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Comparative evaluation of contact angles of three different universal adhesives-an invitro study

Running title : Contact Angles of Different Universal adhesives

1. Dr.Anusha Nadipineni 2. Dr.Vijetha Badami 3. Dr Pavan Kumar Kanumuru 4. Dr Sujathagopal Sooraparaju 5. Dr Swetha Kommavarapu 6. Dr Mounika Pulijala 7. Dr Sahithi Nammaniwar

[1] Department of Conservative Dentistry and Endodontics, MNR Dental college and Hospital, Sangareddy, India, ph: +91 90000 28704, Fax: +08455277688,
E-mail: anushanadipineni123@gmail.com

[2] Department of Conservative Dentistry and Endodontics, MNR Dental college and Hospital, Sangareddy, India, ph: +91 91600 22422, Fax: +08455277688,
E-mail: vijetha.mds@gmail.com

[3] Department of Conservative Dentistry and Endodontics, MNR Dental college and Hospital, Sangareddy, India, ph: +91 96666 68873, Fax: +08455277688,
E-mail: drpavanmds@yahoo.co.in

[4] Department of Conservative Dentistry and Endodontics, MNR Dental college and Hospital, Sangareddy, India, ph: +91 98664 11877, Fax: +08455277688,
E-mail: sujasmiles@yahoo.com

[5] Department of Conservative Dentistry and Endodontics, MNR Dental college and Hospital, Sangareddy, India, ph: +91 94933 52575, Fax: +08455277688,
E-mail: swetha31k@gmail.com

[6] Department of Conservative Dentistry and Endodontics, MNR Dental college and Hospital, Sangareddy, India, ph:+91 95531 81651, Fax: +08455277688,
E-mail: mounikapulijala7@gmail.com

[7] Department of Conservative Dentistry and Endodontics, MNR Dental college and Hospital, Sangareddy, India, ph: +91 94040 64400, Fax: +08455277688,
E-mail: sahithirao690@gmail.com

Correspondence: Dr. Vijetha Badami, Professor, Department of Conservative Dentistry and Endodontics, MNR Dental college and Hospital, MNR Nagar, Narsapur Road,Fasalwadi, Sangareddy,
Telangana 502294, India
E-mail: vijetha.mds@gmail.com, Tel: +919160022422, Fax: +08455277688

ABSTRACT

Context: The development of Universal adhesives has simplified the adhesive protocols in dentistry. Contact angle of adhesives are indicative of bonding ability and can predict longevity of the restoration.

Aims: ***To compare the contact angles of three different universal adhesives and influence of prior acid etching on their contact angles.***

Materials and Methods: 36 caries-free intact extracted human mandibular molars were used. Two sections parallel to the occlusal surface were made beneath the dentin–enamel junction to obtain 2mm thick superficial dentin slices.Three adhesive systems were used- Single Bond Universal (3M ESPE, St Paul, MN, USA), Solare Universal Bond (GC Corporation, Tokyo, Japan) and Tetric N Bond Universal adhesive (Ivoclar Vivadent). Measurement of static contact angle θ* with 1.0 µL droplets of liquid adhesives, was performed by the sessile drop method using Rame Harte Goniometer.

Statistical analysis used: One-way ANOVA analysis followed by post hoc Tukey test to compare contact angles among the groups with respect to both etched and unetched dentin. Independent t- test was used to compare the contact angles of etched and unetched dentin of the respective adhesive groups.

Results: Single Bond Universal exhibited highest contact angle followed by Tetric N Bond Universal and least contact angle was shown by Solare Bond Universal. Prior acid etching had no significant influence on the contact angle of the adhesives.

Conclusions: Solare Bond Universal showed the best wettability followed by Tetric N Bond Universal and Single Bond Universal. Prior acid etching did not improve the wettability.
Keywords: Contact angle. Etching. Self-etch adhesives. Universal bonding agent.

INTRODUCTION

Adhesive restorations aim at producing a stable long term bond to the tooth structure which permits the placement of a more conservative restoration that is critical for the success of esthetic restorative materials in modern dentistry. With rapid progression over the years and changing technologies, dental adhesive systems have evolved from no-etch to total-etch to self-etch adhesives. In recent years, a new family of dentin adhesives has been introduced, which may be used either as etch-and-rinse or self-etch adhesives, known as "multi-mode" adhesives [1].

Contact angle is defined as an angle, measured quantitatively, where the liquid–vapor interface meets a solid surface [2]. If the adhesive has high wettability, there will be an intimate contact with the tooth substrate and adhesive efficiency will be improved [3].

