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Effect of Refrigeration on Tensile Bond Strength of Three Adhesive Systems

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The purpose of this study was to investigate the tensile bond strengths of three adhesive systems applied to dentin at refrigerated and room temperatures. Ninety bovine incisor teeth were obtained, embedded in self-cured acrylic resin, abraded on a lathe under water spray and polished to 400 and 600 grit to form standardized dentin surfaces before randomly assigning to six groups (n=15). The adhesive systems Scotchbond Multi-Purpose, Single Bond and Prime & Bond NT were applied to dentin according to the manufacturers' instructions at refrigerated temperature (4ºC) and at room temperature (23ºC), before bonding resin-based composite (Z 100). The specimens were stored in distilled water at 37ºC for 24 hours and submitted to tensile bond strength tests on a universal testing machine (EMIC DL-2000) at a crosshead speed of 0.5 mm/min. The resulting data were statistically analyzed using analysis of variance and Tukey's test. No statistical differences were found when the adhesive systems were applied at refrigerated and room temperatures. Scotchbond Multi-Purpose and Single Bond had significantly stronger tensile bond strengths than Prime & Bond NT at room and refrigerated temperatures ($p<0.01$). Scotchbond Multi-Purpose and Single Bond were statistically similar. No adverse effects upon tensile bond strength were observed when adhesive systems were taken directly from refrigerated storage.

Key Words: adhesive systems, bond strength, refrigeration, room temperature.

INTRODUCTION

With the introduction of acidic conditioning by Buonocore (1) and composite resin development by Bowen (2), restorative dentistry has advanced significantly. Dental restorative bonding to enamel has become a safe and reliable technique, but bonding to dentin has proven to be more difficult and less predictable. The histologic differences existing between the two tissues make bonding to dentin more complex than bonding to enamel, because the former has higher protein and water concentrations, making it a more humid substrate and much less receptive to the adhesive system (3). Thus, many generations of adhesive systems have been elaborated including the development of the hydrophilic type system

Current methods for dentin bonding use acid conditioners to remove the smear layer before using the superficial dentin as a substrate. The superficial dentin is decalcified and the remaining dentin surface is primarily collagen in nature (4). A primer containing a resin monomer (e.g. HEMA) dissolved in acetone, water, ethanol, or a combination of these solvents, is then applied to the surface. This process is essential for the establishment of a suitable surface to allow adequate wetting and penetration of the resin bonding agents, resulting in hybrid layer formation and a micromechanical bond to the dentin surface (5,6).

Good adhesion is important for the production of well-sealed, long-lasting restorations and many factors such as temperature and relative humidity can affect this adhesion (7). Although most manufacturers

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recommend storage of adhesive materials at room temperature, these materials are usually refrigerated to extend shelf life and, in practice, most dentists take the materials from the refrigerator and use immediately without allowing time for equilibration to room temperature. It is possible that this reduced temperature may have deleterious effects on the efficacy of the bonding agent (8).

Upon refrigeration, the viscosity of the polymeric material increases and penetration of the adhesive into the dentin surface may be reduced, causing decreased bond strength. Refrigeration may influence the evaporation of primer solvents, reducing bond strength and incomplete evaporation of solvents has been demonstrated to adversely affect the sealing ability of adhesive systems (9). In addition, reduced temperatures influence properties related to curing efficiency, such as microhardness and diametral tensile strength, due to the decrease in polymerization (10)

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MATERIAL AND METHODS

Ninety bovine incisor teeth were cleaned of gross debris and stored in distilled water at 4ºC and utilized within a period of 6 months. The root and some parts of the crown were cut-off from each tooth with a double-faced diamond disk (KG Sorensen, São Paulo, SP, Brazil) under water spray to obtain a 5x5-mm piece from the central area of the facial crown. Each tooth section was fixed at the facial surface onto a glass plaque with wax. A metallic cylindrical device was placed on the glass plaque so that the dental surface could be centralized. Self-cured acrylic resin (Jet Classico, São Paulo, SP, Brazil) was then used to fill the metallic cylindrical device. Two mm of acrylic resin and tooth were removed together to expose flat dentin using a lathe (Nardini-ND 250 BE, São Paulo, SP, Brazil) under water spray. Dentin surfaces were ground and polished using 400 and 600 grain sandpaper (Carborundum Abrasivos, Recife, PE, Brazil) on an automated polisher APL-4 (Arotec Ind. Com Ltda, São Paulo, SP, Brazil) to obtain standardized dentin surfaces.

