SEM Study for the Effect of Different Dentin Surface Treatments on Shear Bond Strength to Resin Composite Using Two Total Etch Adhesive Systems

Master Thesis Submitted to Conservative
Dentistry Department
Faculty of Dentistry, Ain-Shams University
In partial fulfilment of the requirements of the master degree in
Operative dentistry

By

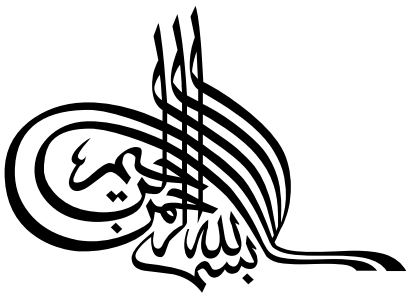
Sherif Mohamed El Hefnawy
B.D.S, Ain Shams University 2001

Supervisors

Dr. Hisham Abd El Wahab Moustafa
Professor of Operative Dentistry,
Faculty of Oral and Dental Medicine,
Ain Shams University

Dr. Farid Sabry El Askary
Assistant Professor of Operative Dentistry,
Faculty of Oral and Dental Medicine,
Ain Shams University

2009



" قَالُوا سُبْحَانَكَ لا عِلْمَ لَنَا إِلَّا مَا
عَلَّمْتَنَا إِنَّكَ أَنْتَ الْعَلِيمُ الْحَكِيمُ "

صدق الله العظيم
سورة البقرة:٣٢

DEDICATION

To my dear Father,

To my dear Mother, Brother, Sister, my Wife and my Daughter Karma

Thank You

Without you I would never be the person who I am now

ACKNOWLEGMENT

My deepest appreciation and gratitude goes to ***Prof. Dr. Hisham Abd El Wahab****, Professor of Operative Dentistry, Faculty of Oral and Dental Medicine, Ain Shams University, for his guidance, sincerity, supervision, unlimited support and help.*

I would like to thank ***Dr. Farid El Askary*** *Assistant Professor of Operative Dentistry, Faculty of Oral and Dental Medicine, Ain Shams University for his encouragement, excellent advice and help.*

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

INTRODUCTION

The success of composite materials as anterior or posterior restorations is controlled to some extent by the composite/dentin bond strength. In fact, bonding to dentin compared to bonding to enamel has led to some clinical problems. Dentin bonding or adhesion refers to "the micromechanical coupling or union of restorative materials to dentin, particularly dental composites, via an intermediary adhesive resin layer. [33]

The alteration of the tooth surface by using chemical materials was one of the different approaches mentioned,[79] that has been included.[11] The use of acid etching as a dentin surface treatment was reported as a step to improve adhesion for a variety of procedures in restorative and preventive dentistry.[68]

The clinical durable effectiveness that confirms the laboratory researches made the three-step etch and rinse adhesives the "gold standard" to compare all new generations with.[26] Although, three-step etch and rinse adhesives showed a good clinical performance, the two-step etch and rinse adhesives behave with less favourable performance.[64]

Because of the non-specific proteolytic and disinfectant properties[53] sodium hypochlorite is widely used in a variety of dental procedures, such as endodontic treatment, chemico-mechanical caries removal[24] and dentin bonding techniques.[23,62,67,70,71,73,86,87] The effect of sodium hypochlorite treatment on the performance of the different adhesives to dentin was extensively evaluated previously.[23,52,62,70,71,73,77,78,86] It was reported that sodium hypochlorite either had no effect[23,52,70,86] positively[68] or negatively affected[52,62,70,71,73,86,87] the performance of the different dentin adhesives. The use of sodium hypochlorite after dentin etching was also showed to

remove the exposed collagen and altered the dentin surface characterization.[24,57,82]

Phosphoric acid is an aggressive treatment that depletes the dentin surface completely from its minerals.[25] On the other hand, EDTA is a mild dentin etchant with mild demineralization ability and mild influence on dentin substrate, which requires the help of the self-etch primer to effectively demineralized dentin.[55]

As the tendency toward the use of simpler and faster adhesives that could affect their clinical effectiveness, the purpose of the this study was to evaluate the effect of two different dentin conditioners (35% phosphoric acid and EDTA) and 5.25% sodium hypochlorite as deproteinizing agent on shear bond strength (SBS) testing of three-step and two-step etch-and-rinse adhesives to dentin.

