Past and New Strategies of Dental Adhesive Systems

Sara Abdulrahman Alfawaz1, Ahmed Mohamed El-Marakby2,3, Razan Saad Aljumaah1, Maram Abdulmohsen Allassaf1, Fadyah Eid Alshalawi1, Mawadah Saleh Alnahdi1, Sarah Mansour Alshaalan1, Maram Khalid Al Sultan1

1Al-Farabi colleges, Riyadh, Saudi Arabia, 2Operative Dentistry Department, Faculty of Dentistry, Al-Azhar University, Assiut branch, Egypt, 3Department of Restorative Dental Sciences, Al-Farabi colleges, Riyadh, Saudi Arabia

Abstract
It is known that clinical success or failure of esthetic restoration like composite restoration rely to great extent on the quality and performance of the bonding system at the interfaces between tooth and restoration. The better adhesive performance depends on many factors but the most important one is proper application. This article discussed variables in the bonding technique, such as over-wetness/over-dryness, over-etching, air-thinning, and solvent evaporation. Actually, the less technique sensitive dental adhesive system may be considered the best for the dentists due to reliable good bonding even under various clinical situations.

Key Words: Dental adhesives, Sclerotic dentin, Dry and moist dentin and solvent evaporation

Introduction
Many recent researches and literatures revealed that success or failure of resin composite restoration in clinical inspections and laboratory observations depends with a great extent on the quality of the adhesive system at the tooth restoration interface. Incremental and layering placement of composite restoration is essentials not only for getting esthetic properties or getting better mechanical performance but also for achieving an effective bonding via adhesive systems. It is clear that the first trials of Buonocore’s to achieve bonding to enamel in 1955 [1] and to dentin in 1956 [2] had a great revolutionary effect in adhesive dentistry. Because of enamel nature and composition, enamel bonding has been predictable, while good bonding to dentin still remain questionable and more challenge. This is due to nature of dentin, its wettability and its heterogeneous composition. All efforts of researchers and manufacturers were directed between the 1960s to early 1990s to create types of adhesive systems that provide reliable bond strength to both enamel and dentin substrates. At the early of 1990s, the fourth generation dentine bonding agent had a good results for bonding to both enamel and dentin with better bond strength than the previous three generations [3]. The technique of application depending on multiple steps, first step was etching enamel with 30% to 40% phosphoric acid followed by sufficient rinsing resulted in surface micro-porosities for reliable mechanical retention [4]. Second step was application of hydrophilic primer without light activation that penetrates the enamel and dentin micro-porosities. The last step is application of hydrophobic adhesive that makes a reliable bond to underlying primed tooth substrates and overlying composite restoration that resulted in a hybrid layer formation as a bonding mechanism [5]. Currently the fourth-generation adhesives known as multistep total-etch or three steps etch-and-rinse adhesives. Some products from this category are still exist in dental market, such as Adper Scotchbond Multi-Purpose Plus (3M ESPE); and OptiBond FL (Kerr) [6]. Although the state of satisfaction presented after many trials of the novel 4th generation adhesive system, the chief complaint of that adhesive system was its technique sensitivity and subsequently time consuming. Simplification was the main cause of production of fifth generation adhesives that minimize time consuming, decrease errors and mishaps during adhesive application and thus improving efficacy. Manufacturers reduce the number of steps from three to two by combining hydrophilic primer and hydrophobic resin into one bottle, eg, Adper Single Bond (3M ESPE), One-Step (Bisco), and OptiBond Solo (Kerr). This group still need etch and rinse as a separate step before application of a one bottle combining hydrophilic primer and hydrophobic resin [7]. As the key word for the millennium was the Simplification, in the late 1990s another group of simplified adhesives has been introduced. In this type of adhesive system there was no need for a separate etching and rinsing step for dentin but the main disadvantage of this type of self-etch adhesive was the need for a separate step of enamel etch in addition to application of self-etch adhesive to dentin prior to composite restoration. The current self-etch adhesives combined both enamel and dentin in one step and simultaneously etch (condition) and prime both of them. According to number of steps, the current self-etch adhesives subdivided into two-bottles and one-bottle systems. In two-bottles system, application of the self-etch primer achieved from one bottle, painting the cavity boundaries and pulpal floor with no need for light curing and then application of hydrophobic resin bonding from the other bottle and light curing for certain time (eg. AdheSE, Prime and Bond SE. (Dentsply) and Excite SE (Viva dent Ivoclar)) [8].