After polishing, the embedded teeth were randomly assigned to 6 groups (n=15) for bonding with three currently available adhesive systems at refrigerated temperature (4ºC) and at room temperature (23ºC). Before the surface treatment, the bonding area was demarcated by placing a piece of vinyl tape (3M Dental Products Division, St. Paul, MN, USA) in the center of the specimen with a 3 mm diameter hole. The adhesive systems Scotchbond Multi-Purpose (3M Dental Products Division, St. Paul, MN, USA), Single Bond (3M Dental Products Division) and Prime & Bond NT (Caulk/Dentsply, York, PA, USA) were then applied following the manufacturers' instructions.

Scotchbond Multi-Purpose: The dentin was treated for 15 s with 37% phosphoric acid and rinsed for 10 s under running tap water. Excess water was removed with a cotton bud, leaving a moist surface. Primer was applied using a saturated brush tip and was dried gently with compressed air for 5 s. The adhesive was applied using a brush tip and the material was cured for 10 s using an XL 1500 curing unit (3M Dental Products Division) at 600 mW/cm^2.

Single Bond: The dentin was treated for 15 s with 35% phosphoric acid and rinsed for 10 s under running tap water. Excess water was removed with a cotton bud, leaving a moist surface. Two consecutive coats of adhesive were applied using a saturated brush tip. After gently air drying for 5 s, the material was cured for 10 s using the XL 1500 curing unit at 600 mW/cm^2.

Prime & Bond NT: 35% phosphoric acid was applied to the dentin surface for 15 s, rinsed for 10 s under running tap water and excess water removed with a cotton bud, leaving a moist surface. Two consecutive coats of adhesive were applied using a saturated brush tip. After gentle air drying for 5 s, the material was cured for 10 s using the XL 1500 curing unit at 600 mW/cm^2.

A 4-mm high metallic cylindrical device, with an orifice of 3 mm in diameter at the base and 5 mm diameter at the top, was placed against the specimen so that the orifice superimposed the treated dentin. Composite resin Z 100 (3M Co.) for Scotchbond Multi-Purpose and Single Bond groups, and composite resin TPH, shade A1, (Caulk/Dentsply) for Prime & Bond NT groups, was inserted inside the orifice in two increments to form an inverted cone of composite resin which provided a grip for the hook used in the tensile bond test. Each increment was cured for 40 s using the XL 1500 curing unit at 600 mW/cm^2.

The specimens were stored in distilled water at

37ºC for 24 h and submitted to tensile bond strength testing with a universal testing machine (EMIC DL-2000, São José dos Pinhais, PR, Brazil) at a crosshead speed of 0.5 mm/min until failure occurred. Tensile bond strength values in MPa were calculated from the peak load at failure divided by the specimen surface area. The results were submitted to analysis of variance and Tukey's test at the 1% significance level.

After the tensile bond strength tests, the fractured surfaces of the specimens were visually examined with a stereomicroscope (Olympus Corp., Tokyo, Japan) at X20 to classify the type of failure that occurred during the debonding procedure. Failures were classified as one of the following: adhesive, if the composite resin cone fractured at the adhesive-tooth interface; cohesive in dentin, if the composite resin cone fractured with a large portion of dentin attached; cohesive in composite resin, if the composite resin cone fractured inside the composite resin; or mixed, a combination of adhesive and cohesive in dentin or cohesive in composite resin.

RESULTS

Mean tensile bond strengths are shown in Table 1. The Scotchbond Multi-Purpose and Single Bond systems demonstrated significantly stronger tensile bond strengths than Prime & Bond NT at both room and refrigerated temperatures ($p<0.01$). No statistical difference was found between the Scotchbond Multi-Purpose and Single Bond. No significant differences were found between the room and refrigerated temperature groups. A trend for improvement was noted with Scotchbond Multi-Purpose and Single Bond at refrigerated temperatures, but the greater strengths obtained were statistically insignificant.

