



**EFFECT OF DIFFERENT CAVITY
DISINFECTANTS ON SHEAR BOND
STRENGTH OF RESIN
COMPOSITES TO DENTIN**
An in vitro study

**BY
NADA FARAG SANUSSI AL-SHUKRI**

Supervisor:

Associat Prof. Dr. Naeima Mohamed Betamar

**This Thesis Submitted in Partial Fulfillment of
Requirements for the Degree of Master of Science in
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Faculty of Dentistry
University of Benghazi
Department of Conservative Dentistry and Endodontics

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NADA FARAG SANUSSI AL-SHUKRI

This Thesis has been approved by the examination committee :

Prof. Dr. Naeima Mohamed Betamar. (supervisor)

Signature:

Dr Samia Senussi Alawjali. (Internal Examiner)

Signature:

Dr Mohamed Hussein Elfoghi. (External examiner)

Signature :

Dr Othman M. Albadry

.....

Head of Graduate Studies

And Training Office

Director of Graduate studies and training

Dr Naeima Mohamed Betamar

Signature:

23/ November /2022

Dr Nagat Hassan Bubteina

.....

Dean of the Faculty



Dedication

*To the soul of my lovely father;
My great mother; beloved brothers; beloved sisters;
my gorgeous husband and lovely kids
For their enormous support*

*To my teachers
For their believe in me
I dedicate my thesis*

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List of Abbreviations

MMPs	Matrix metalloproteinases
CHX	Chlorhexidine digluconate
BAC	Benzalkonium chloride
EDTA	Ethylene diamine tetraacetic acid dehydrate
NaOCl	Sodium hypochlorite
O ₃	Ozon
Bis-GMA	Bisphenol-A and glycidyl methacrylate
Bis-EMA	Bisphenol-A ethoxylated dimethacrylate
UDMA	Urethane di-methacrylate
TEGDMA	Tri-ethylene glycol di-methacrylate
PRIMM	Polymer Rigid Inorganic Matrix Material
Compomers	Polyacid Modified Composite Resins
ORMOCER	Organically Modified Ceramic
MDPB	Methacryloyloxydecyl Pyridinium Bromide
RBCs	Resin bonded composites
SACs	Self-adhesive composites
DIC	Digital image correlation
FEA	Finite element method
C-factor	Configuration factor
ER	Etch-and-rinse adhesive system
SE	Self-etch adhesive system
TE	Total-etch adhesive system
NPG-GMA	N-phenylglycine and glycidyl methacrylate
HEMA	2-Hydroxyethyl methacrylate

4-META	4-Methacryloxyethyl trimellitate anhydride
NCCL	Non carious cervical lesions
RDT	Remaining dentin thickness
N	Newton's
MPa	Mega pascal
MDP	10-methacryloyloxydecyl di-hydrogenphosphate
TBS	Tensile bond strength
SBS	Shear bond strength
I2	Iodine
I2-KI	Iodinepotassium iodide
I2-KI/CuSO4	Potassium iodide/copper sulfate
PVP-I	Providone-iodine
LASER	Light Amplification by Stimulated Emission of Radiation
Er:YAG	Erbium yttrium aluminum garnet
Nd:YAG	Neodymiumdoped yttrium aluminum garnet
Nd:YAP	Neodymium-doped yttrium aluminum perovskite
Er, Cr:YSGG	Erbium chromiumdoped yttrium scandium gallium garnet
KTP	Potassium-titanyl-phosphate
SEM	Scanning electron microscope
DBA	Dentine bonding agents
GP	Group
no.	Number

EFFECT OF DIFFERENT CAVITY DISINFECTANTS ON SHEAR BOND STRENGTH OF RESIN COMPOSITES TO DENTIN (*An in vitro study*)

BY

NADA FARAG SANUSSI AL-SHUKRI

Supervisor: Dr. Naeima Betamar

Abstract

Purpose: The aim of this study was to evaluate the effect of different cavity disinfectants on dentin shear bond strengths of composite resin applied with two different adhesive approaches.

Materials and Methods: eighty caries free third molars were sectioned parallel to the occlusal surface to expose mid-coronal dentin. The specimens were randomly divided into four groups of twenty teeth each. GP1 is control group (no treatment), groups 2,3, and 4 dentin surfaces were treated with the following cavity disinfectants, respectively; 0.12% chlorhexidine solution (CHX), 5% sodium hypochlorite (NaOCl) and 0.15% benzalkonium chloride (BAC). Each group from Gp1 to Gp4 was further divided into two subgroups ($n = 10$ per sup group) according to the adhesive approaches. Ten specimens were bonded with the total-etch approach and the other ten specimens were bonded with self-etching approach. Then resin composite was applied incrementally to the treated dentin surface into cylindrically-shaped Teflon tube (3mm diameter \times 3mm height) then polymerized with LED curing unit. After the specimens were stored in an incubator for 24 h, the shear bond strength was measured at a crosshead speed of 0.5 mm/min. The bond strength data were analyzed with one way analysis of variance ANOVA and independent sample t test.

Results: Statistical analysis found that treated dentin surface with different cavity disinfectant resulted in higher shear bond strength compared with the control group (the lowest SBS value 7.58 ± 0.85) obtained for the untreated dentin surface (control group). Among the cavity disinfectant groups, the highest SBS was recorded for Tantum (13, 39 ± 7.59) group. For the three types of cavity disinfectant, total-etch approach showed higher bond strength than self-etch approach.

Conclusion: Treated the dentin surface with cavity disinfectant before adhesive bonding improved the shear bond strength between resin composite and dentin surface in particular with the total-etch adhesive approach.

Keywords: bond strength, adhesive, composite resin, cavity disinfection.

Chapter 1

INTRODUCTION

1. INTRODUCTION

Cavity preparation is an operative procedure attempts to remove all infected caries dentine prior to placing a restoration. However residual bacteria might be entrapped within the dentinal tubules or the smear layer during and after the cavity preparation, which considers one of a major problem in restorative dentistry (Koshiro et al., 2004; Swift, 2002). Therefore, an effective removal of infected dentin and prevention of microorganisms growth under a restoration leads to prevent the development of secondary caries, reduce microleakage, pulpal inflammation and hence reduce the need for replacing the restoration (Koshiro et al., 2004). With the development and improvement of aesthetic restorative materials, adhesive systems have become essential in clinical applications (Chavesa et al., 2002). Adhesive systems are responsible for the bonding of restorative material to tooth structures. Thus, the longevity of adhesive restoration is directly associated with the effectiveness of adhesive systems (Chavesa et al., 2002).

The key challenge for new dental adhesives is to be simultaneously effective on two dental substrates of conflicting nature. Dentin is considered an intrinsically moist and heterogeneous tissue, which makes adhesion to this tissue more sensitive adhesive technique when compared to enamel (Swift, 2002). The hybrid layer is essential to adhesive dentistry. It is where the dental adhesive system creates a micromechanical bond with demineralized dentinal collagen fibrils (Swift, 2002).

Despite the evolution of adhesive systems, the hybrid layer suffers degradation over time, causing loss of adhesive resistance, which influences the longevity of restorations (Bin-Shuwaish, 2016). The degradation of the adhesive interface is related to several factors, such as oral fluids and bacteria present in situ (Bin-Shuwaish, 2016), leading to degradation of polymers and other organic components. For those reasons, cavity disinfection becomes an important step prior to the restorative procedure. This step is described as cleaning the dental cavity with antimicrobial agents before the use of adhesive systems, making it as innocuous as possible (Bin-Shuwaish, 2016).

Long-term studies have shown that the bond strength of resin bonded to dentine decreased over time due to collagen degradation within the hybrid layer (De Munck et al., 2003; Koshiro et al., 2004; Perdigão et al., 2013) . Therefore, elimination of the

residual bacteria from the cavity surfaces after cavity preparation is of major importance using a disinfectant solution (Koshiro et al., 2004). Matrix metalloproteinases (MMPs) are a group of proteolysis enzymes which are capable of degrading extracellular matrix proteins. Activated MMPs are not fully infiltrated with adhesive resin, and can slowly degrade the collagen fibrils at the resin-dentin bonded interface (Perdigão et al., 2013). Thus, the use of such cavity disinfectants which are MMPs inhibitors is a strategy to prevent degradation of dentine bonds and to increase the longevity of bonded restorations (Perdigão et al., 2013).

Interest in the study of antimicrobial agents and their effects on the pulp originated in the early 1970s with Brannstrom and Nyborg, who emphasized the importance of eliminating bacteria remaining on cavity walls, including dentin and enamel, after caries excavation by means of antibacterial agents, and therefore accordingly it was recommended disinfecting the cavity preparation before inserting the restoration to reduce the incidence of postoperative sensitivity by eliminating viable bacteria and their toxins from the restoration-tooth interface (Brannstrom & Nyborg, 1973). Many chemicals have been tested as cavity disinfectants, including chlorhexidine digluconate (CHX), disodium ethylene diamine tetra-acetic acid dehydrate (EDTA), sodium hypochlorite (NaOCl), ozon (O₃), Er:YAG laser and iodine.

Generally, a potential problem in the use of a disinfectant before dentin bonding agents is the possibility of an adverse effect on the bond strength of the composite resins to dentin (Ercan et al., 2009).

Chapter 2

REVIEW OF THE LITERATURE

2. REVIEW OF THE LITERATURE

2.1 INTRODUCTION

The essential goal of any adhesive restoration is to achieve a tight and long-lasting adaptation of the restorative material to enamel and dentin (Kugel & Ferrari, 2000). The key challenge for new dental adhesives is to be simultaneously effective on two dental substrates of conflicting nature. Some barriers must be overcome to accomplish this objective (Van Meerbeek et al., 2010). While bonding to enamel by micromechanical interlocking of resin tags within the array of micro-porosities in acid etched enamel can be reliably achieved and can effectively seal the restoration margins against leakage (Sato et al., 2018). Bonding effectively and durably to organic and humid dentin is the most puzzling task in adhesive dentistry.

Many dental researchers have attempted to achieve methods for reliable and durable adhesion between resins and tooth structure (Hanabusa et al., 2012; Kugel & Ferrari, 2000; Van Meerbeek et al., 2010). The acid etching transforms the smooth enamel surface into a very irregular surface. After rinsing off the etchant with water and drying the enamel surface with air, a fluid resin is applied on the enamel surface. This resin penetrates into the subsurface, is drawn by capillary action. Monomers in the fluid resin polymerize and become interlocked with the enamel surface (Sato et al., 2018). The formation of resin micro-tags within the enamel structure is the fundamental mechanism of adhesion of resin to enamel (Hanabusa et al., 2012; Van Meerbeek et al., 2010). As opposed to enamel, which is composed of more than 90% of hydroxyapatite and can be dried easily, dentin is an intrinsically wet organic tissue penetrated by a tubular maze containing the odontoplastic process, which communicates with the pulp.

The density of the tubules by unit area is greater close to the pulp than near the dentin-enamel junction (Swift, 2002). The dynamic nature of dentin as a substrate is responsible for inconsistent bond strengths and marginal leakage, which still occur with all resin-based adhesives (Nunes et al., 2001). Whenever tooth structure is prepared with a bur or other instrument, residual organic and inorganic components form a “smear layer” of debris on the surface (Chavesa et al., 2002). The smear layer fills the entrance of dentin tubules to form smear plugs, which decrease dentin permeability by up to 86%. Submicron porosity of the smear plug still allows for

flow of dentinal fluid (de Souza Costa et al., 2002). Although the smear layer acts as a “diffusion barrier” that decreases the permeability of dentin (Özok et al., 2002), it also can be considered an obstacle that must be removed so that resin can be bonded to the dentin substrate (de Souza Costa et al., 2002).

During tooth cavity preparation, the success of restorative treatment can be affected by bacterial remnants in the cavity walls. The bacteria remaining after restorative procedure may survive and multiply, especially in the presence of micro-leakage, which may lead to pulpal irritation (Hiraishi et al., 2009), risk of recurrent caries (Nedeljkovic et al., 2015), postoperative sensitivity, and therefore failure of the dental restoration (Salama et al., 2015). Generally, cavity disinfection is an acceptable approach that may prevent residual potential risk of microorganism on tooth structures (El Wakeel et al., 2015). The use of antibacterial solutions after cavity preparation may be considered a method to reduce the incidence of postoperative sensitivity by eliminating viable bacteria and their toxins from the restoration-tooth interface (Orchardson & Gillam, 2006).

There are varieties of commercial products in dental market for cavity disinfections and have been recommended for clinical use. Long-term studies have shown that the bond strength of resin-bonded dentin decreased over time due to collagen degradation within the hybrid layer (De Munck et al., 2003; Koshiro et al., 2004). However, the effect of the cavity disinfectants on the bond strength has been a controversial issue (Sharma et al., 2011). Some authors reported that disinfection products reduced the bond strength (Sharma et al., 2011). On the other hand other researchers indicated that these procedures may not diminish the bond strength (El Wakeel et al., 2015).

This section reviews the following topics relevant to the study:

- Resin composite.
- Description of dentin bonding systems.
- Adhesion to dentin.
- Factors affect the bond strength to dentin.
- Bond strength testing methods.
- Effect of cavity disinfectants on bond strength.

2.2 RESIN COMPOSITE

2.2.1 Over view of resin composite

Resin composites have been widely used in clinical application for nearly 50 years. Their first introduction into dentistry was dated back to the late 1950s and early 1960s (Stein et al., 2005), and have been gradually improved in their formulations, properties, esthetics and become increasingly popular in dentistry (Samuel et al., 2009).

Composite in materials science is a solid formed from two or more distinct phases (e.g., filler particles dispersed in a polymer matrix) that have been combined to produce properties superior to or intermediate to those of the individual constituents (Sevkusic et al., 2014).

Dental resin-based composites are structures composed of three major components: a highly cross-linked polymeric *matrix* reinforced by a dispersion of glass, mineral, or resin *filler* particles and/or short fibers bound to the matrix by *coupling agents* (Alsharif et al., 2010). Such resins are used to restore and replace dental tissue lost by disease or trauma and to lute and cement crowns and veneers and other indirectly made or prefabricated dental devices (Zhou et al., 2019).

2.2.2 Chemical composition of resin composite

Current direct composites are typically have four major components: a *matrix phase* (forms continuous phase and binds the filler particles that usually contains a dimethacrylate resin); a *dispersed phase* of fillers and tints (reinforcing particles and/or fibers) ; a *coupling phase* that promotes adhesion between the resin matrix to the filler particles (silanes) ; a *polymerization initiators* is added to bring about polymerization of the material that are activated either chemically (by mixing two materials) or by visible light (using a light curing unit), *polymerization activator inhibitors*, and *coloring agents* (Alsharif et al., 2010).

i. Resin Matrix (matrix phase)

The resin is the chemically active component of the composite. It is initially a fluid monomer, but is converted into a rigid polymer by a radical addition reaction (Alsharif et al., 2010). It's based on a blend of aromatic and/or aliphatic dimethacrylate monomers such as (Bis-GMA) bisphenol-A and glycidyl methacrylate

and urethane di-methacrylate (UDMA), to form highly cross-linked strong, rigid, and durable polymer structure. It represents the backbone of composite resin system (Alsharif et al., 2010). The most commonly used monomer for both anterior and posterior resin is Bis-GMA, this resin is commonly referred to as Bowen's resin (Alsharif et al., 2010), after its inventor. Other monomers are urethane di-methacrylate (UDMA), and tri-ethylene glycol di-methacrylate (TEGDMA) (Ferracane, 2013).

Bis-GMA is extremely viscous at room temperature and difficult to blend and manipulate due to hydrogen bonding by hydroxyl groups (Santini et al., 2013). The viscosity of Bis-GMA can be reduced by mixing with diluents and facilitate the addition of fillers (Santini et al., 2013). These diluents are low molecular weight di-methacrylate monomers such as tri-ethylene glycol di-methacrylate (TEGDMA) (Ravi et al., 2013). Addition of the diluents allows greater degree of conversion and more extensive cross-linking to occur between chains providing a matrix that is more resistant to solvents (Santini et al., 2013).

ii. Filler Particles (dispersed phase)

Fillers are the inorganic or organic particles, which are added to improve mechanical properties (Alsharif et al., 2010), such as tensile and compressive strength, modulus of elasticity, abrasion, resistance, radiopacity, esthetics and handling (Ravi et al., 2013). Various transparent mineral fillers are employed to strengthen and reinforce composites as well as to reduce curing shrinkage, water sorption and thermal expansion (Ravi et al., 2013). Commonly used fillers are silicon dioxide, boron silicates and lithium aluminum silicates. In some composites, quartz is partly replaced with heavy metal particles like zinc, aluminum, barium, strontium or zirconium. Barium and strontium are the most common elements used in filler particles to increase radiopacity (R. Wang et al., 2018).

iii. Coupling Agents

Coupling agent binds filler particles to the organic resin (R. Wang et al., 2018). Interfacial bonding between the matrix phase and the filler phase is provided by coating the filler particles with silane coupling agents (R. Wang et al., 2018), to improve mechanical and physical properties, as well as the silane reduces hydrolytic

breakdown and allows stress transfer between the filler and the matrix (Alsharif et al., 2010; Rebholz-Zaribaf & Özcan, 2017). The most commonly used coupling agent is an organo-silane such as gamma methacryloxy propyl tri-methoxy silane. The silane agent is a bi-functional molecule with a methacrylate group on one end and a silanol group on the other (Rebholz-Zaribaf & Özcan, 2017).

iv. Photo-initiator Agents

These agents activate the polymerization of composites (Santini et al., 2013). The polymerization process of composite resin starts with releasing free radicals from methacrylate monomer structure which requires an external energy in the form of heat, chemical, or radiant energy (Santini et al., 2013). Free radicals can be generated by chemical activation or by external energy activation (heat, light, or microwave). Because dental composites for direct placement use chemical activation, light activation, or a combination of the two (Rueggeberg et al., 2017). Chemical activation resin consists of two pastes. One paste consists of benzoyl peroxide initiating material, and the other paste consists of tertiary amine activator (Santini et al., 2013). Currently, the dental photo-activator commonly used is camphorquinone, which has canary yellow color which results in yellowish composite restoration (Santini et al., 2013).

v. Inhibitors

These agents inhibit the free radical generated by spontaneous polymerization of the monomers. For example, butylated hydroxyl toluene (0.01%) (Santini et al., 2013). Inhibitors are molecules added to the resin-matrix composite to prevent premature polymerization when the material is exposed to the room light during the dental procedure (Santini et al., 2013). Materials such as hydroquinone, eugenol and oxygen all serve to inhibit or slow polymerization reaction rate if used in large amount; for this reason, a small amount of hydroquinone is used to prevent premature polymerization of the methacrylate and to extend the half-life of the monomer (Santini et al., 2013).

vi. Coloring Agents

Coloring agents are used in very small percentage to produce different shades of composites. Mostly metal oxides such as titanium oxide and aluminum oxides are added to improve opacity of composite resins (Alsharif et al., 2010).

2.2.3 Polymerization Process of Composite Resin

Polymerization is a process in which monomers of a low molecular weight are converted into chains of polymers with a high molecular weight to attain desired properties (Roggendorf et al., 2011) . The polymerization process is vinyl-free-radical polymerization (Roggendorf et al., 2011). The polymerization process of composite resin starts with releasing free radical from methacrylate monomer structure an external energy in the form of heat, chemical, or radiant energy (Rueggeberg et al., 2017). When free radical is formed, the monomer looks for the electron-rich monomer to form covalent bond. The combination of those monomers forms a new polymer (Rueggeberg et al., 2017). Based on the activation energy, composite resin is classified into chemically activated, light activated, dual activated and heat activated composite resins (Rueggeberg et al., 2017).

