Influence of Surface Inclination on the Spreading Velocity of Simplified Adhesive Systems

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ABSTRACT

Aim: Adhesion can be influenced by adhesives spreading. Both slow and fast spreading can be deleterious as they can respectively lead adhesive to partially cover the demineralized substrate or to accumulate on line angles of cavity preparations. Since brands of dentin bonding systems present distinct compositions and thus different behaviors its important to know how fast they spread over dental substrates. The purpose is to determine the influence of surface inclinations on the spreading velocity of simplified adhesive systems.

Materials and methods: Spreading velocities of adhesive systems (Adper Single Bond, Adper Single Bond Plus, Adper Prompt, Prime and Bond 2.1, Prime and Bond NT, One-Up Bond F) were measured on glass slide surfaces inclined at 45° and 90°. The spreading of each drop was observed at a 30 seconds interval. Data was recorded and the values obtained at 30 seconds were analyzed by two-way ANOVA and Student-Newman-Keuls (SNK) tests ($\alpha = 0.05$).

Results: The type of adhesive system and the angle of inclination influenced spreading velocity (p < 0.05). Adper Single Bond Plus and One-Up Bond F exhibited the lowest spreading velocities of all materials tested (p < 0.01). Adhesives spreading were similar on surfaces inclined 45° and 90°, except for Adper Single Bond Plus which spread faster and Prime and Bond NT that spread slower on 90° angled surfaces (p < 0.01).

Conclusion: The materials tested showed complex spreading patterns since the spreading velocities changed only when some specific material/inclination combinations where tested.

Keywords: Dentin-bonding agents, Wetting agents, Surfaceactive agents, Viscosity, Velocity measurement.

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INTRODUCTION

Adhesion is primarily understood as the intimate contact established between a liquid adhesive and a solid adherent.¹ Some factors influence the wetting of a solid by a liquid, i.e. the surface free energy of the solid and surface tension of the liquid, the adherent surface topography and the viscosity of the liquid.¹

Another factor has been considered as a possible influence on the wetting process. Some authors² theoretically studied the effect of gravity on the solid-liquid interface and have shown that the tension interface is enhanced by

gravity when gas is adsorbed. This observation means that the apparent contact angle on rough surfaces can be changed by gravity. Thus, when the gravitational effect is present, it may affect the adhesion and bond strength,³ since it is reported that a low contact angle⁴ and a high contact time⁵ are both necessary to achieve good adhesion.

Generally, laboratorial studies utilize flat specimens that are prepared for adhesive procedures in which specimens are held in a perpendicular position. While using the average clinical adhesive application time (30 seconds) suggested by most manufacturers, it is thought that, when the gravitational effect is present, adhesive can flow along cavity walls before light-curing, which could affect adhesive thickness along these walls, influencing solvent evaporation⁶ and shrinkage stress relief.^{7,8} Since it is impossible to carry out a clinical procedure without the gravitational effect, it is interesting to observe the adhesive spreading process on surfaces with different inclination angles as it is found in clinical trials.

Only few studies have reported the adhesion mechanism between a solid and a liquid using an adhesive system as a wetting agent.^{1,9,10} Most studies use water, which provides a lower contact angle, and thus more efficiently spreads and wets the substrate⁹ when compared with a resin agent. Because bonding systems are the agents for adhesive restorative procedures rather than water, it is important to study their spreading behavior (flowability) on an inclined substrate. Notwithstanding, the spreading of an adhesive system cannot be directly correlated to its wettability on the substrate, its spreading time can be used to presume adhesive impregnation and adhesive thickness along an inclined cavity wall.

Based on the unknown spreading (flowability) of adhesive systems over a solid surface at different angles of inclination, the aim of this current study was to test the null hypothesis that there is no difference on the spreading of six commercial adhesive systems regardless of the surface inclination (45° or 90°).