Pre-treatment of dentin surface by acid etching is used as a surface preparation step to alter the morphology, chemistry and energy characteristics and enhance the adhesion of polymeric resins [4]. The aim of the present study was to evaluate the contact angle of three commercial multi-mode adhesives and also the influence of prior etching on contact angle measurements.

MATERIALS AND METHODS

Sample preparation

36 caries-free intact extracted human mandibular molars were used in this study. Approval was obtained from the Institutional Ethical Committee(IEC/2018/012). The teeth were cleaned of superficial debris and stored in 0.5% Chloramine solution until further processing. Two sections parallel to the occlusal surface were made beneath the dentino–enamel junction to obtain superficial dentin slices of 2 mm thickness which were then briefly polished with sandpaper.

Adhesives systems

Three adhesive systems were compared- Single Bond Universal (SBU)(3M ESPE, St Paul, MN, USA), Solare Universal Bond(SUB)(GC Corporation, Tokyo, Japan) and Tetric N Bond Universal adhesive(TNB) (Ivoclar Vivadent). The dentin specimens were divided randomly into 3 groups (n= 12). Half of the specimens in each group were subjected to acid etching (3M ESPE Scotchbond universal etchant) prior to the application of adhesives.

Contact angle measurement

Measurement of static contact angle θ* was performed using the sessile drop method by placing a drop of each liquid adhesive, in a volume of 1.0 µL using a micro-pipette. A set of 3 images was captured within 2s after the placement of a drop of liquid adhesive on the dentin specimen and subsequent contact angle was measured by Axisymmetric Drop Shape analysis using Rame Harte Goniometer(Model 400-F4). Statistical analysis included the application of one way-ANOVA, Post hoc Tukey and Independent t-test.

RESULTS

The results of the present study illustrates the variations in contact angles observed for the adhesives applied on the flat dentine surfaces. An enlarged view of the contact angle measurement is shown (Figure 1).

One- way ANOVA analysis(Table 1) showed significant difference in contact angle measurements among the groups with respect to both etched and unetched dentin. The post hoc Tukey test(Table 2) was carried out for pairwise comparison and showed significant difference between the groups. Single Bond Universal exhibited highest contact angle followed by Tetric N Bond Universal and least contact angle was shown by Solare Universal Bond ($p < 0.05$)(Figure 2).Comparison of contact angle values of etched and unetched dentin of the three universal adhesives was carried out using Independent t- test(Table-3)

DISCUSSION

Obtaining a successful adhesion is highly dependent on the adaptation and spreading of the liquid adhesive system [5].The wetting behaviour of dentin bonding agents is influenced by two parameters: the physico–chemical characteristics of the adhesive blend and the hydrophilic/hydrophobic ratio of the monomers [6].

Contact angle is one of the common methods to measure the wettability of a material. The simplest way of measuring the contact angle of a sessile drop is with a contact angle goniometer, which allows the user to optically evaluate the shadow image of the liquid drop. The smaller the contact angle, the greater is the adhesive wettability [7].

Universal adhesives represent the latest generation of adhesives in the market, incorporating the versatility of adapting to the clinical situation, by application under different etching modes. The universal adhesives used in the present study are applied in self-etch mode with the goal of eliminating the technique sensitive step of acid etching, as their acidic monomers simultaneously etch and infiltrate the dental substrate, thereby minimizing the discrepancies between hybridized and etched zones in the substrate. However, it has been shown that hybrid layer formed has been very thin or inexistent due to their mild (or ultra-mild) acidity [8].

According to a study by Pashley et al. [9], the thickness of the smear layer has a greater effect on the surface wettability .Therefore, some researchers recommend acid conditioning of the dentin to eliminate the smear layer & increase the microporosity of the intertubular dentin, thus, modifying both the morphology and wettability of the dentin surface considerably [8,10].Taschner.M et.al [5] supported the application of a phosphoric acid etchant before the placement of a one-step self-adhesive bonding system because improved adhesion capability was found on acid-etched enamel and dentine than use of self-etch adhesives alone. However, there is no consensus in literature on the effectiveness of the wettability of dentin after acid etching. Some researchers found that dentin wettability increases after acid etching [6,11] procedure, while others reported no significant alteration [12]or decrease in dentin wettability [16]. Manuel Toledano et al [13] in his study showed that wettability is enhanced for deep dentin with acid etching, but not so for the superficial dentin. Also, J.I. Rosales et al [14] concluded in his study that there was significant decrease in contact angles for deep dentin compared to superficial dentin.