2.2.4 Classification of composite resins:

Composite resins have been classified in different ways, depending on their composition, to make it easier for dentists to identify and use. The most popular classification is based on filler particle size given by Lutz and Phillip 1983 (Lutz & Phillips, 1983), according to this classification composite resins are divided into macro filler composites, micro filler composites and hybrid composites (fillers of different sizes) (Cramer et al., 2011).

More recently, Zhou *et al.*,(2019) classify the dental composites based on their different compositions and performance characteristics (Zhou et al., 2019), into four categories :

1. According to size of filler particles: macrofilled, microfilled, hybrid, and nanohybrid.
2. According to mode of curing: chemically cured, light cured, heat-cured, and dual-cured.
3. According to restorative procedure: direct and indirect.

4. According to clinical application: packable, flowable, polyacid modified, self-adhesive, and finally Bulk-fill.

2.2.4.1 Macrofilled composites

Macrofilled composites use relatively large inorganic crystalline quartz or glass fillers which has got excellent optical properties, and chemical inertness (Ravi et al., 2013). The particles in early macrofilled composites ranged in size from 10 to 100 μm . Currently, typical composites use particles ranging in size from 1 to 10 μm (Ravi et al., 2013). The most common fillers in current macrofilled composites are ground quartz, strontium, or heavy metal glasses containing barium. Quartz the most common filler used in early composites, has excellent esthetics and durability but lacks radiopacity (Ravi et al., 2013).

2.2.4.2 Microfilled composites

Microfilled composites are agglomerates of 0.01- to 0.1 μm inorganic colloidal silica particles embedded in resin filler particles (Siang Soh et al., 2006). The problems of surface roughening and low translucency associated with traditional and small-particle composites can be overcome through the use of colloidal silica particles such as the inorganic filler component, with a mean particle diameter about one tenth of the wavelength of visible light (Sabbagh et al., 2004).

Microfills were developed to provide better esthetics and polish-ability. A smoother surface can be produced due to the smaller size of the silica particles (Ravi et al., 2013). However, mechanical properties such as strength and stiffness are generally inferior to larger quartz or glass filled composites because of the lower filler content, which often limits their use to non-stress-bearing areas (Ravi et al., 2013).

2.2.4.3 Hybrid composites

As the name implies, hybrid composites are formulated with mixed filler systems containing both microfine (0.01 to 0.1 μm) and fine (0.1 to 10 μm) particle fillers in an effort to obtain even better surface smoothness than that provided by the small particle composites while still maintaining the desirable mechanical properties of the latter (M. F. Burrow, 2013). Thus, they are a general utility class of composite that are also suitable for restoring certain high stress bearing area where esthetic

considerations dominate for example, incisal edges and small non-contact occlusal cavities (Braga et al., 2005) .

2.2.4.4 Nanofill composites

In recent years, nanotechnology has been used in the composition of different types of resin composites. The resin-modified photo-polymerizable glass ionomer based on nanotechnology was introduced to the market, providing the benefits of improved surface polish and esthetics (Chen, 2010).

Nowadays, advances in nanotechnology produces composite resin which has nanoparticle of 25 nm and agglomerate nanoparticle 75 nm (Chen, 2010). Zirconium, silica and nanosilica particle is used as filler in nanofilled. Agglomerate particle is silanized so it can bond with resin. Combining nanoparticle with agglomerate nanoparticle increases the filler loading of composite resin up to 79.5% (Chen, 2010). The increasing filler loading occurs because of lower dimension and distribution area of filler particle. Increasing filler loading leads to reduced polymerization shrinkage and increased the mechanical properties of composite resin (Chen, 2010).

2.2.4.5 Flowable Composites

These resins typically have a lower viscosity through a reduced filler loading, which enables the resin to flow readily, spread uniformly, intimately adapt to a cavity form, and produce the desired dental anatomy (Hervás García et al., 2006). This improves the clinician's ability to form a well-adapted cavity base or liner, especially in class II posterior preparations and other situations in which access is difficult (Roggendorf et al., 2011). However, whereas these materials tend to be less sticky during handling than microfills and hybrids, they are inherently inferior in mechanical properties owing to the lower filler loading and higher susceptibility to wear and other forms of attrition (Roggendorf et al., 2011).

2.2.4.6 Condensable (Packable) Composites

Condensable/packable composites have improved mechanical properties and handling characteristics (Peumans et al., 2001). Were developed by adjusting their filler distribution to increase the strength and stiffness of the uncured material and provide a consistency and handling characteristics similar to the amalgam. Main basis

of packable composites is Polymer Rigid Inorganic Matrix Material (PRIMM) (Peumans et al., 2001). Here components are resin and ceramic inorganic fillers which are incorporated in silanated network of ceramic fibers. These fibers are composed of alumina and silicon dioxide which are fused to each other at specific sites to form a continuous network of small compartments (Peumans et al., 2001).

2.2.4.7 Short Fiber Reinforced Composite

Short fiber reinforced composite resin is used as one of dental restoration materials. Adding 5% - 7.5% of short fiber filler into filler particle composite resin with filler loading of 60% wt, reduces polymerization shrinkage by 70% (Bocalon et al., 2016; Riva & Rahman, 2019). This filler increases the physical properties of composite resin, e.g : flexural strength, modulus, and work of fracture. Moreover, filler of short fiber also increases stress bearing in application of posterior dental restoration (Maas et al., 2017). The most commonly used short fiber reinforced type is glass fiber. Various types of poly-metric fiber are also developed as composite resin filler, including poly (vinyl acetate) fibers, polyethylene and aramid fibers, and nylon 6 fibers (Maas et al., 2017).

2.2.5 Recent advances in resin composites:

2.2.5.1 Giomers

Giomer is hybrid of words “glass ionomers” and “composite”. Giomers have properties of both glass ionomers (Fluoride release, fluoride recharge) and resin composite (excellent esthetics, easy polishability, biocompatibility) (Garoushi et al., 2018), used in non-carious cervical lesions, root caries and deciduous tooth caries (Gordan et al., 2007). Filler particles are made of fluoroaluminosilicate glass which have been reacted with polyalkenoic acid before incorporating into resin matrix (Itota et al., 2004).

2.2.5.2 Compomers (Polyacid Modified Composite Resins)

Compomers provide combined advantages of composites and glass ionomer (Nicholson, 2007). Initially the compomers were introduced as a type of glass-ionomers, which offered fluoride release along with improved physical

properties(Nicholson, 2007). But in terms of clinical use and performance, it was considered as a type of composite resin.

2.2.5.3 Organically Modified Ceramic (ORMOCER)

ORMOCER is an organically modified nonmetallic inorganic composite material (Kalra et al., 2012). Composed of organic molecules of methacrylate groups forming a cross-linked matrix, inorganic condensing molecules to make three dimensional network formed by inorganic poly-condensation (Kalra et al., 2012). This makes the backbone of ORMOCER molecules and fillers have higher bond strength, polymerization shrinkage is least among resin based filling material and highly esthetic (Kalra et al., 2012).

2.2.5.4 Antibacterial Composites /Ion-releasing Composites

Since composites show more tendency for plaque and bacteria accumulation in comparison to enamel, attempts have been made to develop caries resistant antibacterial composites (Cheng et al., 2012). For this, following have been tried to incorporate in the composites: Chlorhexidine, Methacryloyloxy Decyl Pyridinium Bromide (MDPB), and Silver.

2.2.5.5 Smart Composite

Smart composites are based on the recently introduced alkaline glass fillers which inhibit the bacterial growth and thereby reduce formation of secondary caries. It was introduced in 1999 under the name Ariston pHc (Vivadent) (Sigmund & Torquato, 1999). In smart composite, micron size sensor particles are embedded during manufacturing process into composite (Lu et al., 2018). These sensors interact with resin matrix and generate quantifiable ions like fluoride, hydroxyl and calcium ions if the pH falls in the vicinity of the restoration. Fall in pH occurs because of plaque deposition in that area (Lu et al., 2018).

2.2.5.6 Low shrinkage composite

Various materials have been developed, tested, and tried for the purpose of minimizing the polymerization shrinkage and associated stresses of resin bonded composites (RBCs). In 2007, a silorane-based composite became commercially

available (Boaro et al., 2010). The silorane molecule presents a siloxane core with four oxirane rings attached that open upon polymerization to bond to other monomers. The oxirane ring opening causes a volumetric expansion that partially compensates the shrinkage resultant from molecular bonding (Boaro et al., 2010).

2.2.5.7 Self-adhesive composites (SACs)

Self-adhesive composites combine the benefits of adhesive and composite technology, as they are claimed to bond to tooth tissue without a separate adhesive step (Mine et al., 2017). These materials contain self-etching and/ or self-adhesive monomers that are able to etch enamel and dentin surfaces or chemically bond to hydroxyapatite. Some studies reported that self-adhesive composites show limited interaction with dentin or enamel (Mine et al., 2017).

2.3 DESCRIPTION OF DENTIN BONDING SYSTEMS

Traditional “drill and fill” approach is fading now because of numerous advancements taking place in restorative dentistry. The principles of adhesive dentistry date back to 1955 when Buonocore (M. Buonocore et al., 1956), using techniques of industrial bonding, postulated that acids could be used as a surface treatment before application of the resins (Kugel & Ferrari, 2000). In the late 1960s, Buonocore suggested that it was the formation of resin tags that caused the principal adhesion of the resins to acid-etched enamel (M. G. Buonocore, 1963). The idea that resin penetrates the micro-porosities of etched enamel and results in a micromechanical bond is well-accepted today. In 1963, Buonocore demonstrated his insight into adhesion dentistry when he discussed the difference in bonding to enamel and to dentin (M. G. Buonocore, 1963), particularly when he referred to Dr. Bowen’s attempts to investigate substances that will displace water from tooth surfaces (Bowen, 1966), with the idea that they could be used as pretreatment for enamel or dentin.

2.3.1 Mechanism of Bonding

The fundamental goal of adhesive dentistry is to create an effective, durable union between tooth structure and the restorative material (da Fonseca et al., 2013). From the clinical point of view the important thing is that the bonding agent should

successfully seal the tubules to prevent post-operative sensitivity due to the hydrodynamic effect, protect the pulp and provide a long-lasting bond between the tooth and the restoration. The adhesion of composite resins to the dentin substrate is based on smear layer treatment (Chavesa et al., 2002), while some adhesive systems require the conditioning of dentin with phosphoric acid, as etch-and-rinse systems (ER), others preserve the smear layer by incorporating it into the adhesive layer, they are the self-etch systems (SE) (Chavesa et al., 2002).

Dentin adhesive molecule has a bi-functional structure: ideally dentin bonding agent should have both hydrophilic and hydrophobic ends. The hydrophilic end displaces the dentinal fluid to wet the surface. The hydrophobic end bonds to the composite resin (Y. Wang & Spencer, 2004).

2.3.2 Classification of Modern Adhesives:

Over the years, there have been numerous classifications of dentin bonding agents that have been advocated by many authorities. Some of them are based on generation, the number of clinical steps and on the modern adhesive strategy (Sofan et al., 2017). The most commonly used classification of adhesives is chronologically based (Kugel & Ferrari, 2000).

2.3.2.1 Based on Generations (Sofan et al., 2017):

- First generation bonding agent.
- Second generation bonding agent.
- Third generation bonding agent.
- Fourth generation bonding agent.
- Fifth generation bonding agent.
- Sixth generation bonding agent.
- Seventh generation bonding agent.

2.3.2.2 Based on Number of Clinical Steps (Sofan et al., 2017) :

- Three steps dentin bonding agents.
- Two steps dentin bonding agents.
- Single step dentin bonding agents.

2.3.2.3 Based on Smear Layer Treatment (Meerbeek et al., 2020) :

- Smear layer modifying agents.
- Smear layer removing agents.
- Smear layer dissolving agents.

2.3.2.1 Classification based on Generations:

The concept of generation was used because of the complexity of bonding agents, the variety of classifications refers to when and in what order this type of adhesive was developed by the dental industry (Sofan et al., 2017) :

i. First Generation Dentin Bonding agents.

In 1956, Buonocore and colleagues demonstrated that use of a glycerol-phosphoric acid dimethacrylate-containing resin would bond to acid-etched dentin (M. Buonocore et al., 1956). This bond was believed to be due to the interaction of this bi-functional resin molecule with the calcium ions of hydroxyapatite. Of course, immersion in water would greatly reduce this bond. Nine years later Bowen tried to investigate the use of N-phenylglycine and glycidyl methacrylate(NPG-GMA) as bi-functional molecule or coupling agent (Bowen, 1965).

These products ignored the smear layer, mechanism of adhesion was deep penetration of the resin tags into the exposed dentinal tubules after etching and the chelating component which could bond to the calcium component of dentin. Since they could chelate with calcium ions of the tooth structure, they formed stronger bonds with enamel than dentin (Bowen, 1965).

ii. Second Generation Dentin Bonding agents.

They were introduced in the late 1970s. Most of the second-generation bonding agents leave the smear layer intact when used but some of them employed the use of mild cleansing agents to remove the smear layer. Thus, improve resin penetration. However, these systems resulted in bond strengths to dentin that were weak and unreliable (Navyasri et al., 2019).

iii. **Third Generation Dentin Bonding agents.**

Third generation was attempted to deal with smear layer and dentinal fluids. These systems employed the concept of conditioning and priming before application of bonding agent (Yoshida et al., 2000). These were applied for:

1. Removal of the smear layer without disturbing the smear plugs.
2. Modifying the smear layer to improve its properties.

The application of third-generation dentin bonding agents involves three steps: etching with an acidic conditioner, priming with a bi-functional resin in a volatile solvent and bonding with an unfilled or partially filled resin. However, these systems resulted in higher bond strengths to dentin (Yoshida et al., 2000).

iv. **Fourth Generation Dentin Bonding Agents**

The complete removal of the smear layer is achieved with fourth generation bonding systems. Fourth generation is characterized by the process of hybridization at the interface of the dentin and the composite resin (Nakabayashi et al., 1982). Hybridization is the phenomenon of replacement of the hydroxyapatite and water at the dentin surface by resin. This resin, in combination with the collagen fibers, forms a hybrid layer. In other words, hybridization is the process of resin interlocking in the demineralized dentin surface. This concept was given by Nakabayashi in 1982 (Nakabayashi et al., 1982). The use of the total-etch technique is one of the main characteristics of fourth generation bonding systems (Navyasri et al., 2019).

Components of Fourth Generation Adhesives:

1. Conditioner (Etchant): Commonly used acids are 37% phosphoric acid (Christensen, 2001), nitric acid, maleic acid, oxalic acid, pyruvic acid, hydrochloric acid, citric acid or a chelating agent, e.g. EDTA. Use of conditioner/etchant causes removal or modification of the smear layer, demineralizes peritubular and inter-tubular dentin and exposes collagen fibrils (Oliveira et al., 2003).

2. Primer: Primers consist of monomers like HEMA (2-Hydroxyethyl methacrylate) and 4-META (4-Methacryloxyethyl trimellitate anhydride) dissolved in acetone or ethanol. Thus, they have both hydrophilic as well as hydrophobic ends which have affinity for the exposed collagen and resin respectively (Van Meerbeek et al., 2001). Use of primer increases wettability of the dentin surface, bonding between the dentin

and resin, and encourages monomer infiltration of demineralized peritubular and intertubular dentin (Van Meerbeek et al., 2001).

3. Adhesive: The adhesive resin is a low viscosity, semifilled or unfilled resin which flows easily and matches the composite resin. Adhesive combines with the monomers to form a resin reinforced hybrid layer and resin tags to seal the dentin tubules (Van Meerbeek et al., 2001).

v. Fifth Generation Dentin Bonding Agents.

Fifth-generation dentin bonding agents were made available in the mid-1990s (Manuja Nair et al., 2014). They are also known as “one-bottle” or “one-component” bonding agents. In these agents the primer and adhesive resin are in one bottle and etchant in separate bottle, the main advantages of these system are: high bond strength, easy to use and reduced postoperative sensitivity (Van Meerbeek et al., 2001).

vi. Sixth Generation Dentin Bonding Agents

These were made available in 2000 (Manuja Nair et al., 2014). In sixth generation etching step is eliminated, because in sixth generation etchant, primer and bonding are available in single solution (Manuja Nair et al., 2014). The separate acid-etching step was eliminated by incorporating an acidic primer that was placed on the enamel and the dentin after tooth preparation (Manuja Nair et al., 2014).

vii. Seventh Generation Dentin Bonding Agents

They achieve the same objective as the sixth generation systems except that they simplified multiple sixth generation materials into a single bottle and unit dose version one-step self-etch adhesive, thus avoiding any mistakes in mixing (Vinay & Shivanna, 2010).

2.3.2.2 Classification Based on Number of Clinical Steps:

At this stage it was proposed a classification of bonding systems, which reflects their essential mode of use (Sofan et al., 2017), rather than historical development:

i. Three-steps: involving etch, prime and bond. These bonding systems are supplied as three bottles :one each from etchant, primer and bonding agent (Silva e Souza Junior et al., 2010).

ii. Two-steps 1: here the steps are etch, then finally prime and bond in a single coating. Bonding systems of this type employ substances in two bottles, one consisting of etchant, and the other of the combined prime and bond formulation (Silva e Souza Junior et al., 2010).

iii. Two-steps 2: for these systems, the two steps are etching and priming combined followed by bonding. It uses two bottles of components, the first containing a self-etching primer and the second the bonding agent. The self-etching primer modifies the smear layer on the surface of the dentine, and incorporates the products in the coating layer (Silva e Souza Junior et al., 2010).

iv. One-step: this uses a single bottle containing a formulation that blends a self-etching primer and bonding agent (Silva e Souza Junior et al., 2010). Clinically, this is the easiest to use, and bond strengths are generally reported to be acceptable, despite the simplicity of bonding operation (Yazici et al., 2007).

2.3.2.3 Classification Based on Smear Layer Treatment

Basically, three adhesion strategies have been employed to modern dentin bonding agents on the basis of their interaction with the smear layer (Meerbeek et al., 2020). These are:

i. Smear Layer Modifying Agents.

In this strategy, bonding agents modify the smear layer and incorporate it in the bonding process (Van Landuyt et al., 2005).

Steps: in these, enamel is selectively etched with 37% phosphoric acid (taking care not to etch dentin) (Frankenberger et al., 2008; Sato et al., 2018). After washing and drying the tooth, primer and adhesive are applied separately or in combination. This results in micromechanical interaction of dentin and bonding system without exposure of collagen fibrils (Sato et al., 2018).

ii. Smear Layer Removing Agents.

These bonding agents completely remove the smear layer employing the total etch concept. They work on the principle of hybrid layer and resin tags (Oliveira et al., 2003; Van Landuyt et al., 2005).

Steps: in these, enamel and dentin are etched simultaneously using an acid (preferably 37% phosphoric acid) (Christensen, 2001; Frankenberger et al., 2008). After washing and drying the tooth surface, primer and bonding agent are applied either separately or in combination (Kenshima et al., 2006).

iii. Smear Layer Dissolving Agents.

These agents partly demineralize the smear layer and the superficial dentin surface without removing the remnants of smear layer or the smear plugs (Van Landuyt et al., 2005). They make the use of acidic primers also termed as self-etch primers or self-etch adhesives which provide simultaneous conditioning and priming of both enamel and dentin. After this, adhesive is applied without washing the tooth surface (Kenshima et al., 2006).

2.4 ADHESION TO DENTIN

Bonding to dentin has been proven more difficult and less reliable and predictable than enamel. This is because of difference in morphologic, histologic and compositional differences between the two (Swift, 2002).

2.4.1 Problems encountered during dentin bonding

Enamel, contain 92 % (volume) inorganic hydroxyapatite, in dentin it is 45% (volume) Dentin contains more water than does enamel (Swift, 2002). In addition, hydroxyapatite crystals have a regular pattern in enamel whereas in dentin, hydroxyapatite crystals are randomly arranged in an organic matrix. Presence of the smear layer (El-Din, 2002), makes wetting of the dentin by the adhesive more difficult.

