

**The Effect of Different Chemical Surface
Treatments on Resin Cement Bond Durability
to a Chair-Side Resin Composite Inlay: An In
Vitro Study**

Thesis

submitted to Operative Dentistry Department, Faculty of
Dentistry, Ain Shams University, in partial fulfillment of the
requirements of the PhD degree in Operative Dentistry.

By

Khaled Mohamed Adel Mohamed

B.D.S., Ain Shams University (2009)

M.Sc., Ain Shams University (2015)

2019

Supervisors

Prof. Dr. Farid Mohammed Sabry El-Askary

Professor of Operative Dentistry
Faculty of Dentistry, Ain Shams University

Dr. Zainab Mohamed Daa El-Din Soliman

Lecturer of Operative Dentistry
Faculty of Dentistry, Ain Shams University

To my family

My Dear Mom & Dad

My Brother & Sister

Thank you for everything, I am here now because of you

To My lovely wife and adorable Son

You are my backbone

I would like to express my deepest gratitude to ***Prof. Dr. Farid Mohammed Sabry El-Askary***, Professor of Operative Dentistry, Faculty of Dentistry, Ain Shams University, for his fatherly advice, his extreme support, encouragement and help throughout this thesis.

Also, I am grateful to ***Dr. Zainab Mohamed Diao El-Din Soliman***, Lecturer of Operative Dentistry, Faculty of Dentistry, Ain Shams University, for her prompt and meticulous efforts throughout this thesis. I am honored to be one of your candidates.

Finally, I would like to thank VOCO GmbH, Germany for supplying the materials used in this study.

List of Contents

List of Tables.....	ii
List of Figures.....	iii
Introduction.....	1
Review of Literature.....	3
Aim of the Study.....	40
Materials and Methods.....	41
Results.....	60
Discussion.....	73
Summary and Conclusions.....	83
References.....	86
Arabic Summary	

List of Tables

<u>Table No.</u>	<u>Table Title</u>	<u>Page</u>
Table 1:	Materials (Lot#), compositions and manufacturers.	41
Table 2:	Levels of the Investigation.	44
Table 3:	Experimental design of the shear bond strength testing.	45
Table 4:	Two-Way ANOVA for the effect of surface treatment, storage time and their interactions on SBS (Mpa).	60
Table 5:	Means \pm Standard Deviations for the effect of surface treatment within each storage time and the effect of storage time within each surface treatment on SBS.	61
Table 6:	Means \pm Standard Deviations for the effect of different treatments on microhardness of resin composite.	71

List of Figures

<u>Figure No.</u>	<u>Figure Title</u>	<u>Page</u>
Figure 1:	Specially fabricated split copper molds 4mmx4mm and 2mmx4mm.	46
Figure 2:	Curing resin composite against polyester strip.	47
Figure 3:	Shade A4 disc surrounded by acrylic resin material and PVC ring.	48
Figure 4:	Application of resin cement.	50
Figure 5:	Resin composite cylinder placement.	50
Figure 6:	Bonded specimens under 500g load applied by specially fabricated loading device.	51
Figure 7:	Specimen setting in the Universal Testing Machine.	52
Figure 8:	Split copper mold 4mmx7mm.	54
Figure 9:	Split copper mold 2mmx7mm.	54
Figure 10:	Nail varnish coated resin composite specimen.	55
Figure 11:	50 wt % ammonical silver nitrate preparation.	56
Figure 12:	Sectioned composite discs.	57
Figure 13:	Polished composite slabs.	57
Figure 14:	Bar chart for the effect of experimental variables	62