In addition to the pH, the solvent chemistry and specific functional monomer types and ratios could also play a vital role in these adhesives [15,16]. SBU adhesive with a pH of 2.7 is considered to be a mild self-etch adhesive [16]. It contains 10-MDP, Vitrebond Copolymer, HEMA, water and dimethacrylate resins with ethanol as solvent. TNB Universal adhesive has a pH of approximately 2.5 – 3.0 and is based on combination of monomers of hydrophilic (HEMA), intermediate (Bis-GMA) and hydrophobic (decandioldimethacrylate/D3MA), MDP nature with an ethanol/water-based solvent system [17]. SUB is usually HEMA-free and has a unique formulation, with two functional monomers, 4-MET and phosphoric acid monomer in an ethanol based solvent system.

Genevie`ve Gre´goire et.al (2011) [18] showed that contact angles differed significantly among the self-etch adhesives but were not correlated with HEMA or solvent presence which correlate with that of the present study where there appears to be no clear relationship between HEMA and surface free-energy (SBU is a HEMA-free adhesive, whereas other two groups contain HEMA).

An important component of the adhesive system that could influence wettability are functional monomers which are acidic molecules that may serve various functions, such as etching tooth substrates , enhancing monomer penetration, and imparting the adhesives with potential for chemical interactions with the dental substrates [19,20]. They have at least one polymerizable group and one functional group that wets and demineralizes the dentin substrate. They are partially responsible for the hydrophobic/hydrophilic behaviour of the bonding resins [21,22,23].

More hydrophilic spacer carbon chain induces more water sorption and better dentin wettability and hydrophobic functional monomers (MDP) are suitable in order to avoid the effects of hydrolytic degradation.The 10-MDP monomer produces a stable bond on interaction with hydroxyapatite [24].The dihydrogenphosphate group from 10-MDP monomer is responsible for the etching and chemical bonding, while its long carbonyl chain provides the hydrophobic properties [25].Presence of ethanol was found to limit the dissociation of the phosphate groups from the 10-MDP monomer that may explain the reduced wetting behaviour of SBU adhesive [19].

The 4-MET functional monomer in SUB presents two carboxylic groups which are responsible for the demineralizing properties and enhancing the wetting. Recently, Yoshida et al showed that 4-MET is able to establish an ionic bond with calcium in hydroxyapatite and the resulting Ca-4MET salt has high solubility and is relatively unstable, contributing to increased wettability [26].

These findings suggest that the presence of functional monomers in the tested adhesives might have shown a significant effect on the wetting behaviour on the dentin substrate. Improved wetting is due to the presence of hydrophilic functional monomers such as 4-MET and poor wetting may be due to hydrophobic monomers such as MDP and D3MA, though the concentration of these monomers in adhesive formulations is not specified by manufacturer.

CONCLUSION

Within the limitations of the present study, prior acid etching of dentin had no significant influence on contact angles of the universal adhesives. However, a significant difference in contact angles among these adhesives was observed that may be attributed to the functional monomers in the formulations of the tested adhesives. Further research is required for a better understanding of the relation of the composition on the physicochemical properties and flow of universal adhesives.

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Figure 1.Measurement of contact angle of adhesive on the dentin surface