Furthermore, the dentin contains dentinal tubules which contain vital processes of the odontoblasts. This makes the dentin a sensitive structure. Moreover, dentin is a dynamic tissue which shows changes due to aging, caries or operative procedures (Perdigão, 2010). Last but not least, fluid present in dentinal tubules

constantly flows outwards which reduces the adhesion of the composite resin to dentin. By etching dentin, the smear layer and minerals content are removed, exposing the collagen fibers (El-Din, 2002; Perdigão, 2010), areas from where minerals are removed are filled with water. This water acts as a plasticizer for collagen, keeping it in an expanded soft state. Thus, spaces for resin infiltration are also preserved (Perdigão, 2010), but these collagen fibers collapse when dry and if the organic matrix is denatured. This obstructs the resin from reaching the dentin surface and forming a hybrid layer (Hashimoto et al., 2002). Therefore, presence of moist dentin is needed to achieve successful dentin bonding (Hashimoto et al., 2002), clinical and in vitro evidence suggests that vigorous application of the adhesive may lead to a more complete penetration of the adhesive solution into the etched dentin collagen network (Zander-Grande et al., 2011).

2.5 FACTORS AFFECT THE BOND STRENGTH TO DENTIN

The bonding mechanism depends on the penetration of the primer and adhesive resin into the conditioned dentin surface in order to create micromechanical interlocking with the dentin collagen (Lopes et al., 2006). The morphological and physical variations in human dentin make it a difficult substrate for the achievement of durable bonds between adhesive resin and dentin. Marshall *et al.*,(2001) stated that the various structural components and properties of dentin could directly affect the adhesive bond (Marshall, Habelitz, et al., 2001). Biological and clinical factors such as dentin permeability, pulpal fluid flow, sclerotic and carious dentin can also affect dentin bonding (Kwong et al., 2002).

2.5.1 Caries-affected dentin and tertiary dentin

The clinically relevant substrates for dentin adhesion include affected dentin, which is located immediately underneath the carious dentin area. Affected dentin is slightly decalcified (Marshall, Habelitz, et al., 2001). Continuous deposition of mineral within the tubules underneath carious lesion process results in tubular obliteration and the formation of sclerosis, and potentially reducing bond strengths (Perdigão, 2010). Another type of clinically relevant dentin that may be found in deep caries lesions is reactionary tertiary dentin, which is formed by odontoblasts in the

pulp chamber wall near the area corresponding to the carious lesion (Marshall, Habelitz, et al., 2001).

2.5.2 Sclerotic dentin in non-carious cervical lesions (NCCL)

Sclerotic dentin is common in areas where dentin has been exposed to the oral environment, such as non-carious cervical lesions. These lesions contain a complex dentin substrate with different ultra-structural layers (Tay & Pashley, 2004), the tubules appear obliterated by crystalline deposits. Etching sclerotic dentin is difficult to achieve therefore these surfaces prevent a proper infiltration of the adhesive material to the underlying dentine and reducing bond strength (Marshall Jr et al., 2000; Tay & Pashley, 2004).

2.5.3 Dentin age

With increasing patient age, in both crown and root aspects of teeth, dentinal thickness increases, while the density of odontoblasts and pulp fibroblasts decreases (Sardella et al., 2005). Primary dentine is more reactive to acid etching than permanent dentin, therefore the bond strength and thickness of the hybrid layer of primary dentine is greater than the permanent dentine (Sardella et al., 2005).

2.5.4 Tooth region and remaining dentin thickness (RDT)

Superficial dentin normally results in higher composite-dentin bond strength than deep dentin (Yoshikawa et al., 2012). The difference in intrinsic moisture has been deemed responsible for the differences in bond strengths between superficial and deep dentin. For example, Suzuki and Finger *et al.*, (1988) reported that bond strengths decreased 30–40% in deep dentin for three dentin adhesives (Suzuki & Finger, 1988), whereas Nakamichi *et al.*, (1983) reported a 50% decrease in bond strength from superficial to deep dentin in bovine teeth (Nakamichi et al., 1983). These differences tend to diminish when the smear layer is left intact, but lower bond strengths occur in deep dentin when the smear layer is removed (Giachetti et al., 2004). As bonding systems became more hydrophilic, the sensitivity of bond strengths to dentin depth has decreased (Giachetti et al., 2004).

2.5.5 Smear layer

Residual organic and inorganic components form a “smear layer” of debris on the surface whenever dentin is prepared with a bur or other instrument. The smear layer fills the orifices of dentin tubules forming “smear plugs, ” and decreases dentin permeability by up to 86% (Koibuchi et al., 2001). The removal of the smear layer and smear plugs with acidic solutions results in an increase of the fluid flow onto the exposed dentin surface. Some studies reported low dentin bond strengths over thick dentin smear layers (Koibuchi et al., 2001; Lee et al., 2010), while others reported no influence (Tani & Finger, 2002; Tay et al., 2000).

2.5.6 Dentin permeability and pulpal pressure

According to the hydrodynamic theory (Orchardson & Gillam, 2006), once dentin is exposed, external stimuli cause fluid shifts across dentin, which activate pulpal nerves and cause pain (Perdigão, 2010). Trans-dentinal permeability is also responsible for the constant wetness of exposed dentin surfaces due to the outward fluid movement from the pulp (Perdigão, 2010). Dentin permeability results in dentin surface wetness, which influences the quality of the adhesive-dentin interface and may decrease the bond strength between resins and dentin. Etch-and-rinse adhesives result in higher micro-permeability compared to self-etch adhesives (Rosales-Leal et al., 2007). The hybrid layer was always 100% infiltrated by pulpal fluid when an etch-and-rinse adhesive was used. However, pulpal pressure had no effect on enamel sealing (Rosales-Leal et al., 2007). Other studies have reported that a simulated pulpal pressure decreases the dentin bond strengths of resin-modified glass-ionomer and etch-and-rinse adhesive materials (Moll et al., 2005; Pereira et al., 2000). However, the use of hydrophobic bonding agents on the acid-etched dentin saturated with ethanol reverses the fluid conductance to the level obtained with the presence of the smear layer (Carrilho et al., 2007).

2.6 BOND STRENGTH TESTING METHODS

Strong, durable bonds between restorative materials and tooth substrate are essential, not only from a mechanical stand point, but also from the biologic and esthetic perspectives (Bin-Shuwaish, 2016). The bonding of resin-based restorative materials to dentin has always been more challenging compared to enamel bonding

(Swift, 2002). In selecting an adhesive system for clinical use, bond strength and sealing ability should play major roles.

A tensile force produces **tensile stress** (Van Noort et al., 1989), a compressive force produces **compressive stress**, and a shear force produces **shear stress**. The tensile and compressive stresses are principal axial stresses, whereas the shear stress represents a combination of tensile and compressive components (L. Wang et al., 2003). Three types of “simple” stresses can be classified: tensile, compressive, and shear. Complex stresses, such as those produced by applied forces that cause flexural or torsional deformation (Van Noort et al., 1989).

2.6.1 Tensile bond strength

A tensile stress is always accompanied by tensile strain, but it is very difficult to generate pure tensile stress in a body, that is, a stress caused by a load that tends to stretch or elongate a body (P. E. Cardoso et al., 1998). In a tensile bond strength test, using extracted human or bovine teeth, the bonded specimen is placed in a universal testing machine and subjected to tensile force perpendicular to the tooth surface (Sano et al., 1994). In this type of test it is difficult to maintain a proper alignment during both bonding and testing, to avoid stress concentrations due to incorrect interfacial geometry. Furthermore tensile test is very sensitive and minute change will have a great influence on the results (Sano et al., 1994; Van Noort et al., 1989).

2.6.2 Micro tensile bond strength

The micro tensile test is designed to load a test specimen along its long axis and the testing machine fixtures often have a toggle or freely rotating attachment that minimizes the misalignment of loaded specimen with the loading axis of the testing machine (Sano et al., 1994). It is essential to improve the specimen geometry and the experiment design to produce a more uniform stress distribution across the adhesion interface during testing. The microtensile bond strength test (μ TBS) method developed by Sano *et al.*, (1994) provides a solution to the previous testing problems (M. Burrow et al., 2002; Sano et al., 1994; Xie et al., 2002). Moreover, this method is considered to be suitable to evaluate the bond performance of small bonding areas (Takahashi et al., 2002; Uno et al., 2001).

2.6.3 Compressive bond strength

When a body is placed under a load that tends to compress or shorten it, the internal resistance to such a load is called a **compressive stress** (Didem & Yalcin, 2014). A compressive stress is associated with a compressive strain. Compressive strength has particularly important role in the mastication process since most of the masticatory forces are of compressive nature (Didem & Yalcin, 2014). The maximum resistance to compression is calculated by the original cross-sectional area of the test specimen and the maximum force applied. A clinically relevant compressive strength value may be based on compressive strength values of natural mineralized tissues (Jandt et al., 2000).

2.6.4 Shear bond strength

This type of stress tends to resist the sliding or twisting of one portion of a body over another. Shear stress can also be produced by a twisting or torsional action on a material shear stress is calculated by dividing the force by the area parallel to the force direction (Van Noort et al., 1989). Adhesion of resin-based composite to dentine has mostly been evaluated by shear bond strength tests. In the shear bond strength test the force is applied parallel to the tooth surface while the bond is broken. However, during the shear test, cohesive failure within the dentine is often observed (Xie et al., 2002). This form of failure does give reliable information on the actual strength of the adhesive bond (M. Burrow et al., 2002), this may be due to large size specimens and non-uniform stress distribution generated during this test (Eren et al., 2013). The load in the shear bond strength of resin composite to dentine will be recorded in Newton's (N) and the shear bond strength will be calculated in Mega Pascal (MPa) taking into account the cross-sectional area of the composite buildup (Eren et al., 2013).

The advantage of this method is that specimens and loading arrangements are quite easy to produce and because of that this test is often used (Eren et al., 2013). However Van Noort *et al.*, (1989) have reported that the data obtained from the bond strength tests largely depended on the actual test set-up that may differ between laboratories for parameters such as specimen geometry, size of surface area and type of composite used (Van Noort et al., 1989).

2.6.5 Mode of failure

The mode of failure is an important aspect of bond strength tests, a detailed inspection of the fractured surfaces can indicate the failure mode of a bonded assembly. Classifying the failure mode of adhesive is critical to understand the cause of the failure of adhesive joints. There are three basic adhesive failure modes namely:

- a) Adhesive/interfacial, i.e., the failure located in the adhesive interface,
- b) Cohesive, i.e., the failure or the fracture located in one of the substrates on either side of interface, either in the composite or tooth structure.
- c) Mixed of the two. i.e. the failure of interfacial and partially cohesive in dentin/composite interface (Uno et al., 2001).

Hassan *et al.*,(2014), reported that the increased percentage of mixed failure on groups of disinfectants was attributed to the increased shear bond strength which clearly was reflected by the mode of failure of the bonding system (Mohammed Hassan et al., 2014). Ceballos *et al.*,(2003) also reported that the major mode of failure in specimens with low bond strengths was adhesive failure, while cohesive fractures in dentin or composite were seen at higher bond strength (Ceballos et al., 2003).

2.7 EFFECT OF CAVITY DISINFECTANTS ON BOND STRENGTH

During tooth preparation to receive a restoration, the success of restorative treatment can be affected by bacterial remnants in the cavity walls (Cheng et al., 2013). Attempts at complete removal of deep carious dentin, by solely mechanical means, may result in pulpal violation and/or gross destruction of the tooth structure and has failed to generate a completely caries free cavity (Cheng et al., 2013; Singla et al., 2011). It has been documented that bacteria remaining after restorative procedure may survive and multiply, especially in the presence of micro leakage, which may lead to pulpal irritation (Hiraishi et al., 2009), risk of recurrent caries (Nedeljkovic et al., 2015), postoperative sensitivity, and therefore failure of the dental restoration (Salama et al., 2015).

In the study of antimicrobial agents and their effects on the pulp originated in the early 1970s by Brannstrom and Nyborg (Bin-Shuwaish, 2016), the authors emphasized the importance of eliminating bacteria remaining on cavity walls, including dentin and enamel, after caries excavation by means of antibacterial agents,

and therefore recommended disinfecting the cavity preparation before inserting the restoration. Thereafter, cleaning the cavity preparation with antibacterial agents to aid in bacterial elimination, began to gain wide acceptance among dental practitioners (Al-Omari et al., 2006). The use of cavity disinfectants eliminates the residual bacteria, but a potent problem is that it may affect the bond strength of composite resins (Koshiro et al., 2004).

Degradation of the exposed collagen fibrils within the hybrid layer is the key factor which is primarily responsible for the deterioration of the adhesive dentine interface (Sinha et al., 2016). This is mainly brought about by the action of Matrix metalloproteinases (MMPs) enzymes present in the dentin which get activated in the presence of zinc and calcium ions when low pH is created by the process of acid etching (Sinha et al., 2016). Certain mechanisms have been advocated to improve the bond strength and the durability of the resin-dentin bond. One of them is inhibition of MMPs (Perdigão et al., 2013). MMPs are a group of proteolysis enzymes which are capable of degrading extracellular matrix proteins. Activated MMPs are not fully infiltrated with adhesive resin. They can slowly degrade the collagen fibrils at the resin-dentin bonded interface (Breschi et al., 2008; Perdigão et al., 2013). Thus, the use of such cavity disinfectants which are MMP inhibitors is a strategy to prevent degradation of dentin bonds and to increase the longevity of bonded restorations (Breschi et al., 2008; Perdigão et al., 2013). There are many products used as cavity disinfectants have been used in clinical dentistry in an effort to reduce or eliminate bacteria during cavity preparation and prior to the placement of dental restorations, such as:

2.7.1 Chlorhexidine (CHX):

Chlorhexidine (CHX) is one of the most widely used antimicrobial agents in oral health and is considered the “gold standard” of oral antiseptics (Matthijs & Adriaens, 2002). Different concentrations and forms of CHX are available: 0.12 to 0.2% mouth rinses, 2% cavity-cleaning solutions, and 0.5 to 1% gels. It has been reported that the 2% solution is the most widely used CHX form in clinical dentistry and dental research (Bin-Shuwaish, 2016). CHX wash, in the form of 2% solution, before composite bonding has been shown to successfully preserve the bond strength, up to 6 months, when etch-and-rinse adhesive systems were used (Francisconi-dos-

Rios et al., 2015; Gunaydin et al., 2016). Manfro *et al.*,(2012) and Breschi *et al.*,(2009) have reported that the bond in the CHX-treated samples was significantly stronger than the non-treated samples after 12 months of aging (Breschi et al., 2009; Manfro et al., 2012).

The preserved bond interface associated with the use of CHX can be explained by the inhibitory ability of CHX to the MMPs found in etched dentin (Almahdy et al., 2012). MMPs in dentin have been shown to play a role in the degradation of the unprotected collagen fibrils within the hybrid layer. Therefore, MMPs inhibitors, such as CHX can play a role in the longevity of the resin bond to dentin (Almahdy et al., 2012; Mazzoni et al., 2015). Sinha *et al.*,(2016) and Boiter *et al.*,(2013) also suggested that the application of 2% chlorhexidine prevents hybrid layer degradation and this procedure has a beneficial effect on maintaining bond strength (Boitor et al., 2013; Sinha et al., 2016). Several studies have reported higher bond strengths of resin composite to dentin when etch-and-rinse adhesive systems, rather than self-etch systems, were used after CHX pretreatment (Ercan et al., 2009). The adverse effect on bond strength of 2% CHX solutions associated with self-etch bonding systems and cements may be explained by the presence of functional monomer, 10-methacryloyloxydecyl dihydrogenphosphate (MDP), in the bonding resin of self-etch adhesive systems, which might have been affected by CHX bonding (Shafiei & Memarpour, 2012). Another factor is the residual moisture of the 2% CHX solution, which contaminates the bonded surface and alters the ability of the hydrophilic resin in the self-etch system to seal the dentin (Hiraishi et al., 2009; Singla et al., 2011). This may also explain why the bond at the tooth-resin interface was not altered by the 1% CHX gel application prior to the use of self-etch adhesive systems, which has been reported in several studies (Ercan et al., 2009; Sharma et al., 2011; Singla et al., 2011). The gel form of disinfectant does not wet the dentin surface and penetrate the dentinal tubules as does the solution form (Bin-Shuwaish, 2016).

However some authors reported no statistically significant differences in the bond strength with self-etch adhesive systems and after 2% CHX application (Bin-Shuwaish, 2016; Mobarak et al., 2010). Arslan *et al.*,(2012) found no significant differences between self-etch and etch-and-rinse in dentin margins (Arslan et al., 2012). There are several adverse reactions of chlorhexidine include contact dermatitis, damage and irritation to oral mucosa, altered taste sensations, and various other

allergic reactions (Pemberton & Gibson, 2012). In addition, one of the major drawbacks also includes discoloration of tooth surface (Arslan et al., 2012).

2.7.2 Sodium hypochlorite (NaOCl):

Sodium hypochlorite (NaOCl) is an effective organic solvent that has been widely used in clinical dentistry as a cleansing agent after having first been used in 1920 in endodontics as an antimicrobial irrigant (H. Spencer et al., 2007). Controversial results on the effect of NaOCl on resin bond have been reported (Arslan et al., 2011). Some authors found that treatment can adversely affect the hybrid layer and therefore the resulting bond strength and microleakage (Osorio et al., 2002; Reddy et al., 2013; Shinohara et al., 2004), while others found no effects on bond strength (Potter et al., 2013; V. d. P. A. Saboia et al., 2006). However, the effect of NaOCl pretreatment on the bond strength of composite resin is believed to depend on the adhesive system used (Ercan et al., 2009; Fawzy et al., 2008).

Ercan *et al.*,(2009) recommended NaOCl disinfectant to be used with etch-and-rinse bonding systems. They found that 2.5% NaOCl pretreatment negatively affected the shear bond strength (SBS) of self-etching bonding systems (Ercan et al., 2009). Fawzy *et al.*,(2008) also reported similar results with a 2-minute application of 5.25% NaOCl, as they found the tensile bond strength (TBS) of the self-etching adhesive to be negatively affected by the NaOCl pretreatment, with no significant effect reported when etch-and-rinse adhesive was used (Fawzy et al., 2008).

2.7.3 Benzalkonium chloride (BAC):

Benzalkonium chloride (BAC) is a mixture of alkylbenzyltrimethyl ammonium chlorides and is a nitrogenous cationic agent containing a quaternary ammonium group with broad antimicrobial activity (Camila Sabatini & Pashley, 2015). Tubulicid (Global Dental Products, Bellmore, NY, USA) is a quaternary ammonium compound with ethylene diamine tetra acetic acid (EDTA) that comes in three forms:

- i. Tubulicid Red contains 1.0% sodium fluoride, which has been recommended by the manufacturer to be used for cleaning without removing the smear layer (Bin-Shuwaish, 2016).
- ii. Tubulicid Blue is used to disinfect the whole tooth or multiple teeth, prior to the cementation of crowns or bridges (Bin-Shuwaish, 2016).

- iii. Tubulicid Plus has been claimed to be a stronger cleaner and used as a root canal irrigant to remove the smear layer and open dentinal tubules (Hülsmann et al., 2003).

As with CHX, BAC has been documented to be an effective MMPs inhibitor that may preserve the adhesive bond of the resin restoration to dentin (Mazzoni et al., 2015; C Sabatini et al., 2014; Camila Sabatini & Patel, 2013; Sharma et al., 2009). Sabatini and Patel in 2013, evaluated the effects of different concentrations of BAC on the preservation of adhesive interfaces by using two etch-and-rinse adhesives (Optibond Solo Plus and All-Bond 3) (Camila Sabatini & Patel, 2013). They reported improvement in the bond strength in groups pretreated with 0.5% BAC and 1.0% BAC and using Optibond Solo Plus, and in groups pretreated with 0.25 and 0.5% BAC and using All-Bond 3. They found that BAC at all concentrations improved bond stability after 18 months (Camila Sabatini & Patel, 2013).

