Factors affecting the Degree of Conversion of Universal Adhesives

Natalie Rosenthal[a], Cristina Fiuza[a], Asmaa Haimeur[b], Rodrigo França[a.b]

[a] Dental Materials Research Lab, Dr. Gerald Niznick College of Dentistry, University of Manitoba, D232 Dentistry Building, 780 Bannatyne Ave, Winnipeg, MB, Canada.

[b] Department of Restorative Dentistry, Dr. Gerald Niznick College of Dentistry, University of Manitoba, D227 Dentistry Building, 780 Bannatyne Ave, Winnipeg, Canada.

Abstract

Universal adhesives are advertised as having a simplified application technique, however all still require multiple steps. This study tested the effect of solvent evaporation, oxygen inhibition layer (OIL) formation, and curing time on the degree of conversion (DC) for the following dental adhesives: All-Bond Universal Adhesive (AB), Prime & Bond Elect Universal Dental Adhesive (PB), iBond Universal (iBU), Scotchbond Universal (SBU), and One Coat 7.0 (OC). Adhesives were rubbed onto a glass slide for 20 s, then air blown for 5 s or 40 s. Some samples had an Epitex strip (Anaerobic) placed over the adhesive before polymerization, while the other group of samples were cured without an Epitex strip (Aerobic). Samples were light cured for 10 s, 20 s, or 40 s (n=4). The DC was measured using Fourier transmission infrared spectroscopy (FTIR). Results were analysed with 3 way ANOVA and Tukey *post-hoc* test (α=0.05). Curing under anaerobic conditions significantly increased the overall mean DC for all tested adhesives. A higher DC can be obtained by increasing solvent evaporation time for AB, iBU, SBU, and increasing curing time for all the adhesives, except iBU. Some universal adhesives benefit from a different application process than the manufacturers' instructions.

Keywords:

Oxygen inhibition layer, Curing time, A. self-etch adhesive, C. Infra-red spectroscopy.

1. Introduction

Ever since composite was introduced to the market in the 1960's [1], the need for efficient adhesion to a dentin substrate has increased, leading to the development of several generations of bonding agents. These generations have been divided into two different approaches, etch-and-rinse (ER) or self-etch (SE) technique [2]. The ER approach involves several steps such as applying phosphoric acid to remove the smear layer, rinsing the acid, drying the prepared tooth, and then applying primer and bond [3]. Among the advantages proposed by the SE technique, simplicity should be highlighted, since in this approach the etching, rinsing, and drying steps are no long required. For clinicians, it is advertised that SE can be applied as "*one step"*, since all three components are all combined into one bottle. In general, this makes the SE procedure more time efficient, less technique-sensitive, and easier [4, 5]. However, the manufacturers' instructions provide detailed descriptions on how to apply their products. Correctly following the steps, such as agitating, rubbing, and drying is important to achieve maximal performance of dentin bonding agents [6, 7].

All universal adhesives have one or more organic solvents, usually ethanol or acetone, to facilitate monomers infiltration [7-9]. Ideally, all water and organic solvent content should evaporate because residual content of these molecules may reduce the degree of polymerization, also known as degree of conversion (DC) [10-12]. An ideal degree of conversion is 65% [13]. A lower DC is associated with a weaker bonding strength [14, 15]. Having a low DC also decreases biocompatibility, since residual monomers can lead to cytotoxicity, therefore reducing the amount of monomer will improve the biocompatibility [14, 16-18].

Another factor that reduces polymerization rate is oxygen interacting with the outermost layer of the adhesive, forming what has been termed the oxygen inhibition layer (OIL) [19]. The

classical concept [20] of the OIL being important for bonding composite to the adhesive layer (where the adhesive's monomer polymerizes with the composite's monomer), has been contradicted in some studies [21, 22]. Also, for other substrates like ceramics and metals no data is available about beneficial effects of OIL. The relationship between DC and OIL are well established in the literature for SE adhesives [21, 23, 24], yet little data is available about OIL specifically about universal adhesives. Another issue is that the adhesive's DC values can be inflated by reducing or eliminating OIL. Some authors recommend removing OIL with an ethanol impregnated cotton pellet [25]; others suggest using the "anaerobic technique", where a plastic matrix strip is placed on top of the adhesive during curing to prevent OIL formation [23, 24]. Both techniques have been criticized for lacking of clinical correspondence [26].

Other studies have noted that the predominant cause for bond failure, from a dental materials perspective, is the adhesive, not the composite [10, 27, 28]. These facts raise questions about how less technique sensitive and easier universal adhesive effectively are. For that reason, this study aims to further investigate the influence of clinical steps such as solvent evaporate, time of light curing, and the presence of the oxygen-inhibition layer, on the DC of universal adhesives. The null hypothesis is that the three factors tested; solvent evaporation time, effect of the OIL, and curing time will not affect DC of universal adhesives.