The examination of the debonded specimens under a stereomicroscope at X20 magnification demonstrated that the majority of failures were adhesive in all three adhesive systems both at room and cold temperatures (Table 2). Of the mixed failures, adhesive and cohesive in dentin failures were more common than the adhesive and cohesive in composite resin failures. No cases of cohesive failure in dentin alone or in composite resin alone were observed.

DISCUSSION

This study used bovine teeth because this substitute substrate has been previously shown to provide a similar bond strength to that of human dentin (11). In addition, these studies use a large number of teeth and the current supply of human molars for testing is now quite limited.

Because several previous studies have reported the influence of temperature and relative humidity on early bond strength to dentin (7,12), the present study investigated the effect of refrigeration of adhesive systems on bond strength.

The adhesive systems tested are widely avail-

Table 1. Mean tensile bond strengths (MPa) of 3 adhesive systems to dentin at room and refrigerated temperatures.

Material	Room temperature	Refrigerated temperature
Scotchbond Multi-Purpose	18.06 ± 4.60 a,A	18.95 ± 4.52 a,A
Single Bond	19.37 ± 3.69 a,A	21.13 ± 4.97 a,A
Prime & Bond NT	10.49 ± 2.44 b,A	10.39 ± 2.48 b,A

Means followed by the same small letter in the column and capital letter in the line indicate no statistical difference at the 99% confidence level (Tukey test, $p<0.01$).

Table 2. Failure mode analysis of the debonded specimens.

	Adhesive	Mixed (adhesive/cohesive in compositive resin)	Mixed (adhesive/cohesive in dentin)
Scotchbond Multi-Purpose			
room temperature	14	0	1
refrigerated temperature	12	0	3
Single Bond			
room temperature	12	1	2
refrigerated temperature	10	1	4
Prime & Bond NT			
room temperature	13	0	2
refrigerated temperature	11	1	3

able. Scotchbond Multi-Purpose is a conventional adhesive system that includes conditioner, primer and adhesive (multiple-bottle system) and requires three or more application steps. Single Bond and Prime & Bond NT are simplified adhesive systems that also include conditioner and primer and adhesive combined in a single bottle (one-bottle system). In the present study Scotchbond Multi-Purpose and Single Bond had significantly stronger tensile bond strengths than Prime & Bond NT at room and refrigerated temperatures ($p<0.01$).

Conditioning of the dentin surface with phosphoric acid at a concentration of 37% (Scotchbond Multi-Purpose) or 35% (Single Bond and Prime & Bond NT) removes the smear layer and smear plugs and opens the dentinal tubules, in addition to demineralizing the intertubular and peritubular dentin (13). These products contain hydrophilic components in the resin (HEMA dissolved in acetone, water, ethanol or a combination of these solvents) and with the introduction of these hydrophilic components, desiccation of the dentin surface after acid conditioning is clearly no longer indicated. For this reason, most manufacturers caution against excessive drying of dentin surfaces. Tay et al. (14) demonstrated the importance of the presence of humidity to avoid matrix collapse of demineralized collagen, favoring infiltration of the adhesive into the intertubular and peritubular dentin to proportion hybrid layer formation and resin tags. In our study, we used a small cotton pellet to remove excess water after the removal of acid by washing, to improve bond strengths (15).

In addition to dentin humidity, the type of solvent used in the primer has been reported to be important for bond strength (16). Scotchbond Multi-Purpose contains only water in its primer, while Single Bond has a combination of water and ethanol and Prime & Bond NT has acetone. The evaporation of these solvents after application to dentin is extremely important (9). The results of the current study indicate that incomplete evaporation of solvents probably did not occur with refrigeration, because no difference was detected in tensile bond strength. Another theoretical disadvantage of refrigeration, the decrease in bond strength due to the adverse effect of refrigeration upon the physical properties of the adhesive materials, seems not to have occurred, because little or no effect could be detected in the bond strength under refrigerated conditions. Under refrigeration, an increase in viscosity could be observed and the materials were slightly more difficult to remove from their vials, although this did not represent difficulty in handling.

