EFFECT OF SURFACE TREATMENTS ON SHEAR-BOND STRENGTH OF GLASS-IONOMER CEMENTS TO SILVER-DIAMMINE-FLUORIDE TREATED SIMULATED CARIOUS DENTINE

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2024

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ABSTRACT

Objectives: This study investigated the effect of different surface treatments on the shear-bond strength (SBS) and failure modes of high-viscosity glass-ionomer cements (HVGIC) and resin-modified glass-ionomer cements (RMGIC) to silver-diammine-fluoride (SDF)-treated simulated carious dentine (SCD).

Material and Methods: One hundred and fifty extracted human premolars were sectioned and pH-cycled for ten days to simulate carious dentine. In Part A, the demineralised specimens were treated with 38% SDF (Riva Star) for 2 mins, washed, stored in deionised distilled water at 37°C for two weeks, and subjected to the following surface treatments (n=14): T1 - no treatment (control); T2 - 10 seconds polyacrylic acid (PAA); T3 - 5 seconds phosphoric acid (PPA); T4 - 5 seconds PPA plus universal adhesive (Zipbond); T5 - 5 seconds PPA plus RMGIC adhesive (Riva bond LC). High Viscosity Glass-ionomer Cements (Riva Self-cure HV [SC]) and RMGIC (Riva Light-cure HV [LC]) restoratives were applied to the conditioned specimens and stored in artificial saliva at 37°C for one week. SBS was subsequently performed with a Universal Testing Machine (a load of 500N and crosshead speed of 1mm/min). Failure modes were appraised using a stereomicroscope coupled with the ImageJ software. Statistical analysis was done with Kruskal-Wallis/ post hoc pairwise comparisons (α=.05) and Chi-square tests with Bonferroni adjustment (α=0.05). In Part B, the interface between the HVGIC and dentine (n=1) was carried out using scanning electron microscopy (SEM) and energy-dispersive X-ray analysis (EDX).

Results: In part A, **t**he highest SBS was observed when SC and LC were restored with T2 and T5, respectively. Significant differences in SBS were as followed: SC - T2, T1 >T5, T3; LC - T5, T4, T3 > T2. For T3 and T5, SBS achieved with LC was significantly greater than SC. SC generally exhibited adhesive failures. Conversely, LC exhibited

mostly adhesive and mixed failure in the material for most surface treatments. In part B, The SEM demonstrated smooth and continuous layer of adhesive in the SC group. The presence of a short and discrete resin tag was seen with thin hybrid layer in phosphoric acid-treated LC groups. The EDX data demonstrated that there were detected fluoride and silver ions levels in all groups. In LC-T5, it was reported that the content of Ag is highly detected (3.30 at%) on the dentinal surface.

Conclusion: The preferred method for surface-treating SDF-treated simulated carious dentine before restoration application is PAA for SC and PPA plus RMGIC adhesive for LC.

Keywords: Silver-diammine-fluoride; Caries; Bond strength; Dentine bonding; Glass-ionomer cements

KEKUATAN IKATAN RICIH TEKNIK PERAWATAN PERMUKAAN BERBEZA PADA KARIS DENTIN YANG DISIMULASI DAN DIRAWAT DENGAN SILVER DIAMINA FLUORIDA

ABSTRAK

Objektif: Kajian ini menyiasat teknik perawatan permukaan ke atas kekuatan ikatan ricih (*Shear -Bond Strength*) dan cara kegagalan pada simen ionomer kaca kelikatan tinggi (HVGIC) dan simen ionomer kaca diubahsuai resin (RMGIC) kepada dentin karies yang disimulasi dan dirawat silver diamina fluorida (SDF).

Bahan dan Kaedah: Seratus lima puluh gigi premolar manusia yang baru diekstrak telah dibelah dan kitaran pH telah dijalankan 10 hari untuk menghasilkan dentin karies tiruan. Dalam bahagian A, spesimen dentin yang telah dinyahmineral dirawat dengan 38% SDF (Riva Star) selama 2 minit, dibasuh, disimpan selama 2 minggu dalam air suling dinyahion pada suhu 37°C, dan dipilih secara rawak dan tertakluk kepada lima kumpulan prosedur rawatan seperti berikut (n=14): Kumpulan T1 – tiada rawatan permukaan (kawalan); Kumpulan T2 - 10 saat asid poliakrilik (PAA); Kumpulan T3 - 5 saat etsa asid fosforik (PPA); Kumpulan T4 - 5 saat PPA dengan pelekat universal (Zipbond); Kumpulan T5 - 5 saat PPA dengan pelekat GIC yang diubah suai resin (Riva bond LC). HVGIC (Riva Self-cure HV[SC]) dan RMGIC (Riva Light-cure HV [LC]) diletakkan pada spesimen yang dirawat permukaan dengan silinder dan disimpan dalam air liur tiruan pada suhu 37°C selama 1 minggu. SBS kemudiannya dilakukan dengan mesin pengujian universal (UTM) (beban 500N dan kelajuan kepala silang 1mm/min). Mod kegagalan dikategorikan dengan penggunaan *stereomicroscope* dan perisian *ImageJ*. Analisis statistik dilakukan dengan ujian *Kruskal-Wallis*/ ujian post-hoc *pairwise comparisons* dan ujian *Chi-square* (α=0.05). Dalam bahagian B, permukaan di

antara GIC dan dentin (n=1) telah diteliti dengan menggunakan mikroskop elektron pengimbas (SEM) dan analisis *energy-dispersive X-ray analysis* (EDX).

Hasil: Dalam bahagian A, SBS yang paling kuat didapati semasa restorasi SC dan LC diletakkan pada permukaan iaitu pada T2 dan T5 masing-masing. Perbezaan ketara dalam SBS adalah seperti berikut: SC - T2, T1 > T5, T3; LC - T5, T4, T3 > T2. Untuk T3 dan T5, SBS yang dicapai dengan LC adalah lebih signifikan dari SC. Kegagalan pelekat didapati kebanyakan di restorasi SC. Sebaliknya, LC mempamerkan kebanyakan kegagalan pelekat dan campuran dalam bahan untuk kebanyakan rawatan permukaan. Dalam bahagian B, SEM menunjukkan lapisan pelekat yang licin dan berterusan dalam kumpulan SC. Tag resin yang dilihat adalah pendek dan berasingan dengan lapisan hibrid nipis dalam kumpulan LC yang dirawat asid fosforik. Data EDX menunjukkan bahawa terdapat perbezaan tahap ion fluorida dan perak. Dalam LC-T5, kandungan Ag dilaporkan bahawa dikesan dengan kadar yang tinggi (3.30 *at%*) pada permukaan dentin.

Kesimpulan: Kaedah pilihan untuk karies dentin yang dirawat SDF sebelum restorasi adalah asid poliakrilik untuk simen ionomer kaca kelikatan tinggi dan etsa asid fosforik ditambah pelekat GIC yang diubah suai resin untuk simen ionomer kaca diubahsuai resin.

Kata kunci: Perak Diamina Fluorida; Karies; Kekuatan ikatan; Ikatan dentin, Simen Ionomer Kaca

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LIST OF SYMBOLS AND ABBREVIATIONS

$CaCl_2$	:	Calcium chloride
DI	:	Distilled water
DS	:	Demineralising solution
H_2O	:	Water
HEMA	:	2-hydroxyethyl methacrylate
HVGIC	:	High-viscosity glass-ionomer cements
K_2HPO_4	:	Dipotassium hydrogen phosphate
KCl	:	Potassium chloride
KI	:	Potassium iodide
KSCN	:	potassium thiocyanate
LC	:	Riva light cure HV
$MgCl_2$	:	Magnesium chloride
mL	:	Milliliter
mm	:	Millimetre
MPa	:	Megapascal
SBS	:	Shear-bond strength
NaF	:	Sodium fluoride
Na_2SO_4	:	Sodium sulfate
NaCl	:	Sodium chloride
NaH_2PO_4	:	Monosodium phosphate
$NaHCO_3$	:	Sodium bicarbonate
NH_4Cl	:	Ammonium chloride

PAA	:	Polyacrylic acid
PPA	:	Phosphoric acid
PS-OCT	:	Polarisation sensitive optical coherence tomography
RB	:	Riva bond LC
RC	:	Riva conditioner
RMGIC	:	Resin-modified glass-ionomer cements
RS	:	Remineralising solution
SAGF	:	Artificial saliva
SBS	:	Shear-bond strength
SC	:	Riva self-cure HV
SCD	:	Simulated carious dentine
SDF	:	Silver-diammine-fluoride
SE	:	Super etch
SEM	:	Scanning electron microscope
SiC	:	Silicon carbide
SnF_2	:	Stannous fluoride
SPSS	:	Statistical package for social sciences
ZB	:	ZipBond
µm	:	micrometer

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Chapter One: Introduction, Aim and Objectives

1.1 Introduction

Caries is prevalent among young children and aging adults. Dental caries is known as biofilm-mediated, sugar-driven, multifactorial dynamic disease (Pitts et al., 2017). This process involves demineralisation and remineralisation of dental hard tissues. Kassebaum et al. (2015) reported the occurrence of root caries among aging adults to be more than younger adults. The current perspective of remineralising demineralised dental hard tissue is increasingly accepted by dental professionals. Currently, non- invasive methods without mechanical removal of carious tissues are accepted options to treat caries. This involves minimally invasive approaches to preserve sound tooth tissue and tissues which has potential to remineralise.

There are a few indications for SDF application (Horst et al., 2016). First, patients with extreme caries risk which has salivary dysfunction. For this group of patients, frequent visit and traditional restoration failed to slow down the caries progression. Second, patients that cannot tolerate standard treatment due to medical or psychological issues. Third, multiple lesions could be treated in one visit, SDF can be used to prevent some new lesions arise or existing lesions to become symptomatic while waiting for treatment completion. Fourth, SDF is suitable for lesion which are hard to treat due to the location of the caries, such as furcation in root caries and recurrent caries at crown margin.

