The influence of absolute humidity on shear bond adhesion

P. J. J. M. Plasmans*, N. H. J. Creugers†, R. J. Hermansen† and M. M. A. Vrijhoef‡
*Department of Oral Function and Prosthetic Dentistry, TRIKON: Institute for Dental Clinical Research, University of Nijmegen, The Netherlands, †3M Medical EBC Laboratory, Borken, Germany, ‡3M Dental EBC Malakoff, France

ABSTRACT
Objectives: The purpose of this study was to investigate the relationship between dentine shear bond strength, using data from experiments performed according to a standard protocol, and the absolute humidity for a new, fourth generation dentine adhesive system.

Methods: Results of seven recently performed adhesion experiments using the same protocol and product were analysed. Groups of 10 human molars were each bonded in a humidity chamber. The temperature and relative humidity conditions varied from 23 to 37°C and 30 to 95%. After bonding of the composite resin a shear bond test at a crosshead speed of 2.0 mm/min was performed.

Results: The bond strength levels of the dentine adhesive system were influenced significantly by extreme temperature and humidity differences. Adhesion levels varied from 27.8 (SD 8.5) MPA to 12.8 (SD 1.4) MPA. Isobond strength curves are a good method to gain insight into the humidity sensitivity of adhesive systems.

Conclusions: Results of in vitro adhesive bonding procedures for a dentine adhesive can be significantly influenced by the absolute humidity levels at which the procedures are performed. Consequently “Materials and Methods” sections of publications should refer to absolute humidity or temperature and relative humidity levels as a relevant parameter. Copyright © 1996 Elsevier Science Ltd.

KEY WORDS: Adhesion, Shear bond, Humidity

INTRODUCTION
Many factors may bear relevance to dentine adhesion(1-3). The influence of dentine wetness is increasingly drawing attention(4-8). Dentine wetness is influenced by intrinsic factors such as dentine location and distance to the pulp, intrapulpal pressure and presence or absence of a smear layer. One possible important extrinsic factor is the influence of air humidity on the bond strength of commercially available dental adhesive systems. Essentially humid conditions result in lower adhesive strength for dentine bonding systems than dry conditions(9).

There are several reports that a new fourth generation dentine adhesive system shows relatively high adhesive strength in comparison with other products tested under the same conditions, when bonded to moist dentine or when bonded under relatively humid operator conditions(10-13). Although several research groups have reported results in a comparable range, some observed differences may be related to test condition variations. It has been suggested that absolute air humidity may explain these differences.

As the amount of vapour changes strongly with the temperature, a more appropriate scale to describe test conditions may be the mass of water vapour per unit of volume air (= def. absolute humidity)(14). It was the aim of this study to investigate the relationship between dentine shear bond strength, using data from experiments performed according to a standard protocol, and the absolute humidity for the new dentine adhesive system.

MATERIALS AND METHODS
For the experiments of this in vitro research, fresh, unrestored human molars were used. According to the standard protocol the teeth were stored in an aqueous 1% solution of Chloramine T (trihydrate) until prepared. The molars were embedded in self-curing epoxy resin using a cylindrical mould. In a polishing and grinding machine (Struers, Erkrath, Germany) the occlusal surface of the embedded sample was ground perpendicular to the long axis using 120-grit SiC paper under running tap water. A smear layer was then created with 320-grit SiC paper. As a result an occlusal...
dentine surface of superficial/intermediate depth was obtained. All teeth were assigned in a random manner to the respective test groups. Before bonding procedures were started (up to 1–1.5 h), the specimens were placed in the humidity chamber to equilibrate them with the conditions of the environment. Also all test specimens were rinsed with distilled water. The sample size for each experiment was n = 10. The bonding agent used in the experiments was 3M Scotchbond Multi-Purpose adhesive (two different batches), and 3M Z100 was used as resin composite (3M Dental Products Division, St Paul, MN, USA). The manufacturer's instructions for use concerning these materials were followed carefully. 3M Scotchbond Multi-Purpose adhesive system consisted of (1) etchant, (2) primer containing methacrylate groups. (3) The adhesive is a light cured BisGMA and HEMA based system. For a range of relative humidities, the absolute humidity h (in g/m²) was calculated from equation (1).

\[ h = M_{svw} \times \frac{RH}{100} \]  

(1)