Based on two *in vitro* studies, Sharma *et al.*, recommended that only etch-and-rinse bonding systems be used when Tubulicid Red is used as a cavity disinfectant (Sharma et al., 2009; Sharma et al., 2011). In contrast to the results of previous studies Türkün *et al.*,(2004) found that Tubulicid Red did not significantly affect the sealing ability of Clearfil SE Bond and Prompt L-Pop (both are self-etched adhesives) (Türkün et al., 2004).

2.7.4 Iodine-Based Disinfectant:

Iodine-based disinfectants are unstable solutions with wide-ranging effects on microorganisms. The antibacterial effects of these agents are attributed to the presence of molecular iodine (I₂) in these solutions (Athanasiadis et al., 2007).

Different iodine solutions have been used for disinfection purposes in clinical dentistry including: Iodine-potassium iodide (I₂-KI), potassium iodide/copper sulfate (I₂ KI/CuSO₄), iodine disclosing/disinfection solution (I₂DDS), and providone-iodine (PVP-I) (Bin-Shuwaish, 2016). Cunningham and Meiers in 1997, compared the effect of 0.11% I₂-KI/CuSO₄ with that of 2% CHX on the SBS of resin-modified glass-ionomer cements (Fuji II LC, Photac-Fil, and Vitremer) to sound dentin. They found that the I₂-KI/CuSO₄ solution significantly lowered the bond strengths of Vitremer and Fuji II LC to dentin. In contrast, CHX did not significantly affect the bond of any of the tested materials to dentin (Cunningham & Meiers, 1997).

Ora-5 (Mchenry Laboratories, Edna, TX, USA) is a commercially available I2-KI/CuSO4-based oral disinfectant composed of 0.3% iodine, 0.15% potassium iodide, and 5.5% copper sulfate. Meiers and Shook (1996) have stated that Ora-5 adversely affects the SBS of composite to dentin when the Syntac adhesive system is used. However, they have not reported negative effects with the Tenure adhesive system (Meiers & Shook, 1996).

2.7.5 Light Amplification by Stimulated Emission of Radiation (LASER):

Multiple kinds of lasers with different applications for hard tissues, soft tissues, light-curing, tooth-whitening, and disinfecting have been used in dental practice (Nazemisalman et al., 2015). These lasers include neodymiumdoped yttrium aluminum garnet (Nd:YAG), (Er:YAG) Erbium yttrium aluminum garnet, neodymium-doped yttrium aluminum perovskite (Nd:YAP), diode, argon (KTiOPO4), erbium chromiumdoped yttrium scandium gallium garnet (Er, Cr:YSGG), and potassium-titanyl-phosphate (KTP) (Nazemisalman et al., 2015). Multiple studies have reported that the use of Er, Cr:YSGG or KTP lasers does not adversely affect the bond strength of the restoration (Arslan et al., 2012; Jhingan et al., 2015; Siso et al., 2009).

2.7.6 Ozone (O3):

Ozone (O₃) is a pale, non-stable gas, naturally produced by the photodissociation of oxygen into activated oxygen atoms, which then react with further oxygen molecules (Naik et al., 2016). Ozone is known to be a strong oxidizer. Hence, it possesses antibacterial activities by disrupting the cell wall and cytoplasmic membrane of bacteria and therefore destruction of the microorganism (Kapdan & Öztaş, 2015). In dental applications, O₃ can be used in one of three forms: Gaseous, water, or oil. Ozone was first used as a disinfectant in clinical practice in the 1920s by Dr Parr. In 1950, Dr Fisch was the first to use ozonated water for dental procedures in Germany (Naik et al., 2016).

Several studies have reported the effect of O₃ on the bond strength of dental composites. Some of these studies have evaluated the effect of O₃ pretreatment on the enamel bond, as in the case of pit-and-fissure sealants, and have reported no effects on enamel bond strength or microleakage (Cadenaro et al., 2009; Celiberti et al., 2006).

On dentin, most of the studies have reported no effect of O3 on the bond strength, regardless of the type of adhesive systems used (Arslan et al., 2011; Cadenaro et al., 2009; Kapdan & Öztaş, 2015).

Chapter 3

AIM OF THE STUDY

3. AIM OF THE STUDY

3.1 Aim of the study

The aim of this in-vitro study is to evaluate the effect of three different cavity disinfectants on shear bond strength (SBS) of resin composite restorative material to dentine using two different adhesive techniques (total-etch and self-etch approach).

3.2 Objectives of the study:

- I)** To assess and determine the shear bond strength (SBS) of resin composite to dentine surface treating with three different cavity disinfectants.
- II)** To assess and evaluate the effect of two types of adhesive bonding techniques; (Total-etch and Self-etch) among each type of cavity disinfectant on the shear bond strength (SBS) of resin composite to dentine.
- III)** To investigate the failure pattern of the testing specimens.

Chapter 4

MATERIALS AND METHODS

4.MATERIALS AND METHODS

4.1 MATERIALS:

- Materials used in the study are three cavity disinfectants, namely:
 1. Chlorhexidine gluconate (Cariax Gigival mouth wash).
 2. Sodium hypochlorite (Sword).
 3. Benzalkonium chloride-based disinfectant (Tantum Verde mouth wash).
 4. Normal saline as a control.
- 8th generation universal bonding agent, (G-Premio Bond) which could be used in:
 1. Total-etch adhesive system approach.
 2. Self-etch adhesive system approach.
- One resin composite restorative material: Nano-hybrid composite resin (Nexcomp).

Material composition, specification and manufacturing are summarized in **Table 4.1**.

Table 4.1: Material composition and specification used in the study.

Material Classification	Main components	Manufacturer	Applications
<i>Otsuka.normal saline IV infusion.</i>	Sodium chloride intravenous infusion BP (0.9% w/v).	Otsuka Pharmaceutical India Private Limited, India.	Soaking the specimens in the attended solution for one minute.
<i>Cariax Gingival Kin Mouth wash.</i>	0.12% Chlorhexidine-gluconate.	Laboratories KIN S.A- Barcelona- Spain	
<i>SWORD® medical group.</i>	Sodium hypochlorite; 5% chlorine-based disinfectant.	Molla Gurani Mah.Oguzhan,Istanboul-Turkey.	
<i>Tantum Verde Mouth wash & gargle.</i>	0.15% Benzalkonium chloride-based disinfectant.	EPICO international pharmaceut. Egypt.	
<i>Fine Etch 37.</i>	37% phosphoric acid.	Ivoclar Vivadent AG, Schaan, Liechtenstein.	Etching was performed for 15 seconds then washing for 15-20 seconds.
<i>G-Premio Bond.</i>	Acetone (25–50%), 2-hydroxy-1, 3-dimethacrylaxopropane (10–20%), methacryloyloxydecyl dihydrogen phosphate (5–10%), 2, 2-ethylenedioxydiethyl dimethacrylate (1–5%), diphenyl (2, 4, 6-trimethylbenzoyl) phosphine oxide (1–5%), 2, 6-di-tert-butyl-p-cresol (<0.5%).	GC Corporation, Tokyo, Japan.	A universal, 8 th generation bonding agent, which could be used in total-etch or self-etch approaches. cured for 10 seconds.
<i>Nexcomp Nano-hybrid.</i>	Resin matrix: Bis-GMA, UDMA, Bis-EMA Filler: zirconia/silica 5–75 nm (filler), 0.6–1.4 µm (cluster) 59.5vol%–78.5wt%.	META BIOMED, Korea (NXC 1805281).	A layer of ±2 mm light cured for 20 s at 600 mW/cm ² .
TEGDMA: Triethylene glycol dimethacrylate, UDMA: Urethane dimethacrylate, Bis-EMA: Bisphenol-A ethoxylated dimethacrylate, Bis-GMA: Bisphenol-A glycidyl dimethacrylate.			



Figure 4.1: Normal saline.



Figure 4.2: SWORD, Sodium hypochlorite.



. **Figure 4.3:** Cariax Gigival, Mouth wash.



Figure 4.4: Tantum Verde Mouth wash & gargle.



Figure 4.5: FineEtch 37 (37% phosphoric acid).



Figure 4.6: A universal, 8th generation bonding agent; (G-Premio Bond).



Figure 4.7: Nano-hybrid composite; (Nexcomp).

4.2 METHODS:

This in-vitro study was conducted following Ertuğrul Ercan *et al.*, methodology published in 2009.(Ercan et al., 2009) This part explained how the experiment was performed in the following sections:

- Specimen preparation.
- Specimens grouping.
- Application of disinfectants and resin composite build up.
- Shear bond strength testing procedure (SBS).
- Assessment of the mode of failure.
- Statistical analysis.

4.2.1 Specimen preparation:

The inclusion criteria included caries free human third molar teeth. However decayed, or damaged teeth during the extraction, and also those teeth that were congenitally affected such as enamel hypoplasia or amelogenesis, dentinogenesis imperfect were excluded.

Eighty extracted caries-free human third molar teeth were collected from different dental practice were scaled with ultrasonic scaller and cleaned of calculus and soft tissue debris by using pumice and rubber cups. After that the teeth were kept in normal saline (0.9 % isotonic saline) in the fridge till the time of using which was no longer than one month after extraction.

Each tooth was embedded in a mould filled with cold-cure acrylic resin up to a level of 0.5mm from the cemento-enamel junction. The roots were embedded inside a cylindrical-shaped mould filled with self-cured acrylic resin (ACROSTONE, EGYPT), till the cervical line with the exposed occlusal surface plane being parallel to the floor. (**Figure 4.8: a, b, and c**)

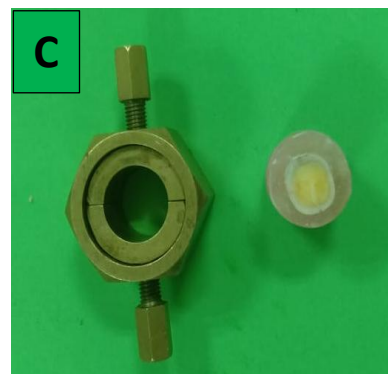
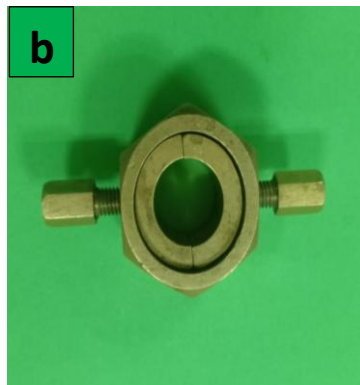
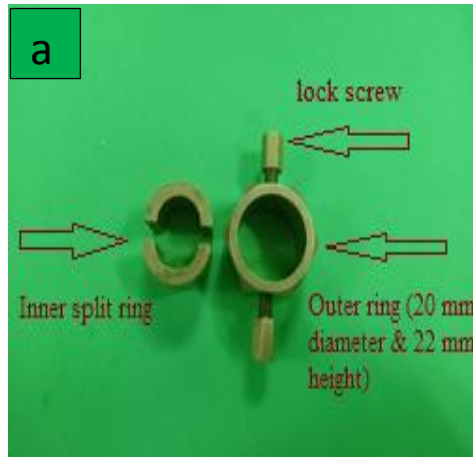


Figure 4.8 (a, b, c): Split brass mould and mounting of extracted teeth in acrylic block:

- a) Non-assembled split brass mould for acrylic block construction.
- b) Assembled Split brass mould for acrylic block construction
- c) Split brass mould with tooth embedded acrylic block after construction.

After completing the polymerization of the acrylic resin, the tooth in the set acrylic resin was removed from the mold and the occlusal enamel of the tooth was cut off perpendicular to the long axis of teeth with a low-speed motorized diamond disk (Ernst Leitz GmbH, Wetzlar, Germany) under water coolant to expose mid-coronal dentine. (Figure 4.9 and 4.10.)

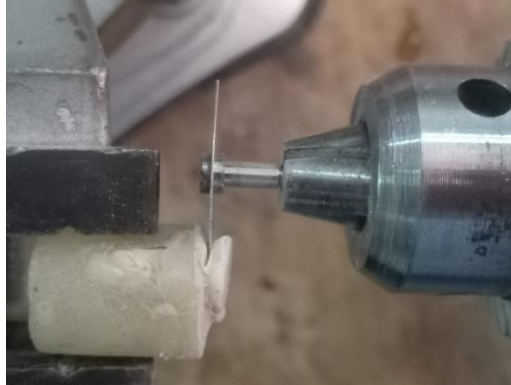


Figure 4.9: Low speed motorized diamond coated disc during cut off the enamel surface to expose mid-coronal dentine.



Figure 4.10: specimens after cutting off the enamel and expose the mid coronal dentin.

4.2.2 Specimens grouping:

Eighty caries-free human third molars were used in this study. Details of number and distribution of testing groups are illustrated in **Figure 4.11**.

The specimens were randomly divided into four main groups (Gp1, 2, 3, and 4), each group contains twenty teeth ($n=20$) according to the proposed dentin surface treatment, as following:

- Gp1: Dentine surface without treatment (normal saline) as a control group (*Otsuka. INDIA*), ($n=20$).
- Gp2: Dentine surface treated with 0.12% chlorhexidine gluconate (*Cariax, Barcelona, Spain*), ($n=20$).
- Gp3: Dentine surface treated with 5% sodium hypochlorite (*SWORD, Istanbul, Turkey*), ($n=20$).
- Gp4: Dentine surface treated with 0.15% benzalkonium chloride (*Tantum Verde, Egypt*), ($n=20$).

Then each group (Gp1-Gp4) was further divided into two subgroups ($n =10$ per subgroup) according to the adhesive approach (dentin bonding agent) as following:

- (TE) Total-etch adhesive system.
- (SE) Self-etch adhesive system.

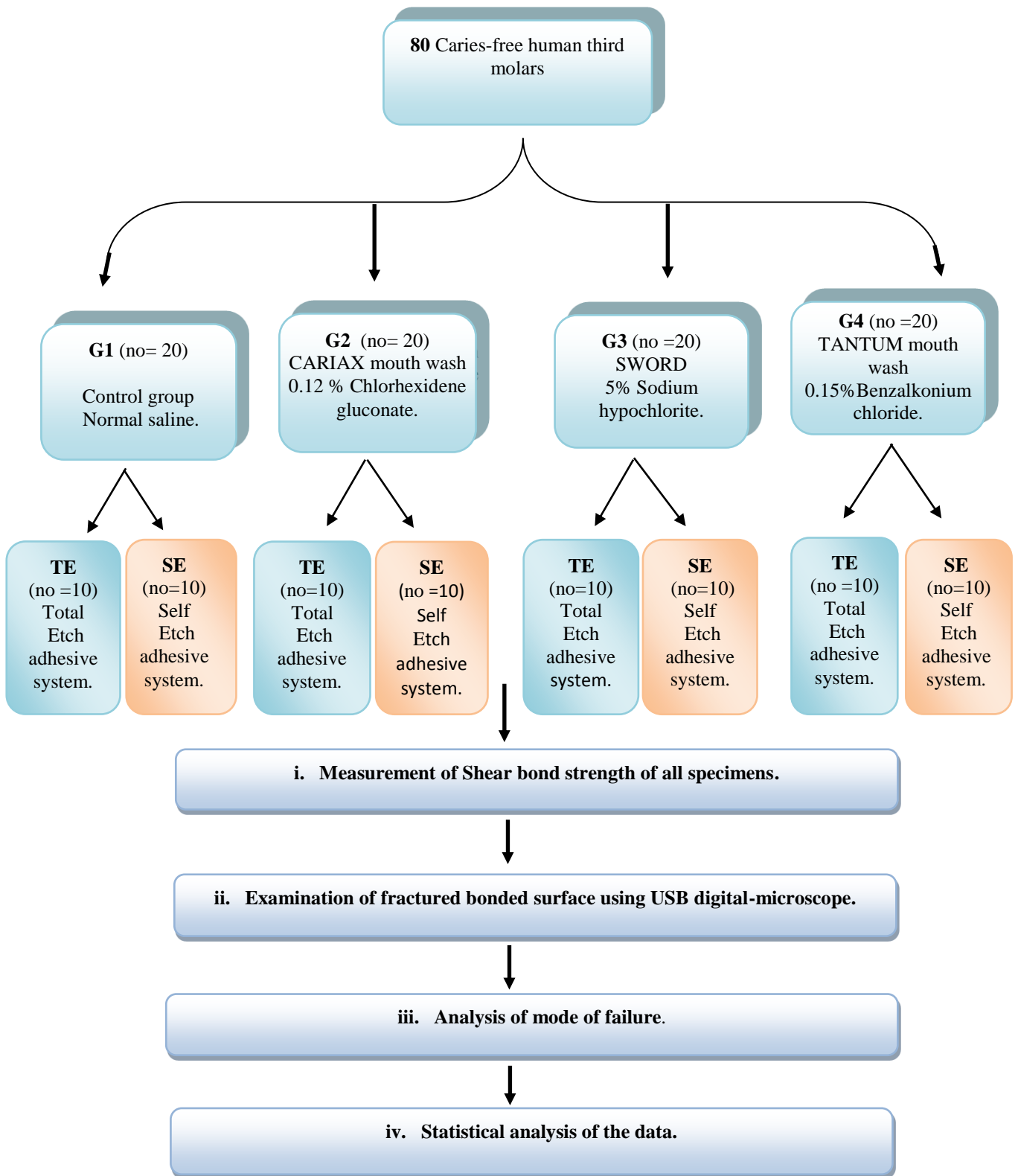


Figure 4.11: Flow chart illustrates number and distribution of testing groups and steps of procedures.

4.2.3 Application of disinfectants and resin composite build up:

The disinfectant solution in every group were applied into the dentine surface through soaking the specimens in the intended disinfectant solution for one minute (**Figure 4.12**), then removed from the disinfectant solution and rinsed with water for 10 seconds, and gently dried with air for 10 seconds.



Figure 4.12: Four groups of specimens are soaking in four different disinfectant solutions.

Each group was then randomly divided into two subgroups of ten teeth each (n=10) according to the adhesive approach used. Ten specimens were bonded with the total-etch approach and other ten specimens were bonded using the self-etching approach. In the total-etch approach, the pretreated dentinal surface was treated with 37% phosphoric acid (Fine Etch 37, Ivoclar Vivadent AG, Schaan, Liechtenstein) for 15 seconds (**Figure 4.13**), rinsed with water for 15-20 seconds, and gently dried with absorbent paper. In the self-etching approach group; the pretreated dentinal surface was treated with the application of the (G-Premio Bond) without prior application of 37% phosphoric acid. This dentin bonding agent (G-Premio Bond) can be used as total-etch or as a self-etch dentin bonding agent as recommended by manufacture instructions for use.

The bonding agent was applied by using a fully saturated brush tip and then gently air-dried for 5 seconds and light-cured for 10 second (**Figure.4.14, 4.15 and 4.16**). Curing of bonding agent was done using a 1,000 mW/cm² strength LED

(Light Emitting Diode-Elipar, 3M ESPE, Germany) light curing unit (**Figure 4.17**).
The light output was checked regulating using a radiometer for each group.



Figure 4.13: Application of acid etching to dentin surface.