However, the application of SDF will form dark, discoloured carious tissue. Knight et al. (2005) reported a reduction of discolouration by the application of potassium iodide (KI) to SDF-treated carious lesion. Lesions which were not restored after the SDF and the cavity was left as a frank cavity. Restoration placement with composite resins or glass ionomer cements (GICs) will help in plaque control, and improve function as well

as appearance (Quock et al., 2012). A Silver-Modified-Atraumatic-Restorative-Technique (SMART) has been proposed using GIC after the application of SDF. (Koizumi et al., 2016) However, SDF forms silver phosphate and calcium fluoride in caries-affected dentin (CAD), thus reducing available calcium and phosphate ions when bonding with GICs (Kucukyilmaz et al., 2016). Nonetheless, prior studies had suggested that SDF does not jeopardize the bonding of GICs to carious dentin (Quock et al., 2012; Selvaraj et al., 2016); Puwanawiroj et al., 2018; Wang et al., 2016). The silver deposits and silver oxide that form on the SDF surface (Knight et al., 2007; Mei et al., 2013) could potentially improve the bonding of GICs due to interactions with the stainless steel metal surface (Fricker, 1998).

1.2 Aim and Objectives of the study

Part A

- To investigate the effect of different surface treatment on the shear bond strength (SBS) of self-cure (SC) and light- cure high-viscosity glass ionomer cements (LC) to SDF-treated carious dentine.
- To establish the failure modes using a stereomicroscope and digital imaging software.

Part B

- To evaluate the dentine-GIC interface using scanning electron microscope/energy dispersive X-ray (SEM/EDX).

- To determine the elements present on SDF-treated simulated carious dentine (SCD) surface after GIC bonding in the study.

1.3 Null Hypotheses

The null hypotheses for this study are:

(a) surface treatment does not influence the bond strength of HVGICs to SDF-treated simulated carious dentine (SCD) and

(b) surface treatment has no effect on the failure modes.

Chapter Two: Literature Review

2.1 Brief Introduction to SDF

Known for their antibacterial properties, silver metals and their salts have been known to exhibit protection against dental caries. Silver compounds have been used for decades to reduce the incidence of caries (Peng et al., 2021).

Silver-diammine-fluoride (SDF) contains fluoride, ammonia, and silver ions. It halts the development of caries and avoids caries formation in the future. Silver-diammine fluoride in its ionic formula is $Ag(NH_3)_2F$. Silver provides antimicrobial activity and hinders the activity of several glycosyltransferases in biofilm formation (Knight et al., 2007; Burgess & Vaghela, 2018; Savas et al., 2015; Mei et al., 2013a).

When SDF is applied to teeth, it penetrates enamel and dentine, increasing the subsurface fluoride stored in the tooth and creating a fluoride reservoir (Rosenblatt et al., 2009). In 1974, Suzuki and colleagues reported that SDF has a better depth of enamel penetration (25μm) and has more fluoride than that delivered by other fluoride products such as Sodium Fluoride (NaF) or Stannous Fluoride (SnF_2). The penetration of silver fluoride in sound tooth can goes up to 100μm (Knight et al., 2010). Meanwhile, in the dentine surface, the penetration depth can go up to 500-1200μm (Sayed et al., 2019).

It was found that there are “silver microwires” formed, which are filamentous and continuous (Seto et al., 2020). They have lengths between 50 - 2100μm and are 0.25 - 7.0μm in diameter. These filled the voids in lesions and permeated through surrounding dentinal tubules, thus providing a reservoir of silver in the tubules to inhibit bacterial growth. This increased its resistance to recurrent caries (Shah et al., 2014; Shimizu, 1974). The silver densities will increase the hardness of the lesion and prevent fluid flow through tubules. Remineralisation of the decayed surface by SDF and the 3abilities of the

"microwires" can dispense forces from the lesion and increase the hardness of dentine (Seto et al., 2020).

Besides that, both silver (Ag) and fluoride (F) ions are antibacterial (Mei et al., 2013a). Silver phosphate interacts with thiol groups of nucleic and amino acids in bacteria. Thus, the bacteria couldn't carry out metabolic and reproductive functions, leading to bactericidal effect. In addition, SDF is able to interact with the biofilm by interfering with the synthesis of glucan by interrupting the glycosyltransferase enzymes. Glucan forms the major constituent of plaque. This could inactivate the bacterial growth of cellular polysaccharides. Treatment using SDF reduces the demineralisation process by exerting an inhibitory effect on cariogenic biofilm formation. Furthermore, studies done by Mei et al. (2012) have shown that with SDF, calcium (Ca) and phosphate (P) ions are retained at the dentine surfaces. Silver-diammine-fluoride also hardens the caries lesions and inhibits matrix metalloproteinase activity. A study by Delbem et al. (2006) showed that SDF can withstand chemical acid challenges, reducing the solubility of tooth tissue and promoting enamel remineralisation. A caries-resistant layer could be created by the remineralisation of caries-infected teeth at the base of a restoration (Knight et al., 2010).

Potassium iodide (KI) application following SDF treatment was developed as a strategy to overcome the discolouration issue. An in-vitro study found that incorporating potassium iodide into SDF during application reduced tooth discolouration (Knight et al., 2005).

The highest fluoride concentration (44800 ppm fluoride ion) is found in 38% SDF, and is about twice the amount of 5% fluoride varnish (Table 2.1). SDF is recommended for patients with high caries risk, hard-to-treat lesions, and those with behavioural or

medical difficulties, e.g., severe early childhood caries or xerostomia. It is also suggested for those in community settings or without access to dental care (Judy et al., 2017). It is easy to apply, minimally invasive, and a low-cost method which is easy to use in paediatric settings.

Fluoride concentration	**Parts per million (ppm)**
38% Silver-diammine-fluoride	44800ppm
5% fluoride varnish	22600ppm
APF (in office)	12300ppm
NaF_2 (Rx)	9000ppm
CPP ACP with Fluoride	900ppm

Table 2.1: Comparison of common fluoride products (Judy & Young, 2017*)*.

2.1.1 Recent FDA Approval

The US Food and Drug Administration (FDA) announced SDF product as a Class II medical device for desensitizing teeth in the USA in 2014. This means that it is a medical device with a moderate to high risk that requires special controls. Caries lesions were treated using off-label application of SDF.

Safety margin would be a concern in gaining clearance in FDA. The lethal dose (LD50), when tested on the rat studies by oral and subcutaneous administration, was defined at 520mg/kg and 380mg/kg, respectively. Take a 10kg child as an example, the relative safety margin is: 380mg/ kg LD50/ 0.95mg/kg dose, which is 400- fold of safety margin. According to Horst et al. (2016), the recommended limit is one drop (25µL) per 10kg for each visit with weekly intervals at most.

2.1.2 Mechanism of actions

The mechanism of actions of SDF is associated with tooth tissue, interaction with bacteria and biofilm.

2.1.2.1 Interaction with tooth tissue with promotion of remineralisation

The major tooth component, hydroxyapatite ($Ca_{10}(PO_4)_6(OH)_2$) could interact with SDF/$Ag(NH_3)_2F$ and forms mainly calcium fluoride(CaF_2), silver phosphate(Ag_3PO_4) and ammonia monohydrate($H_{10}N_2O_2$) (Suzuki et al., 1974). It was suggested that silver phosphate is responsible for the increased hardness of the arrested lesion (Yamaga et al., 1972).

In carious teeth, the product fluorohydroxyapatite is formed from the reaction of SDF with hydroxyapatite through reprecipitation reactions (Lou et al., 2011). This is because CaF_2 is broken down into calcium ions and fluoride ions. During cariogenic challenges, CaF_2 will serve as a reservoir for fluoride ions, which are released due to reduced concentration of hydrogen phosphate ($HPO_4^{(2-)}$) at acidic pH levels (Roll and Saxegaard, 1990).

SDF reacts with hydroxyapatite (1) leading to the subsequent reactions (2,3) as shown in Figure 2.1. (Peng et al., 2012)

$$Ca_{10}(PO_4)_6(OH)_2 + Ag(NH_3)_2F \rightarrow CaF_2 + Ag_3PO_4 + NH_4OH \quad (1)$$

$$CaF_2 \rightarrow Ca^{2+} + 2F^- \quad (2)$$

$$Ca_{10}(PO_4)_6(OH)_2 + 2F^- \rightarrow Ca_{10}(PO_4)_6(F)_2 + 2OH^- \quad (3)$$

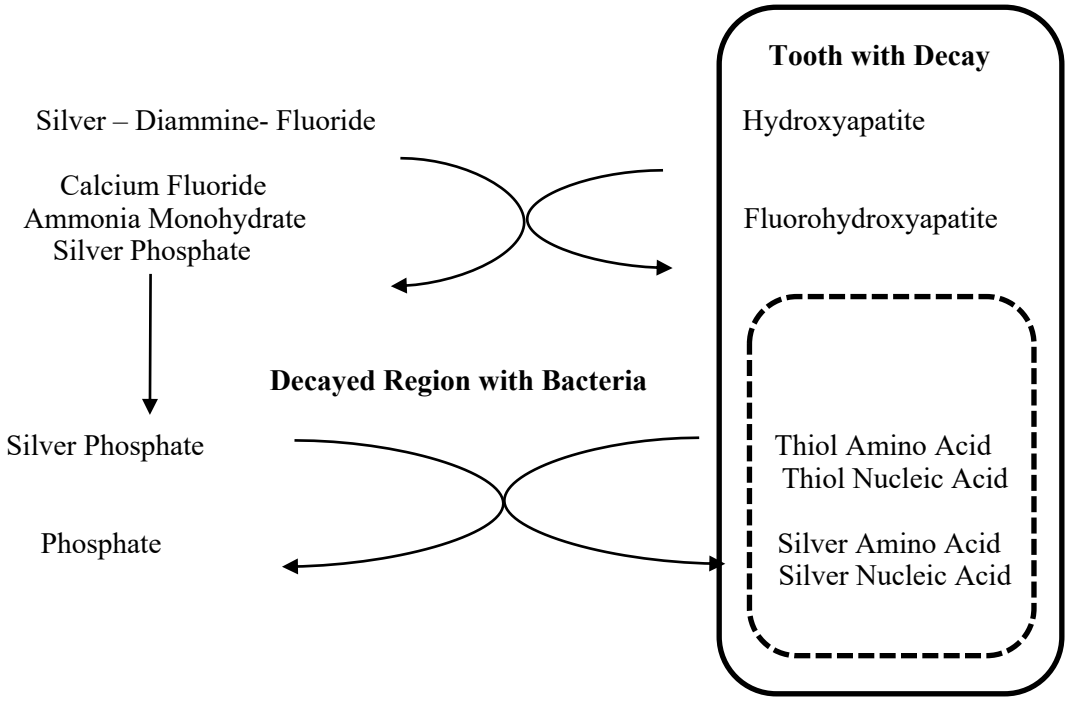


Figure 2.1 The reactions of fluoride, silver nitrate and silver-diammine-fluoride on teeth and bacteria. (Rosenblatt, 2009)

Silver phosphate (Ag_3PO_4) is formed as one of the main products. The compound penetrates the tubules to block the lumen, partially or totally. It could inactivate cariogenic bacteria once it is in contact with them due to its antimicrobial effect. Silver ions penetrate into dentine surface and remains, whereas phosphate reservoir aids in remineralisation. Silver compounds stains lesion black (Willershausen et al., 2015). The other end product is ammonium hydroxide (NH_4OH), this keeps pH elevated and provides antimicrobial activity (Jennings et al., 2015). Both of the end products precipitate the formation of arrested caries.