RH stands for the relative humidity, while \( M_{svw} \) corresponds with the amount of saturated water vapour at the temperature concerned (in g/m³). \( M_{svw} \) is given by equation (2), which is based on data from the Handbook of Chemistry and Physics (1988–1989).

\[ M_{svw} = 0.025t^2 + 0.075t + 5.67 \quad (r = 0.999) \]  

(2)

The temperature and relative humidity (RH) conditions at which the experiments were performed are as indicated in Table I. The conditions were either ambient (Experiment no. 1 and 2) or controlled by means of an air-tight compartment that was held at preset levels of temperature and RH (Humidity chamber model 2215, Kottermann, Uetze-Häningen, Germany). Bonding procedures for Experiments 3, 4, 5, 6 and 7 were carried out in this humidity chamber. For these experiments the samples were placed inside the humidity chamber to obtain the required conditions. Access to the samples was gained through air-tight sleeves. After acid etching (15 s), rinsing (10 s) and drying (15 s) the primer was applied (15 s). After application of the bonding agent (10 s) and light-curing for 10 s this was followed by placement of the resin composite material. The resin composite material was inserted in cylindrical PTFE (Poly Tetra Fluor Ethylene or Teflon®) moulds lined with gelatine capsules (internal diameter 4.7 mm, area 17.3 mm²) and cured in one layer of 2.5 mm for 60 s using a Visilux 2 (3M Dental Products, St Paul, MN, USA).

After fabrication of the bonded samples all specimens were stored in water at 37°C for 23 h. The gelatine capsules dissolved in this period ensured easy release of the outer PTFE mould.

Before shear bond testing the samples were removed from the water and allowed to stand for 1 h to reach standard test conditions (\( T = 23^\circ C \pm 2^\circ C; \) RH = 50 ± 5%). Shear bond strength values were obtained using the wire loop test on a universal tensile tester (Houndsfield H10KM, Croydon, UK) at a crosshead speed of 2.0 mm/min.

**RESULTS**

The results of seven different experiments are shown in Table I. Stepwise multiple regression analysis showed that the temperature had no statistically significant influence on the bond strength. Therefore, only absolute humidity was taken into account. This resulted in a relationship between dentine shear bond strength \( (S_{\text{shear}}, \text{MPa}) \) and absolute humidity \( (h, \text{g/m}^2) \), represented by equation (3).

\[ S_{\text{shear}} = 28.4 - 0.398h. \]  

(3)

Linear regression analysis per definition provides a linear equation, while a fixed B and a coefficient A are calculated. If these are included in the general equation of a line \( (Y = Ax + B) \), equation (3) develops. The linear regression coefficient of this function is \( r = 0.97 \). The relationship is depicted in Fig. 1. The 95% confidence intervals have also been included in this figure. The variation in bonding experiments is dependent on many factors such as operator differences, standardization of the film thickness of the adhesive and application technique of the composite resin material. The statistical analysis shows that the variation was sufficiently controlled to obtain an acceptable coefficient of correlation. Therefore, no further attempt was made to further standardize parameters influencing the experimental variation.

Graphically, the calculations result in the iso-curves as shown in Fig. 2. These iso-bond strength curves are normalized for absolute humidity.

**Table I.** The average adhesion to dentine in shear mode as a function of the humidity and temperature. Standard deviation in parentheses.

<table>
<thead>
<tr>
<th>Exp.</th>
<th>Batch</th>
<th>Temperature</th>
<th>Relative</th>
<th>Absolute</th>
<th>Adhesion</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2AC</td>
<td>23</td>
<td>30</td>
<td>6.17</td>
<td>27.8 (8.5)</td>
</tr>
<tr>
<td>2</td>
<td>2AB</td>
<td>25</td>
<td>40</td>
<td>9.22</td>
<td>23.6 (8.5)</td>
</tr>
<tr>
<td>3</td>
<td>2AC</td>
<td>25</td>
<td>76</td>
<td>17.52</td>
<td>19.6 (5.4)</td>
</tr>
<tr>
<td>4</td>
<td>2AC</td>
<td>27</td>
<td>95</td>
<td>24.49</td>
<td>19.7 (3.3)</td>
</tr>
<tr>
<td>5</td>
<td>2AC</td>
<td>35</td>
<td>95</td>
<td>37.65</td>
<td>13.7 (3.0)</td>
</tr>
<tr>
<td>6</td>
<td>2AB</td>
<td>35</td>
<td>95</td>
<td>37.65</td>
<td>12.1 (4.3)</td>
</tr>
<tr>
<td>7</td>
<td>2AC</td>
<td>37</td>
<td>95</td>
<td>41.76</td>
<td>12.8 (1.4)</td>
</tr>
</tbody>
</table>
DISCUSSION

