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Is the Ethanol Wet-bonding Technique a Promising One?

¿Es Prometedora la Técnica de Adhesión Húmeda en Etanol?

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SOUZA JÚNIOR, M. H. S.; FERREIRA, R. O.; ARAÚJO, J. F.; BARROS, T. A. F.; BRAGA, E. M. F. & LORETTO, S. C. Is the ethanol wet-bonding technique a promising one? ***Int. J. Odontostomat., 9(3)***:463-468, 2015.

ABSTRACT: The ethanol wet-bonding technique (EWBT) was introduced in an attempt to overcome the problems caused by high hydrophilicity and/or incomplete penetration of most commercially available adhesive systems. This strategy provides better conditions for the inter-diffusion of hydrophobic dentin monomers. Today, there are many EWBT protocols, which yield bonding interfaces with minimal degradation and longer durability compared with commercial hydrophilic adhesive systems. The aim of this review is to discuss in greater detail the EWBT, focused on the following aspects: dentin saturation, hydrophobic primer preparation, inactivation of metalloproteinases (MMPs), dentin biomimetic remineralization and the clinical perspectives of this technique. The present review on the EWBT provides support for a better understanding of the behavior of dentin when exposed to dehydration and hydrophobic monomer interaction. Moreover, additional studies are suggested to investigate the long-term stability of this type of hybrid layer.

KEY WORDS: dentin, dentin-bonding agents, ethanol.

INTRODUCTION

The wet-bonding concept, introduced by Kanca (1992), increased the strength of resin-dentin bonds, allowing good sealing of dentin and significantly decreasing post-operative pain (Pashley *et al*., 2011). In this bonding approach, organic solvents such as acetone and ethanol added to the hydrophilic monomers displace the water molecules from the demineralized collagen matrix, which is then in part replaced by these monomers (Kanca; Guimarães *et al*., 2012).

The presence of water within the collagen network plays some important roles such as preserving the interfibrillar spaces among the exposed collagen fibrils and allowing the diffusion of adhesive monomers, which allows the sealing of restoration interface (Malacarne *et al*., 2006). However, this process requires the presence of a hydrophilic monomer blend that produces polymers that can absorb 5–12% water (Malacarne *et al*.), resulting in plasticization that lowers the adhesive interface mechanical properties, and a 30–40% decrease in bond strength has been observed after 3–6 months of in vitro aging (Hosaka *et al*., 2009).

Hydrophobic monomers, when added to the adhesives, can produce polymers that are more resistant over time owing to their increased stiffness and resistance to hydrolysis (Guimarães *et al*.; Breschi *et al*., 2008). Thus, the durability of the bonded interface is increased compared with that when hydrophilic-rich adhesives are applied (Breschi *et al*.). However, the diffusion of hydrophobic monomers within demineralized dentin is not simple. Air- or blow-drying dentin after rinsing does not remove enough water to allow for favorable interactions between the hydrophobic monomers and the tissue (Figs. 1 and 2). To replace the residual water in the demineralized dentin matrix prior to using adhesives, specific sequences and concentrations of ethanol solutions must be used (Grégoire *et al*., 2013).

The ethanol-wet bonding technique (EWBT) was introduced in an attempt to overcome the problems caused by incomplete penetration of most adhesive systems and the high hydrophilicity observed in commercially available adhesive systems. Most of the EWBTs have proposed dentin saturation by increasing

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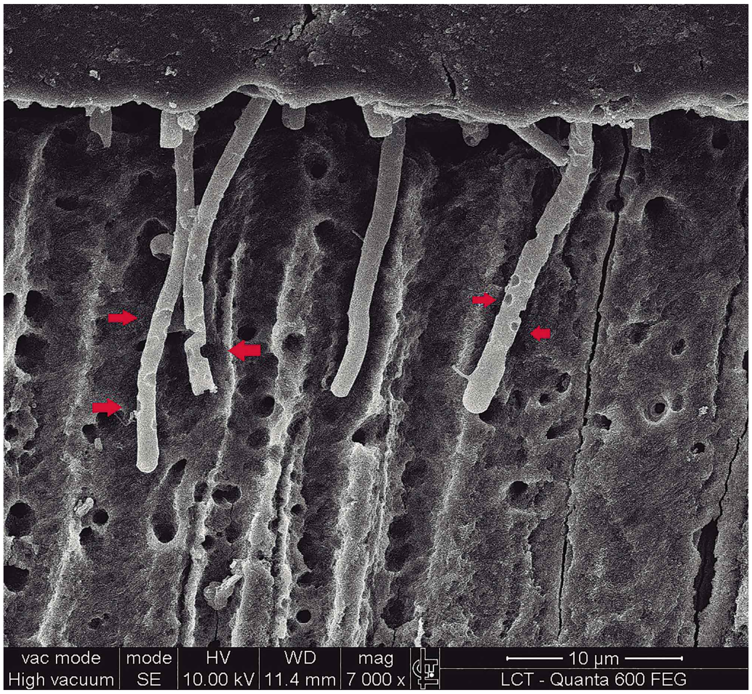