2.1.2.2 Interaction with bacteria and biofilm

Silver-diammine-fluoride is an effective antibacterial result when compared to other chemomechanical caries removal gel, such as chlorhexidine, enzyme based

Papacarie and NaOCl based Cariosolve (Hamama et al., 2015). Moreover, when comparing the application of SDF/KI to the chemomechanical gel, the intratubular bacterias' viability was significantly reduced.

2.1.2.3 Reduction in destruction of the collagenous organic matrix

The application of SDF will activate the collagenase inhibition to reduce the destruction of the collagen matrix in dentine (Mei et al., 2013c). The matrix metalloproteinases (MMPs) will promote enzymatic degradation of dentine. MMPs can be present in the dentine matrix or in the saliva. The inhibition factor that is provided by SDF is more effective than 10% NaF, which has the equivalent concentrations of fluoride and silver ions (Mei et al., 2012).

2.1.3 Efficacy for caries arrest and prevention

2.1.3.1 Efficacy of SDF in arresting and preventing dental caries.

In the study by Knight et al. (2007), he found that demineralised dentine when treated with SDF/KI is better able to resist further demineralisation by Streptococcus mutans than non-demineralised dentine.

Another in-vitro study was conducted using a computer-controlled artificial mouth which simulates real mouth conditions for temperature, humidity, and saliva flow rate. Dental caries was arrested by 38% SDF in minimising the loss of mineral content, slowing down Type I collagen destruction and decreasing the dentine demineralisation process. Moreover, it can inhibit the growth of cariogenic biofilms, due to its silver and fluoride ions in high concentration (Mei et al., 2013b).

The effectiveness of SDF at 38% concentration was recommended for childhood caries prevention and arrest (Zhao et al., 2017a). It was noted that direct application of SDF into a cavitated caries lesions without prior removal of the carious tissue was able to arrest the caries and no further progression was noted after 2-3 years (Yee et al., 2009; Llodra et al., 2005).

Studies done by Chu et al. (2008a) and Mei et al. (2014) on carious primary teeth of preschool children showed that application of 38% SDF annually was significantly useful in preventing new caries and arresting existing caries than three-monthly applications of sodium fluoride varnish.

Llodra et al. (2005) conducted a 36-month randomised clinical trial design, at a Cuban primary school, with 7 visits and a sample size of n = 452. The sample size was divided into two groups; the first received application of 38% SDF every 6 months on decayed deciduous teeth surfaces and on the occlusal surfaces of any first permanent molars that had erupted. The second group served as a control group with no treatment. Placement of SDF was done for deciduous teeth without any caries removal. The teeth surface was classified as healthy, with inactive caries (cavity with hard floor or walls) with active caries (presence of cavity with soft floor or walls). However, the carious tissue was removed in permanent teeth using excavators. 373 children (82.5%) completed the 36-month follow-up. SDF was more effective for caries reduction in deciduous teeth (80%) and first molars (65%) than the control group.

The effectiveness of SDF was compared with temporary restorations in caries arrest capability. SDF may be a better solution in slowing down initial occlusal caries in erupting permanent first molars, when compared to other non-invasive approaches like cross tooth-brushing technique (CTT) and glass ionomer fissure sealants (GIC) (Braga et al., 2009). In this study, patients included were aged 5 to 7 years old, with a sample size of 66. Teeth included in the study were first molars with occlusal active initial caries

without cavitation. The findings were recorded at baseline and at the following intervals: 3, 6, 12, 18, and 30 months. Active lesions were generally reduced in all groups. However, after 3 and 6 months, use of 10% SDF showed a significantly higher capacity to arrest caries than CTT and GIC.

Dos Santos et al. (2012) studied the properties of caries arrest of 30% SDF with GIC as an interim restorative technique (IRT). The sample size was large (n=1016) and clinical data was recorded at baseline, 6 months, and 12 months. The inclusion criteria was children aged 5 – 6 years old that had primary teeth with active lesions (ICDAS Score 5). They were then assessed using a few criteria: active caries lesions in the SDF group, failure of the sealant in the IRT group, Miller criteria, and ICDAS code. Silver-diammine-fluoride was placed without removal of carious tissue. It was discovered that caries arrest in 30% SDF was 1.73 times more effective than an IRT after 6 and 12 months (p<0.05).

The efficacy of annual and semi-annual topical application of 38% SDF in arresting active dentine caries in deciduous teeth was compared with that of yearly application of GIC (Zhi et al., 2012). The target group was children aged 3 - 4 years old with a sample size of n = 212. This group presented with active caries in dentine that did not involve the pulp, as per visual and tactile inspection. SDF was placed after carious tissue was removed with hand instruments. The group that received SDF application twice a year resulted in greater caries arrest rates than the groups with SDF and GIC application once a year.

Duangthip et al. (2016) examined the efficacy of three topical fluoride treatment protocols: weekly 30% SDF for three times, yearly 30% SDF, and weekly 5% sodium fluoride (NaF) varnish for 3 times. It was performed on patients aged 3 to 4 years old who had at least one tooth with untreated active dentinal caries that did not affect the pulp. Without removing any carious tissue, SDF was used. Groups that received 3 times weekly SDF treatments exhibited greater caries arrest rates at 6 and 12 months than the other

treatment groups that received yearly SDF and weekly NaF varnish application. At 18 months, however, the group treated with yearly SDF application had a greater rate of caries arrest (40%) than the other treatment groups.

There are several concentrations of SDF available in the market: 10%, 12%, 30%, and 38%. Concentration of 38% has been recommended for the prevention and treatment of dental caries in children (Horst et al., 2016).

Sayed et al. (2019) reported that the penetration of the silver ions into demineralised dentine can further penetrate into the underlying mineralised dentine, when it was stored more than 24 hours. When it was stored for 24 hours, the discoloration was confined only at the superficial layer. The discolouration caused by SDF in the dentine was extending deeper after 2 weeks and 1 year.

In summary, SDF is effective in prevention and arresting caries when compared to interim restorative technique, fluoride varnish and cross- brushing technique. At concentrations of 30% or 38%, SDF exhibits caries arrest properties as an alternative treatment in the deciduous and permanent dentition.

2.2 Staining of carious dentine

The silver ions contained in SDF solution may result in black discolouration of dentine as shown in Figure 2.2 (Rosenblatt et al., 2009). The stains are due to oral sulphides reacted with free silver ions to form silver sulphide and silver phosphate precipitation (Chu et al., 2008b).

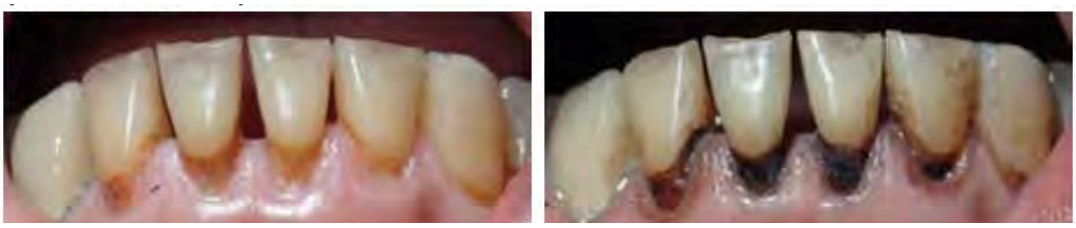


Figure 2.2: Pre- and post-operative SDF on a xerostomic patient (without restoration) (Bendit & Young, 2017)

Knight et al. (2007) discovered that SDF followed by KI application was antibacterial It could reduce the black staining associated with SDF application. Silver ions adsorb on any protein surface and form strong bonds with denatured proteins. The silver ion bonding will bind specifically to decayed collagen and stain the carious lesion with intrinsic pigmentation, whereas only surface protein staining occurs in healthy tissue. The oxides that are bound to the tissue are not able to be polished or washed away. Pardue (2018) mentioned that the presence of silver oxide bound to diseased collagen indicated the efficacy of SDF. If the surface did not turn grey/black, the silver did not bind and the antimicrobial effect will be only transient.

SDF when reacts with KI, form a bright yellow precipitate, silver iodide (AgI). Silver iodide is a yellowish precipitate with low solubility, which is easily rinsed away by water and air due to the silver iodide can break down into metallic silver and iodine when being exposed to light (Vinh et al., 2017). As a result, curing light can cause instant darkening on SDF-treated areas.

Silver-diammine-fluoride delivers the antimicrobial silver ions, but the excess silver ions are precipitated as silver sulphite (Ag_2S). Zhao et al. (2017a) suggested that SDF will not be useful in arresting caries after the application of KI solution to SDF, since the excess silver ions were removed (Zhao et al., 2017a). It was shown that the degree of dentinal demineralisation increased the rate of dentinal discolouration after the

application of SDF (Sayed et al., 2018). Vinh et al. (2017) reported that regardless of the restorative material used, all teeth treated with only SDF showed darkening and a reduction in lightness value. All KI-treated teeth were lighter than SDF-treated teeth as shown in Figure 2.3.