The relationship between dentine bond strength and absolute humidity is evident in terms of significance of its linear relationship (Fig. 1). However the cause and effect relationship remains unknown because this experiment was not designed to elucidate this. It can be speculated that the amount of water absorbed to the dentine surface increases with higher absolute humidity, which then results in a deterioration of the dentine bond quality. This may account for the variability in bond strength values obtained with in vitro testing. More research is needed to find out whether the deterioration of the bond strength is related to a changed interaction between dentine and the primer, primer and adhesive, or adhesive and composite. This new research should concentrate on varying levels of humidity and the different steps of the bonding procedure.

Climatological differences around the world are substantial. Consequently different bond strength values may be obtained in different climate zones. As a result the clinical practice and performance may be affected as well. From a clinical point of view, it is evident that the rubber dam still may give the best results for the dentine adhesive investigated. This was also suggested by Nikaido et al. after their study on the effect of an artificial oral environment with relative humidity from 30 to 90% on the adhesion of resin to dentine. In a recent study it was shown that the temperature in the mouth, in case rubber dam is not used, ranges from 26-29°C and that RH ranges from 78-94%. Further, this study shows that the relative air humidity and temperature are almost equal to ambient conditions when, for clinical procedures, rubber dam is used. However, ambient conditions may significantly influence the dentine bond strength and practical be of more importance in certain parts of the world. Generally the best results will be obtained under rubber dam; however, even without using this preferred method of isolation, acceptable bond strength values will be obtained under mild climatic conditions. An exception might be under tropical conditions without proper conditioning of the dental surgery. Figure 2 makes it clear that under normal surgery conditions, the bond strength of the dentine adhesive system used is relatively independent of the circumstances chosen. Under oral conditions, without rubber dam, the absolute humidity ranged from 19-27 g/m³. For the adhesive system investigated this implies a shear bond strength ranging from 18 to 21 MPa. Adhesion levels are dependent on absolute humidity. Air humidity, however, is measured in degrees of RH. Thus, Figs. 1 and 2 essentially provide the same information. However, to show graphically the relation between adhesion and RH in generally known conditions (temperature and RH), a contour plot is provided in Fig. 2. The four contour lines show the bond strength at fixed absolute humidity levels and varying temperatures. In this way the corresponding RH can be deducted.

In selecting the test method, shear tests would seem to have an advantage over tensile tests, in that they appear to be more likely to produce failure at the tooth-composite interface. As all seven experiments were carried out with the same wire loop method it is possible to compare these results, although the wire loop test produces somewhat higher figures as, for example, in blunt knife or sharp knife methods. It should be stressed here that the results of this study are not representative for commercial products in general. The product chosen for investigation is an exception, in that high air-humidity has been shown to result in much lower bond strength for the majority of the commercial dentine adhesives. In this way the material is used as a control for itself.

In a survey of "Material and Method" sections of bond strength articles in five international dental jour-
nals (Amer Dent J, Dent Mat, Dtsch Zahnart. Z; J Prosth Dent and Quint Int published in 1992), 27 papers were screened. In these publications no information was given as to temperature, or relative or absolute humidity levels at which the bonding procedures were performed. Apparently, bond strength values are reported without any reference to differences in absolute air humidity (or temperature and RH). This underlines the urgent plea of Stanley for a standardized bonding (adhesion) test (17). Actual differences may account for different test results for apparent comparable testing conditions. In some cases it might explain differences between clinical results.

CONCLUSIONS

Results of in vitro adhesive bonding procedures for a fourth generation dentine adhesive following a well defined protocol are highly influenced by the absolute humidity levels at which the procedures are performed. These levels are partly dictated by climatological conditions. Consequently, it appears that either absolute humidity or temperature and RH levels should be referred to as relevant test parameters in the “Materials and Methods” sections of publications. This need for specification of test parameters is in sharp contrast with the current policy not to mention these at all. An evaluation at both low and high absolute humidity levels is recommended. This work concerned one selected dentin adhesive system. More work should elucidate the situation with regard to other products available. In this respect iso-bond strength curves are a good method for gaining insight on the humidity sensitivity of adhesive systems.

References