For more simplification, some manufacturers collected all components in one uni-dose (eg. Adper L-pop 3M ESPEE) that contain the two parts separated by diaphragm in one packet, Application is done by dispensing the self-etch primer to be mixed with the hydrophobic bond and both are then dispensed to get out as one solution into the cavity boundaries and pulpal floor. The other one bottle system may belong the seventh generation that has the following characters: one bottle, one step, self-etch adhesive system. Some manufacturer give it the name” All in one“ adhesive system. Examples are G-Bond and i-Bond [9]. The latest trend in dental adhesives is universal bonding. Some examples are Scotchbond Universal (3M ESPE), Prime and Bond Elect (Dentsply Sirona), ClearfilTM Universal Bond (Kuraray), and All-Bond Universal (Bisco). The main advantage of that...
category is that Universal adhesives can be used in all modes either etch rinse, selective enamel etching mode and self-etch mode [10]. Another advantage may be that is not only confined bonding to tooth structure substrates (Enamel and Dentin) but also can make chemical bonding to other substrates such as zirconia or ceramics [9,11,12]. It may be important for clinicians to select ideal adhesive products [13]. This article will discuss current dental adhesives based on technique sensitivity, common mishaps in bonding procedures, and how to avoid those.

**Controversial in Bonding Procedures**

**Bonding to carious and sclerotic dentin**

Bonding to sclerotic and caries-affected dentin still a challenge. Thick hyper-mineralized surface layers and obliterated dentinal tubules that may contain crystalline deposits are features found in Sclerotic dentin. On the other hand, occluded dentinal tubules with precipitation of minerals, areas with high mineral loss and disrupted collagen matrix network are features most commonly found in caries-affected dentin. In general, for both caries-affected and sclerotic dentin, researches have shown that etch-and-rinse adhesives were more effective than self-etch adhesives [14-16]. If it is possible to avoid over-etching of sound dentin, it will be great to extending etching time or using more concentrated phosphoric-acid in etch-and-rinse technique to increase bond strengths of sclerotic and caries-affected dentin [17].

**Contamination**

Technique sensitivity is the most accepted expression about the procedures done before resin composite restoration placement. Any errors during acid etching step or bonding application will lead to degree of failure to subsequent steps. Moisture contamination is one of the most common cause of decreasing bond strength or even complete de-bonding (adhesive or cohesive failure). There was difference in the reported research results. These may be due to differences in contamination period, the design of theses and type of the materials undergo the experiments. When saliva contamination occurred, rinsing off the saliva and drying before repeating the adhesive procedures recovered the bond strength of self-etch and universal adhesives eg. AdheSE and Scotchbond Universal [18-21]. For the total-etch adhesive, one study reported that blotting the surface and applying the primer (without re-etching) recovered the bond strength after saliva contamination [22].

**Over-etching dentin/under-etching enamel**

Finding a balance between under-etching enamel and over-etching dentin is important. The Proper acid etching technique is achieved by using ortho-phosphoric acid with 37% concentration and with 20 seconds application time on enamel and 15 seconds on dentin. Better marginal integrity and less gap formation in case of enamel etching may be achieved with etch-and-rinse technique than do with self-etch primer/adhesives [23]. Dentin is more sensitive to undergo undesirable morphological changes due to over-etching that can resulted from increasing the concentration of acid or long duration application. Partial demineralization of dentin for achieving resin infiltration into the collagen network forming a hybrid layer is very important for better hybridization. Over-etching will lead to formation of a zone of weakened dentin under the hybrid layer that will increase the ability to time dependent bio-degradation [5]. Although self-etch adhesives may lead to under-etch of the enamel, however this technique may solve the problem of over-etching dentin by simultaneously demineralizing and infiltrating the exposed collagen network [8]. Some few approaches may overcome the over-etch/under-etch problem:

For the etch-and-rinse or total-etch systems eg. ScotchBond Multipurpose, SingleBond Plus, Optibond FL, Optibond Solo Plus, most of manufacturers’ instructions focusing on making difference in period of etching between enamel and dentin. While hard enamel composition needs extended application time to 20 seconds, dentin bio-structures may need only 15 seconds acid-etch. Some others advise clinicians to make etching for enamel margins only 15 seconds and dentin for only 10 seconds. After that dentin is rinsed thoroughly for 15 seconds, and blot dry.

Excessive hydrophilic nature that may be incorporated in most types of seventh generation one-step self-etch adhesives (All-in-one) with exception of some newer products; lead to clinically poor performance even though they are more acidic for effective enamel etching [12,24].