Fig. 1. Poor hydrophilic monomer shows some areas in which the interaction with moist tissue was not achieved (Red arrows).

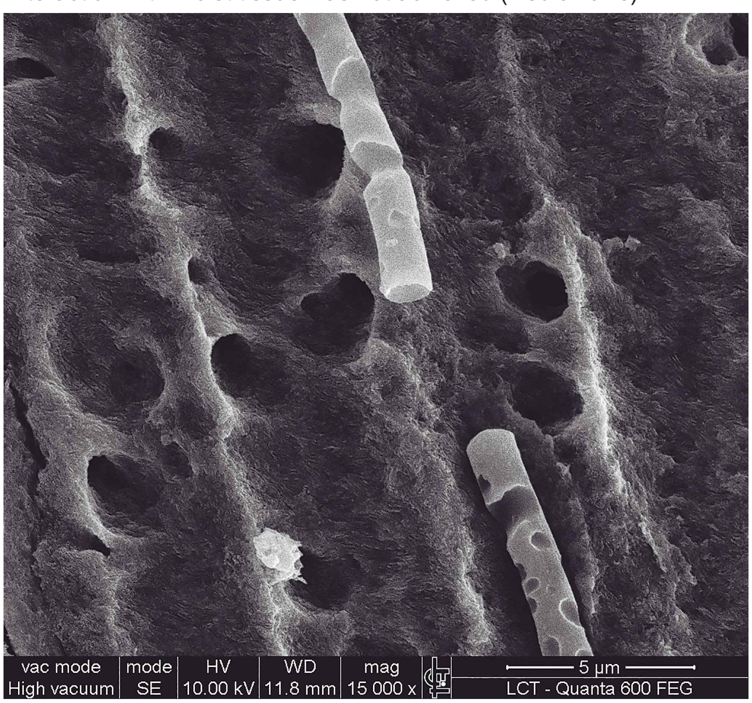

Fig. 2. Closer view of the non-interaction areas between adhesive/moist tissue.

the concentration of ethanol solutions prior to the application of relatively hydrophobic monomers to produce a more stable and hydrolysis-resistant hybrid layer (Pashley *et al*., 2007; Sadek *et al*., 2008, 2010a, 2010b). Simpler techniques have also been proposed to reduce the time required to complete the entire procedure, normally by employing one application of 100% ethanol (Hosaka *et al*.; Nishitani *et al*., 2006; Cadenaro *et al*., 2009; Sadek *et al*., 2010c).

Another aspect mentioned in the literature is that when ethanol solutions are used to dehydrate etched dentin, collagen fibrils shrink. This shrinkage increases the interfibrillar spaces, which are associated with reduced hydrophilicity of the collagen matrix (Pashley *et al*., 2011). These conditions may allow the inter-diffusion of higher-molecular-weight molecules present in the hydrophobic monomer formulations; thus, an improved matrix collagen seal may be expected.

Some laboratory studies have demonstrated higher adhesive interface bond strengths when ethanol solutions are used to dehydrate dentin prior to primer and adhesive application compared with the strengths achieved using conventional adhesive techniques (Hosaka *et al*.; Sadek *et al*., 2010a, 2010b, 2010c; Nishitani *et al*.; Cadenaro *et al*.). Theoretically, hydrophobic resins lead to lower water absorption and smaller plasticization effects, resulting in more durable bonding (Pashley *et al*., 2011; Malacarne *et al*.; Breschi *et al*.; Shin *et al*., 2009).

Another issue discussed in the literature regarding the EWBT is the primer preparation. Some studies have suggested experimental solutions based primarily on Hoy's solubility parameters to determine the miscibility of the monomers and solvents, as well as the interdiffusion between the monomers and solvent blends and the ethanol-saturated dentin (Hosaka *et al*.; Sadek *et al*., 2008, 2010b; Cadenaro *et al*.). Conversely, there are studies that use commercial bonding systems in which the adhesives were diluted in various concentrations of ethanol (Sauro *et al*., 2011).