2. Materials and Methods

A total of five adhesives were used in this study. Their chemical compositions, manufacturers' information and recommended methods of application are in Table 1. Four universal adhesives; Prime & Bond Elect Universal Dental Adhesive (PB), iBond Universal (iBU), Scotchbond Universal (SBU), All-Bond Universal Adhesive (AB), and a seventh generation adhesive, One Coat 7.0 (OC), were tested.

Table 1. Adhesive composition and method of application based on manufacturers' instructions.

Adhesive Brand	Manufacturer	Chemical composition	Manufacturer Instructions
Prime & Bond Elect Universal Dental Adhesive (PB)	Dentsply Caulk, Milford, DE, USA	Monomer: PENTA Solvent: Acetone Other: Mono-,di-, and trimethacrylate resins, diketone, organic phosphine oxide, stabilizers, cetylamine hydrofluoride, water, mono- and di-methacrylate resins, catalyst, camphorquinone, initiators	-Agitate for 20 s -Air dry for 5 s -Cure for 10 s
iBond Universal (iBU)	Heraeus Kulzer, Hanau, Germany	Monomer: UDMA Solvent: Acetone Other: 4-MET, glutaraldehyde, water, camphorquinone, stabilizer	-Rub for 20 s -Air dry until film no longer moves -Cure 10 s
Scotchbond Universal (SBU)	3M ESPE, St Paul, MN, USA	Monomer: 10-MDP Solvent: Ethanol Other: dimethacrylate resin, HEMA, polyalkenoic acid copolymer, filler, water, camphorquinone, silane	-Rub for a minimum of 20 s -Air dry for 5 s -Cure 10 s
All-Bond Universal Adhesive (AB)	BISCO Inc, Schaumburg, IL, USA	Monomer: MDP, Bis-GMA, Solvent: Ethanol Other: water, camphorquinone	-Apply 2 coats -Rub in for 10-15 s per coat, do not cure in between coats -Cure 10 s
One Coat 7.0 (OC)	Coltene Whaledent, Altstätten, Switzerland	Monomer: Methacrylate Solvent: Ethanol Other: Camphorquione, water	-Brush in for 20 s -Air dry for 5 s -Cure 10 s

PENTA: dipentaerythritol penta acrylate monophosphate; UDMA: Urethane dimethacrylate; 4-MET: 4-methacryloxyethyltrimellitate; 10-MDP: 10-methacryloyloxydecyl dihydrogen phosphate; HEMA: 2-hydroxyethyl methacrylate; Bis-GMA: bisphenol A glycidylmethacrylate

On a glass slide previously cleaned with ethanol, a single drop of adhesive was dispensed using the bottle's spout held at 90 degrees. All samples were mixed, agitated, and rubbed into the glass slide with a clean microbrush for 20 s over an area of 3.14 cm^2 outlined on the glass slide. The different protocols tested is represented in Figure 1 below.

