بسم الله الرحمن الرحيم

﴿ قَالُوا سُبْحَانَكَ لاَ عِلْمَ لَنَا إِلاَّ مَا عَلَّمْتَنَا إِنَّكَ أَنتَ الْعَلِيمُ الْحَكِيمُ ﴾

صدق الله العظيم

سورة البقرة - الآية (٣٢)

Effect of Temperature of Single-step Self-etch Adhesives on Bonding to Tooth Substrates

Thesis

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By

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<u>Dedication</u>

To my precious parents, my beloved husband and to my sweet daughter

For their endless love, patience, care, and support

*First and foremost, I would like to thank "**ALLAH**" for his great blessings; without which I would never have accomplished this work.*

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This research was designed to study the effect of temperature whether refrigerated or ambient of three self-etch adhesive systems on their micro-shear bond strength to tooth substrates (enamel and dentin).

This study was designed to test the effect of temperature of the single-step self-etch adhesive systems on their micro-shear bond strength to enamel and dentin. Three types of single-step self-etch adhesive systems were selected for this study. Despite they are supplied by three different manufacturers, the three adhesives have the same storage recommendations, which suggested the refrigeration of the adhesive system to ensure prolonged shelf life.

All adhesives were selected within the same pH range, categorized as mild self-etch adhesives. However, they differed in solvent content; iBond is acetone/water based, while Bond Force and Adper Easy One are alcohol/water based. The modulus of elasticity of resin composite has been shown to possibly influence the results of bond strength measurements ***(Atash and Van den Abbeele, 2005)***. For this reason, one resin composite (Tetric Evo-Ceram) was used to minimize the possible influence of the filling material upon the bond strength of the adhesive.

In this study, every effort was exerted to standardize the methodology and to mimic the clinical conditions as closely as possible. In an attempt to standardize enamel and dentin substrates used in this study, only freshly extracted, non-defective upper central incisors were used. Central incisors were selected to obtain sufficient enamel width incisally as well as the dentin cervically. Phosphate buffer solution containing sodium azide was used for storage of the freshly extracted teeth at 4ºC. Sodium azide is a known anti-bacterial that inhibits the growth of the non-oral flora ***(Kitasako et al., 2000)***. This storage medium does not influence the chemical and physical properties of human tooth substrate ***(Özok et al., 2002)***.

In order to assure that even amount of labial enamel was ground, the incisal half of the crown was supported to be in one level with the cervical half during embedding of teeth. This was done by fixing the tooth with a piece of pink wax to the center of the drawn ring on the glass slab. In addition, to standardize the amount of labial reduction, a PolyVinylChloride ring of 1mm height was placed on the glass slab coinciding with the drawn circle before embedding; the ring was then removed protruding the set embedding material by 1mm above the mould margins. Upon grinding, only this 1mm was removed, thus exposing evenly ground superficial cervical dentin and incisal enamel.

As the adhesive's volume may influence the temperature of the adhesive, it had to be equalized within the bottles of the three utilized single-step self-etch adhesives. This was done by aspiration of liquid adhesive with a graduated insulin syringe inserted through the bottle cap. This ensured that the adhesive bottle was sealed during the whole procedure to prevent solvent evaporation; since any loss of volume induced by solvent evaporation will alter the adhesive's proportions, which will affect the final results of the adhesive ***(Perdigão et al., 1991)***.

The refrigerated temperature used in this study was 5ºC as suggested by ***Alexandre et al., in 2008*** and ***Reis et al., in 2009***. Many authors suggested 4ºC as the refrigerated adhesive temperature, however, 5ºC is more realistic since the refrigerator temperature is usually adjusted at 4ºC±0.5 and even if the adhesive was applied immediately after removal from the refrigerator, the time required to apply the adhesive will cause a slight increase in temperature. The time needed for every utilized adhesive system to reach 5ºC±1, and then to reach the ambient temperature were determined via a digital logger. The Digital

Temperature Logger used in this study has a high degree of accuracy (±1.1ºC). It was thus selected for accurate determination of adhesive systems' temperature as well as the oro-dental temperature. Also, it has two K-type thermocouples which enabled to represent the time dependant refrigerated adhesive temperature changes at the same time with that of the ambient temperature changes.

