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PREHEATING IMPACT ON THE DEGREE OF CONVERSION OF SINGLE BOTTLE ADHESIVE SYSTEMS: AN IN VITRO STUDY

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ABSTRACT:

Purpose: to evaluate and compare preheating impact on the degree of conversion of single bottle adhesive systems

Materials and methods: The single bottle adhesive systems used in the study was Adper Easy One, XP Bond and Optibond Solo. The degree of conversion was evaluated for each adhesive system at different temperatures (25° c = room temperature, 60° c = preheated). 60 disk shaped samples (6mm x 1mm) were prepared using each adhesive system. One drop of each adhesive system was placed into the mold followed by solvent evaporation for 10 sec with the use of uncontaminated air spray. Then the mylar strip was placed over the mold and the adhesive was photoactivated for 20 seconds with a light emitting diode. After photoactivation the sample was removed from the mold and stored dry in light proof containers at 37°c for 24 hours. The degree of conversion was analyzed using fourier transform infrared total reflectance spectroscopy. The absorption spectra of nonpolymerized and polymerized adhesives was obtained.

Results: Adper Easy One had the highest mean value, whereas XP Bond provided the lowest mean value of degree of conversion, regardless of the adhesive temperature.

Conclusion: preheating the single bottle adhesive systems increased the degree of conversion for all the systems tested. Adper Easy One had the highest value for degree of conversion at all the temperature's tested.

Keywords: Preheating, Adper Easy One, Xp Bond, Optibond Solo, Degree Of Conversion (DC)

INTRODUCTION:

The development of adhesive systems has changed the concepts of bonding in dentistry. Today, adhesive systems are used in direct procedures for reattachment of fractured fragments, corrections in tooth morphology, restoration of anterior and posterior cavities, fissure sealing, indirect procedures involving cementation of root canal posts and indirect ceramic and composite crowns.[1] Contemporary adhesive systems are categorized as etch-and-rinse (ER) and self-etch (SE) systems based on the management of the smear layer substrate.[2]

Successful adhesion to enamel and dentin is a fundamental requirement prior to the placement of dental materials and is directly dependent on the quality of the hybrid layer. Hence, any approach to prolong the lifetime of

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adhesives might focus on improving the stability of the bonding interface of these biomaterials to tooth tissues.[3] Optimal monomer infiltration into the demineralized substrates and the achievement of a high degree of conversion (DC) are crucial factors in establishing long-lasting bonds.[4]

A low DC of dental adhesives is associated with low bond strength values and mechanical properties, high monomer elution, increased permeability, and phase separation.[5] Moreover, reduced DC even accounts for the possible continuous etching of the tooth substrate due to suboptimally polymerized acidic monomer in self-etch adhesives. Thus, obtaining a high DC of adhesive systems is a crucial factor in the long-term stability of the hybrid layer.[4]

Although manufacturers recommend storing adhesive materials at room temperature, dentists still use the traditional practice of refrigerating materials to extend its shelf life. It has been shown that when refrigerated materials are used immediately, without allowing time to reach the room temperature, hence their efficacy is reduced. In this refrigerated condition, the overall conversion of monomers is lowered and the adhesive viscosity increased, which reduces the penetration of adhesives into acid-etched dentin, resulting in a decrease in resin dentin bond strength. Refrigeration also affects solvent vapor pressure inhibiting the evaporation of the solvents from the adhesive layer.[6]

Methods have been proposed to increase the degree of conversion of resin based dental materials and one such method is preheating before photoactivation. Heating the material to high temperatures decreases viscosity and increases the radical mobility which favors higher degree of conversion.[7]

Therefore this study was undertaken to analyze and compare the degree of conversion of preheated and non-preheated single bottle adhesive systems. The nulls hypothesis was stated that preheating would have no impact on the DC of the adhesive systems.

MATERIALS AND METHODS:

The single bottle adhesive systems used in the study were Adper Easy One (3M ESPE, St Paul, MN, USA), XP Bond (Dentsply/Caulk, Milford, DE, USA) and OptiBond Solo (Kerr Corporation). The degree of conversion was evaluated for each adhesive system at different temperatures (25^{o} C = room temperature : 60^{o} C = preheated)

TABLE 1 : Composition, Manufacturer of the Adhesive Systems used in the Study

Adhesive System	Composition (% by weight)	Manufacturer
Adper Easy One	Phosphoric acid acrylate (<11), Bis-GmaHEMA (<15) Dimethacrylates (<53), alcohol (<20)	3M ESPE St Paul,USA
XP Bond	HEMA (25-50), methacrylate (10-25), tert-butyl alcohol (10-25), acrylates (10-25)	Dentsply/Caulk, Milford, DE, USA
OptiBond Solo	Bis-Gma, BDMA, HEMA, silica, hexafluorosilicate Ethanol	Kerr Corporation

PREHEATING OF THE ADHESIVE SYSTEM:

Each bottle of the adhesive system was kept in an oven (incubator) at 25°C or 60°C two hours before starting the adhesive procedure. Before starting the adhesive procedure, the temperature was checked with a thermometer for each adhesive system.[7]

SPECIMEN PREPARATION :

60 disk shaped samples (6mm X 1mm) were prepared using each adhesive system. One drop of each adhesive system was placed into the mold followed by solvent evaporation for 10 sec with the use of uncontaminated air spray. Then the mylar strip was placed over the mold and the adhesive was photoactivated for 20 seconds with a light emitting diode (Coltolux, Coltene/Whaledent, Allastatten, Switzerland). The 8 mm diameter tip of the LED covered the sample during photoactivation. An irradiance of 1264 mW/cm^2 was distributed over the top surface of each sample. After photoactivation the sample was removed from the mold and stored dry in light proof containers at 37°C for 24 hours.