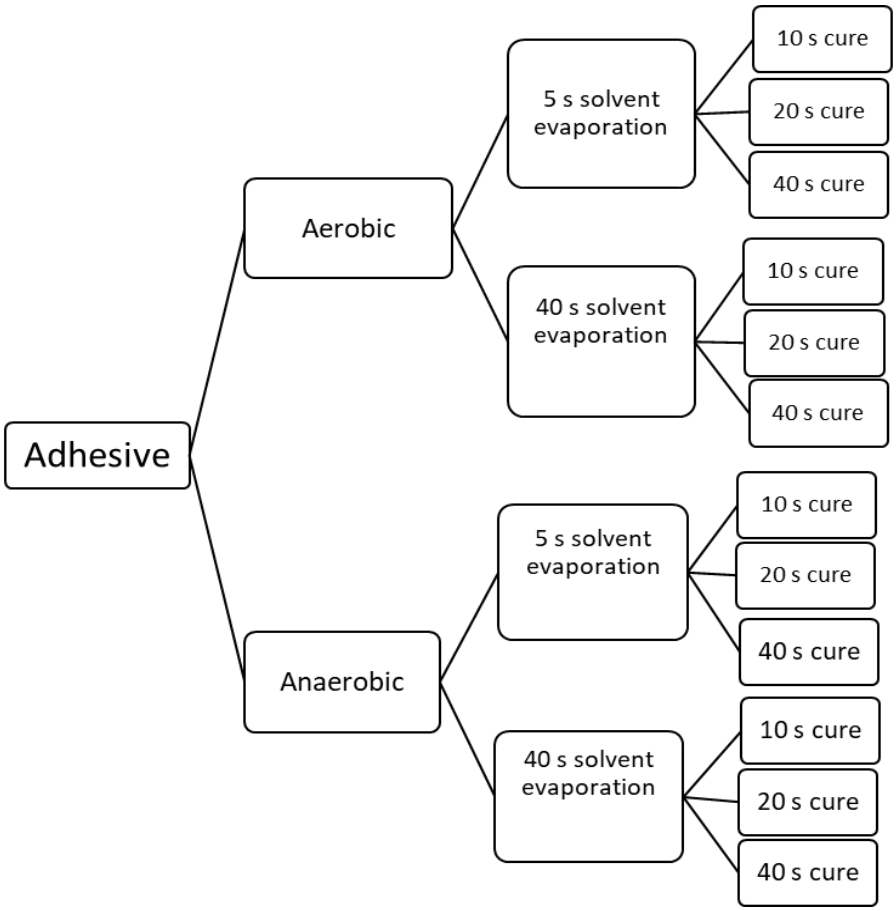

Figure 1. Schematic of factors applied to each adhesive.

To test the effect of solvent evaporation, samples had air blown from a syringe for either 5 s, which is the recommended time, or 40 s. Then to test the effect of the OIL, some samples had an Epitex translucent finishing strip (GC America Incorporated, Alsip, IL, USA) placed over the drop of adhesive before polymerization to minimize the effect of the OIL (anaerobic technique) and the other group of samples were cured without an Epitex strip being placed (aerobic technique). All samples were cured for either 10 s, as recommended by the manufacturers, 20 s, or

40 s using a light-emitting diode (LED) light curing unit (Valo Cordless, Ultradent, South Jordan, UT, USA), with a potency of 1000 mW/cm^2. All samples were cured at a distance of 8.0 mm, this distance was standardized by resting the curing light on a glass slab. To minimize the effect of operator variability, all samples were prepared and tested by a single operator.

2.1 Attenuated Total Reflection-Fourier Transformed Infra-Red (ATR-FTIR) analysis.

The effect of the clinical steps on the DC for each sample was determined by using ATR-FTIR (Thermo Scientific Nicolet 6700, Waltham, MA, USA). For each experimental condition, four samples were produced (n=4). The carbonyl peaks at 1700 cm^{-1} and the aliphatic double carbon bond at 1640 cm^{-1} were used to calculate the DC for the universal adhesives; PB, iBU, AB and SBU, while the aromatic peaks at 1610 cm^{-1} and the aliphatic double bond peaks at 1640 cm^{-1} were used for OC [15]. In order to calculate the DC for each sample, an unpolymerized sample was also prepared under the same conditions except it was not cured. The DC was calculated using equation 1 below.

$$1- \frac{[(\text{unpolymerized 1640 cm}^{-1}\text{ peak}) / (\text{unpolymerized 1700 cm}^{-1}\text{ peak})]}{[(\text{polymerized 1640 cm}^{-1}\text{ peak} / (\text{polymerized 1700 cm}^{-1}\text{ peak})]} \times 100 = \text{DC} \quad (1)$$

To increase the accuracy, all samples were prepared at the testing site and subjected to FTIR analysis immediately after curing. To ensure the sample did not polymerize further during FTIR analysis, the FTIR machine was covered to prevent any penetration of ambient light.

All the data from each adhesive was tabulated and submitted to statistical analysis using 3 way ANOVA and Tukey *post-hoc* test (α=0.05), and the analysis was performed using OriginLab softwear version 2017 (Northampton, MA, USA). The three factors studied were: solvent evaporation time, anaerobic effect (presence of OIL), and curing time.

3. Results

The three experimental conditions tested when analyzed independently from each other, proved to be statistically significant for the adhesives used in this study. When analyzed with the three factors interacting with each other, the significance drops for all but SBU. Letting the solvent evaporate for 40 s was statistically significant for AB, iBU, and SBU, but not significant for PB and OC. The overall results indicated that curing time was statistically significant for AB, PB, OC, and SBU, but not for iBU.