REVIEW OF LITERATURE

Effect of Phosphoric Acid Etching

The dentinal surface is covered with smear plugs in tubule orifices which decrease dentin permeability and protect the pulp, but only temporarily, because saliva dissolves it, and the dentin liner is also insufficient. Removing the smear layer increases dentin permeability more than twenty times. Smear layer, however, is still not an ideal bonding mediator because shear bond strength to underlying dentin is only 5 MPa, and that is not a sufficient anchor of restoration

Drummond et al 1996[31] carried out a study to evaluate the effect of the surface treatment of dentin on shear dentin-bonding: using phosphoric acid, and no treatment. All teeth were stored in distilled water and tested in a shear at a cross head speed of two mm/min. The specimens were loaded in static or cycled for 1000 cycles. On samples with etched dentin surfaces, the push-out test did no significant difference in measured bond strength when compared with results from the planar test. The bond strength resulting from cyclic fatigue of the etched specimens was approximately 51% of the static loading value. 10% and 32% phosphoric acid had the same effect for dentin bonding. Finite-element analysis indicated that the traditional shear test produced flexure of the specimen and high tensile stress magnitudes within the resin bonding layer. The push-out test produced high compressive stresses along composite circumference of the punch. Shear stresses in the resin bonding layer were nearly similar at the same loading element contact force.

Chaves 2002[18] evaluated the tensile bond strength of two self-etching adhesive systems and one one-bottle system applied on dentin

surfaces after different smear layer treatments. Results showed the mean bond strength for etch and rinse adhesive was significantly higher than self-etching systems. No significant differences were observed among smear layer treatments within the same dentin adhesive. It was concluded that: bond strength values were significantly different according to the adhesive system used. Different smear layer treatments prior to bonding did not affect tensile bond strength within the same bonding agent

Fawzy 2005[36] evaluated the surface structure of deep and superficial dentin after acid etching procedure including; surface area, surface roughness, dentinal tubule diameter and collagen fibrils diameter and structure. In addition, the degree of surface hydration was characterized by probing the nano-scale surface adhesive force. The specimens used were of approximately two mm thickness one superficial dentin disk and one deep dentin disk were from each tooth. The superficial dentin disks were prepared from the dentin below the DEJ. Then the exposed dentin surfaces, which approximately 2 mm below the DEJ, were considered as deep dentin.

The dentin disks were etched for 15 seconds with 35% phosphoric acid, rinsed and excess water was removed by gentle blotting with absorbent paper, leaving the dentin surface visibly moist. Specimens were then mounted for viewing in an atomic force microscope (AFM), to evaluate the surfaces of phosphoric acid etched deep and superficial dentin disks were characterized by AFM in hydrated state.

The total surface area of etched intertubular dentin was lower in deep dentin than in superficial dentin. In contrast, the increase in surface area of etched intertubular dentin/ $1\mu m^2$ projected area was higher for deep dentin. No differences in mean roughness height and collagen fibrils diameter were found between deep and superficial etched intertubular dentin. However, collagen fibrils in deep intertubular dentin exhibited more demarcated axial

periodicity banding. The nanoscal surface adhesive force was higher in deep dentin than in superficial dentin. The amount of interfibrillar spaces and the degree of surface hydration of acid demineralized intertubular dentin increase with dentin depth. The structural integrity of collagen fibrils is more preserved in deep intertubular dentin than in superficial intertubular dentin after acid etching.

De Souza et al 2005 [28] evaluated the effect of dentin deproteinization on microtensile bond strength (μTBS) of four total-etch adhesive systems. The ultra structure of the resin– dentin interfaces was also examined using scanning electron microscopy. They concluded that bonding performance on deproteinized dentin surfaces depended on the characteristics of each adhesive system, as well as the adhesive dentin specificity to the oxidant effect of sodium hypochlorite. The incorporation of fillers in the adhesive, a possible self-etching action, and the presence of a volatile solvent (acetone) were the main factors for a better union between the adhesive system and deproteinized substrate

