally, 10-MDP shows an important property that is present in few monomers used in adhesive dentistry which is the ability to bond chemically with tooth structure through ionic bonding to calcium ions present in hydroxyapatite crystals[25,26].

These new products of adhesives were called "Universal" due its ability of being used for the placement of both indirect and direct restorations in addition to its compatibility with light-cure, self-cure and dual-cure resin-based cements. Moreover, it is able to bond to different types of substrates such as; zirconia, porcelain, metal and composite[27].

I.1. Etch-and-rinse mode

Acid etching of enamel with phosphoric acid increases resin-enamel bond strength which was firstly demonstrated by Buonocore [28]. He believed that; microscopic surface area

available for resin retention simply increases with acid etching. However one of Buonocore students "John Gwinnett" who was trained as electron microscopist expressed the ability of adhesive resin to penetrate into the acid-etched enamel prisms forming an envelope surrounding the apatite crystallites[29] making them acid-resistant. Resin infiltration into the acid-etched enamel produced a new structure which was neither enamel nor resin but hybridization of the two components, and this was the first hybrid layer that wasn't introduced yet.

The true hybrid layer formed in acid-etched dentin was firstly established by Nakabayashi et al [30] in which he firstly used transmission electron microscope to observe the hybrid layer and later he used scanning electron microscope following argon ion beam etching to demonstrate it[31]. Nakabayashi's group was the first to determine that acid-etched dentin could be infiltrated by resin forming a new complex composed of collagen fibrils reinforced with resin-matrix. Fusayama caused an evolution of etch-and-rinse mode of application of adhesives[32] when he announced the revolutionary concept of total-etching of cavities (i.e., simultaneous etching of dentin and enamel).

This new concept was resisted by the European and American dentists because they thought that adverse pulpal reactions would be induced if we etched dentin using 40% phosphoric acid but later investigations explained that acid-etching dentin with thickness more than 0.5mm showed no adverse pulpal reactions in case of good sealing of dentin against bacteria[33] .

Bonding with adhesives starts by acid-etching the enamel and dentin which allows in increasing the permeability of resin to both [29,34]. Phosphoric acid 37 wt. % is used for etching enamel and dentin resulting in complete surface