MATERIALS AND METHODS

Six adhesive systems (Table 1) were applied on 45° or 90° inclined glass slide surfaces ($25 \times 75 \times 1 \text{ mm}$) (Corning, Monterrey, Mexico) to simulate some possible clinical

inclinations of cavity walls during restorative procedures, and their spreading was measured.

The test was performed using a protractor and a base to support the glass slides. A metallic millimeter ruler was placed on the back of each glass slide to allow the spreading of adhesive systems to be measured (Fig. 1).

Ten microliter adhesive drops were placed onto the upper half of the glass slide with a micropipette (Pipetman, Gilson Medical Electronics SA, Villiers Le Bel, France). The spreading of each drop was observed at a 30-second interval

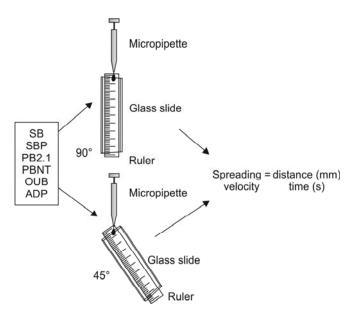


Fig. 1: Scheme showing experimental conditions used

and data was recorded as millimeters per second (mm/s). For each adhesive system, the spreading was measured five times on the same glass slide. The glass slide was changed for each adhesive system tested. Before measurements, the glass slides were cleaned with household detergent to remove any contaminants, rubbed with acetone-soaked gauze and dried with compressed air. Groups and experimental conditions are summarized in Figure 1. None of the adhesives were light-cured. The experiments were carried out at both room temperature $(25 \pm 2^{\circ}C)$ and relative humidity ($50 \pm 5\%$). The spreading velocity data obtained from the 30-second time interval were plotted for the different adhesive systems and surface inclinations. Data were analyzed by a two-way ANOVA test, where the tested variables were the adhesive and the glass slide inclination. Multiple post hoc comparisons between pairs of means were performed using the Student-Newman-Keuls test (SNK). For all analysis, statistical significance was stated at $\alpha = 0.05$.

RESULTS

The means of the spreading velocities for the experimental conditions and respective standard deviations for the 30-second period are shown in Table 2. Two-way ANOVA indicated a statistically significant influence of the adhesive variable (p < 0.001). Post hoc tests showed that, although SBP and OUB spread similarly (p > 0.05); they exhibited the lowest spreading velocities (p < 0.01) among all of the

Table 1: Adhesi	ve systems studied with their respective compositions and batch numbers	
Adhesive system	Composition	Batch number
Adper single bong (SB) (3M ESPE Dental Products, St Paul, MN, USA)	BISGMA, HEMA, Polyalkenoic acid copolymer, dimethacrylates, camphorquinone, ethanol (30-40%), water (2-8%)	1FH
Adpter Single Bond Plus (SBP) (3M ESPE Dental Products, St Paul, MN, USA)	BISGMA, HEMA, polyalkenoic acid copolymer, dimethacrylates, silane treated fillers (Nanofillers-10-20%), camphorquinone, ethanol (25-35%), water (<5%)	005AA
Prime and Bone 2.1 (PB 2.1) (Dentsply industria e Comércio Ltda, Petropolis RJ, Brazil)	Elastomeric BISGMA-diisocyanate adduct, UEDMA, BISDMA, PENTA, photoinitiators, stabilizers, cetylamine hydrofluoride, acetone (60-85%)	9875
Prime and Bone NT (PBNT) (Dentsply industria e) Comércio Ltda, Petropolis RJ, Brazil)	Polymerizable di and trimethacrylate resing, UDMA, PENTA, nonofillers (amorphous silicon dioxide with a particle size of about 7 nm), photoinitiators, stabilizers, cetylamine hydrofluoride, acetone (<70%)	030600019 7
One Up Bond F (OUB) (Tokuyama Corp., Tokyo, Japan)	MMA, HEMA, coumarin dye, methacryloyloxyalkyl acid phosphate, methacryloxyundecane dicarboxylic acid (MAC-10), multifunctional metacrylic monomer, 16% of fluoramine silicate glass filler (inside liquid B), photoiniciator (acrylborate catalyst), water (10%-inside liquid B)	455661C
Adper Prompt (ADP) (3M ESPE Dental Products, St Paul, MN, USA)	Part A: Di-HEMA phosphate, BISGMA, Ethyl 4-Dimethyl Aminobenzoate, camphorquinone Part B: water (70-80%), HEMA	133511