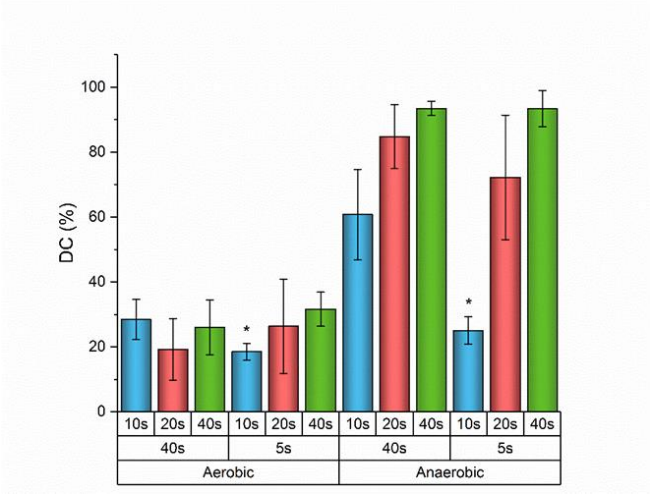

Figure 2. DC mean (%) for AB under aerobic or anaerobic conditions, solvent evaporation for 40 or 5 s, and curing times of 10, 20, or 40 s. Asterisk signifies manufacturer's instructions.

For AB, the DC mean under anaerobic conditions was 71.6 % (± 26.2), which presented to be over twice the DC for aerobic conditions which was 25.0% (± 9.0). When comparing solvent evaporation time, the DC was higher with 40 s of solvent evaporation when curing for 10 s. As seen in figure 2, under aerobic conditions the mean results for solvent evaporation and curing time were not significant. The highest DC for AB was obtained under anaerobic conditions when cured for 20 or 40 s. When the solvent evaporated for 5 s and cured for 10 s, it resulted the lowest DC mean for both aerobic and anaerobic groups.

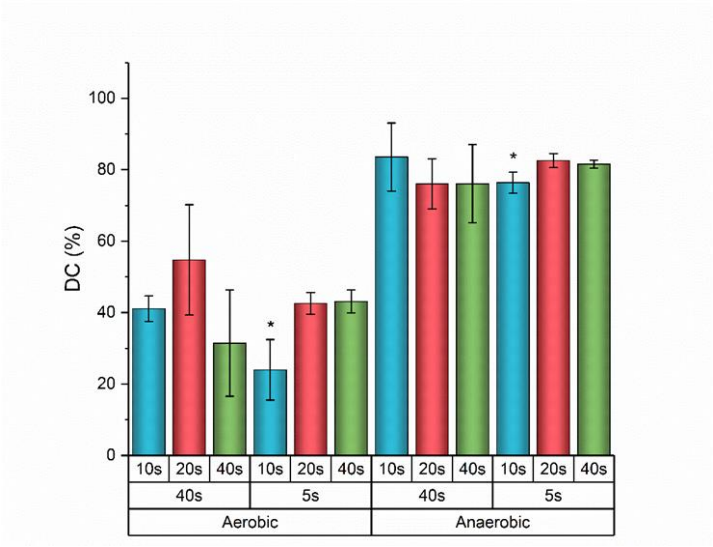

Figure 3. DC mean (%) and for PB under aerobic or anaerobic conditions, solvent evaporation for 40 or 5 s, and curing times of 10, 20, or 40 s. Asterisk signifies manufacturer's instructions.

For PB, the results showed a consistently high DC when anaerobic conditions were used regardless of the time of solvent evaporation and time of cure, but the anaerobic results were not statistically significant compared to each other. The increasing from 5 s to 40 s in the solvent evaporation time seems did not produce major improved in DC values.

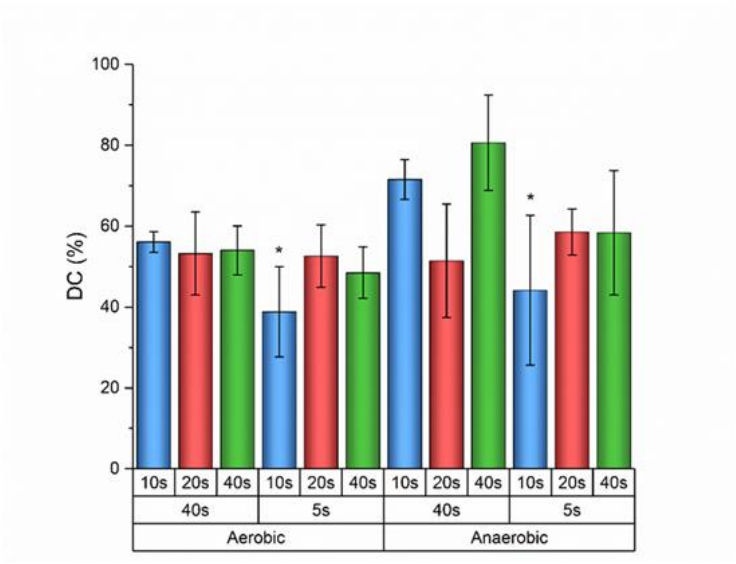

Figure 4. DC mean (%) and for iBU under aerobic or anaerobic conditions, solvent evaporation for 40 or 5 s, and curing times of 10, 20, or 40 s. Asterisk signifies manufacturer's instructions.