DC ANALYSIS:

The DC of conversion was analyzed using Fourier Transform Infrared Total Reflectance Spectroscopy (Spectrum 100, PerkinElmer, Shelton, CT, USA) at 24° C under 64% relative humidity.[4] The absorption spectra of nonpolymerized and polymerized adhesives were obtained from the wavenumber region between 4000cm^{-1} and 650cm^{-1} , with 32 scans at 4cm^{-1}. The DC(%) was calculated with the following equation

DC(%) = 100 X (1 – [R polymerized/R nonpolymerized]) cm^{-1}. The final DC values were analyzed using two way analysis of variance (ANOVA) considering adhesive system X temperature and Tukey's test. Where R represents the ratio between the absorbance peaks at 1638 and 1608.

RESUTS:

Two way ANOVA showed a statistically significant differences between materials and temperature ($p<0.05$). Multiple comparisons among the groups are listed in in Table 2. All three adhesive systems exhibited higher mean DC values at 60°C than they did at 25°C. Adper Easy One had the highest mean value, and XP Bond provided the lowest mean value, regardless of the adhesive temperature.

Table 2:

DC MEAN PERCENTAGE AT DIFFFERENT TEMPERATURES (%)		
MATERIALS	TEMPERATURE	
	25°C	60°C
OptiBond Solo	45.32	57.44
XP Bond	40.50	48.62
Adper Easy One	47.63	61.81

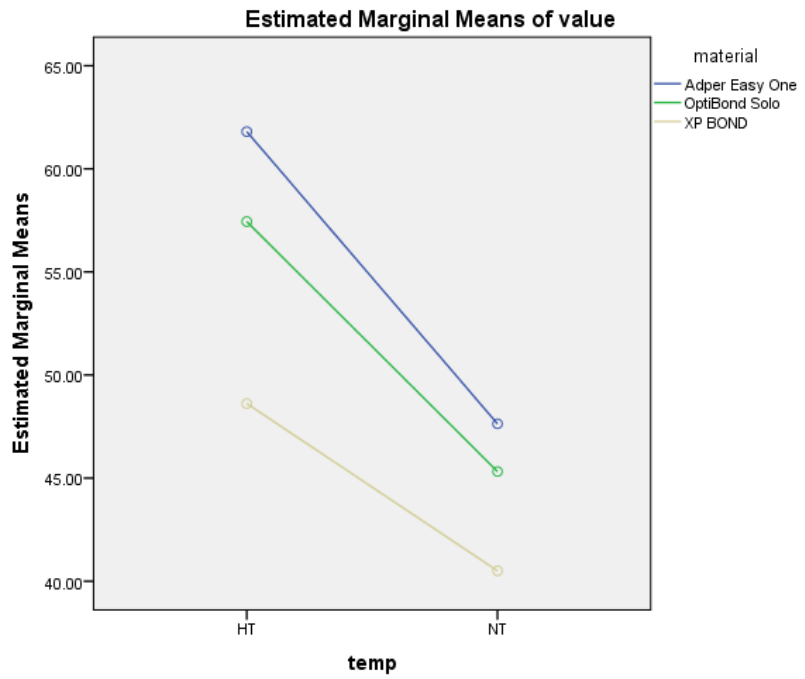
Tests of Between-Subjects Effects

Dependent Variable: value

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	1570.384[a]	5	314.077	1.845	.142
Intercept	75662.456	1	75662.456	444.510	.000
temp	986.707	1	986.707	5.797	.024
material	536.199	2	268.099	1.575	.228
temp * material	47.478	2	23.739	.139	.871
Error	4085.170	24	170.215		
Total	81318.010	30			
Corrected Total	5655.554	29			

a. R Squared = .278 (Adjusted R Squared = .127)

Two-way analysis of variance suggests that temperature has statistically significant effect on the dependent variable, when considered across all materials, as indicated by P-value of 0.024 (P < 0.05). The effect of material was statistically insignificant (P=0.228).



Material	Normal temperature	High temperature	P-value*
Adper Easy One (n=10)	47.63 (17.23)[aA]	61.81 (16.58)[aA]	0.2217
OptiBond Solo (n=10)	45.32 (15.06)[aA]	57.44 (5.37)[aA]	0.1508
XP Bond (n=10)	40.50 (11.63)[aA]	48.62 (7.64)[aA]	0.2342
P-value**	0.745	0.196	

Similar upper case letters indicate row wise statistical insignificance, while similar lower case letters indicate column wise statistical insignificance.