In this article, different dentin saturation protocols and preparations, the use of hydrophobic primers, and the clinical possibilities of the EWBT are discussed in greater detail.

RESULTS AND DISCUSSION

Thirty articles were included. This discussion focused on the aspects related to the EWBT, including dentin saturation, hydrophobic primer preparation, inactivation of MMPs, dentin biomimetic remineralization, and the clinical perspectives of the technique.

1. Dentin saturation. The first laboratory techniques that proposed to focus on dentin saturation using the ethanol wet-bonding approach involved the application of increasing concentrations of ethanol (50–100%) for a duration ranging from 20 s to 3.5 min (Hosaka *et al*.; Sadek *et al*., 2008; Tay *et al*., 2007). The full chemical dehydration protocol suggests the application of solutions with increasing concentrations of ethanol as follows: a single 30-s application of 50%, 70%, 80%, or 95%, and three applications of a 100% ethanol solution for 30 s each. However, this laboratory procedure takes a long time, which makes the technique unattractive and clinically inappropriate. Sadek *et al*. (2008) showed poor results with one application of 100% ethanol for 30 s. However, Tay *et al*. did not observe a difference in the bond strength between the EWBT with one application of a 100% ethanol solution for 20 s and the conventional etch-and-rinse strategy (Sauro *et al*.).

Indeed, Nishitani *et al*., used one application of 100% ethanol for 20 s to saturate dentin before using different experimental adhesives diluted in 50% ethanol and better results in the μ-tensile bond strength tests were observed compared with the results for the same adhesive formulations applied on water-saturated dentin or air-dried dentin. The same protocol with the dentin saturation time increased to 60 s was used by Hosaka *et al*. In their study, an increase in the bond strength and durability was observed for the EWBT, and, according to the authors, a higher resin uptake and better resin sealing of the collagen matrix was possible, which may minimize endogenous collagenolytic activity.

2. Hydrophobic primer . To achieve hydrophobic monomer inter-diffusion in the naturally hydrophilic dentin, it is necessary to have previous dentin saturation and hydrophobic monomer dilution with solutions of ethanol. The preparation of ethanol-based hydrophobic primers may vary. Some studies used experimental blends and considered Hoy's solubility parameters to determine the degree of hydrophilicity of the solvated comonomer blends (Nishitani *et al*.), *i.e.*, to obtain a good affinity between the dentin and monomer blends. The previously mentioned dentin treatment using ethanol solutions and the dilution of monomers in ethanol, results in a favorable interaction between the substrate and primer.

As we noted, there are several different proposals on dentin saturation. Similarly, different methods are reported for monomer solvation. The majority of laboratory studies diluted the monomers in 50% ethanol (Sadek *et al*., 2008; Sadek *et al*., 2010a, 2010c; Cadenaro *et al*.); however, less diluted solutions have also been proposed (Nishitani *et al*.). The less concentrated monomer blends show some advantages such as the ability to replace the residual water content within the collagen fiber spaces (Sadek *et al*., 2008). Conversely, the evaporation of excess ethanol from resin tags composed of more than 30% ethanol may compromise the interface sealing because of the high residual ethanol content and shrinkage (Pashley *et al*., 2007; Cadenaro *et al*.). Dickens & Cho (2005) stated that it is difficult to evaporate the excess solvent if the ethanol/comonomer blend contains over 30% solvent. Cadenaro *et al*. used different monomer blends (hydrophobic x hydrophilic) diluted in 10% ethanol, and found that the degree of conversion was not affected. Indeed, when the most hydrophobic resin was bonded to ethanol-saturated dentin cured for 60 s, a reduced permeability of 81.2% was observed. More hydrophilic resins were also used and their permeability when bonded to ethanol-saturated dentin was as high as 92.8%. However, owing to the presence of hydrophilic monomers, higher sorption of water may take place; this plasticizing effect would reduce the stiffness of the resin, resulting in a decrease in bond strength.

The EWBT has been clinically observed in non-carious cervical lesions (NCCL) at 12 months of service compared with a three-step etch-and-rinse system using the commercially available dental adhesive SBMP and the mild self-etching adhesive Adper Easy One (Araujo *et al*., 2013). For the EWBT, the hydrophobic primer was prepared using SBMP adhesive diluted in 10% ethanol. The original formulation of the SBMP adhesive is HEMA, Bis-GMA, TEG-DMA, polyalkenoic acid copolymer, and ethanol. This blend is mainly hydrophobic; however, a hydrophilic monomer (HEMA) is present.