Previous studies considered the body temperature (37ºC) as the temperature of the substrate at which it receives the adhesive system. However, for the present investigation, a pilot study was done to determine the oro-dental temperature aiming to record the temperature of the cavity prepared to receive the adhesive system within the human oral cavity, which was found to be 35ºC±1. It was also found that incubation of the teeth at 35ºC for 10 minutes prior to bonding resulted in sustaining this temperature during the bonding procedure. In addition, the ambient temperature was determined via a pilot study and it was found that 24ºC is the average clinic temperature.

Bond strength measurements have become a well recognized method to analyze an important part of the *in vitro* performance of dental adhesives. It has supported the development of improved bonding systems and techniques. Since a development of a micro-bond test by ***Sano et al., in 1994***, many micro-bond tests have been performed using tensile method ***(Shimada et al., 2002)***. The information collected from the micro-bond tests is an effective method in terms of testing small areas of tooth structure, thus minimizing the effect of dentin surface irregularities ***(El-kholany et al., 2005)***. Thus, it uses a small bonding area (≈1mm^2) to provide nearly a flat interface that is required for ideal bond strength testing. However, for testing micro-tensile bonds, trimming of the specimen is an indispensable step for making small specimens which might induce stresses at the interface and is labour extensive ***(Sano et al., 1994*** and ***Shimada et al., 2002)***. This step is not required in micro-shear test. Furthermore, the bonded area can be determined prior to the placement of resin composite. Thus, the obtained resin specimens were bonded to the specified and selected area ***(Sattabanasuk et al., 2005)***. In the current study, a double adhesive tape was used to aid in localization of the area of adhesive system application and in supporting the polyethylene irises during bonding and polymerization of resin composite.

In this study, a specially designed attachment jig was used for micro-shear bond strength testing ***(Mobarak et al., 2010)***. This was utilized to aid in adjusting the position of the composite micro-cylinders of each specimen in relation to the wire and the center of the load cell (all as straight as possible) during debonding of the specimen and to ensure that the force acting on the adhesive bond is perpendicular to the direction of composite micro-cylinders in order to maintain the desired orientation in shear stress ***(Shimada et al., 2002)***.

The results of the current study showed that, regarding the effect of temperature on adhesive bond strength, there was no statistically significant difference in micro-shear bond strength (µSBS) between the refrigerated and ambient temperature for the three tested adhesive systems.

Refrigeration of adhesive systems is expected to positively affect the bond strength through preventing their chemical deterioration, since some components of these products are unstable in high temperatures, such as HEMA, MDP, BIS-GMA and DMA (***Kubo et al., 1991*** and ***Salz et al., 2005)***. This deterioration before application of adhesive system can result in higher adhesive degradation over time, thus decreasing adhesive durability ***(Nishiyama et al., 2006*** and ***Sadr et al., 2007)***. Also, if the adhesive is stored at room temperature, any temperature increase elevates the vapor pressure in the solution and facilitates evaporation of the solvent. This evaporation can quickly reduce the concentration of water into the mixture, reducing the demineralization capacity of these adhesives by dispersion of the smear layer and demineralization products ***(Jacobsen et al., 1995)***.

On the other hand, it has been reported that low temperatures during application of an adhesive can lead to a difficulty in solvent and water evaporation ***(Carvalho et al., 2005)***. This decreases the bond strength due to the presence of a high amount of residual solvent and water inside the adhesive layer ***(Pashley et al., 2004)***. Also, the reduction of adhesive viscosity, due to low temperature, can impair dissolution of the smear layer and substrate demineralization. In addition, as the viscosity of the polymeric material increases, penetration of the adhesive into the dentin surface may be reduced, causing decreased bond strength ***(Tay et al., 1995)***. Also, reduced temperatures influence properties related to curing efficiency, such as micro-hardness and diametral tensile strength, due to the decrease in polymerization ***(Bausch et al., 1981)***.

However, in the current study, there was no significant difference between the refrigerated and the ambient temperatures of the three tested adhesives. It seems that the adverse effects of refrigeration was not encountered, since the time necessary to apply the adhesive material to the substrate was probably enough to attenuate the low temperature and