DISCUSSION:

DC of dental adhesives is an important parameter as low mechanical properties and monomer elution are related with low percentage of monomer to polymer conversion within resin-based materials. Lower degrees of conversions are associated with increased permeability, phase separation and higher water sorption of suboptimally cured adhesives.[8] The traditional method of using of low temperature adhesive systems results in difficulty in substrate wetting since the viscocity of the material is reduced. This affects the diffusion of solvated comonomers into the acid-etched demineralized dentin matrix.[6] Hence the method of preheating the adhesive system prior to polymerization was suggested as increased temperatures decreases viscosity and enhances radical mobility, resulting in additional polymerization and higher conversion (Nie *et al.*, 1998; Lovell *et al.*, 2001). The collision frequency of unreacted active groups and radicals increases with elevated curing temperature when below the glass transition temperature (Bausch *et al.*, 1981). Furthermore, as composite temperature is raised, free volume increases, giving trapped radicals increased mobility, resulting in further conversion.[9] Hence, the nulls hypothesis tested in the study, that higher temperature would not affect the DC of the adhesive systems was rejected as increased temperature provided higher DC of all adhesive systems tested (table 2).

AD Loguercio et al in his experimental study showed that preheating the single bottle adhesive system did not enhance the DC.[6] This result can be attributed to the fact that the adhesive system were heated at 50^{0}C. In addition to this the 10 second air application used to evaporate the solvent substantially decreased the temperature, leading to reduced DC. For this reason temperature of 60^{0}C was used in the study.

Dental bonding agents are composed of a mixture of hydrophilic primers and hydrophobic adhesive resins either dissolved in acetone, ethanol, water or a combination of both.[4] Acetone has a boiling point of (56.5°C) which is much less as compared with that of ethanol (78.5°C) and water (100°C). This suggests that when the adhesive system is heated to a temperature of 60^{0}C, faster solvent evaporation will occur with the acetone based adhesives. Faster solvent evaporation impairs ideal monomer infiltration to dentin leaving a thin adhesive layer which is more susceptible to polymerization inhibition by oxygen. As a result this can decrease the bond strength of adhesives. For the above reasons only alcohol based adhesive systems were used in our study.[7]

Adper Easy One showed the highest DC among the adhesive systems used, irrespective of the temperature. This can be explained because of the different photoinitiators, which differs from the other adhesive systems. Adper Easy One contains camphorquinone (CQ) and co-initiators like ethyl-4-

[dimethylamino]benzoate (hydrophobic) and hydrophilic photoinitiators like trimethylbenzoldiphenyl phosphine oxide (TPO). Thus the inclusion of TPO has shown to have a higher reactivity and improving the overall DC.[8] The results of our study are in accordance with the studies conducted by Cadenaro M *et al*[8] and Vale *et al*[7] which showed that presence of TPO in the adhesive system resulted in a higher DC.

XP Bond showed lowest value of DC. XP Bond has tert-butyl alcohol (2-methyl-2-propanol) as a solvent, which consists of C4 body with an alcohol group surrounded by three methyl groups. This makes it totally miscible with water and polymerizable resins. Although ethanol and tert-butyl alcohol have similar vapor pressures, tert-butyl alcohol presents better stability towards chemical reaction with monomers, hence presenting better DC at higher temperatures. The presence of water softens the polymer by swelling the network and reducing the frictional forces between the polymer chains. After the relaxation process, the unreacted monomers trapped in the polymer network are released at a rate that is controlled by the swelling and relaxation capacities of the polymer. Thus, a high amount of permeability will facilitate fluid transport in and out of the network leading to enhanced water uptake and elution. These affirmations may explain why XP Bond presented lowest DC, regardless the temperature.[7] to improve the degree of conversion of XP Bond, the use of prolonged evaporation times (60 seconds), with or without air steam, has been observed to be effective.[4]

The method used in this study to warm the adhesive systems may not be feasible clinically. However, single dose adhesive systems can be warmed in a few minutes by means of a device used for resin-based composite application that is, Calset Compule Heater (AdDent Inc, Danbury, CT, USA).

The use of preheated ethanol/water based adhesive systems seems to facilitate solvent evaporation, which translates to improving the DC. However further studies evaluation the impact of preheating other etch-and-rinse and self –etching systems on the DC are necessary.

Limitations: The in-vitro study cannot stimulate the clinical conditions like difference in the substructure of dentin, moisture control, c- factor and polymerization shrinkage due to composite material used which ascertain the success of the restoration in the oral cavity. Long term evaluations are needed to assess the influence of these properties on the bond strength.

CONCLUSION:

Preheating the single bottle adhesive systems to 60^0C improved the DC of all the adhesive systems used. Thus the hypothesis stating that preheating would not affect the DC of adhesive system was rejected. Preheating enhanced molecular mobility as the temperature increased resulting in more highly cross linked polymer network with improved mechanical and physical properties

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