One aspect of this procedure must be addressed here: the use of a commercially available product due to ethical considerations. Ideally experimental groups would be treated with neat hydrophobic monomer blends diluted in different concentrations of ethanol. However, the use of such experimental solutions in patients could not be justified to the ethics committee (Araujo *et al*.). Figures 3 and 4 show images taken after the in vitro evaluation of the simplified ethanol technique in an on-going study of our group; a good seal was obtained for dentin coated with the three-step dental adhesive system (SBMP) modified for the EWBT after 18 months of storage in water. It is possible to observe a good interaction after aging between the monomer blend and the tubule demineralized walls.

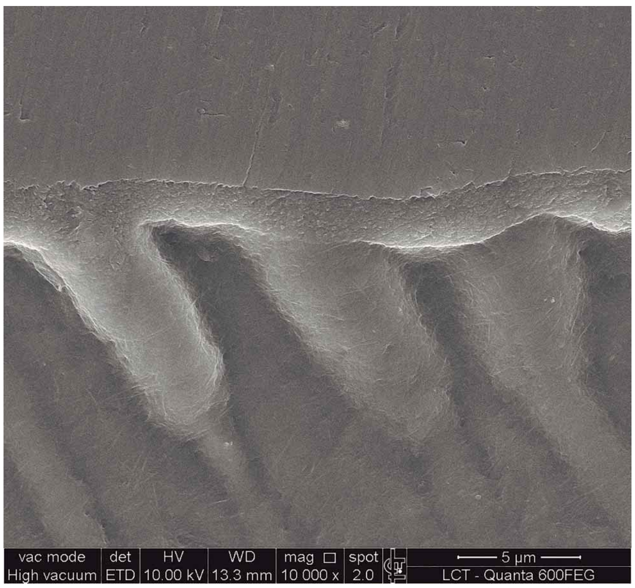

Fig. 3. Non carious lesions restored with the EWBT after 18 months storage. The interface between adhesive system an dentin walls was well preserved.

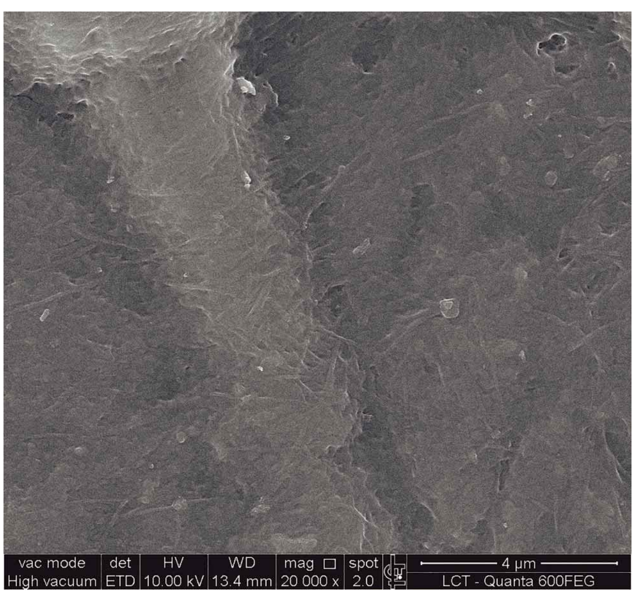

Fig. 4. A closer view of the areas, previously mentioned, showing a good sealing after 18 months.

3. Inactivation of MMPS and biomimetic remineralization of the hybrid layer. The longevity of adhesive interfaces is partially related to the presence of MMPs and their ability to cleave collagen peptide chains (Perdigão *et al*., 2013). Acid etching activates MMPs and non-infiltrated collagen networks may undergo degradation processes (Pashley *et al*., 2004). As MMPs become inactive in the absence of water, one may speculate that the replacement of water by an organic solvent such as ethanol might improve the longevity of the bonding interfaces (Perdigão *et al*.). However, ethanol was not considered as effective as the chlorhexidine (CXD) in inhibiting MMPs (Tezvergil-Mutluay *et al*., 2010; Liu *et al*., 2011).