Manufacturers' information

adhesives tested. Prime Bond 2.1 (PB2.1) spread faster than all materials (p < 0.01), except when compared to Prime and Bond NT (PBNT) (p = 0.103). SB presented spreading means higher than those of SBP and OUB but lower than those of ADP, PBNT and PB2.1 (p < 0.01). ADP spread faster than SBP, OUB and SB (p < 0.05) but similarly to PBNT (p = 0.158). These results are shown on Graph 1.

The glass slide inclination did not affected spreading velocity (p = 0.843), but there was a significant influence of the interaction between adhesive and inclination on it (p < 0.001). The SNK post hoc test showed that SBP applied on a 45° inclined surface showed the slowest spreading velocity (p < 0.001). PB2.1 and PBNT applied respectively on 90° and 45° inclined surfaces spread faster than all materials (p < 0.01), except they are compared with PB2.1 and ADP at a 45° inclination (p > 0.05).

The spreading velocity of SBP increased when the inclination was changed from 45° to 90°. Conversely, PBNT had its spreading velocity decreased with the change in surface inclination (p < 0.01). The other materials did not show significant differences in spreading velocities when surface inclination was altered from 45° to 90° (p > 0.05). Graph 2 graphically represents the results of spreading velocity found in this current study at the 30-second time interval for all adhesives applied on both 45° and 90° inclined surfaces.

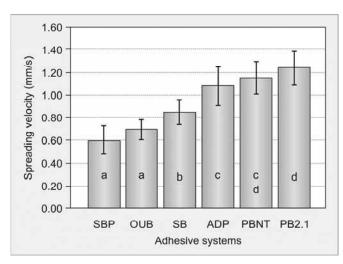
DISCUSSION

When an adhesive system is applied on a cavity preparation, it should cover all surfaces, infiltrating into the microporosities created by acid etching. Moreover, the material should be able to spread uniformly over enamel and dentin, creating a film of uniform thickness and providing the formation of a homogeneous hybrid layer and reliable bonding. However, some authors have suggested that, in class V cavities, a low viscosity adhesive could spread too fast on the cavity walls, leading to very low contact times and yielding low bond strength values.³ Thus, since an adequate contact angle⁴ and proper contact time⁵ between adhesive and adherent is necessary for promoting good etching and priming for a substrate, adhesive spreading may affect the adhesive layer homogeneity and adhesion quality, especially when inclined cavity walls are considered.

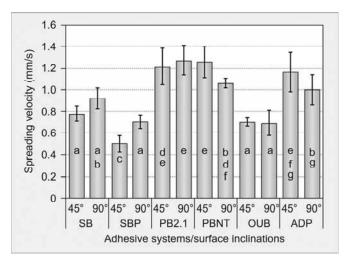
It is known that the type of substrate¹⁰ and the adhesive composition¹¹ influence its wettability. In this present study, the rationale behind the use of glass slide surfaces as the test substrate, instead of dentin or enamel, was to control the substrate variations and better understand the spreading mechanism of each adhesive system without the variations of the dental surfaces.