Regarding two step self-etch adhesive which has mild acidity, it is preferable not to finish the enamel margin and cavity walls with an extra-fine diamond bur that may result in thinning of the smear layers, which is preferable to be thick for good performance of the self-etch adhesives [9]. A five-year clinical study revealed that however, slight discoloration and small marginal defects were observed in enamel margins, the selective enamel etching with phosphoric acid prior to use two step self-etch adhesive was improved the marginal integrity. The main disadvantage of this technique was incorporation the etch-and-rinse step in the self-etch procedure that make the technique to be time consuming and less simplified. Although some author believed that mild two-step self-etch adhesives may be the gold standard for dentin adhesion, some studies revealed that dentin bond strength was decreased when dentin was accidentally etched during selective enamel etching [25-27].

Regarding the Universal adhesives, even though they are less technique sensitive and could be performed well in either self-etch, selective etch, or etch-and-rinse modes.it will be better to wait for confirmation the results of long-term clinical studies to become available before complete trust to new techniques and materials. Results of 18-month clinical studies using most commercially available universal adhesives revealed variation with dentin when pre-etching with phosphoric acid or not [10,11]. High content of water and mainly hydrophilic nature of Universal adhesives play an important role in increasing resin-dentin interface permeability and the bond stability would be long-term affected. Selective etch mode may be more predictable and has a better results at this point of view. The etched dentin surface may be strongly attached with apatite crystals to provide stable bonds to acidic monomers [28,29].
Effect of aggressive dry or excessive wet

After acid etching of enamel and dentin, some authors were preferred to make over-dry of enamel surface for achieving enamel frosted appearance that was considered as an indication for good etching of enamel surface. Numerous studies revealed that adverse effect of enamel over-drying was still limited when compared to dentin [9]. Over-dry of dentin surface may lead to collagen collapse and evaporation of fluids inside dentinal tubules that aggressively decrease bond strength especially for adhesives where solvents were acetone based. On the other hand over-wet may lead to over-wet phenomenon that give manifestation of water globule and blister formation that resulted in dilution of liquid of adhesives. It is important to make a balance between proper dryness and wetness of tooth substrate after acid etching and prior to adhesive application. In case of using etch and rinse technique, dentist should perform acid etching with optimum time for both enamel and dentin then rinsing with water and wait and see to select the next step according to the type of solvent inside the adhesive he will use; If this type contain water/ethanol as a solvent, dentin surface should left gentle dry because the water presented in the solvent will re-wet the dry dentin surface. If tooth was re-wetted before application of that type of adhesive, water globule and blister like voids will formed as a result of Over-wet phenomenon. Scotchbond Multipurpose and Optibond FL are examples of adhesives contained hydrophilic primer that are less sensitive to over-drying/over-wetting [30]. On the other hand, if the type of adhesive that will be used contained acetone or ether as a solvent, tooth should be left wetted or moisten enough. If the tooth substrate was aggressively dried, acetone based adhesive will lose up to 66% of its efficacy and only performed 33%. This may be due to the mechanism of action of acetone that replaced and occupied the water sites within the dentin and then evaporate leaving adequate spaces for the remaining adhesive component to penetrate. So if there is no water resulting from over-dry, there will be no penetration to the full depth of dentinal tubules and no intermingling with collapsed collagen network. This technique of acetone based adhesives called “wet bonding technique”. Because they no longer require the etching, rinsing, and drying steps, Self-etch adhesives are not sensitive to over-drying/over-wetting. Some recent researches revealed that Universal adhesive system also has the advantage of less sensitivity to both over-drying/over-wetting [30,31].

Thickness of adhesive layer

After acid etching procedures, enamel and dentin pores (interprismatic enamel, dentinal tubules, inter-tubular and peritubular dentin) become ready to receive the upcoming adhesive liquid. Complete penetration of adhesive liquid into a full length of pours in enamel and tubules in dentin make a strong resin tags. Better intermingling between hydrophilic part of adhesive and collagen network in dentin ensure well formation of thick hybrid layer. The manufacturers advise dentists to follow different strategies to ensure achievement of strong resin tags and hybrid layer formation. One of them is forcing the liquid of adhesive into open pores using air of three way syringe for few seconds. Another strategy is painting the cavity walls with multiple coats of adhesive to avoid hazards of adhesive tearing due to viscosity. Some others recommended vigorous rubbing of adhesive with application brush. A thick layer adhesive may be better than thin ones because it saturates the exposed collagen network and create a thick resin layer can resist water permeability and fluid dissolution rather than thin one. Adhesives with high acetone content (such as All-Bond) are more vulnerable to having a thin bonding layer, and, hence, are more technique sensitive than water based and water/ethanol-based adhesives [9]. The problem become more complex with highly hydrophilic adhesive systems. This category contains seventh generation all-in-one adhesives and Universal bond that have the ability for being too thin film [8]. Technique of multiple coats may be useful also in case of self-etch adhesives as it increases etching ability with an additional supply of fresh acidic monomers [32]. Some researches revealed that clinical performance can be improved by Adding a hydrophobic resin layer on the top layer but this has the disadvantage of more time consuming and technique sensitivity [10,26].