In summary, treatment of potassium iodide solution after SDF could help in reducing the discoloration caused by SDF.

2.3 Bonding to SDF/KI treated dentine

Problems with SDF including tooth discoloration and bonding to restorative materials which may be needed to mask discoloration and minimize food trap. Placement of SDF to caries-affected dentine (CAD) is usually effective; however, the bond strength of caries-affected dentine is usually lower than that of sound dentine. The lower bond strengths have been mainly attributed to the obliteration of dentine tubules by acid-resistant mineral crystals, thicker zone of exposed collagen after the application of the adhesive system, and the reduced hardness of the CAD (Marquezan et al., 2009). Infiltration of SDF into the dentinal tubules might block the penetration of the bonding agent into the dentine (Koizumi et al., 2016). Healthy dentine is usually the substrate for conducting bond strength studies; however, in the clinical scenario, the dentine bonding and surface pre-treatment is applied to sclerotic and demineralised dentine rather than only on healthy dentine (Wang et al., 2019).

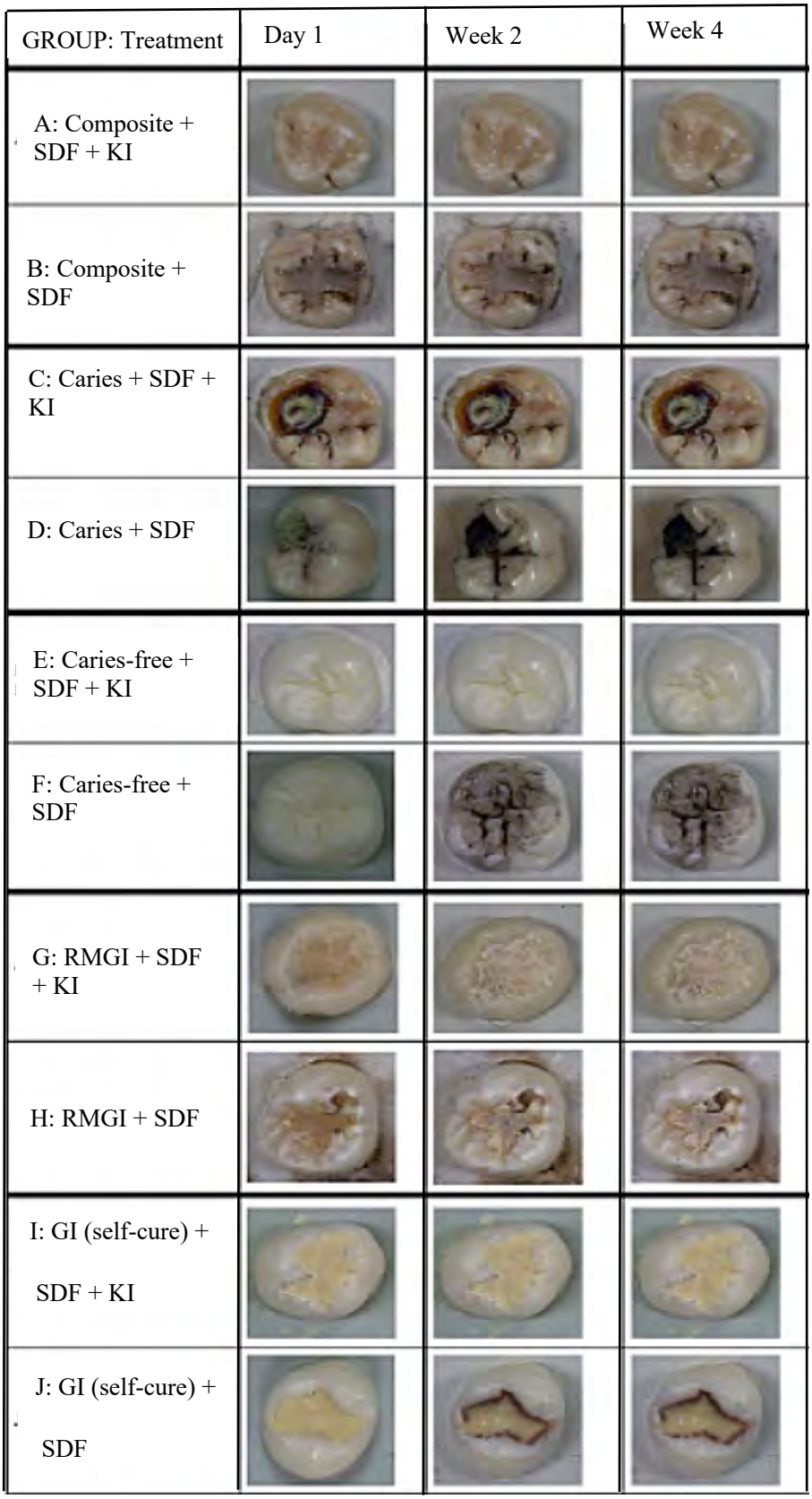


Figure 2.3: Photos from each SDF treatment group at different time period (Vinh. et al., 2017)

Silver particles extend into the dentine tubules and could interfere with subsequent placement of adhesive tooth-coloured restorations into the SDF-treated surface. There were studies to investigate whether SDF would reduce the bond strength of tooth-coloured restorations. Chemical cavity preparation is one of the strategies to improve SDF penetration into affected dentine. Pre-treatment with ethylenediaminetetraacetic acid (EDTA) to remove superficial hydroxyapatite from affected dentine may allow SDF to penetrate deeper into the dentinal tubules (Horst et al., 2016).

Selvaraj et al. (2016) proposed that pre-treatment of non-carious dentine surfaces with SDF/KI reduces nanoleakage at the resin-dentine interface; there was no reduction in bond strength with either an etch and rinse or self-etching bonding system when tested with a transmission electron microscope. Quock et al. (2012) discovered no significant changes to bonding strength of the non-carious dentine when pre-treated with SDF. In another study carried out by Kucukyilmaz et al. (2016), the bond strength of caries-affected dentine treated with SDF to resin composite was shown to have been reduced, while the bond strength to non-carious dentine was unaffected.

Koizumi et al. (2016) suggested a decreased bond strength in another study when the sound dentine was treated with resin modified GIC (RMGIC), self-etch adhesives, and etch-and-rinse after treatment with SDF/KI. Bond strength to dentine was observed to be the same with no changes in another study when using GIC after the application of SDF/KI (Selvaraj et al., 2016; Quock et al., 2012), provided that the precipitates were thoroughly washed out prior to applying the GIC (Knight et al., 2016). Yamaga et al. (1993) found that immediate placement of GIC after applying SDF increased bond strength. Furthermore, the SDF prevents shear bond strength from deteriorating over time. Gupta et al., (2019) has suggested that application of SDF/KI followed by rinsing with

water will increase the SBS of sound dentine to RMGIC. However, SBS on demineralised dentine was not tested earlier on.

Table 2.2 summarised the currently available studies on bond strength of restorative materials with SDF-treated dentine.

Table 2.2 Previous studies of bond strength of restorative materials with SDF-treated dentine.

Author(s)/ Year	Type of study	Intended test	Materials tested	Treatment protocol	Changes in bond strength
Yamaga et al. (1993)	In-vitro study	Adhesiveness of GIC containing tannin-fluoride preparation (HY agent) to dentine — an evaluation of adding various ratios of HY agent and combination with application of diammine silver fluoride	GIC bond-normal and SDF dentine	Shear bond strength & percentage of cohesive failure of GIC to SDF treated non-carious dentine (n=7), 1 day and 1 month comparison 20kg load was applied with shear speed of 1mm/min, chart speed of 50mm/min	SBS of GIC to SDF group significantly higher at 1M than GIC to normal dentine group.
Knight et al. (2006)	In-vitro study	Effect of SDF/KI on bond strength of auto cure GIC to dentine	1) Etch-and-rinse 2) Applying polyacrylic acid 3) Etching, apply SDF/KI, wash off and air drying 4) Etching, applying SDF/KI and drying the end products.	10 non-carious third molars were sectioned into 4 dentine surfaces treated using the 4 testing methods: 1) Etch-and-rinse 2) Applying polyacrylic acid 3) Etching, apply SDF/KI, wash off and air drying 4) Etching, applying SDF/KI and drying the reaction products. Then, it was followed by placement of GIC. Shear bond strength test were carried out.	Etched SDF/KI followed by washing and air-dried exhibits the highest bond strength when compared to etched SDF/KI, air-dried.
Quock et al. (2012)	In-vitro study	Effect of SDF on Microtensile Bond Strength to Dentine	Self-etch and etch-and-rinse adhesives	42 non-carious molars dentine pre-treated with 38% SDF, were treated either ER or SE. Tested using microtensile bond test.	No significant changes
Koizumi et al. (2016)	In-vitro study	Effect of SDF/KI on Microtensile Bond Strength to Dentine	Etch-and-rinse, self-etch adhesives, and resin modified GIC (RMGIC)	80 molars dentine pre-treated with 38% SDF/KI, were treated either ER, SE or RMGIC . Tested using microshear bond test.	Decreased bond strength

Table 2.2, continued

Author(s)/ Year	Type of study	Intended test	Materials tested	Treatment protocol	Changes in bond strength
Selvaraj et al. (2016)	In-vitro study	Microshear bond strength and nanoleakage of SDF/KI treated teeth to composite.	Etch-and-rinse and self-etch adhesives	72 non-carious third molars dentine pre-treated with 38% SDF/KI, were treated either etch-and rinse (ER) or self-etch (SE). Tested using microshear bond test.	No significant changes
Wang et al. (2016)	In-vitro study	Effects of silver-diammine-fluoride on microtensile bond strength of GIC to dentine	GIC with normal and artificial caries affected dentine that has been treated with SDF with addition of light cure	Microtensile bond strength of 24h/7days SEM/ EDX for interfacial analysis 6 study groups: a) artificial demineralised for three days; b) demineralised, applied SDF, then precipitate was washed and air dried; c) demineralised, applied SDF, 60s light-cured, then precipitate was washed and air dried; d) 3 days stored in DI water; e) 3 days stored in DI water, applied SDF, then precipitate was washed and air dried; f) 3 days stored in DI water, applied SDF, 60s light-cured, then precipitate was washed and air dried.	1. Artificial carious dentine has a significant higher bond strength value, compared to normal dentine at 7 days compared to 24 hours. 2. The SDF or SDF light-cured pre-treatments have no significant impact on the bond strength of GIC and dentine 3. All treatment groups experience an increase in bond strength in all subgroups, regardless of SDF placement.