The combination of the EWBT with CXD was evaluated to determine the bond strength after 24 h, 9 months, and 18 months (Sadek *et al*., 2010c). The reduction of the bond strength in the EWBT with and without CXD was significantly less than that observed for water wet-bonding. It has recently been reported that the EWBT is associated with the possibility of precluding the process of hybrid layer remineralization using biomimetic analogs (Perdigão *et al*.; Tay & Pashley, 2009; Niu *et al*., 2014; Kim *et al*., 2010). In the present research, changing the moisture condition by replacing water with ethanol showed that the absence of water on the specimens treated with meticulously performed EWBT does not result in the remineralization of the hybrid layer, because water plays a pivotal role in the maintenance of collagen molecules, and consequently, in hybrid layer remineralization (Kim *et al*.).

However, Kim *et al*. also showed that the possibility of remineralization can be restricted to water-contaminated ethanol wet-bonded specimens in badly executed EWBT protocols or specimens that were bonded to deep dentin without tubular occlusions (Sadek *et al*., 2007). Nevertheless, until now the possibility of biomimetic remineralization has only been observed in laboratory trials, and none in clinical situations (Perdigão *et al*.; Tay & Pashley; Niu *et al*.; Kim *et al*.).

4. clinical perspectives. Although the EWBT seems to bring some long-term benefits, the time needed to replace water with 100% ethanol within an acid-etched dentin is relatively long and not clinically attractive (Sadek *et al*., 2010c). Therefore, current adhesive research is directed toward the development of an adhesive that has a good bonding efficiency with enamel and dentin and low technical sensitivity.

According to this objective, the self-etching adhesives have become the choice of many researchers and practitioners to minimize, the shortcomings between the bonding agents and the dental substrates.

Since last few years, 10-methacryloyloxydecyl dihydrogen phosphate (10-MDP) is being described as a good approach to dental bonding owing to the good outcomes obtained (Pashley *et al*., 2011; Inoue *et al*., 2005; Yoshirara *et al*., 2013; Nikaido *et al*., 2011). Thus, improved results involving bond strength (Inoue *et al*.); marginal sealing by chemical interactions between collagen fibrils, hydroxyapatite, and monomers (Inoue *et al*.; Yoshirara *et al*.); low nanoleakage; and remineralization of the hybrid layer (Nikaido *et al*.) have made 10-MDP more commercially attractive to companies developing new products based on this monomer already known as a “gold standard”.

In conclusion the present review of the EWBT provides:

1) An improved understanding of the behavior of dentin after undergoing dehydration, *e.g.*, inactivation of MMPs and preservation of bond integrity;

2) Support for further studies to investigate the long-term stability of the hybrid layer composed of hydrophobic monomers diluted in ethanol for considering the integrity of resin tags, interfacial nanoleakage, remineralization of the hybrid layer “untouched” by the resin monomers, and remineralization of the etched dentin that surrounds the bonding interface.

This review is an important guide to researchers and clinicians searching for long-lasting adhesive interfaces.

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SOUZA JÚNIOR, M. H. S.; FERREIRA, R. O.; ARAÚJO, J. F.; BARROS, T. A. F.; BRAGA, E. M. F. & LORETTO, S. C. ¿Es prometedora la técnica de adhesión húmeda en etanol? ***Int. J. Odontostomat., 9(3)***:463-468, 2015.

RESUMEN: La técnica de la adhesión húmeda en etanol (TAHE) se introdujo en un intento de superar los problemas causados por la alta hidrofilicidad y/o la penetración incompleta de la mayoría de los sistemas adhesivos disponibles comercialmente. Esta estrategia ofrece mejores condiciones para la interdifusión de monómeros dentinarios hidrofóbicos. Hoy en día, hay muchos protocolos TAHE que producen las interfaces de unión con mínima degradación y mayor durabilidad en comparación con los sistemas adhesivos hidrofilicos comerciales. El objetivo de esta revisión es discutir con más detalle la TAHE, explicando los siguientes aspectos relacionados: la saturación de la dentina, la preparación del primer hidrofóbico, la inactivación de las metaloproteinasas (MMP’s), remineralización biomimética de la dentina, y las perspectivas clínicas de esta técnica. La presente revisión sobre la TAHE proporciona soporte para una mejor comprensión del comportamiento de la dentina cuando es expuesto a la deshidratación y la interacción con monómero hidrófobo. Además, se sugieren estudios adicionales para investigar la estabilidad a largo plazo de este tipo de camada híbrida.

PALABRAS CLAVE: dentina, recubrimientos dentinarios, etanol.

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