Solvent evaporation time

Many literatures concluded that the longer the time of solvent evaporate, the higher the bond strength obtained. The bonding of adhesives is improved with extended solvent-evaporation time. Dental adhesives should contain solvents in their components. Solvent may be water, ethanol, acetone or water/ethanol. Solvent complete evaporation plays an important role in achievement of optimum polymerization. As mentioned before, manufacturers recommended different strategies to insure better resin infiltration into the collagen network. In addition to previously mentioned method, manufacturers may advise agitating the adhesive on the dentin/enamel surfaces [32]. Better resin infiltration can be achieved through agitation by vigorous rubbing and increasing evaporation time. Luque et al. reported that when the evaporation times were extended from 5 seconds or 10 seconds to 25 seconds, nano-leakage formation were reduced and higher bond strength were achieved when examine universal adhesives in etch-and-rinse mode [33]. When the bonding layer is thick, sufficient time for solvent evaporation becomes more essential. To achieve uniform adhesive layer, some manufacturers advise gentle air blowing and avoid aggressive air usage that may lead to thinning and weakening of adhesive layer. On the other hand, Some author recommended the use of aggressive compressed air and discussed its effectiveness in improving the bond strength by forcing the bonding liquid into full depth of tooth pores and better evaporation of water and organic solvent [34,35].

Adhesives and light curing

Many light curing devices are available in dental market. In general, tungsten halogen light curing devices had better light intensity than traditional types of LED (light emitting diode) but both are less than Laser types and Plasma arc devices. The latter two types are rarely used by most of dentists due to limited indications and high cost. New generations of LED devices are more commercial and most accepted for use with different types of adhesives and resin composite restorations. Some authors considered LED devices is more reliable in case
of application of adhesives in deep cavities due to its cold cure effect that is not found in case of halogen light that can make thermal insult for pulp. Manufacturers recommended that proper time of cure is very important to achieve strong bond strength. This is a role regardless the type of adhesive system used [36-38].

**Shelf life and effect of solvent evaporation**

Many efforts were exerted to put monomers of different chemical structures and different behaviors (hydrophilic or hydrophobic nature) together in one bottle. These different components can gradually change by time. Adhesive shelf life refers to the period in which adhesive remain performed effective optimum bond. Hydrolysis of the components of Seventh generation adhesives (All-in-one or One-step self-etch adhesives) may be a reason for limited shelf life [39]. Evaporation of solvent is the main cause for short shelf life of adhesives after being used. Acetone based adhesives lose more than 50% of its bond strength after continuous opening and repeated use of the adhesive due to evaporation of the highly volatile acetone solvent that resulted in increasing the viscosity if the adhesive liquid and ill infiltration and poor penetration of adhesive to the full length of tooth pores [40]. Regarding Universal adhesives, Pongprueksa et al. reported in their study a complete impairment of bond strength when evaporation of ethanol solvent is more than 50% in case of ethanol based universal adhesive. This may be reason for recommendation for wide range use of (Uni-dose) package adhesive [41].

**New trends regarding adhesives**

It was clear that all efforts of manufacturers and researchers have directed to examine the morphology of enamel and dentin substrates and the physical nature and chemical properties of adhesive systems. The new trends focused on new strategies like the role of biologic factors in long term efficacy of adhesive/tooth interface. Some researchers concluded that nano-leakage and biodegradation within the hybrid layer due to hydrolysis of exposed collagen network may be caused by the effect of endogenous enzymes eg. cysteine cathepsins and matrix metalloproteinases. So there is a trend to incorporate some special enzyme inhibitors into dental adhesive systems. Another trial was focused to the role of chlorhexidine if applied before adhesive and its good effect in delaying and decreasing the tendency of degradation of dentin collagen network that lead to poor loosely hybrid layer and later on partial or complete de-bonding [42]. Other innovative approaches of contemporary researches are thinking to improve the bond stability through the biomimetic re-mineralization of dentin and to incorporate some bio active materials into newly dental adhesive system [43-45].

**Conclusion**

Dental adhesives that are performing well under non ideal clinical conditions contribute to reliable bonding. Understanding the weakest points of each adhesive and employing meticulous techniques of bonding are essential for resin composite restorations success.

**References**


43. Tjäderhane L. Dentin bonding: can we make it last? *Operative Dentistry.* 2015; **40**: 4-18.