Table 2.2, continued

Author(s)/ Year	Type of study	Intended test	Materials tested	Treatment protocol	Changes in bond strength
Kucukyilmaz, et al. (2016)	In-vitro study	Effect of SDF and SiF with/without laser irradiation on MTBS on sound and caries-affected dentine	1. Self-etch bonding agent (Clearfil SE Bond, Kuraray, Tokyo, Japan) 2. Placement of 3M Filtek Z250.	96 caries free molars stored in 0.5% thymol solution at 4˚C, used within one month of extraction. Creation of artificial caries by pH-cycling procedure.	Reduced bond strength with SDF treated caries-affected dentine compared to normal dentine.
Puwanawiroj et al. (2018)	In vitro study	Microtensile Bond Strength Between Glass Ionomer Cement and Silver Diammine Fluoride-Treated Carious Primary Dentine.	Forty natural carious molars were treated with 38 percent SDF, and the control, deionized water before GIC were placed.	Microtensile bond strength testing was carried out. The failure mode was assessed with a stereomicroscope under 40X magnification	Bond strength between GIC and carious primary dentine was not affected by SDF application
Gupta et al. (2019)	In vitro study	Effect of silver diamine fluoride- potassium iodide and 2% chlorhexidine gluconate cavity cleansers on the bond strength and microleakage of resin-modified glass ionomer cement.	Freshly extracted non-carious molars were tested for bond strength and microleakage while using SDF-KI and CHX as cavity cleansers in resin-modified glass ionomer cement (RMGIC) restoration.	Shear bond strength was evaluated using a universal testing machine. Rhodamine-B dye penetration was viewed under a fluorescent microscope to evaluate the microleakage of RMGIC.	SDF-KI application had shown a drastic increase in the bond strength of RMGIC.

Table 2.2, continued

Author(s)/ Year	Type of study	Intended test	Materials tested	Treatment protocol	Changes in bond strength
Ng et al. (2020)	In vitro study	Shear Bond Strength of Glass Ionomer Cement to Silver Diamine Fluoride-Treated Artificial Dentinal Caries	Permanent molars were sectioned and demineralised to create artificial carious lesions. In five groups, the demineralisation of dentin, application of SDF, use of conditioner, and elapsed time between the placement of SDF and restoration were tested.	Differences in SBS were tested using an UltraTester machine.	Bond strength did not increase significantly after placement of SDF before the GIC was placed to dentine lesions. Treatment with SDF and use of conditioner did not statistically affect the SBS of GIC to demineralised dentin. Improved retention of GIC was noted when SDF was left for a week prior to GIC placement.
François et al. (2020)	In vitro study	Shear bond strength and interfacial analysis of high-viscosity glass ionomer cement bonded to dentin with protocols including silver diammine fluoride	Sound dentine specimens were used in the study. Surface treatments were divided into 6 groups, n = 22: i) water; ii) polyalkenoic acid; iii) SDF; iv) SDF + potassium iodide (KI); v) SDF + KI + polyalkenoic acid; vi) SDF + KI + two weeks of storage in water + polyalkenoic acid	SBS were carried out after 48 h, and 2 samples were cut and subjected to environmental scanning electron microscopy (E-SEM) and energy-dispersive X-ray (EDX) analysis.	No significant differences in SBS on the adhesion of HVGIC were found when SDF was placed on the sound dentine.

SDF: silver-diammine-fluoride; SiF: ammonium hexafluorosilicate; GIC: glass ionomer cement; MTBS: microtensile bond strength; CAD: Caries-affected dentine

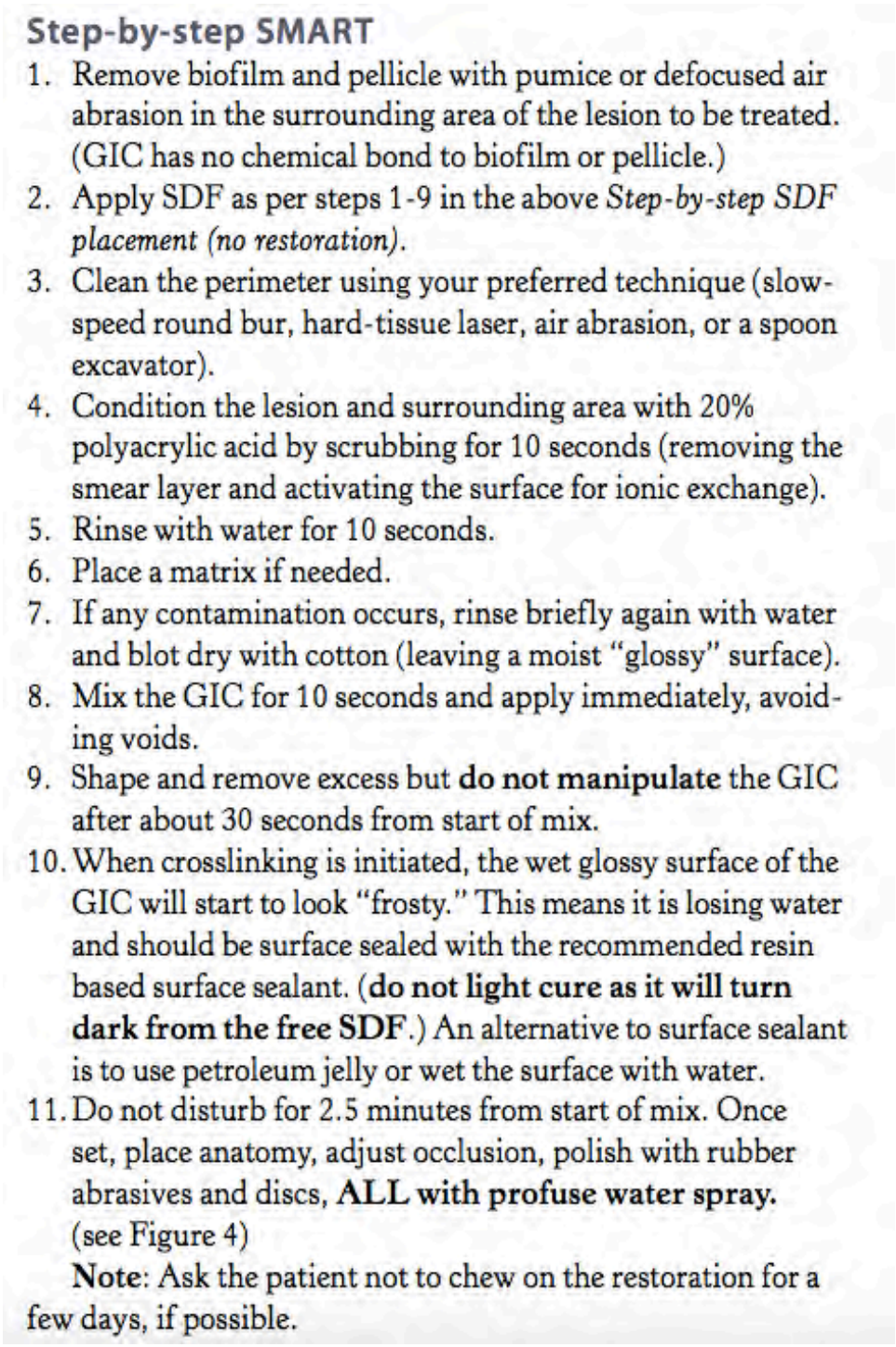
2.4 Ion Uptake in Demineralised Dentine

Fluoride concentration levels and penetration depth into the dentine are significantly increased when AgF was applied prior to the placement of auto-cure glass ionomer cement. (Knight et al., 2006). The penetration of the various elements into demineralised dentine was measured using electron probe microanalysers and relative percentage weights. Fluoride penetration into dentine was greater than 250μm although the demineralisation solution creates 150μm depth of demineralisation in dentine (Knight et al., 2006).

2.5 Dental Restoration- glass ionomer cements

Glass ionomer cements (GIC) are widely used as restorative materials in dentistry. With time, the conventional glass-ionomers, when interacting with dentine, will form an ion-enriched interfacial zone. The setting reaction of the glass ionomer cements is of alumino-silicate glass powder, when mixed with the polyalkenoic acid, forming metal ion (calcium and aluminium) polyalkenoates. The set material is formed by unreacted glass spheres surrounded by a silicaeous gel and incorporated in metal polyakenoates. Upon mixing the liquid and powder, an acid-base reaction occurs. The acids in the mixture react with the glass particle forming acid-base reaction and releases metal ions. Polyalkenoic acid chains are crosslinked by the metal ions. The newly set cement has an immature outer surface; if this dries out, micro-cracks can form on the cement surface, leading to a chalky appearance (Wilson and McLean, 1988). It could also absorb water, causing loss of network crosslinking ions and swelling during the initial setting (Lohbauer, 2010).

A Silver-Modified-Atraumatic-Restorative-Technique (SMART) procedure (Figure 2.4) has been mentioned by Bendit & Young (2017) by using GIC after placement of SDF.



Step-by-step SMART

1. Remove biofilm and pellicle with pumice or defocused air abrasion in the surrounding area of the lesion to be treated. (GIC has no chemical bond to biofilm or pellicle.)
2. Apply SDF as per steps 1-9 in the above *Step-by-step SDF placement (no restoration)*.
3. Clean the perimeter using your preferred technique (slow-speed round bur, hard-tissue laser, air abrasion, or a spoon excavator).
4. Condition the lesion and surrounding area with 20% polyacrylic acid by scrubbing for 10 seconds (removing the smear layer and activating the surface for ionic exchange).
5. Rinse with water for 10 seconds.
6. Place a matrix if needed.
7. If any contamination occurs, rinse briefly again with water and blot dry with cotton (leaving a moist "glossy" surface).
8. Mix the GIC for 10 seconds and apply immediately, avoiding voids.
9. Shape and remove excess but **do not manipulate** the GIC after about 30 seconds from start of mix.
10. When crosslinking is initiated, the wet glossy surface of the GIC will start to look "frosty." This means it is losing water and should be surface sealed with the recommended resin based surface sealant. (**do not light cure as it will turn dark from the free SDF**.) An alternative to surface sealant is to use petroleum jelly or wet the surface with water.
11. Do not disturb for 2.5 minutes from start of mix. Once set, place anatomy, adjust occlusion, polish with rubber abrasives and discs, **ALL with profuse water spray.** (see Figure 4)

Note: Ask the patient not to chew on the restoration for a few days, if possible.