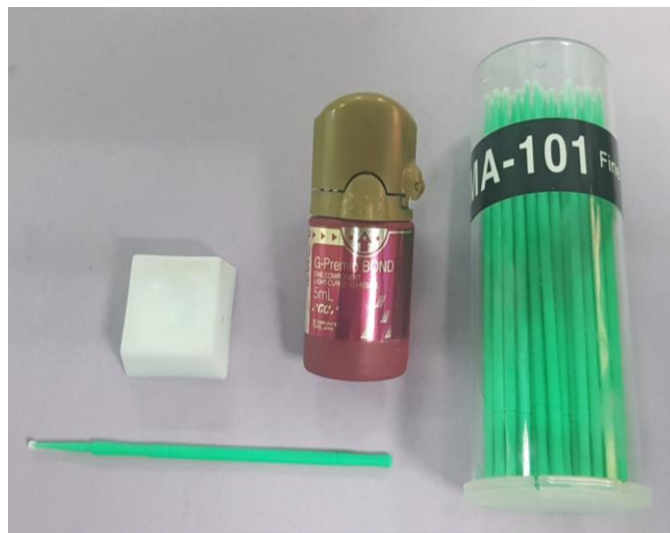


Figure 4.14: A universal, 8th generation bonding agent (G-Premio Bond), dispensing well and brush applicator.



Figure 4.15: G-Premio Bond bonding agent in the dispensing well.

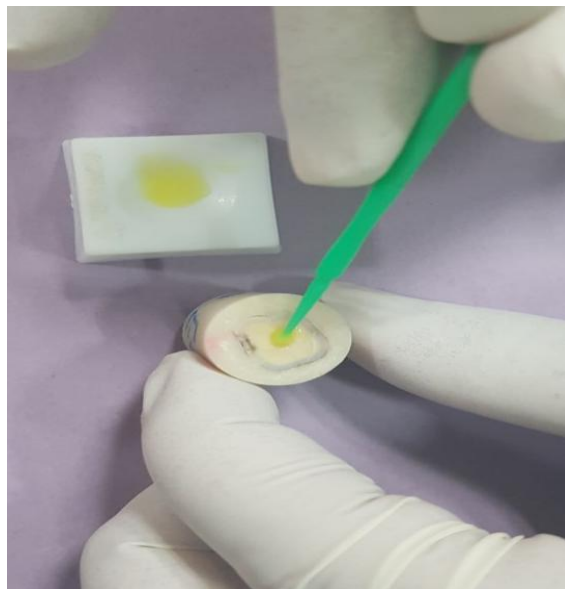


Figure 4.16: Application of the (G-Premio Bond) bonding agent to dentin surface.

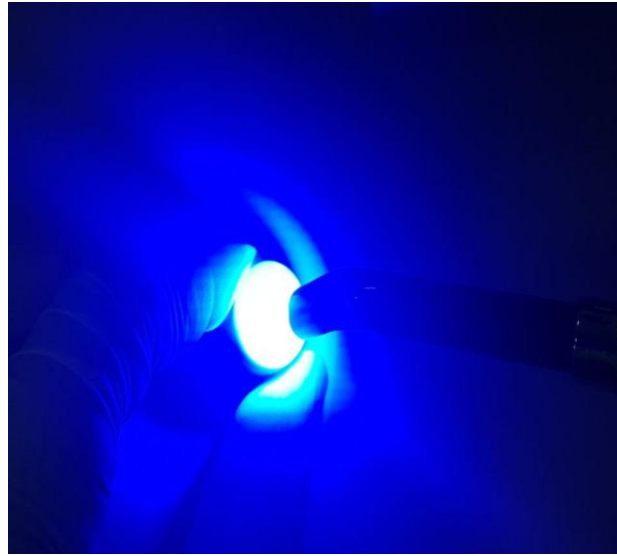


Figure 4.17: Curing of bonding agent after application to dentin surface.

After the application of the adhesive bonding agent, the Nanohybrid resin composite (Nexcomp Nano-hybrid, META BIOMED, Korea) was carefully applied to the treated dentine surface by placing the material into cylindrical-shaped Teflon tube with an internal diameter of 3 mm and a height of 3 mm. Composite was placed incrementally. Each increment should not exceed 2mm in thickness (**Figure 4.18**). Each incremental layer was light cure for 20 seconds with the same light curing unit. The specimens were then stored for 24 hours in humid environment, in incubator at 37 °C in 100% humidity.



Figure 4.18: Nanohybrid resin composite application to the treated dentine surface by placing the material into cylindrical-shaped Teflon tube.

4.2.4 Shear bond strength testing procedure (SBS):

After storing in an incubator at 37°C in 100% humidity for 24 hours, the circular interface shear test was designed to evaluate the bond strength. All specimens were individually and horizontally mounted on a computer-controlled materials testing machine (Model 3345; Instron Industrial Products, Norwood, USA) with a load cell of 5 kN. Specimens were secured to the lower fixed compartment of testing machine by tightening screws. Shearing test was done by compressive mode of load applied at liner substrate-resin interface using a mono-bevelled chisel shaped metallic rod attached to the upper movable compartment of testing machine traveling at cross-head speed of 0.5 mm/min. The load required to de-bonding was recorded in Newton (N) (**Figure 4.19**). The data were recorded using computer software (Bluehill Lite; Instron Instruments).

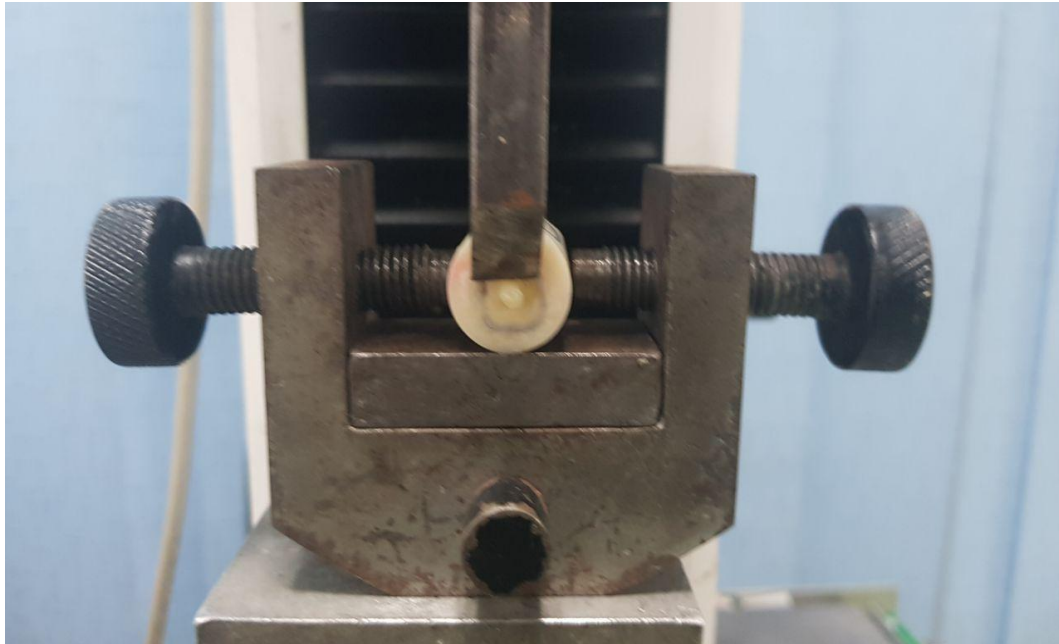


Figure 4.19: Shear bond strength test: Specimen mounted onto testing machine during testing procedure.

4.2.4.1 Shear bond strength calculation:

The load at failure was divided by bonding area to express the bond strength in Mega Pascale (MPa):

$$\tau = P / \pi r^2$$

Where; τ =shear bond strength in MPa,

P =load at failure (N)

π =3.14 and r =radius of resin composite disc (mm)

4.2.5 Assessment of the mode of failure:

The mode of failure is an important aspect of bond strength tests. After bond testing procedure, seven specimens were randomly selected from each group for examination of the fractured bonded surface as a representative for each testing group. The procedure was performed using a USB digital-microscope (U500x Digital Microscope, Guangdong, China), at magnification of x35. The images were captured (**Figure 4.20**) and transferred to an IBM personal computer equipped with the Image-tool software (Image J 1.43U, National Institute of Health, USA) to determine failure mode pattern according to the following categorization;

- Adhesive failure: means the failure/fracture located at the adhesive interface.
- Cohesive failure: means the failure/fracture located at one of the substrates on either side of the interface, either in the composite or dentine substrate.
- Mixed failure: means a mixture of fracture of adhesive and partially cohesive in dentin/composite within the same fractured surface (Inoue et al., 2001; Rodrigues Junior et al., 2010).

The images were taken with the following image acquisition system:

Digital camera (*U500x Digital Microscope, Guangdong China*) with 3 Mega Pixels of resolution, placed vertically at a distance of 2.5 cm from the specimens. The angle between the axis of the lens and the sources of illumination is approximately 90°. Illumination was achieved with 8 LED lamps (Adjustable by Control Wheel), with a color index close to 95 %. The images were taken at maximum resolution (2272 1704 pixels) and connected with an IBM compatible personal computer using a fixed magnification of 90X. The images were recorded with a resolution of 1280 × 1024 pixels per image.

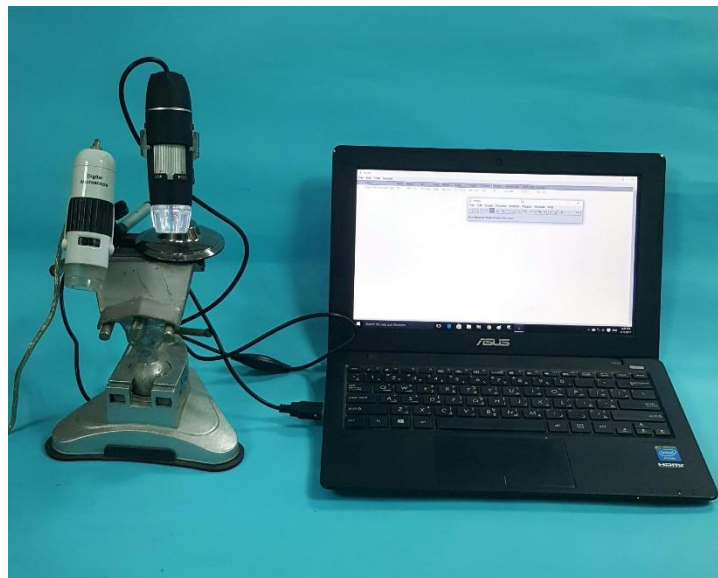


Figure 4.20: USB digital-microscope.

4.2.6 Statistical analysis:

Data was analyzed using SPSS software version 25. Data was first uploaded on excel sheet and grouped according to the type of disinfectant solution. Comparisons of average SBS was conducted using one-way analysis of variance ANOVA test and independent sample t test. All statistical tests were conducted at 0.05 significance level.

Chapter 5

RESULTS

5. RESULTS:

This in-vitro study was conducted to evaluate the effect of three different cavity disinfectants on shear bond strength (SBS) of resin composite restorative material to dentin surface treated with total-etch and self-etch dentin bonding approaches. The statistical analysis of data was performed and the results were as follow:

5.1 Result of shear bond strength (SBS) of resin composite to dentine surface treated with three different cavity disinfectants:

Graph 5.1 shows comparison of average shear-bond-strength (SBS) of resin composite to dentine surface treated with three different cavity disinfectants. The average SBS for the control group was 7.58 ± 0.85 , while the average value of SBS were; Sword (10.70 ± 3.83), Cariax (10.73 ± 3.64), and Tantam (13.39 ± 7.59). It is clear from the **graph 5.1** that SBS is higher for disinfectant groups than that for control group (normal saline). The SBS for Tantam group was higher than that of Sword and Cariax groups ($p=0.002$). (**Figure 5.1**) The raw data of bond strength of all tested groups are shown in (**appendix I-IV**).

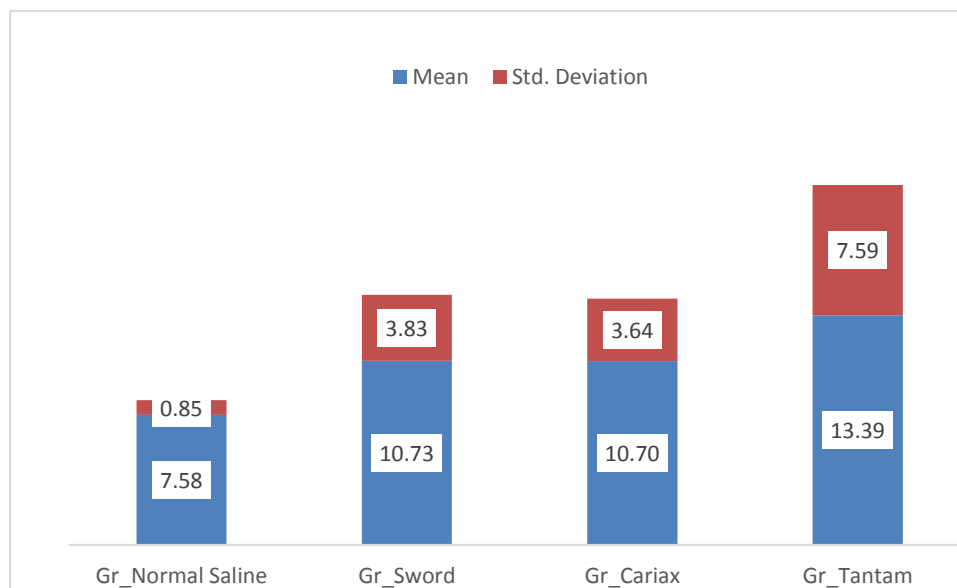


Figure 5 1: Comparison of average shear-bond-strength (SBS) by disinfectant group.

Graph 5.2 shows comparison of average shear-bond-strength (SBS) of resin composite to dentine according to the type of adhesive bonding approach (self-etch VS total-etch). The average values for SBS were 9.4 ± 3.76 for self-etch and 11.79 ± 5.79 for total-etch. The statistical analysis using independent sample t test demonstrated that SBS was higher among total-etch group as compared to self-etch group. This difference was statistically significant ($p=0.031$). (**Figure 5.2**)

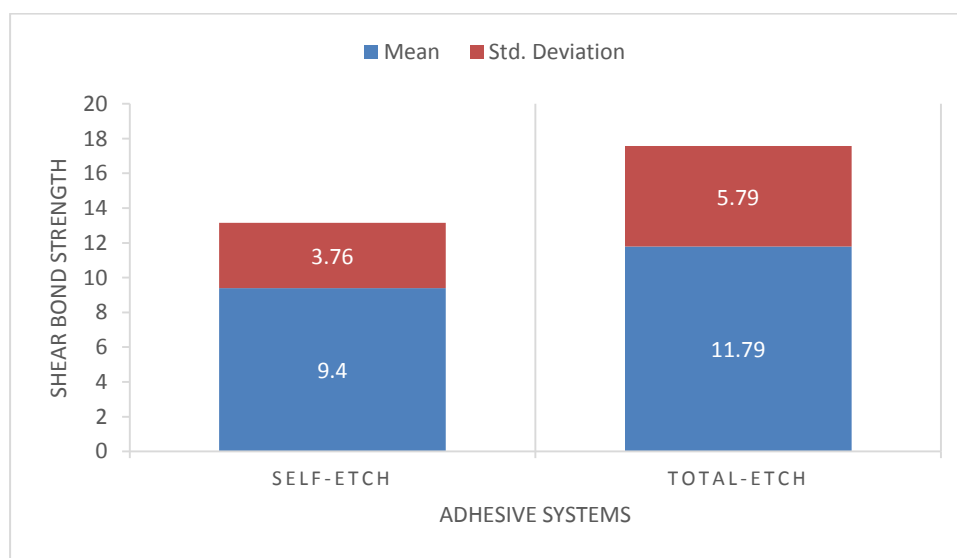


Figure 5.2: Comparison of average shear-bond-strength (SBS) by type of adhesive system.

5.2 Assessment and evaluation of the effect of two types of adhesive bonding approaches; (Total-etch and Self-etch) among each type of cavity disinfectant on shear bond strength of resin composite to dentin:

Graph 5.3 and Table 5.1 present the comparison between self-etch and total-etch adhesive approaches according to different types of cavity disinfectants. For control group, the difference was negligible with nearly equal values for mean and standard deviation ($p=0.717$). SBS for self-etch was 7.51 ± 0.93 and for total etch was 7.65 ± 0.80 . For the three types of disinfectants, total-etch adhesive approach showed higher SBS with Tantam group ($p=0.000$), and Sword group ($p=0.710$). On the other hand, self-etch demonstrated statistically significant higher SBS with Cariax group ($p=0.031$). The highest value of SBS was observed in Tantum with total-etch (19.49 ± 6.11), followed by Cariax with self-etch adhesive approach (12.42 ± 4.51),

then Sword with total etch (11.07 ± 3.45). The lowest value of SBS was reported with self-etch and Tantam (7.29 ± 1.25).

Table 5.1: Comparison of average shear-bond-strength (SBS) by type of adhesive system.					
Group		N	Mean	Std. Deviation	P value
Gr-Normal saline	Self-Etch	10	7.51	0.93	0.717
	Total-Etch	10	7.65	0.80	
Gr_SWORD	Self-Etch	10	10.40	4.33	0.710
	Total-Etch	10	11.07	3.45	
Gr_Cariax	Self-Etch	10	12.42	4.51	0.031
	Total-Etch	10	8.99	1.03	
Gr_Tantam	Self-Etch	10	7.29	1.25	0.000
	Total-Etch	10	19.49	6.11	

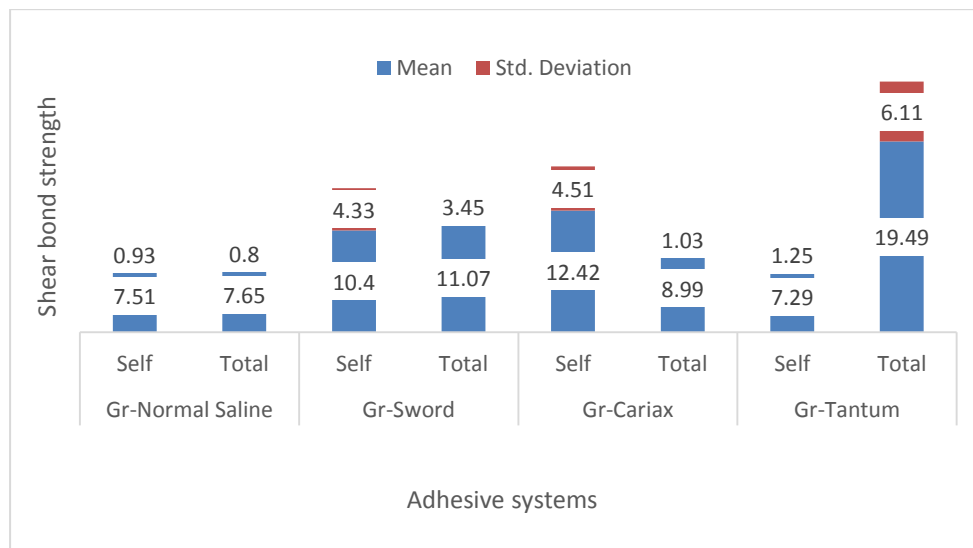


Figure 5.3: Comparison of average shear-bond-strength (SBS) by type of adhesive System.

5.3 Result of the failure pattern of the testing specimens:

Examination of fractured bonded surfaces of all tested specimens revealed that the mode of failure was either mixed or adhesive failure pattern. Examples of the adhesive and mixed failure patterns are shown in **Figure 5.4 (a and b)** and (**appendix IX- XVI**).