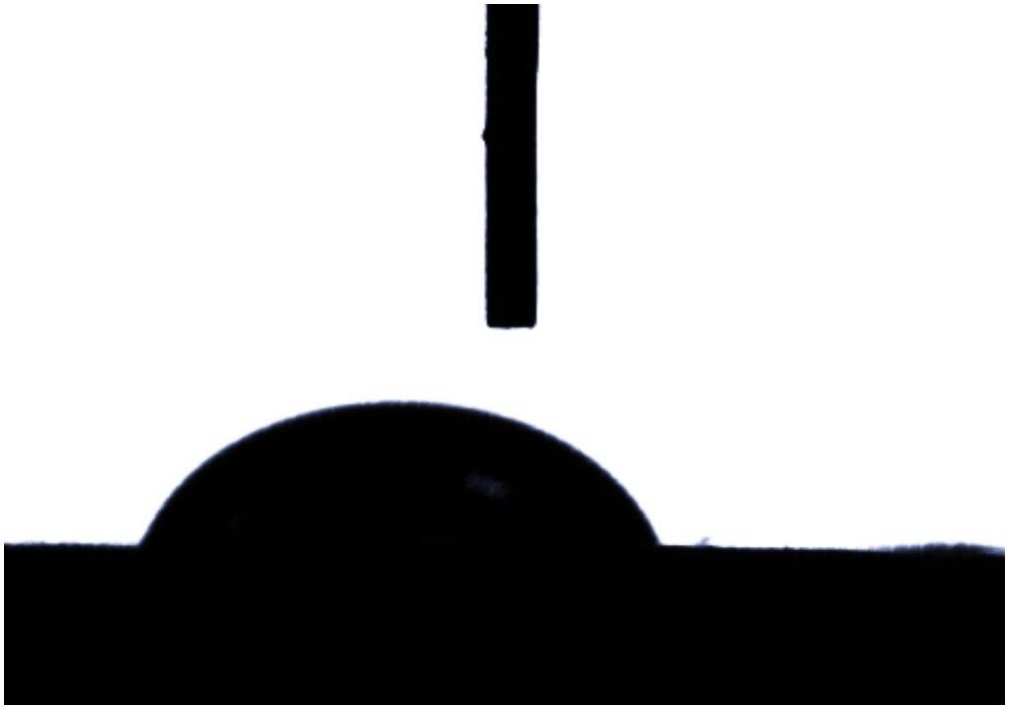

Table 1: Contact angle values of the three different universal adhesives on etched and unetched dentin (One- way ANOVA analysis)

	DESCRIPTIVES							
	ETCHED				**UNETCHED**			
Group	Mean	Std. Deviation	Std. Error	Sig.	Mean	Std. Deviation	Std. Error	Sig.
Single Bond Universal	72.900	2.5464	1.0396	**0.000***	73.267	8.2007	3.3479	**0.000***
Solare Universal Bond	38.250	4.2917	1.7521		42.033	3.6528	1.4912	
Tetric N Bond Universal	55.100	3.7513	1.5314		59.167	10.0604	4.1071	
***p<0.05 (Statistically significant)**								

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Table 2: Pairwise comparisons of contact angle values of the three different universal adhesives on etched and unetched dentin (Post- hoc Tukey test):

Multiple Comparisons							
		ETCHED			**UNETCHED**		
		Mean Difference	Std. Error	Sig.	Mean Difference	Std. Error	Sig.
Single Bond Universal	Solare Universal Bond	34.6500*	2.0810	.000	31.2333*	4.4945	.000
	Tetric N Bond Universal	17.8000*	2.0810	.000	14.1000*	4.4945	.018
Solare Universal Bond	Tetric N Bond Universal	-16.8500*	2.0810	.000	-17.1333*	4.4945	.005
***p<0.05 (Statistically significant)**							

Figure 2 : Graph showing the mean value of contact angles(measured in degrees) of the three universal adhesives on etched and unetched dentin

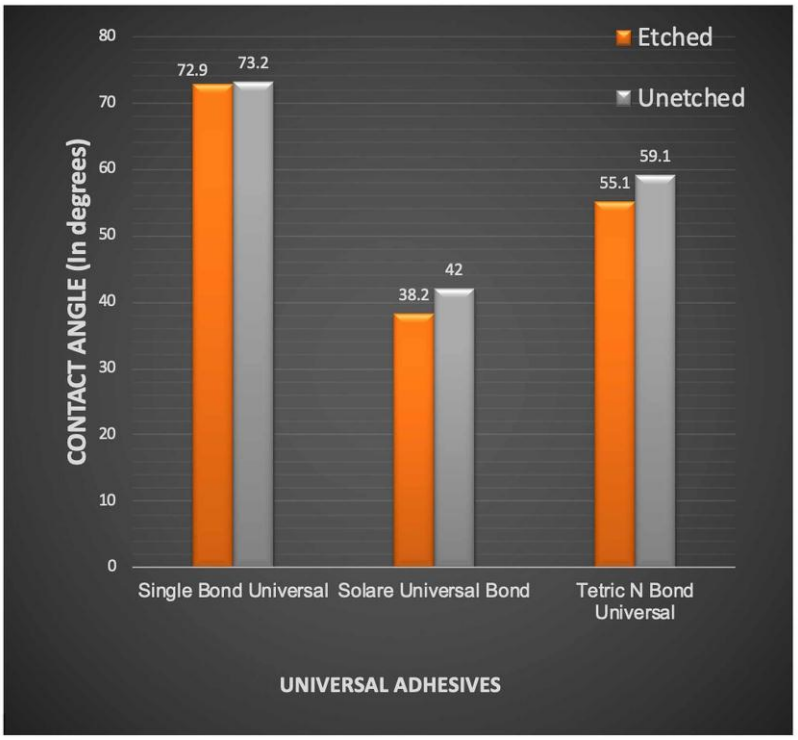

Table 3:Comparison of contact angle values of etched and unetched dentin of the three universal adhesives (Independent t- test)

Group	Sig. (2-tailed)	Mean Difference	Std. Error Difference	95% Confidence Interval of the Difference	
				Lower	Upper
Single Bond Universal	**0.919**	-0.3667	3.5056	-8.1776	7.4443
Solare Universal Bond	**0.131**	-3.7833	2.3008	-8.9098	1.3431
Tetric N Bond Universal	**0.375**	-4.0667	4.3834	-13.8334	5.7001