Figure 2.4: The step-by-step SMART protocol by Bendit & Young (2017).

2.5.1 Adhesion of GIC to Dentine

Chemical bonds form when GIC adheres to dentine. One type of chemical bond is chelation bonding with calcium and phosphate in tooth structure. The next type of bond

is the static electrical ion bridge reaction, or the hydrogen bonding of the cement mixture's carboxyl group (-COOH) with carboxyl groups, carbonyl groups, amino groups, or imino groups of organic collagens, or with hydroxyl groups, hydrogen ions, or metallic ions in the tooth surface (Yamaga et al., 1993). The last type of bonding is the polyalkenoic acid adsorption onto hydroxyapatite surfaces and collagen.

Properties of GIC include a lower stiffness, more prone to elastic deformation, more vulnerable to wear, as well as lower fracture toughness compared to resin-based composites. It is being used in Atraumatic Restorative Technique (ART). This technique is intended to enable dentists to carry out restorations in areas distant from electrical sources. In addition, ART relies on hand instruments for opening tooth cavities, removing carious dentine, and mixing the material. Fluoride release of GIC is throughout the lifetime of the restoration. When the intraoral fluoride concentration is raised the "reservoir effect" of the GIC will able to take up fluoride (Nakanuma et al., 1998).

Light-cure GIC (LC) is also known as resin-modified GIC (RMGIC), is a "command" setting GIC that aids in tackling the low early mechanical strength of conventional GIC and the moisture control during clinical operation. The resin component allows light-curing, autocuring, or both.

Zhao et al. (2017a) found that resin-modified GIC had a higher SBS to demineralised dentine, which could be attributed to the monomer, 2-hydroxyethyl methacrylate (HEMA), which is added into this product. Due to the lack of smear layer in the demineralised dentine surface, the hydrophilic HEMA can penetrate into the exposed collagen fibre network. It is the resin addition to the GIC that increases the dentine bond strength. This GIC has chemical as well as micromechanical adhesion to

dentine (Zhao et al., 2017b). It has better strength, wear resistance, and aesthetics than conventional glass ionomers.

2.5.2 Rationale of Using GIC after SDF

The lesions in the studies were not restored following the SDF, and the cavity was left as a frank cavity (Zhi et al., 2012; Yu et al., 2018). However, placement of a restoration facilitates plaque control by eliminating plaque-retentive areas and improves the area's cleanability.

High viscosity GIC (HVGIC) is the most appropriate material for ART. There are two varieties of HVGIC, namely self-cure and light-cure. It has` higher compressive strength, due to the fine glass particle size and it uses a higher powder to liquid (P/L) ratio. It has excellent packability for ease of handling. When compared to conventional GIC, HVGIC shows superior mechanical properties, as shown in Table 2.3, reduced wear and restoration fracture when use in load-bearing posterior teeth. This is due to the mixture of conventional GIC structure with ultrafine and highly reactive glass particles, with a higher-molecular-weight polyalkenoic acid to improve the mechanical and wear properties (Khoroushi & Keshani, 2013). A four-year study done by Gurgan et al. (2015), when evaluating the HVGIC for the restoration of posterior teeth, it exhibited successful clinical performance.

Self-cure GIC adheres to enamel and dentine surface by chemical bonding by ionic displacement of calcium and phosphate with polyacrylate ions. Chemical bonding by adhesion occurs at the wetting stage. The cement is able to form a close contact with the surface, aided by hydrophilic dentine surface and cement. Formation of hydrogen bonds between the free carboxylate groups of the cement and the layer of tightly bound water

on the surface of the tooth. The hydrogen bonds will be gradually replaced by genuine ionic bonds formed between calcium ions in the tooth and carboxylate groups from the polymer within the cement. This would form an ion-exchange layer/ intermediate layer (Wilson AD, 1974; Yoshida et al., 2000).

Light- cure HVGIC (LC) could be used in sandwich technique and as base for resin composite instead GIC due to the improved bond strength to resin composite because of its chemical bonding (Farah et al., 1998). It can strengthen the bond between teeth and resin-based composites, if a resin composite is required for the more esthetic and it has higher masticatory force. LC achieved adequate bond strength to withstand contraction forces from overlying resin composite (Deepa et al., 2016). The lower carboxylic acid and water content of the hybrid cement reduces its potential to wet enamel or dentine, resulting in a higher microleakage percentage than conventional glass ionomers. If hybrid ionomer in LC contains nonreactive filler particles, less carboxylic acid is available for bonding to tooth structure, thus a dentine bonding system or surface conditioning is needed. LC have two mechanisms for dentine bonding: chemical bonding, plus enhancing the features of micromechanical interlocking as there is the addition of small portions of resin components - hydroxyethyl methacrylate (HEMA) - into the liquid portion. The binding of GIC to the dentine layer could be impaired by the smear layer (Nakanuma et al., 1998). The LC might contain a high molecular weight resin-modified component, therefore it was preferentially retained on the acid-etched dentine surface, within minimal diffusion (around 500nm) into the underlying demineralised collagen. The HEMA component can diffuse into the interfibrillar spaces, but these spaces might not be completely infiltrated by high molecular weight PAAs in chemically cured GICs.

Calcium aluminate GIC/ hydraulic cement is another alternative. However, a clinical trial showed high failure rate on the calcium aluminate cement restorations when

it was being used in load bearing area (Phillips' Science of Dental Materials 12th edition). This cement is used specifically for dental procedures involving dental pulp or root system. The precipitation of hydroxyapatite, which is also called biomineralisation can shield dental tissue from underlying cement. The cements also adsorb ions that stimulate cytokines, this contributes to healing of the dental pulp or in tissue surrounding the root of a tooth (Primus et al., 2021).

Table 2.3: Properties of restorative GICs (Xie et al., 2000)

Materials	Compressive Strength (MPa)	Diametral Tensile Strength (MPa)
Type II	196-251	18-26
Cermet	176-212	19-22
High-viscosity	301	24
Hybrid	202-306	20-48

2.5.3 Ways to improve adhesion in GIC

Diffusion from the cement ions and ions from the tooth can move into the interfacial zone and create an ion-exchange layer or interfacial zone. This layer could be observed using scanning electron microscopy. Interfacial zone can contain both calcium

and the ion from GIC, results from the ion movement from both the cements and the tooth. This will help the cement and the tooth to adhere strongly.

Despite GIC self-adhesive properties, the quality of attachment between GIC and tooth substrates can be influenced by the use of pre-treatments. Pre-treatments, such as the application of an etchant or conditioner, may alter the surface characteristics of the tooth and enhance the bond between the tooth and the GIC. These pre-treatments are often employed to improve the bond strength and clinical performance of GIC restorations.

2.5.3.1 Increase bond strength of GIC

In order to increase the bond strength of GIC, we can do a few modifications to the dentine surface. Citric acid treatment removes residue from the tooth surface, exposes the dentinal tubules, and forms a mechanical interlocking to enhance the bond between cement and dentine. However, the removal of the calcium and phosphate-rich smear layer reduces the interfacial interaction between cement and dentine due to the decrease of surface minerals (Yamaga et al., 1993).

Usage of pumice paste on SDF-treated dentine before placement of resin, can reduce the negative effect of SDF on the bond strength (Braz et al., 2020). Removal of dentinal smear layer can improve the bond strength and the retention rate of GIC to dentine. The acquired pellicle on the tooth surface may interfere with the bonding of restoration.

2.5.3.2 Addition of Surface Treatment

Bonding of GIC could be improved if the surface is pre-treated with a weak polyalkenoic acid conditioner (Ramachandran et al., 2018). The pre-treatment would increase in adhesion property of aluminosilicate polyacrylic acid glass.

Dentine has higher water content, but less mineral phase for bonding when compared to enamel. It has fluid-filled tubules which could impair the bonding interface between the dentine and cement. Glass ionomers that are self-cure and light-activated are hydrophilic and capable of creating adhesive bonds and wetting the freshly sliced dentine surfaces (Mount and Hume, 2005; Ogata et al., 2001).

Smear layers consists of shattered hydroxyapatite, as well as denatured collagen in mineral phase. The cutting of tooth surface forms smear layer, which is 1-2 μm in thickness and it has a strong bond with the underlying dentine. Dentine bonding in Resin-Modified GICs involves addition of small portions of resin components – HEMA – into the liquid portion. The binding of GIC to the dentine layer could be impaired by the smear layer (Nakanuma et al., 1998). The removal of the smear layer will create a uniform surface for bonding and prevents any blocking of dentinal tubules. This allows the GIC to penetrate the surface. The attachment to the teeth is via micro-mechanical attachments when the cement sets (Koibuchi et al., 2001).

37% phosphoric acid is an acid stronger than 25% polyacrylic acid. Phosphoric acid can help in removing the smear layer and promote micromechanical adhesion by demineralisation of the tooth substrate on the intertubular and peritubular dentine. Conditioning with polyacrylic acid, eliminates the majority of the smear layer while causing little or no damage to the sound tooth structure. It increases surface areas and exposes microporosities by partially demineralising the exposed layer of the tooth and opening up the dentinal tubules. This enables the formation of hybrid layers. Surface conditioning of dentine surfaces with a weak acid treatment in resin-modified glass-ionomer resulted in higher bond strengths than phosphoric acid pre-treatment (Pereira et al., 2002).