Spreading is strongly related to viscosity. Viscosity is the capacity of a liquid to resist spreading.¹² With regard to

inclined surfaces (n = 5). The values are representative of the 30s time interval Bonding systems									
Surface inclination	SB	SBP	PB2.1	PBNT	OUB	ADP			
45° 90°	0.78 (0.06) 0.92 (0.09)	0.50 (0.07) 0.70 (0.06)	1.21 (0.16) 1.27 (0.13)	1.25 (0.14) 1.05 (0.04)	0.70 (0.03) 0.69 (0.11)	1.16 (0.18) 1.00 (0.13)			



Graph 1: Mean values and standard deviations of spreading velocity for the adhesive systems studied at 30 seconds. Different letters inside the boxes indicate statistically significant differences at $\alpha = 0.05$



Graph 2: Spreading velocity means and standard deviations for the adhesive systems tested and the two surface inclinations evaluated at the 30 seconds interval. Different letters inside the boxes indicate statistically significant differences at $\alpha = 0.05$

dentin bonding systems, many factors can influence their viscosity, thus affecting adhesion.^{1,10,12-14} The more viscous an adhesive system, the more difficulty it has of wetting a substrate.^{12,15} Monomers with longer chains, consequently with high molecular weights, are usually more viscous than others who posses shorter chains and low molecular weights.¹⁶ In this current study, SBP and OUB resulted in the lowest spreading velocities of all the adhesive systems (Graph 1). This result can be explained by the composition of these materials. SBP is a bis-GMA based adhesive. Bis-GMA is a very viscous substance because it has a high molecular weight, a large rigid section, and it is capable of hydrogen bonding to its neighbors due to the presence of the hydroxyl group and carbonyl oxygen.^{12,16} Moreover, SBP is a nanofilled adhesive, containing approximately 10 to 20% silica fillers (see Table 1). Nanofillers are added to adhesive systems to increase their viscosity.^{17,18} However, the nanometer-sized silica can be only added to adhesives in small concentrations due to their unfavorable surface area to weight ratio.¹⁹ OUB is also a filled adhesive, with approximately 16% microfillers in its composition (see Table 1) which probably contributed its lower spreading velocity registered in this present research.

As opposed to SBP and OUB, PB2.1 and PBNT presented the highest spreading velocities means (see Graph 1). Despite the presence of fillers in PBNT (see Table 1), the type of solvent (acetone) may have facilitated the spreading. It has been shown that adhesive systems that contain higher vapor pressure solvents, like PB2.1 and PBNT, spread faster than others.²⁰ Acetone is able to diminish the surface tension and the density of the adhesives that contain it.²⁰ Also, the amount of solvent in the PB2.1 and PBNT compositions (<70% for PBNT and 60 to 85% for PB2.1; see Table 1) probably lowered their viscosity.

Another factor which can influence the spreading capacity of a liquid is the inclination of the surface where it will be applied. The flow of a liquid down an inclined surface is a complicated phenomenon that involves a threedimensional distortion of a free surface and movement of both advancing and receding contact lines; it is strongly related to the volume and the inclination of the surface.²¹ Less than 90° inclined surfaces (considering the ground plane) will minimize the gravitational effect. A gravitational force acting on the liquid will be attenuated in this situation, since the gravitational force will be reduced, with a normal component of the force acting on the Y-axis perpendicular to the surface.²² On the other hand, on 90° inclined surfaces, the weight of the liquid is the force that will induce it to spread (Normal = 0; acceleration = gravity), because the liquid will be in a free fall, if both the surface and air

frictional forces are not considered.²² Thus, theoretically, a liquid in standardized conditions of temperature and pressure trends to spread faster on 90° inclined surfaces rather than on 45° ones. For SBP, this really happened, since this material presented higher spreading velocities on 90° surfaces. Contrary to this trend, SB, OUB, PB2.1 and ADP presented similar spreading velocities on both inclinations (see Graph 2). It is likely that the solvent evaporation compensated for the surface inclination effect. It is expected that the spreading velocity of the adhesive would be actually higher on 90° inclined surfaces than on 45° ones, at least in the first seconds of the spreading process. This faster spreading could also cause a faster solvent evaporation, enhancing the viscosity of the adhesive and diminishing the spreading velocity after completion of the 30-second period. Within the limitations of the current study, the spreading velocities of the adhesives at times below and above 30 seconds were measured. Future studies should be done to confirm or deny these statements. For SBP, it is hypothesized that it behaved differently from SB, OUB, PB2.1 and ADP because SBP has 10 to 20% fillers in its composition, which probably led to an increment of the adhesive mass in comparison to the other materials. It is known that the mass of a body is directly proportional to its weight,^{21,22} which is, as stated above, the force acting on liquids sliding down 90° inclined surfaces. Therefore, considering that SBP has a greater mass than the other materials, it could spread faster on 90° surfaces than over 45° inclined ones. This hypothesis should also be investigated further.