As seen in figure 4, iBU was less affected by the OIL, because under aerobic conditions this adhesive had high DC values with a mean DC of 50.54% (± 9.10), while the anaerobic conditions had 60.78% (± 3.42). Also the solvent evaporation time had no significant effect on the DC. The highest DC was obtained under anaerobic conditions when the solvent had 40 s to evaporate and cured for 40 s, and this result was statistically different from the 10 s recommend by the manufacturer.

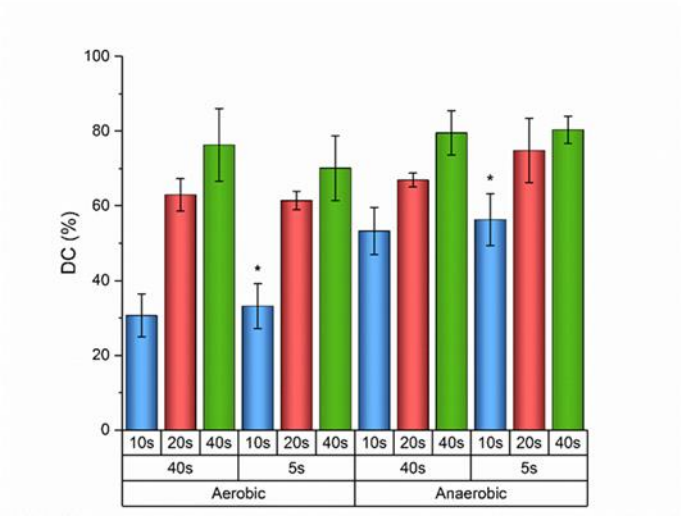

Figure 5. DC mean (%) and for OC under aerobic or anaerobic conditions, solvent evaporation for 40 or 5 s, and curing times of 10, 20, or 40 s. Asterisk signifies manufacturer's instructions.

For OC a trend was observed, an increase in the curing time increased the DC regardless of solvent evaporation time and the OIL presence. Although the manufacturer recommends 10 s for curing time, leaving the curing-light on for 40 s may double the DC values, even for aerobic conditions.

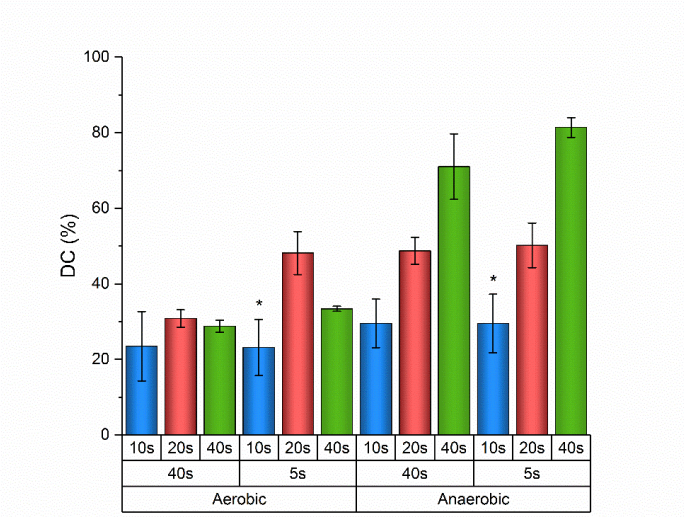

Figure 6. DC mean (%) and for SBU under aerobic or anaerobic conditions, solvent evaporation for 40 or 5 s, and curing times of 10, 20, or 40 s. Asterisk signifies manufacturer's instructions.

For SBU, curing time increased the DC when under anaerobic conditions. The highest DC was achieved when cured for 40 s under anaerobic conditions regardless of the solvent evaporation time. Lengthening the solvent evaporation time did not increase the DC for SBU.

4. Discussion

Simplicity has been the concept behind the proposed use of universal adhesives, because with 5 s of solvent evaporation and 10 s of light curing, a strong and durable bond could be produced. Therefore, the question that motivated this study was: how these different universal adhesives, with varying chemical components, can have almost identical manipulation recommendations? In other words, does following the manufacturer's recommendations lead to maximizing the material's properties? Or is the focus on minimizing application time to make it more attractive? This study's results showed that for some adhesives, there is a mismatch between the proposed solvent evaporation time and curing time recommended by manufacturers and those

where high DC values were obtained. In particular, AB, OC, and SBU benefited from increasing the curing time. Some of the results have large standard deviations, which can be explained by how the adhesives are heterogeneous materials, so each drop tested could have a slightly different percentage of individual components.