2.5.4 GIC Maturation time

In order to reach maximum bond strength, GIC needs time to mature. In conventional GIC, it could between 24 hours to 1 week to reach full maturation, whereas in light-cure GIC, the resinous portion would set immediately upon light curing but not the GI portion. The setting reaction stops when GIC has reached the maximum bond

strength, and the bond strength gradually decreases subsequently (Yamaga et al., 1993). The ions in GIC crosslink the polyalkenoic acid chains once mixed. The cement hardens as a result of the metal ion crosslinking and the neutralisation of the polyalkenoate molecules. This happens in a short amount of time, usually 2 - 5 minutes after mixing, and the cement can then be finished. Therefore, it has lower SBS if the GIC was tested immediately after placement.

Cohesive failure mostly occurs when GIC was subjected to macro SBS, because of low early wear resistant when the glass ionomer matrix is being developed. Bonding between GICs and the tooth is achieved through chemical bonding.

2.6 Bond strength testing

The definition of bond strength is “force per unit area required to break a bonded assembly with failure occurring in or near the adhesive/adherend interface” according to ISO 29022:2013. It is usually presented as a number or value to show the strength of a bond (Armstrong, 2009).

The bond strengths are solely useful for comparing bonding agents' levels of efficacy; they have no direct bearing on what may occur in a clinical setting. This is a result of a current absence of knowledge regarding the exact interfacial stresses that an occlusal load creates in a filling restored tooth (Van Noort et al., 1989).

The value of nominal stress at failure, or the fracture load per unit area of bonded surface, is the value reported by conventional tests used for evaluating the bond strength of dental bonding agents. In reality, the achievement of a critical stress locally at the most

vulnerable location—typically the edge—of the bonded area determines the nominal strength. However, no direct or practical connection between the two measurements of strengths is generally achievable due to the non-uniform distributions and the sensitivity to the particulars of the test, especially in the case of the shear bond test (Van Noort et al., 1989).

2.6.1 Types of bond strength test

There are a few bond-strength tests for measuring the efficacy of the adhesiveness of the enamel and dentine. The bond strength could be measured using the micro or macro test set-up, depending on the bond size area. The macro test usually comes with the bond area larger than 3mm^2 and measured in “shear”, “tensile”, or “push out” method. The micro test ultilises a smaller bonded area at about 1mm^2 or less (Van Meerbeek et al., 2010).

The traditional SBS test has been criticised for failing to accurately reflect "true" bond strength. In reality, the adherend, adhesive, and adherent may not fully be in "brittle shear" mode in the conventional shear condition. Macro bond strength testing, in comparison to micro tests, is simpler to carry out. Shear bond strength does not require additional sample testing. Bond strength results corresponded to the bonding area. Increase in bonding area resulted in decrease in bond strength value (Braga et al., 2010).

2.6.1.1 Shear-Bond Test

In the SBS test, the substrate is adhered to the cylindrical adherent material. A tool - which can be a wire loop, chisel, shear blade, or metallic tape - is placed at the instrument crosshead to load test. The load speed is usually constant at 1.0mm/min. The force will be recorded by the load transducer attached to the crosshead. The force will be recorded until the sample fractures.

F = N/A, where F is the shear-bond strength (MPa), N is the maximum force exerted on the specimen (in Newtons), and A is the size of the bonding area (mm^2).

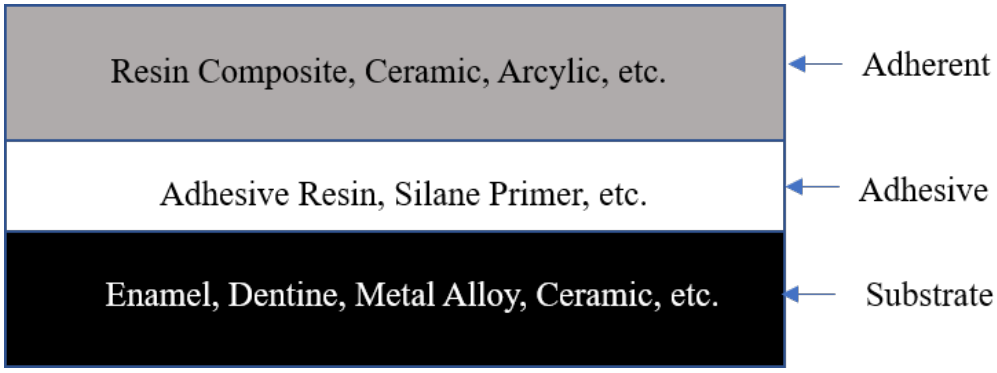


Figure 2.5: The schematic drawing regarding the adherent, adhesive and substrate layer.

In shear bond test, the terms "adherent," "adhesive," and "substrate layer"are used to describe specific components in the bonding process as described in Figure 2.5. The adherent is the substance that the bonding agent is applied. This is the layer that is receiving the adhesive. Secondly, the adhesive is the material that is applied between the adherent and the substrate to create a bond. It is the bonding agent that facilitates the attachment of the two surfaces. The properties of the adhesive play a crucial role in determining the strength and durability of the bond. Lastly, the substrate layer is the material that forms the base or foundation to which the adherent is applied. It is the

underlying surface that the adhesive bonds with. Any material that needs to be bonded together can serve as the substrate layer, such as a tooth structure, a metal surface, or any other material that requires bonding.

GIC-to-dentine macro shear-bond strength is typically in the range of 2 - 7MPa, according to previous research (Ismail et al., 2021; Braga et al., 2010), however the bond strength of dentine to LC is higher than conventional GIC (14 - 22 MPa) (Koizumi et al., 2016; Gupta et al., 2019). Smaller sized specimens may have higher reading in SBS due to reduced likelihood of a critical-sized defect present and alignment relative to the applied load (Söderholm, 2009).

The study proposed by Cheetham et al. (2013) indicates the potential utility of the mould-enclosed shear-bond strength (ME-SBS) test. The mould would be used to enclose the test stub, and provides a more consistently distributed loading weight for the adherent as close as possible to the adhesion zone. Heterogeneous stress can be removed using the ME-SBS test. By using ME-SBS, the removal of the mould is not required, thus reducing the differences between independent measurements.

Jin et al (2016) proposed a new idea of a lever-induced mould-enclosed shear-bond strength (LIME-SBS) test with an enclosed mould, with cylindrical adherent bonded to the substrate with/without adhesive. In shear-bond strength testing, a fulcrum is added at the midpoint between the bonded interface and the load. The load would be applied to the mould far away from the adhesion zone. The model using LIME-SBS could provide even stress distribution to the bonding surface, allowing for the best evaluation of shear.

The common way to prepare the macro shear-bond strength testing involves the preparation of specimen by placing the adhesive on the dentine surface, which the

cylinder mould will be placed on the tooth surface before the adherent being placed into the cylindrical mould. In the top design in Figure 2.6, usually this will cause the specimens to be debonded at a larger area than what we intended to see at the adhesive region. The Ultradent jig (top), which is the representative of SBS, and the SDI rig (bottom), which is the representative of ME-SBS (Van Meerbeek et al. (2010). In the bottom design, the specimens could be debonded at a specific area demarcated at the adhesive region.

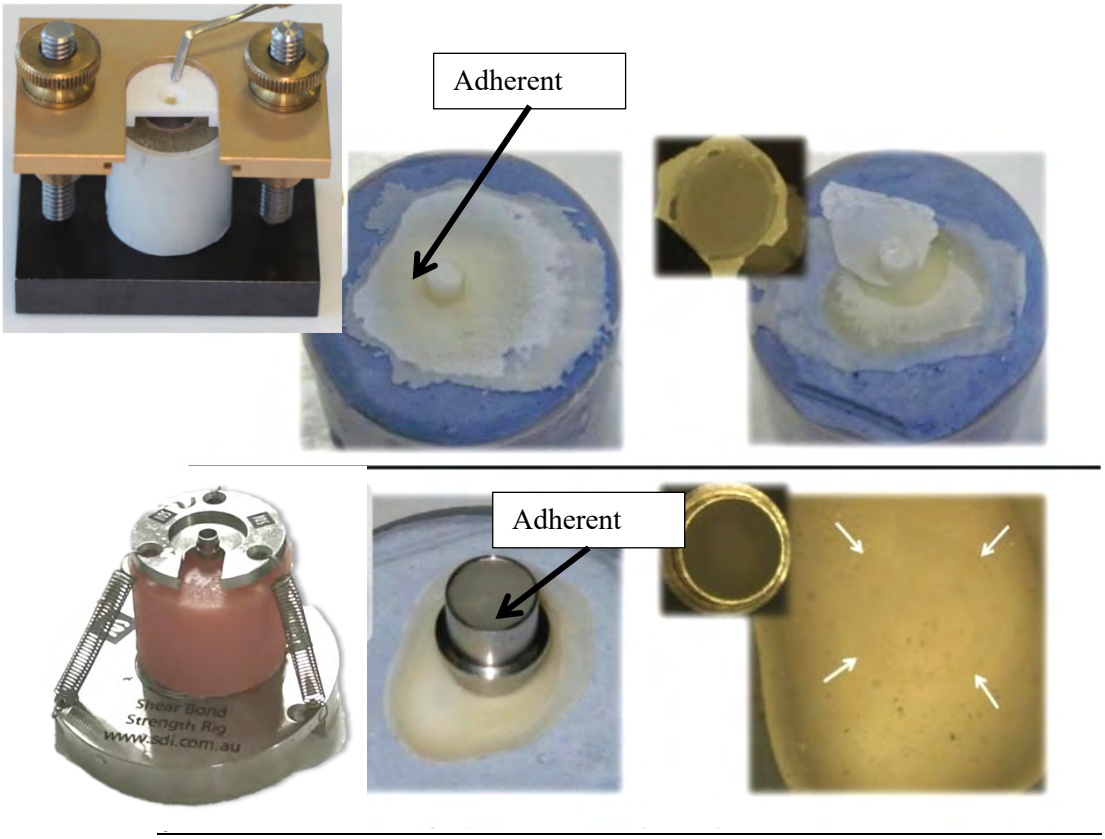


Figure 2.6: The Ultradent jig (top) and the SDI rig (bottom) (Van Meerbeek et al., 2010)

2.6.1.2 Tensile Bond Test

In the tensile bond test (TBS) (Figure 2.7), samples are prepared to be broken by tensile stress working at a 90° angle (perpendicular) to the tooth surface. Calculation of

TBS is comparable to SBS; the force at fracture over the bonded cross-sectional area is used to calculate the TBS. Active or passive gripping methods can be used to hold the specimens.