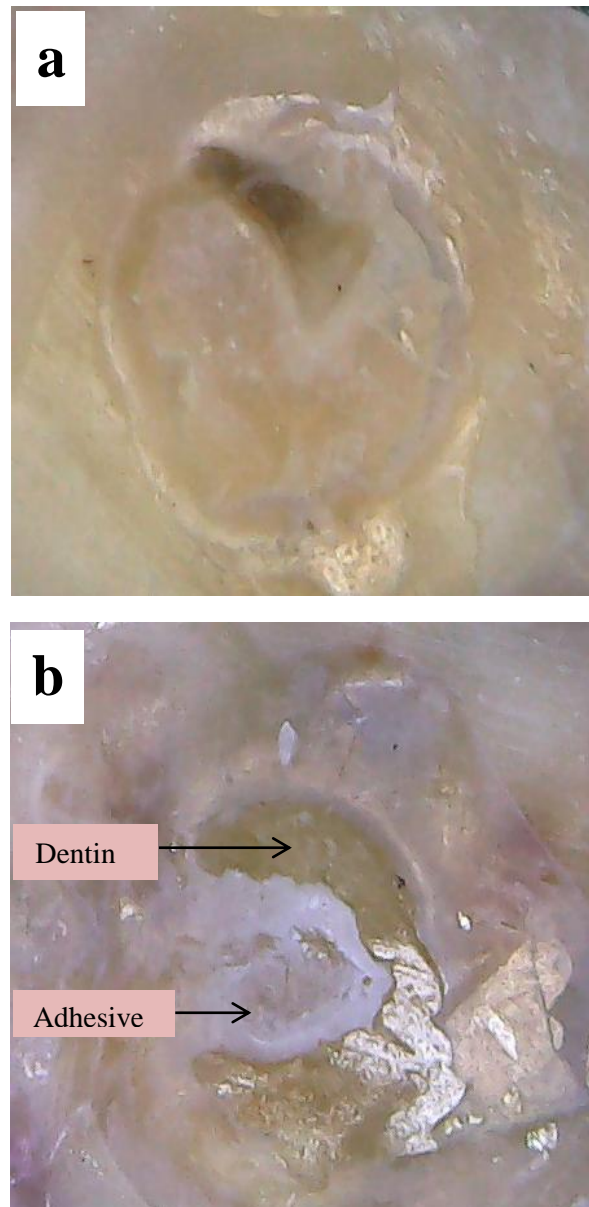


Figure 5.4: Failure patterns: a) adhesive failure pattern. b) mixed failure pattern.

In **figure 5.4 (a)**: the failure located at the adhesive interface, where the adhesive material covers the entire dentin surface. Whereas **figure 4.5 (b)** shows a mixture of failure patterns in the adhesive materials and dentin surface.

Figure 5.5 shows the distribution of frequencies of failure pattern (Mixed VS Adhesive) according to self-etch and total-etch adhesive approaches. It is evident that the mixed failure is higher than adhesive failure in both types. In total-etch adhesive approach, the mixed failure was observed on 17 out of 28 samples. Likewise, in self-etch adhesive approach, the mixed failure was observed on 19 out of 28 samples. The failure pattern data are shown in (**appendix V-VIII**).

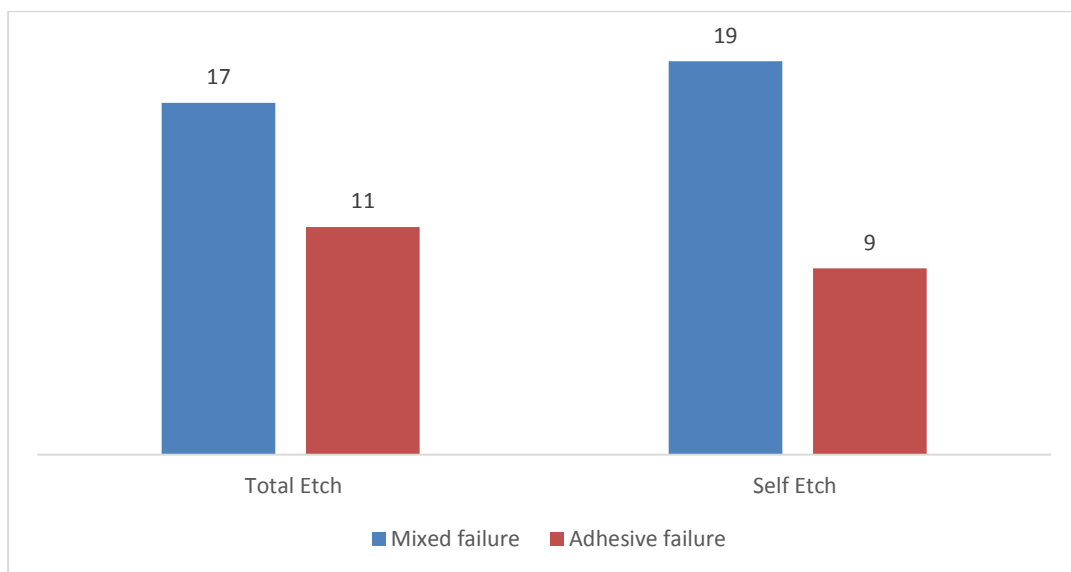


Figure 5.5: The distribution of frequencies of failure pattern (Mixed vs Adhesive) according to adhesive system.

Figure 5.6 shows the distribution of frequencies of failure (Mixed VS Adhesive) according to study group. Overall, mixed failure was higher than adhesive failure except for Tantam group. In Cariax group, there was no adhesive failure with total-etch subgroup. The highest failure was of the mixed type and observed in 7 samples of Cariax group treated with total-etch adhesive approach. The lowest mixed failure was seen in the self-etched Tantam group (1 sample). The most common adhesive failure was reported among self-etch Tantam group (6 samples). The least common adhesive failure was observed in self-etch saline group (1 sample).

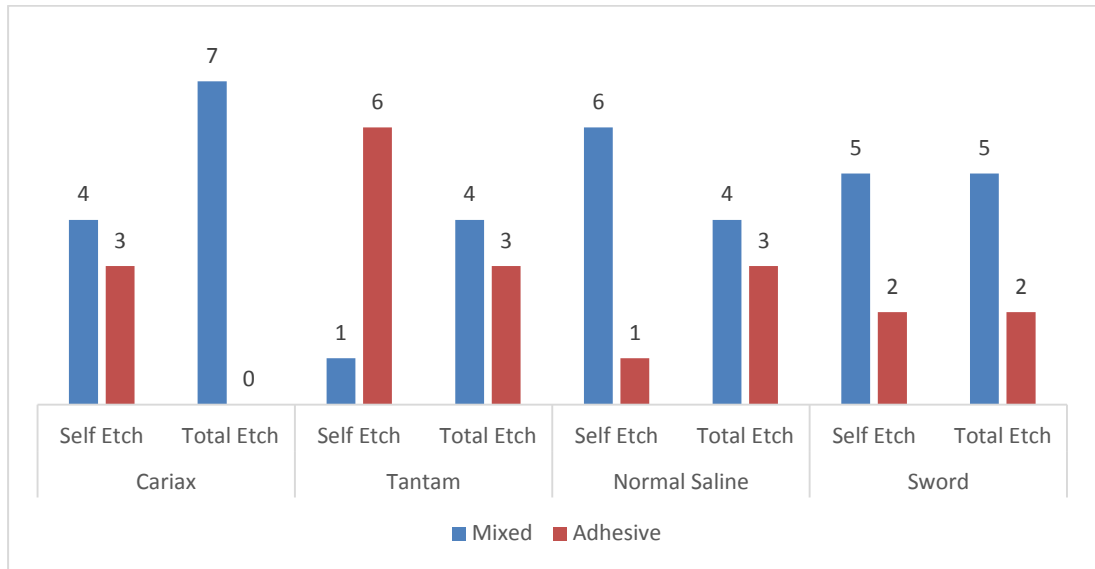


Figure 5.6: The distribution of frequencies of failure (Mixed vs Adhesive) according to study group.

Chapter 6

DISCUSSION

6. DISCUSSION

6.1 Discussion

Cavity prepared for restoration is never completely free from microorganisms/sterile, no matter whichever method of caries removal is followed, always a few microorganisms are left behind in cavity. These microorganisms may be viable and are capable of causing secondary caries in presence of microleakage, hence leading to failure of treatment (Hiraishi et al., 2009). Sterilization of prepared cavity is one of G.V.Black's instructions. He has advocated surgical sterilization of dentinal walls, however bond strength is multifactorial in nature, having many variables affecting it (Turkun et al., 2006).

Conventional removal of carious tissue and cavity preparation procedures do not guarantee the complete elimination of oral cariogenic bacteria that might be entrapped within the dentin tubules or the smear layer, which may induce secondary caries or pulpal inflammation. Therefore the success in the elimination of bacteria during cavity preparation and prior to the insertion of a restoration may increase the longevity of the restoration (Say et al., 2004). For these reasons, eradication and elimination of the bacteria from the cavity surfaces after caries excavation is of major importance. Disinfectant solutions are commonly used to reduce or eliminate bacteria from the cavity preparations. On the other hand, these disinfectant agents might affect the bonding ability of resin materials to tooth substrate (Ercan et al., 2009).

Interest in the study of antimicrobial agents and their effects on the pulp originated in the early 1970s with Brannstrom and Nyborg , who emphasized the importance of eliminating bacteria remaining on cavity walls, including dentin and enamel, after caries excavation by means of antibacterial agents, and therefore recommended disinfecting the cavity preparation before inserting the restoration (Brännström & Nyborg, 1973). The efficacy of these disinfectant solutions has been reported in a number of studies (Owens et al., 2003; Pappas et al., 2005; Say et al., 2004). Long-term studies have shown that the bond strength of resin-bonded dentin decreased over time due to collagen degradation within the hybrid layer (De Munck et al., 2003; Koshiro et al., 2004; Camila Sabatini & Patel, 2013). The use of cavity disinfectants eliminates the residual bacteria, but a potent problem is that it may affect the bond strength of composite resins (Koshiro et al., 2004).

Therefore, the aim of this in-vitro study was to compare the effect of the most commonly used cavity disinfection materials in dental clinic on shear bond strength of nanohybrid composite resin with either a self-etching or a total etch (etch-and-rinse) dentin bonding system.

In the present study, eighty caries free third molars were sectioned parallel to the occlusal surface to expose mid-coronal dentin. The specimens were randomly divided into four groups of 20 each. In group 1, the specimens were not treated with any cavity disinfectants and served as control. From groups 2 to 4, dentin surfaces were treated with the following cavity disinfectants, respectively; 0.12% chlorhexidine solution (CHX), 5% sodium hypochlorite (NaOCl) and 0.15% benzalkonium chloride (BAC). The specimens were then randomly divided into 2 subgroups including ten teeth each to evaluate the effect of different bonding approaches. Dentin bonding systems were applied to the dentin surfaces and the composite build-up were created. After the specimens were stored in an incubator for 24 hs, the shear bond strength was measured at a crosshead speed of 0.5 mm/min. The bond strength data were analyzed with one way analysis of variance of ANOVA test and independent sample t test.

We selected these three cavity disinfectants because they are different in their composition, popular and preferred by most of the dentists in daily practice and available in the market. The specimens were fabricated to a specific dimension to standardize the specimen preparation. We selected the nanohybrid composite because this is composite has esthetic properties required for anterior restorations and mechanical properties for posterior restoration, most frequently used, available in dental clinic.

Shear bond strength is the resistance to forces that slides restorative material past tooth structure and for that the major dislodging forces at tooth restoration interface have shearing effect (Suresh & Nagarathna, 2011). Evaluation of shear bond strength (SBS) is important as the restoration is subjected to shear stress during mastication, and because of the fact that major dislodging forces at the tooth restoration interface have shearing effect (Suresh & Nagarathna, 2011). Shear bond strength tests have been widely used, mainly because of ease of specimen preparation and simple test protocol.

In the present study we found that treated dentin surface with disinfectant solutions such as (normal saline as control group, chlorhexidine gluconate (CHX),

sodium hypochlorite (NaOCl) and benzalkonium chloride (BAC)), positively affect the shear bond strength (SBS) of resin composite to dentin. Increased the bond strength was observed when used with the total -etching adhesive approach than with the self-etching adhesive approach. In addition, we found that treated the dentin surface with 0.15% Benzalkonium chloride-based disinfectant recorded the highest shear bond strength values followed by 5% sodium hypochlorite and then 0.12% chlorhexidine groups while the control group showed the lowest shear bond strength in total-etch adhesive approach. The self-etch adhesive approach showed the highest shear bond strength values with 0.12% chlorhexidine groups, followed by 5% sodium hypochlorite and then 0.15% Benzalkonium chloride-based disinfectant.

One of the variables that affect the shear bond strength to dentine is cavity disinfectant, in our study we found that bond strength increased with total etch adhesive. In this context Ercan *et al.*,(2009) suggested that the cavity disinfectants such as 2.5% NaOCl and 2% CHX solution decreased the bond strength when used with self-etching bonding system, whereas no adverse effect was observed when used with the etch and rinse bonding system (Ercan *et al.*, 2009). The authors suggested that, it is better to use an etch and rinse adhesive when NaOCl and CHX solutions are used as a cavity disinfectant. However, in this study they used different type of composite restoration and different concentration of disinfectant solutions.

Benzalkonium chloride (BAC) has been commonly used as cavity disinfectant in clinical practice because of its disinfecting action and wettability property (Sharma *et al.*, 2009). It has been stated that after deproteinization of dentine, it may exhibit a very porous structure with many irregularities and anastomoses and hence promote the bond strength, but controversial results still exist (P. Spencer *et al.*, 2000; Y. Wang & Spencer, 2003). As with CHX, BAC has been documented to be an effective MMP inhibitor that may preserve the adhesive bond of the resin restoration to dentin (Almahdy *et al.*, 2012; C Sabatini *et al.*, 2014; Camila Sabatini & Patel, 2013; Sharma *et al.*, 2009). Results of the present study are in line with those of Sharma *et al.*, (2011), who suggested that when benzalkonium chloride-based, and chlorhexidine solutions are used as a cavity disinfectant, an etch-and-rinse bonding system should be preferred. As CHX and BAC have excellent rewetting capacity and a strong affinity to tooth structure, which is thought to improve the bond strength to dentin (Sharma *et al.*, 2011). However in study done by Camila Sabatini & Patel (2013) to evaluate the effects of different concentrations of benzalkonium chloride (BAC) on the

preservation of adhesive interfaces created with two etch-and-rinse adhesives, the authors revealed that the groups treated with 0.5% BAC and 1.0% BAC demonstrated significantly higher shear bond strength than the control group, who concluded that the benzalkonium chloride, at all concentrations, inhibited dentin proteolytic activity, which seems to have contributed to the improved bond stability (Camila Sabatini & Patel, 2013), which is in agreement with our results.

chlorhexidine has been advocated to improve the bond strength and the durability of the resin-dentin bond. One of them is inhibition of Matrix metallo-proteinases (MMPs). The results obtained in current study are in support to the study conducted by Sinha *et al.*,(2016), whom showed that 2% chlorhexidine had improved bond strength as compared to the control group for immediate and delayed SBS (Sinha *et al.*, 2016). In addition, another study done by Boitor *et al.*,(2013), who suggested that application of 2% chlorhexidine prevents hybrid layer degradation and this procedure has a beneficial effect on maintaining bond strength (Boitor *et al.*, 2013). Chlorhexidine showed improved bond strength from that of the control group by blocking of the dentin MMPs activation (Dionysopoulos, 2016; Singla *et al.*, 2011). The previous studies are consistent with study done by Stanislawczuk *et al.*,(2011) , who suggested that the use of CHX was found to be effective to reduce the degradation of dentin bonds over a 2-year (Stanislawczuk *et al.*, 2011). The preserved bond interface associated with the use of CHX can be explained by the inhibitory ability of CHX to the matrix metallo-proteinases (MMPs) found in etched dentin. Matrix metallo-proteinases in dentin has been shown to play a role in the degradation of the unprotected collagen fibrils within the hybrid layer (Almahdy *et al.*, 2012).

With its perfect antibacterial activity, CHX has also been used as a good cavity disinfectant for many years (Bin-Shuwaish, 2016). Because it has a rewetting capacity and a strong affinity to tooth structure (Pilo *et al.*, 2001), it seems that CHX would improve the bond strengths of the adhesive to dentin (Pilo *et al.*, 2001), which is in agreement with our study. However, el-Housseiny and Jamjoum (2001), reported that application of chlorhexidine before acid etching did not significantly affect the bond strength of total etch dentin bonding agent to dentin (El-Housseiny & Jamjoum, 2001). In other studies, it was shown that applying chlorhexidine before acid etching did not significantly affect the bond strength (El-Housseiny & Jamjoum, 2001; Hebling *et al.*, 2005; Perdigao & SWIFT JR, 1998; Say *et al.*, 2004), which are in

contrary with our study. The explanation for this could be due to the fact that chlorhexidine was not washed off the dentin debris remained on the dentin surface, and in the tubules, that may account for decrease in bond strength (Perdigao & SWIFT JR, 1998). These results could be due to the difference in the protocol of applications of cavity disinfectant, concentration of solution, type of dentin bonding agents and composite restoration used.

The NaOCL may be beneficial for adhesive system performance. Although it has been proposed that the dentin substrate after deproteinization as it exhibits a remarkably porous structure with multiple irregularities and anastomoses could promote bond strength. In the current study, the results showed improvement of bond strength of total etch adhesive with 5% NaOCL cavity disinfectant, which was not in line with the study, done by Aries *et al.*, (2005) who found that the 10% NaOCl had no effect on bond strength when the total etch technique was used. The explanation of this could be related to the high concentration of NaOCL used, that had an effect on collagen removal property of NaOCL that promote the bond strength (Arias *et al.*, 2005). Some authors found that this NaOCl pretreatment can affect the hybrid layer and therefore the resulting bond strength adversely (Osorio *et al.*, 2002; Reddy *et al.*, 2013; Sanae Shinohara *et al.*, 2004), while others found that no effects on bond strength (Potter *et al.*, 2013; V. d. P. A. Saboia *et al.*, 2006). However, the effect of NaOCl pretreatment on the bond strength of composite resin is believed to depend on the adhesive system used (Ercan *et al.*, 2009; Fawzy *et al.*, 2008).

Whereas, NaOCl solution can remove the exposed collagen fibrils and smear layer on dentin surface. This surface with rich hydroxyapatite crystals increased the bonding of the adhesive systems (Elkassas *et al.*, 2014). Our results revealed that the highest value of SBS was observed in 0.12% CHX followed by 5% NaOCL with self-etching approach, which is not in line with study done by Reddy *et al.*, (2013) who suggested that pretreatment with 2% CHX and 2% NaOCL, had a negative effect on the shear bond strength of self-etching bonding systems. The explanation of this could be related to concentration of cavity disinfectants used, protocol of disinfection of dentine surface which is the dentine surface treated for 20 seconds and air dried for 10 seconds, and the type of composite material used (microhybrid composite) (Reddy *et al.*, 2013).

The total-etch technique relies on the removal of the smear layer and exposure of the collagen matrix by acid etching step, that improve impregnation of adhesive

and infiltration of the resin monomers through the demineralized interfibrillar spaces (Proença et al., 2007). However, Gwinnett *et al.*, (1992), who examined the effect of two commercially available disinfecting agents (2% CHX and 1% BAC) on the shear bond strength of hybrid composite to dentin using a fourth generation dentin bonding agent, and suggested that these cavity disinfectants had no effect on the bond strength when the etch-and-rinse technique was used (Gwinnett, 1992). The explanation of this result could be related to concentration and duration time of disinfectants (20 seconds), type of composite and adhesive materials used. In addition, from 1992 till recent years, great improvements have occurred in the adhesive dentistry including the physical and mechanical properties of adhesive and resin composite materials. However several studies have reported higher bond strengths of resin composite to dentin when etch-and-rinse adhesive systems, were used rather than with self-etch systems, after CHX and NaOCL pretreatment (Ercan et al., 2009; Mobarak et al., 2010; Sharma et al., 2009), which supported the results of our study.