- on shear bond strength.
- Figure 15:** Bar chart represents the percentage of different types of failure in all experimental groups. **63**
- Figure 16:** Representative Microscopic image of type 1 failure (adhesive). A: shade A4 composite disc and B: shade A1 composite cylinder. **63**
- Figure 17:** Representative Microscopic image of type 2 (mixed) failure A: shade A4 composite disc B: shade A1 composite cylinder. **64**
- Figure 18:** Representative Microscopic image of type 3 failure (cohesive in composite disc). A: shade A4 composite disc and B: Shade A1 composite cylinder. **64**
- Figure 19:** Representative Microscopic image of type 3 failure (cohesive in composite cylinder) A: shade A4 composite disc B: shade A1 composite cylinder. **65**
- Figure 20A-C:** Representative SEM micrographs (500x) for NT, AE+S and HP respectively. C1: Resin composite A1, RC: Resin cement and C2: Resin composite A4. **66**
- Figure 21A-B:** SEM micrograph (500x) for 1-year HF, which showed silver nitrate deposits along the Resin composite/Resin cement/Resin composite. **66**

- interface (arrow). C1: Resin composite A1, RC: Resin cement and C4: Resin composite A4.
- Figure 22A-B:** SEM micrograph (500x,3000x respectively) for 1-year HF+S, which showed traces of silver nitrate deposits along the Resin composite/Resin cement/Resin composite interface (arrow). C1: Resin composite A1, RC: Resin cement and C2: Resin composite A4. **67**
- Figure 23A-C:** 1-year representative SEM micrographs for NT, AE+S and HP respectively. C1: Resin composite A1, RC: Resin cement and C2: Resin composite A4. **67**
- Figure 24A-C:** 24 hours representative SEM micrographs (4000x) for the adaptation of Resin-composite-to-resin composite interface for HF, AE and PP+S respectively. C1: Resin composite A1, RC: Resin cement and C2: Resin composite A4. **68**
- Figure 25A-C:** 1-year representative SEM micrographs (4000x,1000x,1500x respectively) for the adaptation of Resin-composite-to-resin composite interface for HF, AE and PP+S respectively. C1: Resin composite shade A1, RC: Resin cement and C2: Resin composite A4. **69**
- Figure 26A-J:** Surface topography after different surface treatments. A: NT 500x; B: NT 3000x; C:AE **71**

500x; D:AE 3000x and E: HF 500x; F: HF 3000x;
G: HP 500x; H: HP 3000x I: PP 500x; J: PP 3000x
respectively (G: Grinding groove; White arrows:
Detached/Eroded filler particles; Black arrow:
Smear layer).

Figure 27: Bar chart representing the Microhardness values of resin composite after surface treatment. **72**

Direct restorative technique is the most commonly used for restoring both anterior and posterior teeth.¹ However, with direct placement of composite restorations, polymerization shrinkage generates stresses at the composite-tooth interface, which may compromise the integrity of the bond.² In large posterior cavities, especially in those with cervical margins located in dentin, the lack of moisture control in addition to polymerization shrinkage could produce marginal defects and gaps,³ marginal discoloration, post-operative sensitivity, secondary caries and pulpal irritation.⁴ In addition, poor anatomical and proximal contact were other technical drawbacks that compromised direct restoration longevity.⁵

Due to such drawbacks, indirect composite restorations were introduced in 1980's. The continuous developments in resin composites have made the possibility to fabricate esthetic indirect adhesive restorations that aimed to overcome the shortcomings of direct resin composite restorations.⁶ However, indirect restorative techniques need multiple visits to perform. To counteract the drawback of the multiple visits of the indirect technique, a chair side indirect resin composite system was introduced, featuring a die silicon material that sets in few minutes and can be used immediately to construct the indirect resin composite restoration eliminating the need of multiple visits and extra laboratory work.

The bonding effectiveness of the luting cement and restorative material was significantly affected by the material surface treatments.^{7,8,9,10} Increased percentage of inorganic fillers (%vol) greatly optimized their mechanical and physical properties⁷ and affected their surface roughness and morphology in response to the different surface treatments employed.

On this basis, different chemical treatments were used to improve the bonding of resin composite materials to resin cement.^{11,12,13,14} The rationale of chemical treatments was to create a rough fitting surfaces that allowed better adhesion.

The effect of aging of resin composites in the oral cavity was shown to be a very complex mechanism. Thermal changes, food, beverages, saliva and biofilm resulted in biodegradation of resin composites.^{15,16} A previous study¹⁷ showed that, aging of bonded resin composites specimens, irrespective of the mechanism of aging process employed, negatively affected the bond strength between luting cement/tooth/resin composite materials.