PBNT exhibited a very peculiar behavior. This material presented a lower spreading velocity on the 90° inclined surface than on the 45° surface (see Graph 2). PBNT has acetone as a solvent, which has a higher vapor pressure as well a lower boiling point than water, ethanol or the mixture of both.^{16,19} As the force of gravity acts in its full acceleration on these 90° angled surfaces, the adhesive solvent most likely evaporated faster (this material probably forms a very thin layer, because acetone is almost 70% of its composition). As stated before, fillers are capable of making the adhesive more viscous, so, in this case, after the solvent had been evaporated, the filler loading effect made the adhesive so viscous that the gravity force was not high enough to surpass the internal forces of the liquid plus the air/surface attrite, so this material spread slower despite the gravity effect. In the case of PB2.1, which has also acetone as the main solvent but spread equally on both inclinations, it is speculated that although the acetone had evaporated, thus elevating adhesive viscosity, this material does not have fillers in its composition; therefore, the gain in viscosity was not enough to change the adhesive's spreading on

90° inclined surfaces. These raised hypotheses deserve further investigation.

This study showed that the spreading velocity of the adhesive systems tested is material dependent and that modification of the surface inclination alone is not capable of altering this property. Despite the different performances of each adhesive system, the results of this study may predict that, if some adhesives are applied onto an inclined cavity wall, they may spread rapidly enough and possibly accumulate at line angles of the cavity, even with a short clinical time of 30 seconds that recommended by most of the manufacturers. Also, the way that an adhesive spreads may yield different thickness of an adhesive layer along the cavity wall, impairing the evaporation of the solvent before light curing⁶ and influencing the stress relief during resin composite shrinkage.^{7,8} Moreover, if a thick layer of adhesive accumulates in the outline of the cavity, it may facilitate adhesive plasticizing due to water sorption and may influence failure of the adhesive interface.^{23,24} To overcome all these problems, it seems reasonable that these materials would be applied on cavity walls by gentle brushing to permit an adequate spreading, especially when water/ethanol-based filled adhesives are used, like SBP. Otherwise, solvent rich, especially acetone-based adhesives, such as PB2.1 and PBNT should be applied at least in two layers over the surface, to create a film with an adequate thickness to permit saturation of the demineralized zone with resin monomers.²⁵ In fact, some researchers have found higher bond strength means when PBNT was applied in two layers.²⁶ Several authors have shown improved bond strength using this protocol, mainly when all-in-one adhesives are employed.^{6,25,27}

Although this current work did not study the entire adhesive process, it gives some knowledge about adhesive spreading on inclined surfaces and it presumes how this spreading could occur clinically in a cavity preparation. The commercially available adhesive systems do not have equal compositions, thus they provide different adhesion mechanisms. Within the limitations of this study, the null hypothesis must be rejected, since surface inclinations affected the spreading velocities of the adhesives. The null hypothesis was rejected. The surface inclinations (45° and 90°) influenced spreading velocities for SBP and PBNT. The influence of surface inclinations on the spreading velocity was material-dependent. Knowing how different adhesives spread along inclined surfaces, professionals can minimize possible adhesive failures, such as partial infiltration on the demineralized substrate or adhesive accumulation on line angles of cavity preparations.

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