Hagge et al. (8) investigated the shear bond strength of adhesive systems applied at refrigerated and room temperatures and found no differences between Prime & Bond and All-Bond 2, but found the shear bond strength of Scotchbond Multi-Purpose to be much greater at refrigerated temperature (13.14 MPa) than at room temperature (5.52 MPa). They did not offer a satisfactory explanation for the improved bonding ability of refrigerated Scotchbond Multi-Purpose.

Upon visual inspection, the types of failures observed in this study demonstrated that the effect of refrigeration did not cause variations in the mode of failure. Adhesive failures occurred more commonly in all adhesive systems tested, both at refrigerated and room temperatures. Mixed failures occurred in few specimens and the adhesive and cohesive in dentin type failure occurred more frequently than the adhesive and cohesive in composite resin type. Hagge et al. (8) observed that mixed failures were most frequent and that there was an improvement in this type of failure in refrigerated adhesive systems when submitted to shear bond strength. The differences in the mode of failures found between publications may be explained by the methodologies used, i.e., tensile versus shear bond strength. Van Noort et al. (17) reported that the stresses at the interface between the adhesive and the substrate are far from homogeneous and are highly dependent on the test geometry and loading configuration adopted. In the shear bond tests, there is a tendency for cohesive failures to occur in dentin due, in part, to the mechanism of the test (18). It is important to emphasize that it is difficult to compare the results obtained in different studies because a series of factors can lead to varying results. Such factors include the dentin substrate and the operator’s influence and demonstrate the need for methodology standardization so that results can be compared more easily (19,20). In the current study, a small improvement in the occurrence of mixed failures in the refrigerated adhesive systems was observed, but was not significant.

The results of this study suggest that no adverse effects occur when adhesive systems were used directly from refrigerated storage. As this study used only three adhesive systems, extrapolation of these findings can-not be made to other systems. Further investigation under similar conditions using other adhesive systems would be beneficial.

RESUMO

Spohr AM, Correr Sobrinho L, Consani S, Sinhoreti MAC, Borges GA. Efeito da refrigeração na resistência à tração de três sistemas adesivos. Braz Dent J 2001;12(2):75-79.

O objetivo deste trabalho foi avaliar a resistência à tração sobre a dentina de três sistemas adesivos quando estes se encontravam refrigerados e à temperatura ambiente. Noventa dentes bovinos foram embutidos em resina acrílica autopolimerizável, desgastados em um torno mecânico sob refrigeração à água, polidos com papel abrasivo de granulação 400 e 600 para obter uma superfície plana em dentina, e divididos aleatoriamente em seis grupos (n=15). Os sistemas adesivos Scotchbond Multi-Purpose, Single Bond e Prime & Bond NT foram aplicados sobre a dentina de acordo com as instruções dos fabricantes, estando os mesmos refrigerados (4ºC) ou à temperatura ambiente (23ºC), seguido da união da resina composta Z 100. Os corpos-de-prova foram armazenados em água destilada a 37ºC por 24 horas e submetidos ao teste de resistência à tração em máquina de ensaio universal (EMIC DL-2000) com velocidade de 0,5 mm/min. Os valores obtidos foram submetidos à análise de variância e ao teste de Tukey. Não houve diferença estatística nos valores de resistência de união quando os sistemas adesivos encontravam-se refrigerados ou à temperatura ambiente. Scotchbond Multi-Purpose e Single Bond demonstraram resistência à tração significativamente superior em relação ao Prime & Bond NT na temperatura ambiente e refrigerada ($p<0,01$). Nenhuma diferença estatística foi encontrada entre o Scotchbond Multi-Purpose e Single Bond. Nenhum efeito adverso foi observado na resistência à tração quando utilizados os sistemas adesivos imediatamente após a remoção da armazenagem sob refrigeração.

Unitermos: sistemas adesivos, resistência de união, refrigeração, temperatura ambiente.

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