Accordingly, it was found beneficial to evaluate the effect of different chemical treatments, on bond durability, nanoleakage and surface microhardness between the chair-side inlay and resin cement.

I. Introduction to Resin composite

For many years, amalgam has been the most widely used restorative material, which was always characterized as technically easy to use and clinically predictable, with favorable mechanical properties, in spite of some disadvantages.¹⁸ These disadvantages include a lack of adhesion to dental tissues and lack of reinforcement of the remaining tooth structure. Jagadish and Yogesh reported that teeth with complex preparations restored with amalgam experienced cusp fractures due to microcrack propagation under fatigue loading.¹⁹

As an alternative to amalgam, direct adhesive restorative techniques with resin composite have been proposed, since these materials bond to tooth structure, and hence increase its fracture resistance.²⁰ Adhesive dentistry has many considerable advantages in the treatment of weakened tooth structure. The possibility of establishing adequate adhesion between tooth structure and restorations through adhesive materials may eliminate the need for extended cavity preparations. As a result of the increasing demand for a universal restorative material indicated for all types of direct restorations, including posterior teeth, a new category of resin composites was developed, which is the Nano-filled composites. Nano-composites show high translucency, superior polish, and polish retention together with maintaining physical properties and wear resistance equivalent to those of several hybrid composites.²¹

Filled resin composites, in the past were limited for the restoration of anterior teeth. Many studies have been undertaken to investigate the filler phases, resin compositions, and curing conditions as an attempt to improve the mechanical properties of filled composites. However, further

significant improvements are needed in order to extend the use of filled composites to high stress-bearing applications such as direct posterior restorations involving multiple cusp build ups and indirect restorations, such as inlays and onlays.²² Nowadays, with the current improvement in the material, it's use has been widened to include posterior intra-coronal area, extra-coronal restorations, even complete crowns and fixed partial dentures.²³

Indirect restorations, including inlays/onlays were introduced in the hope of overcoming problems associated direct resin composites, which needs to be inserted using conventional incremental techniques. These disadvantages included fractures in high stress-bearing areas due to their limited degree of conversion and all the well documented consequences of polymerization shrinkage stress. It was hoped that the use of the indirect technique would improve the load-bearing capacity of the resin composites by raising the degree of conversion obtained by laboratory post-curing of the restoration.²²

It was reported that extra-oral polymerization of resin composite followed by cementation appear to improve the marginal fit and minimize contraction stress. The mechanical properties of the resin composites were also improved by post-cure heat treatment, although such improvements were modest and sometimes not statistically significant. The relatively high brittleness and low load-bearing capacity of resin composites still hinder their use in large stress-bearing restorations. There is a considerable need for improving the mechanical properties, especially load-bearing capacity and wear resistance of such materials, while maintaining their superior esthetic properties.²⁴

II. Direct versus indirect resin composite restorations

Resin composites have been widely used as tooth-coloured direct restorative materials. These restorations are well accepted clinically, and clinical studies show satisfactory results, even for large and extensive restorations.²⁵ Many of the problems associated with the direct placement of large posterior resin composite restorations can be paved with the use of an indirect composite technique. Patient, dentist and material-related factors seem to affect the life span of bonded restorations.²⁶ The negative effects of polymerization shrinkage, besides wear resulting in loss of anatomic form and interproximal contact, were often cited as common causes of failure of direct posterior composite restorations. Marginal opening with secondary caries, fracture of the restorations, marginal deterioration, discoloration are now the principle modes of failure of this type of restorations.

The indirect technique allows the production of restorations in the laboratory with appropriate proximal contours and contacts and control of the anatomic form. The first generation of commercial indirect resin composite inlay systems was introduced on the market in 1980s by Touati and Mörmann. Based on a light-cured resin composite, the restoration was formed directly in the inlay cavity. Following an initial cure and removal of the inlay from the cavity, the inlay was post-cured in a heat and light oven at 110⁰C. The post-curing at a high temperature resulted in a higher stress relaxation and conversion compared to the direct-placement of the restoration, this could improve material's wear resistance and hardness.