Literature already shows that the presence of the solvent can decrease polymerization, since the monomers are positioned further apart [10]. Therefore, letting the solvent evaporate longer, brings the monomers in greater proximity to each other and increases the bond strength [29]. When there was less than 10 s of solvent evaporation time, other studies found a lower average bond strength [28, 29]. Some studies showed that letting the solvent evaporate for longer than the manufacturer's recommendations increased the bond strength [28, 30]. In the study done, solvent evaporation time was chosen to be 40 s because a previous study done found 20 s to have no significant difference in bond strength for some adhesives, so doubling the solvent evaporation time was done to see if this would result in significant differences [30]. The solvents in the adhesives tested were ethanol (SBU, AB, OC) and acetone (PB, iBU). Ethanol has a vapor pressure of 42.75 mm Hg at 20˚C [31] while acetone's is 185 mm Hg at 20˚C [32]. These differences in vapor pressure should result in adhesives with ethanol requiring more solvent evaporation time than adhesives with acetone. Yet with such volatile solvents, letting the solvent evaporate for a prolonged period did not yield significant results for each adhesive under aerobic conditions.

Photopolymerization begins by the initiator molecule, which usually is camphorquinone (CQ), becoming activated by light. The direct effect of the time of light curing on DC is well established in literature [2, 15, 33-35]. It is expected that when more energy from light is provided to CQ more monomers will become polymerized. However, this is not what happen for some experimental conditions of this study. For example, increasing four times the energy did not

improve their DC values for iBU and PB under anaerobic conditions. The reasons for this are currently unclear, and further studies would be required to understand this phenomenon. Meanwhile, this finding could be a clinical advantage for these adhesives, because prolonged curing time may result in clinical complications. The heat generated from prolonged light curing, can cause water convective movement in the dentin tubules [4, 36]. This movement increases the water content at the adhesive-dentin interface which accelerates the rate of degradation [36]. Water may bring matrix metalloproteases to the interface, which also contributes to bond degradation [37, 38].

In this study, the effect of OIL was tested comparing the polymerization of the adhesives in two conditions: the aerobic condition, when the adhesive is exposed to air, which mimics the adhesive after being applied on the tooth surface; and the anaerobic condition, when air is no longer present on adhesive surface, which mimics the condition when the first layer of composite is applied on top of the adhesive. For all tested adhesives, when OIL was present, the DC values were far lower than when OIL formation was prevented. Also, some results from this study showed that in aerobic conditions there is no significant difference for 40 s over 5 s for each adhesive tested, but under anaerobic conditions better results was found with 40 s of solvent evaporation time. This finding could indicate that OIL presence could mask the potential improvement brought from the other factors. Curing under anaerobic conditions proved to have the greatest effect on increasing the DC for AB, PB, and SBU. Other studies have also confirmed the OIL as significant [26, 39] and that removal of the OIL increased shear bond strength as well [25]. The opposite is also argued that the OIL is desirable since the monomers can polymerize with the layer of composite placed [40]. Then another study found the OIL to not be significant [41]. It is worth noting that the OIL contains a lower concentration of CQ compared to the polymerized layer

underneath, which will influence the ability for the OIL to be cured under the restorative material [25]. The presence of the OIL may be what is preventing other factors from improving the physiochemical properties of the universal adhesive.

5. Conclusion

With the limitation of this study, it is possible to conclude that letting the solvent evaporate for 40 s increased the DC for AB, iBU, and SBU, but showed no significant difference in the results for PB and OC. Curing under anaerobic conditions significantly increased the overall DC mean for all tested adhesives. The overall results for AB, OC, and SBU indicated a higher DC when the curing time was longer than 10 s. iBU showed no significant difference with respect to curing time. The null hypothesis has been rejected, since the three factors tested did affect the DC.

Acknowledgement

This study was funded by the University of Manitoba, Dr. Gerald Niznick College of Dentistry Research Grant.

There was no conflict of interest for this study.

References

[1] Bowen R.L., Dental filling material comprising vinyl silane treated fused silica and a binder consisting of the reaction product of Bis phenol and glycidyl acrylate., Patent No: 3,066,112, (1962).

[2] Van Landuyt K.L., Snauwaert J., De Munck J., Peumans M., Yoshida Y., Poitevin A., et al., Systematic review of the chemical composition of contemporary dental adhesives, Biomaterials, 28 (2007) 3757-85.

[3] Buonocore M.G., A Simple Method of Increasing the Adhesion of Acrylic Filling Materials to Enamel Surfaces, Journal of dental research, 34 (1955) 849-53.