One of the advantages of self-etching adhesives is that dentin conditioning and priming occur simultaneously, resulting in the formation of a strong void-free hybrid layer (Jacques & Hebling, 2005), while other studies found that etch-and-rinse adhesive showed higher bond strength than self-etch adhesives (Ceballos et al., 2003; Oliveira et al., 2003). In contrast, Giriappa and Chandra (2008) , showed that the self-etching adhesive had higher mean shear bond strength value than total etch adhesive (Giriappa & Chandra, 2008). The smear layer and smear plugs should be dissolved to overcome the main problems during using self-etching adhesives, to assure a better monomer penetration, which enhances the bond strength to dentin (Cardoso et al., 2002). In this context, findings of the present study were not in line with result done by Hassan *et al.*, (2014), who suggested that the surface treatment of dentin with 2% CHX and 4% NaOCL solutions before bonding application has a great effect on shear bond strength between resin composite and dentin surface especially with self-etch adhesive. In their study, groups treated with cavity disinfectants recorded statistically significant higher shear bond strength for self-etch bonding agent than the etch-and-rinse bonding agent (Mohammed Hassan et al., 2014). The explanation for the obtained results could be related to the fact that the self-etching adhesives have higher pH values than the phosphoric acid used and are not rinsed away (Mohammed Hassan et al., 2014), as well as concentration of disinfectants solutions, and duration of time of application. The authors attributed the increased

bond strength to the elimination of collagen layer which was removed by application of NaOCl leading to a better penetration of the adhesive into inter-tubular dentin (Marshall, Yücel, et al., 2001). This increase in bond strength may be also due to removal of smear layer by NaOCl. Complete removal of smear layer might enhance the bonding to dentin as it facilitates the penetration of resin monomer leading to complete infiltration of the demineralized layer by numerous resin tags (Arias et al., 2005).

On the other hand, results of the present study found that the self-etch adhesive demonstrated statistically significant higher SBS with 0.12 % CHX cavity disinfectant group, which is not in line with study done by Suma *et al.*, (2017), who reported that the use of commercially available cavity disinfectants such as 2% chlorhexidine gluconate with self-etch adhesive would significantly lower the SBS of composite to dentin when compared with control group (Suma et al., 2017). The explanation of this result could be related to the residual moisture of the 2% CHX solution, which contaminates the bonded surface and alters the ability of the hydrophilic resin in the self-etch system to seal the dentin (Hiraishi et al., 2009; Singla et al., 2011), concentration of disinfectant solution and protocol of dentin surface treatment (Suma et al., 2017). However, in some studies, the CHX solution exerted an adverse effect on shear bond strength when used with a self-etch adhesive system (Campos et al., 2009; Ercan et al., 2009).

Development of nanocomposite resin, using advanced methacrylate resin has esthetic properties required for anterior restorations and mechanical properties for posterior restoration (Mitra et al., 2003). In this context, regardless of composite type or bonding agent, our results were in line with previous results done by Hassan *et al.*, (2014), who found that the type of resin composite used can affect the recorded shear bond strength values. The highest mean shear bond strength was recorded for nanohybrid composite bonded to dentin specimen using etch-and-rinse adhesive, while the lowest mean shear bond strength was recorded for micro-hybrid composite bonded to dentin specimen using self-etch adhesive (Mohammed Hassan et al., 2014).

Different mechanical tests have been proposed to assess the bonding performance of restorative materials (Shimada et al., 2002). Testing in shear mode is a relatively simple, reproducible and widely accepted test (Suresh & Nagarathna, 2011). Although this mode of testing has been met with some criticism, it is still being used to evaluate the bonding potential of adhesives to dental structure (Suresh &

Nagarathna, 2011). However, regardless to type of adhesive, the current study disagreed with study done by Korkmaz *et al.*,(2010), who reported that the nano-restorative materials tested in their study exhibited significant differences in SBS, even when tested with the same adhesive (Korkmaz et al., 2010), the authors suggested that the highest bond strength was recorded with the nano-composite when the self-etch adhesive was used, meaning that the composite formulation also had an impact on bond strength. Materials containing more resin components in their composition may exhibit improved bond strength performance with evidence of mechanical interlocking (Korkmaz et al., 2010).

In the present study examination of fractured bonded surfaces of all tested specimens revealed that the mixed failure is higher than adhesive failure in both types of adhesive approach, which is disagreed with previous study done by Reddy *et al.*, (2013) who observed that the fractured specimens in the groups treated with CHX and NaOCL presented mostly adhesive failures. The speculation of this could be related to testing methodology, mechanical properties of materials used and size of specimens (2 mm height, 5 mm internal diameter) (Reddy et al., 2013). On the other hand, our results were in agreement with the results of Mohammed Hassan *et al.*, (2014), they reported that the increased percentage of mixed failure for groups of disinfectants (Mohammed Hassan et al., 2014). The increased percentage of mixed failure on groups of disinfectants was attributed to the increased shear bond strength which clearly was reflected by the mode of failure of the bonding system. This result was in agreement with other studies (Dalli et al., 2010; Di Hipólito et al., 2012; Mohammad et al., 2011).

Controversial results still exist from various laboratory results (V. Saboia et al., 2000; Y. Wang & Spencer, 2003). It has been observed that depending on the testing methodology, restorative materials used, protocol of dentin surface treatment, and the adhesive system used.

6.2 LIMITATIONS OF THIS IN-VITRO STUDY

There are several limitations of this in-vitro study; the experiment had been done in laboratory in Cairo-Egypt. Due to Corona pandemic in 2020, several difficulties have been experienced, difficulty in traveling due to closure of airport, and difficulty in sample collection due to closure of clinics in Corona pandemic, that delayed starting the experiment.

Chapter 7

CONCLUSIONS AND RECOMMENDATIONS

7. CONCLUSIONS AND RECOMMENDATIONS

7.1 Conclusion:

Within the limitation of this in-vitro study, it can be concluded that:

- The surface treatment of dentin with the three-cavity disinfectant, before bonding positively affects the shear bond strength (SBS) between resin composite and dentin.
- All tested disinfectant solutions, i.e., 0.12% chlorhexidine gluconate, 5% sodium hypochlorite and 0.15% benzalkonium chloride, significantly increased the bond strength to dentin especially with total-etch approach compared with self-etch adhesive approach.
- In total-etch adhesive system, the highest value of SBS was observed with application of 0.15% benzalkonium chloride, followed by 5% sodium hypochlorite, and the lowest value of SBS was reported with 0.12% chlorhexidine gluconate cavity disinfectant.
- In self-etch adhesive system, the highest value of SBS was observed with application of 0.12% chlorhexidine, followed by 5% sodium hypochlorite, and the lowest value of SBS was reported with 0.15% benzalkonium chloride.
- The examination of fractured bonded surface of all tested specimens, revealed that the mode of failure was either mixed or adhesive failure pattern.
- Regarding the self-etch and the total-etch adhesive bond approaches, it was observed that the mixed failure pattern was higher than the adhesive failure in both types, except for the benzalkonium chloride group.
- The highest mixed failure mode was reported in total-etch, chlorhexidine group, and the lowest mixed failure was reported with self-etch benzalkonium chloride group.

7.2 Recommendations:

- The application of disinfectants after cavity preparation and before the restoration is gaining acceptance. This study opens the perspective further research of the use of cavity disinfectants in dentistry as behaves in the oral environment after cavity preparation.
- Future, long term clinical study is recommended to evaluate performance of cavity disinfectants with different concentration and different duration of application time on shear bond strength of composite to dentin.
- Further studies to evaluate the effect of the cavity disinfectants on shear bond strength by using different types of resin composites.

Chapter 8

REFERENCES

8. REFERENCES

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APPENDICES

APPENDIX I

Shear bond strength values in Sword group

Gr_Sword	Self-etch				total-etch		
	Shear Bond Strength (Mpa)				Shear Bond Strength (Mpa)		
2	5.5			1	8.8		
3	10.9			5	9.8		
4	9.8			6	7.4		
9	6.5			7	9.1		
10	19.9			8	18.3		
12	8.6			11	14.5		
13	11.02			14	10.7		
	7.65				8.1		
	8.76				9.9		
	15.4				14.05		
Average	10.403			Average	11.065		

APPENDIX II

Shear bond strength values in Cariax group.

Gr_Cariax	Self-etch				total-etch		
	Shear Bond Strength (Mpa)				Shear Bond Strength (Mpa)		
16	5.6			15	9.23		
18	14.8			17	9.1		
20	19.6			19	6.9		
23	18.9			21	10.6		
24	9.6			22	10.3		
26	9.7			25	8.7		
28	7.8			27	8.2		
	12.6				8.75		
	13.35				9.25		
	12.2				8.9		
Average	12.415			Average	8.993		

APPENDIX III

Shear bond strength values in Normal saline group

Gr_Normal Saline						
	Self-etch				total-etch	
	Shear Bond Strength (Mpa)				Shear Bond Strength (Mpa)	
29	8.6			31	7.1	
30	7.6			35	8.6	
32	7.8			36	6.6	
33	6.4			37	6.8	
40	8.6			38	8.6	
34	5.9			39	7.9	
41	7.1			42	8.02	
	8.2				6.85	
	6.75				7.41	
	8.1				8.6	
Average	7.505			Average	7.648	

APPENDIX IV

Shear bond strength values in Tantium group

Gr_Tantam						
	Self-etch				total-etch	
	Shear Bond Strength (Mpa)				Shear Bond Strength (Mpa)	
43	5.9			44	8.6	
46	6.8			45	25.5	
47	5.8			50	28.3	
49	8.11			51	26.3	
48	6.02			52	19.8	
55	6.991			53	17.2	
56	9.5			54	12.4	
	7.7				18.45	
	8.805				19.35	
	7.25				18.95	
Average	7.2876			Average	19.485	

APPENDIX V

Mode of failure in Sword group

Gr_Sword	Self-etch			total-etch		
	Shear Bon	Failure mode		Shear Bon	Failure mode	
2	5.5	Mixed		1	8.8	Mixed
3	10.9	adhesive		5	9.8	Mixed
4	9.8	Mixed		6	7.4	adhesive
9	6.5	adhesive		7	9.1	adhesive
10	19.9	Mixed		8	18.3	Mixed
12	8.6	Mixed		11	14.5	Mixed
13	11.02	Mixed		14	10.7	Mixed
	7.65				8.1	
	8.76				9.9	
	15.4				14.05	
Average	10.403		Average	11.065		

APPENDIX VI

Mode of failure in Cariax group

Gr_Cariax	Self-etch			total-etch		
	Shear Bond Strength (Mpa)			Shear Bon	Failure mode	
16	5.6	Mixed		15	9.23	Mixed
18	14.8	Mixed		17	9.1	Mixed
20	19.6	adhesive		19	6.9	Mixed
23	18.9	Mixed		21	10.6	Mixed
24	9.6	Mixed		22	10.3	Mixed
26	9.7	adhesive		25	8.7	Mixed
28	7.8	adhesive		27	8.2	Mixed
	12.6				8.75	
	13.35				9.25	
	12.2				8.9	
Average	12.415		Average	8.993		

APPENDIX VII

Mode of failure in Normal saline group

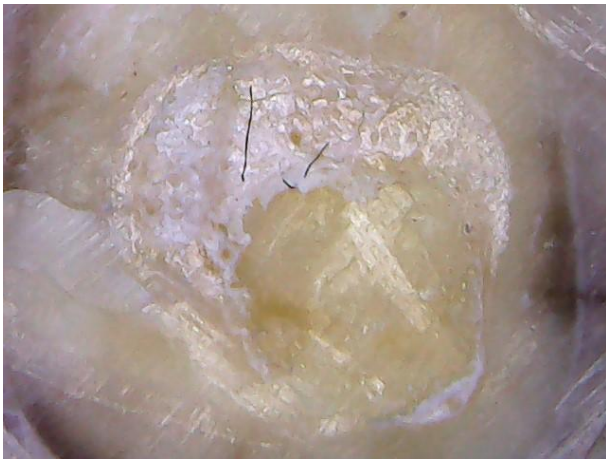
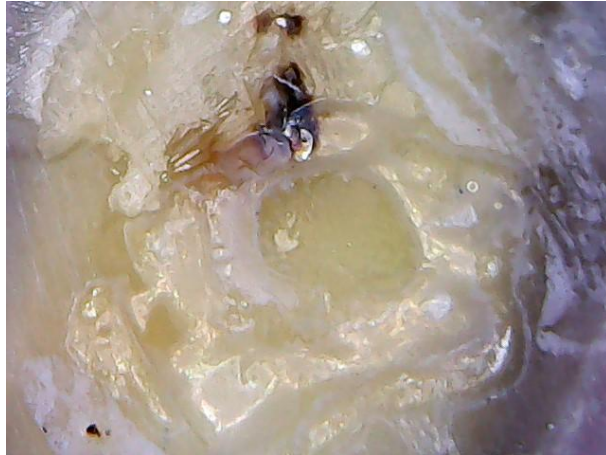
Gr_Normal Saline							
	Self-etch				total-etch		
	Shear Bon	Failure mode			Shear Bon	Failure mode	
29	8.6	Mixed		31	7.1	Mixed	
30	7.6	Mixed		35	8.6	adhesive	
32	7.8	Mixed		36	6.6	Mixed	
33	6.4	Mixed		37	6.8	adhesive	
40	8.6	Mixed		38	8.6	Mixed	
34	5.9	Mixed		39	7.9	Mixed	
41	7.1	adhesive		42	8.02	adhesive	
	8.2				6.85		
	6.75				7.41		
	8.1				8.6		
Average	7.505			Average	7.648		

APPENDIX VIII

Mode of failure in Tantum group

Gr-Tantam							
	Self-etch				total-etch		
	Shear Bond Strength (Mpa)				Shear Bond Strength (Mpa)		
43	5.9	adhesive		44	8.6	adhesive	
46	6.8	adhesive		45	25.5	adhesive	
47	5.8	adhesive		50	28.3	adhesive	
49	8.11	Mixed		51	26.3	adhesive	
48	6.02	Mixed		52	19.8	adhesive	
55	6.991	Mixed		53	17.2	adhesive	
56	9.5	Mixed		54	12.4	Mixed	
	7.7				18.45		
	8.805				19.35		
	7.25				18.95		
Average	7.2876			Average	19.485		

APPENDIX IX



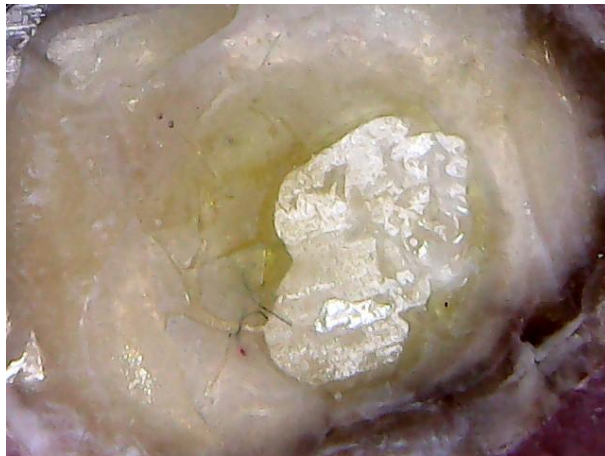
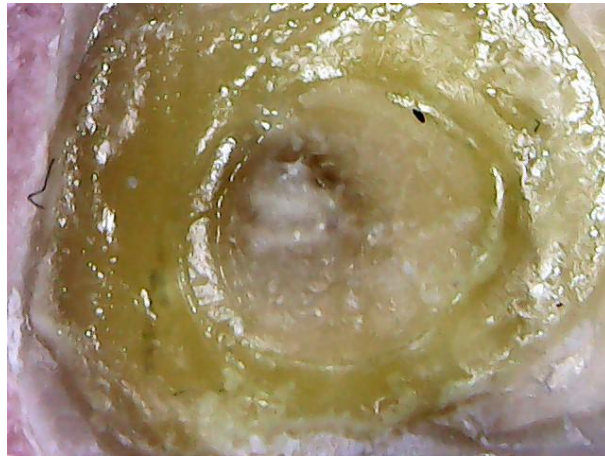
Mode of failure patterns of normal saline group with Self-Etch approach.

APPENDIX X



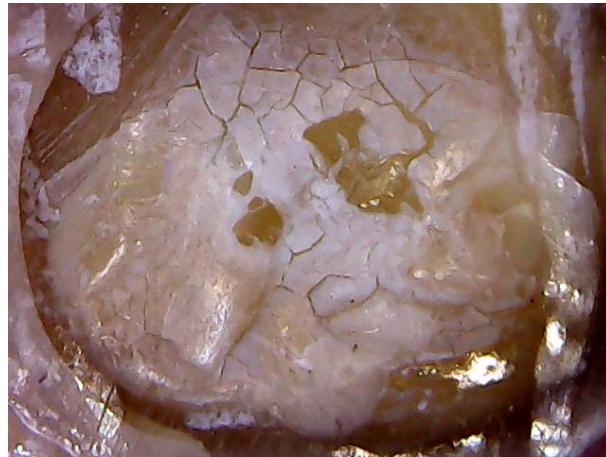
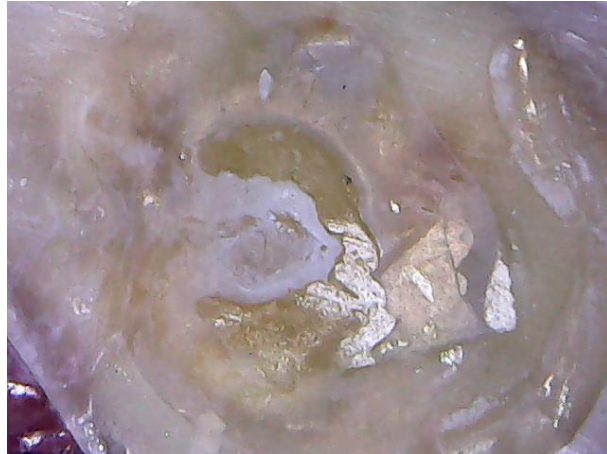
Mode of failure patterns of normal saline group with Total-Etch approach.

APPENDIX XI



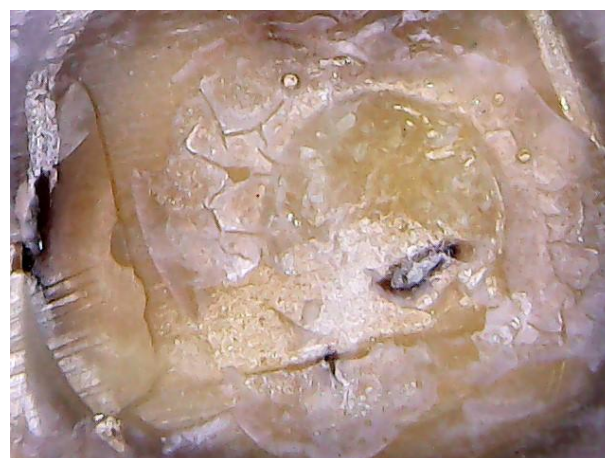
Mode of failure patterns of Sword group with Self-Etch approach.

APPENDIX XII



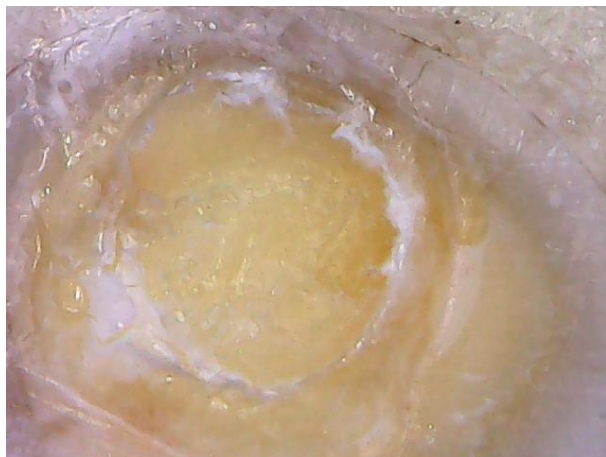
Mode of failure patterns of Sword group with Total-Etch approach.