Toledano et al 2005 [81] evaluated the effect of different chemical dentin pre-treatments performed for resin bonding. Dentin discs of superficial (SD) and deep dentin (DD) were obtained by transversally sectioning the crown. Knoop was measured after different treatments: (1) polished up to 4000 grit, (2) polished and etched (37% ortophosphoric acid for 15 s), (3) resin (Single Bond -SB-) infiltrated dentin after acid etching, (4) polished, etched and treated with 5% NaOCl for 2 min, (5) resin infiltrated (SB) after etching and NaOCl treatment. Results showed that Dentin KHN decreased on both SD and DD after acid etching. NaOCl treatment after acid etching did not affect on SD, but KHN of DD was lowered. Resin infiltration increased KHN but did not recover the initial values in both SD and DD. In conclusion dentin treated with either H3PO4 or NaOCl caused marked reduction of its

surface hardness and subsequent resin infiltration was not capable to restore it.

Van Landuyt et al 2006[88] evaluated bond strength of two-step self-etch adhesive Clearfil SE Bond bonds equally effective to enamel/dentin either with or without prior etching with phosphoric acid. Bur-cut enamel/dentin surfaces prepared from human molars were partially split in two halves by cutting a shallow groove. One half was first etched with 40% phosphoric acid, while protecting the other half by holding a razor blade in the groove. The C-SE was applied according to manufacturer's instructions, after which the surface was built up using Z100. After 24-h water storage, micro-specimens were prepared with the interface circularly constricted using a Micro-Specimen Former, prior to micro-tensile bond strength measurement. In addition, interfaces of C-SE with enamel/dentin prepared with and without beforehand acid etching were examined by Feg-SEM and TEM.

The results showed that etching increased the bonding effectiveness of C-SE to enamel. A clearly more micro-retentive surface was revealed by TEM and Feg-SEM when enamel was etched. Phosphoric-acid etching prior to C-SE application on dentin decreased the mTBS to dentin. TEM provided indications of a low quality hybrid layer after beforehand phosphoric-acid etching. It was concluded that Using C-SE, additional etching with phosphoric acid to improve bonding effectiveness should be limited to enamel.

Chinelatti et al 2007[20] evaluated the effect of phosphoric acid etching and air abrasion on the enamel and dentin using Scanning Electron Microscopy (SEM), the surface topography and the morphology of the adhesive interfaces of enamel and dentin after these treatments were examined. Teeth were taken to the sectioning machine and the occlusal overlying enamel surface was eliminated with the water-cooled diamond saw

at low-speed to prevent fracture or overheating. Discs were polished with #600-grit SIC papers for 30s to produce a standardized smear layer. The enamel–dentin discs were randomly assigned into three groups according to the surface treatment: I-35% phosphoric acid; II-air-abrasion; III-air-abrasion followed by 35% phosphoric acid. After surface treatment, discs were divided in two: one hemi-disc was separated for surface analysis; the other hemi-disc received the Single Bond/Filtek Z-250 restorative system.

Results indicated that phosphoric acid application on enamel led to the formation of microporosities, the peripheral tissues of the prisms were dissolved and the cores were intact. In dentin it was noticed presence of open tubules and absence of smear layer; distinction between peritubular and intertubular dentin was provided by different contrasts .Analysis of the adhesive/enamel interface disclosed the presence of a hybrid layer with resinous tags and at the adhesive/dentin interface it was observed clear formation of a homogenous hybrid layer with thickened tags in the superficial peritubular part, as well as tags deeper than10 lm from the surface and when the surface treatment was performed by air-abrasion, irregularities were observed at the enamel surface; microcracks and occluded tubules at dentin surface and lack of hybrid layer at adhesive/dentin interface. The air-abrasion treatment followed by acid etching provided an enamel etching pattern similar to the acid etching; microfissures and open tubules at dentin surface, and formation of hybrid layer at adhesive- dentin interfaceperitubular part (asterisks), as well as tags deeper than 10 mm from the surface

Dusevich et al 2007 [32] evaluated the different effects of etching using phosphoric acids and EDTA Specimens of human dentin were treated with 14% EDTA for 15 sec, 30 sec and 60 sec and observed immediately in wet condition with a field emission ESEM. After a 15 sec treatment the dental tubules were not fully open, which indicated insufficient removal of the