[4] Chen C., Niu L.N., Xie H., Zhang Z.Y., Zhou L.Q., Jiao K., et al., Bonding of universal adhesives to dentine--Old wine in new bottles?, Journal of dentistry, 43 (2015) 525-36.

[5] Tezvergil-Mutluay A., Pashley D., Mutluay M.M., Long-Term Durability of Dental Adhesives, Current Oral Health Reports, 2 (2015) 174-81.

[6] Masarwa N., Mohamed A., Abou-Rabii I., Abu Zaghlan R., Steier L., Longevity of Self-etch Dentin Bonding Adhesives Compared to Etch-and-rinse Dentin Bonding Adhesives: A Systematic Review, The journal of evidence-based dental practice, 16 (2016) 96-106.

[7] Gregoire G., Sharrock P., Prigent Y., Performance of a universal adhesive on etched and non-etched surfaces: Do the results match the expectations?, Mater Sci Eng C Mater Biol Appl, 66 (2016) 199-205.

[8] Ekambaram M., Yiu C.K.Y., Matinlinna J.P., An overview of solvents in resin–dentin bonding, International Journal of Adhesion and Adhesives, 57 (2015) 22-33.

[9] Nagpal R., Manuja N., Pandit I., Effect of Ethanol Wet Bonding Technique on the Durability of Resin-Dentin Bond with Contemporary Adhesive Systems, The Journal of clinical pediatric dentistry, 39 (2015) 133-42.

[10] Luque-Martinez I.V., Perdigao J., Munoz M.A., Sezinando A., Reis A., Loguercio A.D., Effects of solvent evaporation time on immediate adhesive properties of universal adhesives to dentin, Dental materials : official publication of the Academy of Dental Materials, 30 (2014) 1126-35.

[11] Yiu C.K., Pashley E.L., Hiraishi N., King N.M., Goracci C., Ferrari M., et al., Solvent and water retention in dental adhesive blends after evaporation, Biomaterials, 26 (2005) 6863-72.

[12] Elliott J.E., Bowman C.N., Effects of Solvent Quality during Polymerization on Network Structure of Cross-Linked Methacrylate Copolymers, The Journal of Physical Chemistry B, 106 (2002) 2843-7.

[13] Heymann H.O., Swift E., Ritter A.V., Sturdevant's Art and Science of Operative Dentistry 6th ed., in: J D. (Ed.), Elsevier Mosby St. Louis, Missouri, 2013, pp. 64-5.

[14] Hass V., Dobrovolski M., Zander-Grande C., Martins G.C., Gordillo L.A., Rodrigues Accorinte Mde L., et al., Correlation between degree of conversion, resin-dentin bond strength and nanoleakage of simplified etch-and-rinse adhesives, Dental materials : official publication of the Academy of Dental Materials, 29 (2013) 921-8.

[15] Navarra C.O., Breschi L., Turco G., Diolosa M., Fontanive L., Manzoli L., et al., Degree of conversion of two-step etch-and-rinse adhesives: In situ micro-Raman analysis, Journal of dentistry, 40 (2012) 711-7.

[16] Van Landuyt K.L., Krifka S., Hiller K.A., Bolay C., Waha C., Van Meerbeek B., et al., Evaluation of cell responses toward adhesives with different photoinitiating systems, Dental materials : official publication of the Academy of Dental Materials, 31 (2015) 916-27.

[17] Aranha A.M., Giro E.M., Hebling J., Lessa F.C., Costa C.A., Effects of light-curing time on the cytotoxicity of a restorative composite resin on odontoblast-like cells, J Appl Oral Sci, 18 (2010) 461-6.

[18] dos Santos R.L., de Sampaio G.A., de Carvalho F.G., Pithon M.M., Guenes G.M., Alves P.M., Influence of degree of conversion on the biocompatibility of different composites in vivo, The journal of adhesive dentistry, 16 (2014) 15-20.