APPENDIX XIII



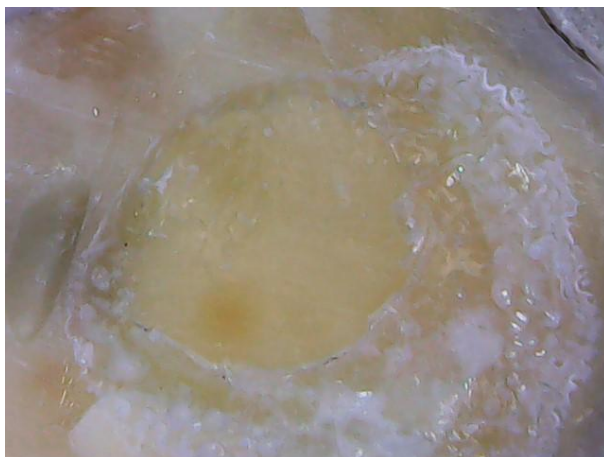
Mode of failure patterns of Cariax group with Self-Etch approach.

APPENDIX XIV



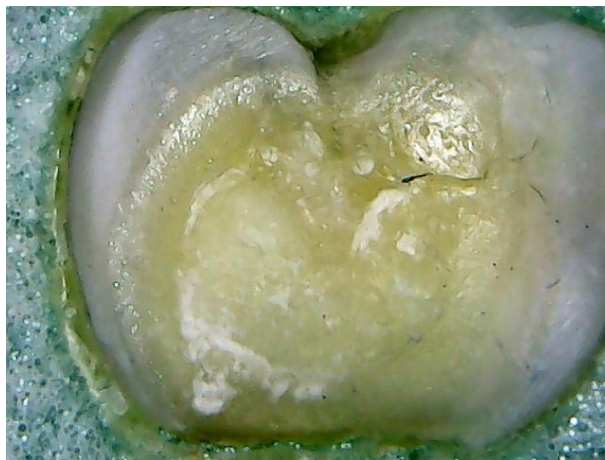
Mode of failure patterns of Cariax group with Total-Etch approach.

APPENDIX XV



Mode of failure patterns of Tantium group with Self-Etch approach.

APPENDIX XVI



Mode of failure patterns of Tantum group with Total-Etch approach.

**Effect of Different Cavity Disinfectants on Shear
Bond Strength of Resin Composite to Dentine
(An *in vitro* study)**

تأثير مطهرات التجويف المختلفة على قوة الرابطة للحشوات
الضوئية لعاج الاسنان
(دراسه في المعمل)

BY

**NADA FARAG SANUSSI AL-SHUKRI
(BDS, 2007-2008)**

Thesis proposal

**Submitted in Partial Fulfillment of Requirements
For The Degree of Master of Science in Conservative
Dentistry and Endodontics**

Supervisor: Dr Naeima Betamar

Faculty of Dentistry

University of Benghazi

Benghazi – Libya

1. INTRODUCTION

Cavity preparation is an operative procedure attempts to remove all infected caries dentine prior to placing a restoration. However residual bacteria might be entrapped within the dentine tubules or the smear layer during and after the cavity preparation, which considers one of a major problem in restorative dentistry.¹ Therefore, effective removal of infected dentine and prevent growth of microorganisms under a restoration leads to decrease pulp sensitivity and pulpal inflammation, reduce microleakage and therefore prevent the development of secondary caries, and hence reduce the need for replacing the restoration. Long-term studies have shown that the bond strength of resin bonded to dentine decreased over time due to collagen degradation within the hybrid layer.¹⁻³ Therefore, elimination of the residual bacteria from the cavity surfaces after cavity preparation is of major importance using a disinfectant solution.¹ Many chemicals have been tested as cavity disinfectants, including chlorhexidine digluconate (CHX), disodium ethylen diamine tetraacetic acid (EDTA) dihydrate, sodium hypochlorite (NaOCl), Ozon (O3), Er:YAG laser and iodine. However, there is concern about the use of cavity disinfectants with dentine bonding agents, since they may have an adverse effect on the bond strength of the resin composites to dentine.

Matrix metalloproteinases (MMPs) are a group of proteolytic enzymes which are capable of degrading extracellular matrix proteins. Activated MMPs are not fully infiltrated with adhesive resin, and can slowly degrade the collagen fibrils at the resin-dentin bonded interface.² Thus, the use of such cavity disinfectants which are MMP inhibitors is a strategy to prevent degradation of dentine bonds and to increase the longevity of bonded restorations. There are many products used as cavity disinfectants such as:

i) Chlorhexidine digluconate (CHX):

which has been widely used as an antimicrobial agent as well as for disinfection before the placement of restorations, and is considered the “gold standard” of oral antiseptics.⁴ It has been used as an oral antimicrobial agent since the 1970s.⁵ CHX wash, in the form of 2% solution used before composite bonding has been shown to successfully preserve the bond strength up to 6 months when etch-and-rinse adhesive systems were used.^{6, 7} Manfro et al⁸ and Breschi et al⁹ have

reported that the bond in the CHX-treated samples were significantly stronger than the non-treated samples after 12 months of aging. Further studies have reported higher bond strengths of resin composite to dentine when etch-and-rinse adhesive systems used rather than when self-etch systems were used after CHX pretreatment.^{10, 11}

ii) **Sodium Hypochlorite (NaOCl) Solution:**

is the most commonly used antimicrobial agent in clinical dentistry.¹² Application of NaOCl over the smear layer covered dentine would eliminate its collagen phase resulting in reduction in the smear layer compactness.¹³ Depending on the testing methodology and the adhesive system composition, the application of sodium hypochlorite may increase, decrease, or have no effect on bond strengths.^{14, 15} Ercan et al¹¹ recommended NaOCl disinfectant to be used with etch-and-rinse bonding systems, since they found that 2.5% NaOCl pretreatment negatively affected the shear bond strength (SBS) of self-etching bonding systems.

iii) **Benzalkonium Chloride (BAC):**

is a mixture of alkylbenzyltrimethyl ammonium chlorides and is a nitrogenous cationic agent containing a quaternary ammonium group with broad antimicrobial activity.¹⁶ It has been described as a strong antibacterial agent against microorganisms like *S. mutans*, *Streptococcus Salivarius*, and *S. aureus*. It was an effective MMP inhibitor that may preserve the adhesive bond of the resin restoration to dentine.¹⁷

iv) **Iodine-based Disinfectants:**

The antibacterial effects of these agents are attributed to the presence of molecular iodine, it has been reported to disclose and eliminate bacteria presence in the dental plaque.¹⁸

v) **Disodium ethylene diamine tetraacetic acid (EDTA):**

is a weak acid and has a disinfectant and demineralizing effect, and has been widely used to dissolve the mineral phase of dentin without altering the structure of dentin collagen.

vi) **Ozone (O₃):**

is known to be a strong oxidizer, hence it possesses antibacterial activities by disrupting the cell wall and cytoplasmic membrane of bacteria and therefore destruction of the microorganism.¹⁹ Majority of the studies have reported no effect of O₃ on the bond strength, regardless of the type of adhesive systems used.¹⁹⁻²²

vii) **Lasers “Light Amplification by Stimulated Emission of Radiation”:**

Laser irradiation causes expansion of intratubular water of the bacterial cell and has thermal and photodisruptive effects on bacteria leading to cell growth impairment and lysis.²³ The effectiveness of the erbiumdoped yttrium, aluminum, and garnet lasers (Er,YAG laser) as an antimicrobial agent as well as smear layer remover has also been documented. Several studies have reported that the use of Er,Cr:YSGG or potassium-titanyl-phosphate (KTP) lasers does not adversely affect the bond strength of the restoration.^{10, 24-26}

Successful adhesion between tooth-colored restoration and tooth structure is essential. Bonding of resin composite to enamel has been a simple and uncomplicated procedure compared with bonding to dentine. However, bonding to dentine has been less reliable, and represents a greater challenge. Nowadays, a variety of dentine bonding systems are available that allow for good bond strengths between resin composite and dentine as high as that between resin composite and enamel, all these dentine bonding agents try to achieve a long-lasting and reliable bond between the restorative material and the dentine surface.

Adhesion of resin-based composite to dentine has been evaluated by tensile, micro-tensile and shear bond strength testing methods. However, shear bond strength tests have been often used. In the shear bond strength test the force is applied parallel to the tooth surface while the bond is broken. In addition, during the shear test, cohesive failure within the dentine is frequently observed. This form of failure does give reliable information on the actual strength of the adhesive bond which could be due to the large size specimen and the ^{non}-uniform stress distribution generated during this test.

2. AIM OF THE STUDY:

The aim of this in-vitro study is to evaluate the effect of three different cavity disinfectants on shear bond strength (SBS) of resin composite restorative material to dentine applied with two different adhesive systems.

2.2. Objectives of the study:

- IV)** To evaluate and determine the shear bond strength (SBS) of resin composite to dentine surface treating with three different cavity disinfectants.
- V)** To assess and evaluate the effect of two types of adhesive bonding systems; (Total-etch and Self-etch) among each type of cavity disinfectant on the shear bond strength (SBS) of resin composite to dentine.
- VI)** To investigate the failure pattern of the testing groups

3. MATERIALS AND METHODS:

3.1 MATERIALS:

- Materials used in the study are three cavity disinfectants, namely:
 5. Chlorhexidine gluconate,
 6. Sodium hypochlorite,
 7. Benzalkonium chloride-based disinfectant.
 8. Normal saline as control group.
- The two adhesive systems are:
 1. Total-etch adhesive system,
 2. Self-etch adhesive system.
- One resin composite restorative material: Nanohybrid composite resin.

3.2 METHODS:

This study will be conducted following Ertuğrul Ercan et al. methodology.¹¹ The methods include:

3.2.1. Specimen preparation:

Eighty extracted caries-free human third molar teeth will be collected from different dental practice will be cleaned of calculus and soft tissue debris.²⁷ The teeth crown will be sectioned horizontally with a low-speed diamond disk saw under water coolant to expose mid-coronal dentine. The sections of the teeth including the roots will be embedded inside a cylindrical-shaped mold filled with self-cured acrylic resin till the cervical line with the exposed occlusal surface plane being parallel to the floor.

3.2.2 Specimens grouping:

Eighty caries-free human third molars will be used in this study. Details of number and distribution of testing groups are illustrated in Figure 1.²⁷ The specimens will be randomly divided into four main groups (GP1, 2, 3, 4), each group contains twenty teeth ($n=20$) according to the proposed dentine surface pretreatment, as following:

Gp1: Dentine surface without pretreatment, as a control group (normal saline), ($n=20$).

Gp2: Dentine surface pretreatment with 2% chlorhexidine gluconate, ($n=20$).

Gp3: Dentine surface pretreatment with 2.5% sodium hypochlorite, (n=20).

Gp4: Dentine surface pretreatment with benzalkonium hydrochloride, (n=20).

Then each group (Gp1-Gp4) will be further divided into two subgroups ($n = 10$) per subgroup according to the type of adhesive system (dentin bonding agent) used, as following:

(TE) Total-etch adhesive system.

(SE) Self-etch adhesive system.

3.2.3 Application of disinfectants and resin composite build up:

The disinfectant solution in every group will be applied into the dentine surface using a disposable syringe, left for seconds, then rinsed with water for 10 seconds, and dried.

Each group was then randomly divided into two subgroups of ten teeth each ($n=10$) according to the bonding agent used, so the pretreatment dentinal surface of each specimen will be applied with bonding agent either a total-etch adhesive system or a self-etch adhesive system as recommended by manufacture instructions for use.

After the application of the adhesive system, the Nanohybrid resin composite will be carefully applied to the treated dentine surface by placing the material into cylindrical-shaped split Teflon mold with an internal diameter of 3 mm and a height of 3 mm. Composite will be placed incrementally in 2 layers of 1.5mm each. Each layer is light cure for 20 seconds with the same light curing unit, and then specimens will be stored for 24h in humid environment.

3.2.4 Shear bond strength testing:

After storing in an incubator at 37°C in 100% humidity for 24 h, all the specimens will be individually mounted on a universal testing machine and the shear bond strength will be measured at a crosshead speed of 1 mm/min. the load in the shear bond strength of resin composite to dentine will be recorded in Newton's (N) and the shear bond strength will be calculated in Mega pascal (MPa) taking into account the cross-sectional area of the composite buildup.

3.2.5 Assessment of the mode of failure:

After the testing procedure, the fractured/deboned surfaces were observed visually and examined by means of a dissecting microscope with different magnifications to determine the modes of failure. Then three specimens from each group will be examining under scanning electron microscope (SEM) to examine and locate the failure pattern. The failure mode will be divided into adhesive, cohesive and mixed failure pattern.

3.2.6 Statistical analysis:

For all testing groups; the bond strength data obtained for the corresponding subgroups will be statistically analyzed using one-way analysis of variance ANOVA. Mean and standard deviations will be calculated for each testing group. Tukey's multiple comparison tests will be further computed to identify differences between the sub groups. Statistical significance will be set in advance at the 95% probability level.

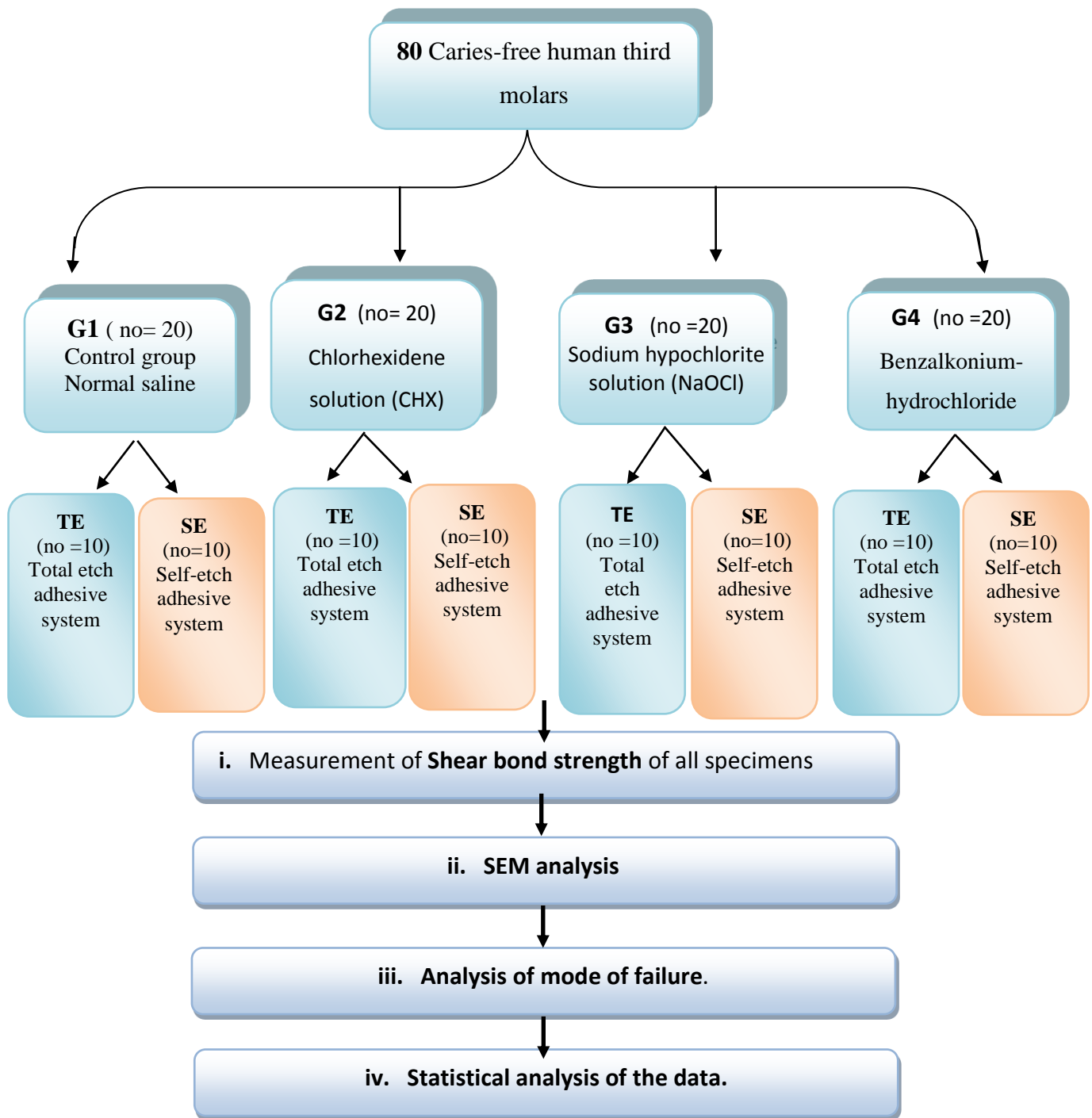


Figure 1: flow chart illustrates number and distribution of testing groups and steps of procedures.

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تأثير مطهرات التجويف المختلفة على قوة الرابطة الحشوات الضوئية لعاج الاسنان

(دراسة معملية)

اعداد:

ندى فرج سنوسي الشكري

تحت إشراف:

أ. د. نعيمة محمد بالتمر

الملخص

الغرض: الهدف من هذه الدراسة هو تقييم تأثير مطهرات التجويف المختلفة على قوة رابطة العاج للراتنج المركب المطبق بطريقتين مختلفتين من المواد اللاصقة.

المواد والطرق: تم قطع ثمانين ضرساً ثالثاً خالياً من التسوس بالتوازي مع سطح الإطباق لفضح عاج منتصف الإكليل. تم تقسيم العينات عشوائياً إلى أربع مجموعات من عشرين سنّاً لكل مجموعة. المجموعات 2 و 3 و 4، تم معالجة أسطح العاج بمطهرات التجويف التالية، على التوالي؛ 0.12% محلول الكلورهيكسيدين (CHX)، 5% هيبوكلوريت الصوديوم (NaOCl) و 0.15% كلوريد البنزلكونيوم (BAC). تم تقسيم كل مجموعة من Gp1 إلى Gp4 إلى مجموعتين فرعيتين ($n = 10$ عدد 10 لكل مجموعة فرعية) وفقاً لأساليب اللصق. تم ربط عشر عينات بنهج اللصق الكلي وتم ربط العينات العشر الأخرى بنهج اللصق الذاتي. ثم تم وضع مركب الراتنج بشكل تدريجي على سطح العاج المعالج في أنبوب تفلون أسطواناني الشكل (قطر 3 مم × 3 مم ارتفاع) ثم بلمرة بوحدة معالجة LED. بعد تخزين العينات في حاضنة لمدة

24 ساعة، تم قياس قوة رابطة القص (SBS) بسرعة تقاطع 0.5 مم / دقيقة. تم تحليل بيانات قوة الرابطة مع تحليل التباين (ANOVA) واختبار t المستقل للعينة.

النتائج: وجد التحليل الإحصائي أن سطح العاج المعالج بمطهرات تجويف مختلفة نتج عنه قوة رابطة قص (SBS) أعلى مقارنة بمجموعة التحكم بغض النظر عن نوع طريقة اللصق. كانت أقل قيمة (SBS) (0.85 ± 7.58) التي تم الحصول عليها لسطح العاج غير المعالج (مجموعة التحكم). من بين مجموعات مطهر التجويف، تم تسجيل أعلى SBS لمجموعة Tantum (7.59 ± 13.39). بالنسبة للأنواع الثلاثة من مطهرات التجويف، أظهر نهج اللصق الكلي قوة رابطة أعلى من نهج اللصق الذاتي.

الاستنتاجات: معالجة سطح العاج بمطهرات التجويف قبل الربط اللاصق يحسن قوة رابطة القص (SBS) بين مركب الراتنج و سطح العاج على وجه الخصوص من خلال نهج اللصق الكلي.

الكلمات المفتاحية: قوة رابطة القص (SBS) ، اللاصق، الراتنج المركب، مطهر التجويف.



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قدمت من قبل:

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تحت إشراف:

أ.مشارك.د. نعيمة محمد بالتمر

قدمت هذه الرسالة استكمالاً لمتطلبات الحصول على درجة الماجستير في

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