[19] Bijelic-Donova J., Garoushi S., Lassila L.V., Vallittu P.K., Oxygen inhibition layer of composite resins: effects of layer thickness and surface layer treatment on the interlayer bond strength, Eur J Oral Sci, 123 (2015) 53-60.
[20] Yamaji A., Tsujimoto A., Asaoka T., Matsuyoshi S., Tsuchiya K., Takamizawa T., et al., Effect of oxygen inhibition in two-step self-etch systems on surface free energy and dentin bond strength with a chemically cured resin composite, Journal of oral science, 56 (2014) 201-7.
[21] Suh B.I., Oxigen-inhibited layer in adhesion dentistry, Journal of Esthetic and Restorative Dentistry, 16 (2004) 316-23.
[22] Endo T., Osada T., Finger W.J., Hoffmann M., Kanehira M., Komatsu M., Effect of oxygen inhibition of self-etching adhesives on enamel-dentin polymer bond, J Adhes Dent, 9 (2007) 33-8.
[23] Faria-e-Silva A.L., Lima A.F., Moraes R.R., Piva E., Martins L.R., Degree of conversion of etch-and-rinse and self-etch adhesives light-cured using QTH or LED, Operative dentistry, 35 (2010) 649-54.
[24] Moraes R.R., Faria-e-Silva A.L., Ogliari F.A., Correr-Sobrinho L., Demarco F.F., Piva E., Impact of immediate and delayed light activation on self-polymerization of dual-cured dental resin luting agents, Acta Biomater, 5 (2009) 2095-100.
[25] Tsujimoto A., Barkmeier W.W., Takamizawa T., Latta M.A., Miyazaki M., Influence of the Oxygen-inhibited Layer on Bonding Performance of Dental Adhesive Systems: Surface Free Energy Perspectives, J Adhes Dent, 18 (2016) 51-8.
[26] Robertson L., Phaneuf M., Haimeur A., Pesun I., França R., Degree of Conversion and Oxygen-Inhibited Layer Effect of Three Dental Adhesives, Dentistry Journal, 4 (2016) 37.
[27] Rosa W.L., Piva E., Silva A.F., Bond strength of universal adhesives: A systematic review and meta-analysis, Journal of dentistry, 43 (2015) 765-76.
[28] Fu J., Saikaew P., Kawano S., Carvalho R.M., Hannig M., Sano H., et al., Effect of air-blowing duration on the bond strength of current one-step adhesives to dentin, Dental materials : official publication of the Academy of Dental Materials, (2017).
[29] Lee Y., Park J.W., Effect of moisture and drying time on the bond strength of the one-step self-etching adhesive system, Restor Dent Endod, 37 (2012) 155-9.
[30] Jacobsen T., Finger W.J., Kanehira M., Air-drying time of self-etching adhesives vs bonding efficacy, Journal of Adhesive Dentistry, 8 (2006) 387-92.
[31] ScienceLab, Material Safety Data Sheet Ethyl alcohol 200 Proof MSDS, Houston, TX, 2013.
[32] Axiall, LLC, Material Safety Data Sheet: Acetone, Plaquemine, LA, USA, 2013.
[33] Aguiar T.R., de Oliveira M., Arrais C.A., Ambrosano G.M., Rueggeberg F., Giannini M., The effect of photopolymerization on the degree of conversion, polymerization kinetic, biaxial flexure strength, and modulus of self-adhesive resin cements, J Prosthet Dent, 113 (2015) 128-34.
[34] Baek. C.-J., Hyun. S.-H., Lee. S.-K., Seol. H.-J., Kim. H.-I., Kwon. Y.H., The Effects of Light Intensity and Light-curing Time on the Degree of Polymerization of Dental Composite Resins, Dental Materials, 27 (2008) 523-33.
[35] Catelan A., Mainardi Mdo C., Soares G.P., de Lima A.F., Ambrosano G.M., Lima D.A., et al., Effect of light curing protocol on degree of conversion of composites, Acta odontologica Scandinavica, 72 (2014) 898-902.
[36] Tay F., Pashley D., Suh B., Hiraishi N., Yiu C., Water Treeing in Simplified Dentin Adhesives– Déjà Vu?, Operative dentistry, 30 (2005) 561-79.
[37] Tjaderhane L., Nascimento F.D., Breschi L., Mazzoni A., Tersariol I.L., Geraldeli S., et al., Optimizing dentin bond durability: control of collagen degradation by matrix metalloproteinases and cysteine cathepsins, Dental materials : official publication of the Academy of Dental Materials, 29 (2013) 116-35.
[38] Perdigao J., Reis A., Loguercio A.D., Dentin adhesion and MMPs: a comprehensive review, J Esthet Restor Dent, 25 (2013) 219-41.

[39] Velazquez E., Vaidyanathan J., Tritala K V., Houpt M., Shey Z., Hagen S.V., Effect of primer solvent and curing mode on dentin shear bond strength and interface morphology, Quintessence intemational, 34 (2003) 548-55.
[40] Truffier-Boutry D., Place E., Devaux J., LeLoup G., Interfacial layer characterization in dental composite, Journal of Oral Rehabilitation, 30 (2003) 74–7.
[41] Suh B.I., Oxygen-inhibited layer in adhesion dentistry, Journal of esthetic and restorataive dentistry, 16 (2004) 316-23.