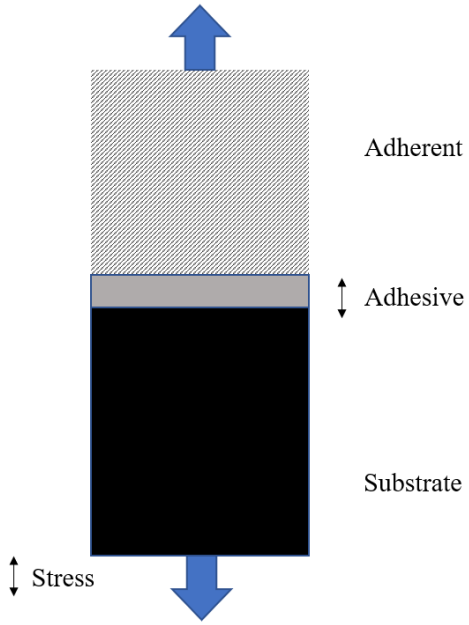


Figure 2.7: Tensile test of a bonded assembly

In the active gripping method, specimens are mechanically fastened to the gripping device using glue or clamps, whereas in the passive gripping method, specimens are positioned in the testing apparatus without the use of glue or clamps (Sirisha et al., 2014).

The test apparatus should ensure that the substrate and adhesive material are aligned during the test, which means that the tensile force can be applied at a 90° angle to the planed substrate surface. One issue with this test is in maintaining alignment both during bonding and testing to avoid stress concentration due to incorrect interfacial geometry. Furthermore, the TBS requires fine sectioning of the specimens which may lead to initial/eventual cracks on the specimens.

2.7 Factors affecting bond strength

2.7.1 Variables associated to specimen preparation

Bond strength can be affected by substrates (enamel/dentine). A study by Yazici et al. (2007) showed that there are significant differences in SBS between enamel and dentine. Glass ionomer cement has a higher bond strength to enamel than to dentine. This is because the cement interacts with the hydroxyapatite mineral phase, instead of the collagen in the dentine (Wilson & Mclean, 1988). Dentine bond strength increases over several days, reaching 80% bond strength after 15 minutes (Lin et al.,1992; Powis et al., 1982).

2.7.1.1 Dentine Depth and Permeability

The reduction of dentine thickness increases its permeability, thus decreasing the shear-bond strength. The removal of the smear layer results in a significant increase in the permeability. Superficial dentine has less water content than deep dentine, due to the bigger diameter and number of tubules per unit area. Sharafeddin et al. (2020) believed that SBS in superficial dentine is higher than deep dentine. Caries-free dentine will show uniform permeability. Tooth surfaces must be kept wet, since drying the tooth surface for several minutes may cause damage to the bonding surface, as dentine is sensitive to dehydration.

2.7.1.2 Smear Layer

The smear layer is made up of an organic film composed of apatite particles ranging in size from 0.5 to 15μm on all surfaces, though it is not always continuous (Eick et al., 1970). When the tooth surface is cut by hand or rotary instruments, smear layers

are formed (Gwinnett, 1983). In clinical scenarios, bacteria and saliva contamination might form a biofilm layer (White et al., 1989). A smear layer covering the bonding surface also has the drawbacks of a weak bond to the underlying dentine and being brittle (Pashley & Henry, 1991).

The removal of the smear layer prior to bonding or the use of bonding agents that can penetrate beyond the smear layer while incorporating it are the two main options for overcoming low bond strengths due to the smear layer's limited strength (Pashley et al., 1997). Both methods have been proven to be effective (Van Meerbeek et al., 2003).

A smear layer is commonly prepared by grinding a tooth with silicon carbide (SiC) paper for practical reasons. SiC paper with a grit of 600 is frequently used. Tooth structure is cut by rotary or manual instruments during cavity preparation, resulting in denatured collagen and altered surfaces. When dentine was cut by diamond bur and carbide rotary instruments, according to Brännström et al. (1979), the diamond cut dentinal surfaces appear to have more irregularities and a thicker smear layer. A study by El-Askary et al. (2008) showed that dentine and glass-ionomer adhesion, are proved to be independent of the smear layer thickness. With the interposition of a smear layer, bonding of GIC could be achieved even without the usage of dentine conditioner (Inoue et al., 2004).

2.7.1.3 Substrate Location

Higher bond strength to the occlusal dentine was observed than buccal dentine. To reduce variation and produce a uniform dentine structure, it is recommended that superficial dentine be used, as close to enamel as possible. As a result of that, substrate location must be specified when bond strength is studied (Sirisha et al., 2014).

2.7.1.4 Storage condition and time

Storage media such as distilled water can be used. However, the storage medium must be replaced weekly to limit bacterial growth (Liebermann et al., 2017). This is so as the intense bacterial contamination typically results in an additional adverse impact on surface properties. 0.5% aqueous solution of chloramine-T could be used as preservative, and it need to be stored in distilled water for at least 2 h to reduce the chloramine concentration before restoratives are used (Rolland et al., 2007). Aldehydes and other preservatives that react with dentine or inhibit radical polymerisation (phenols, for example) must not be used. Teeth that have been extracted for more than six months prior to testing may experience dentinal protein degeneration, according to the ISO/TS 11405.

2.7.2 Variables related to testing specimen properties

2.7.2.1 Bonding area

A clear delimitation of the bonding area is important, according to ISO/TR 11405, and the diameter of the bonded surface must be stated when comparing bond strengths. The specific value for bond area wasn't mentioned by ISO/ TR11405. In static tests, the test specimen will be stationary when bond strength testing is carried out. Sano et al. (1994) reported decreased tensile bond strength when the bonding area increased, with specimens with rectangular bonding areas between 0.25-11.65mm^2. Another report by El-Askary et al. (2012) showed increased in shear-bond strength when smaller surface areas of bonding area where used.

2.7.2.2 Properties related to various types of GIC

In comparison to a conventional GIC, LC provides higher bond strength to teeth. The methacrylate polymerisation of resin-based components creates more shrinkage of hybrid ionomers during setting. There are two mixing methods of GIC- which is capsule-mixed versus hand-mixed glass ionomer cements. Optimal clinical results can only be achieved with correct mixing technique, mixing time and powder-to-liquid ratio. The compressive strength of the capsule- mixed GIC were more superior than the hand-mixed GIC, suggestive that capsule-mixed GIC might be more superior on teeth surfaces where occlusal forces are higher (Arnold et al., 2022).

2.7.2.3 Operator skill and technique sensitivity

Individual operators would affect the outcome, and there are significant interactions exist between products, substrates, and operators. Differences in individual skill are always expected. This is because GIC may be difficult to handle and can lead to inadequate adaptation to cavity and leads to gaps. Bonifácio et al. (2013) reported no operator influence in the survival rate of proximal-ART restorations using GIC. The proper use of hand instruments when inserting the adherent to the dentine is also important to ensure there's minimal to no gap on the cavity wall.

2.7.2.4 Storage medium before Shear-bond Strength Test

Regarding the storage medium after bonding of GIC prior to the SBS test, storage of GIC in artificial saliva solution has shown better strength as compared to storage in deionised water (Bali et al., 2015). Furthermore, artificial saliva simulates the oral cavity,

which has a complex chemistry of the oral environment. GIC may release more fluoride in deionised water than artificial saliva (Mallakh and Sarkar,1990). The reason for storing in deionised distilled water (DI) is to maintain the SDF humidity and because it exhibits a thin, smooth, regular coating with some occlusion of dentine tubules (Peng et al., 2021).

2.7.3 Variables of influence related to testing mechanics

2.7.3.1 Type of loading

There are several types of configurations used to load the shear force, which include wire loops, knife-edges, and points (DeHoff et al., 1995). The use of a knife-edged chisel causes significant stress concentration at the bonded interface, whereas the use of a wire loop results in a better stress distribution at the area's edge (Braga et al., 2010).

A wire loop configuration uses the smallest orthodontic ligature wire possible (0.2mm) to provide better adhesive interface engagement and remove the substrate evenly. The wire loop can be placed at an equal radius from the adhesive interface, with the point of contact being the curved surface created by the wire. The repeated wire ductile failure could be the limitation for wire loop technique as it deforms and stretches prior to actual specimen loading. The knife- edged chisel was the traditional loading method proposed by ISO standards, despite having several concerns regarding stress concentration at a specific point on the bonded interface, leading to complex representation of stresses and underestimated bond strength values (Ismail et al., 2021).

2.7.3.2 Crosshead speed

The speed at which the sample is placed is loaded up to its breakage is essential for in-vitro test. ISO/TS 11405 recommended crosshead speed between 0.75 ± 0.30 mm/min. Low speed may capture more reliable data, meanwhile high speed could develop abnormal stresses during the mechanical testing leading to cohesive fracture. A study done by Naves et al. (2016) showed that the failure load of premolars remained unaffected by varying crosshead speeds in the range of 0.5–5.0 mm/min. Majority of studies utilised crosshead speed of 0.5 or 1.0mm/min (Scherrer et al., 2016). Braga et al. (2016) suggested that comparing load rates among testing assemblies that have various compliance levels is more significant and less challenging than comparing crosshead speeds.

2.8 Failure mode

Fractured specimens can be classified as cohesive, adhesive, or mixed. If more than 75% of the bonding area is exposed dentine, this is considered adhesive failure. Cohesive failure in materials is defined as more than 75% of the bonding area is covered with remnants of GIC. Mixed failure would be the combination of both adhesive and cohesive failures, involving between 25% and 75% of the bonding area (Francois et al., 2020). Compared to macro-sized samples, microshear samples displayed higher adhesive failure percentages. This might be explained by the smaller specimen diameter, which lowers the possibility of complicated stress formation and yields more adhesive rather than mixed/ cohesive failure (Ismail et al., 2021). A microscopic examination of the fracture surfaces can reveal the assembly's failure mode. Stereomicroscopes are frequently used to view magnifications of 10 – 50X.

The fillings are categorized as displaying cohesive failure, when cracks in glass ionomer cement (GIC) are observed close to the interface with the tooth structure. Cracks that occurs in the GIC-tooth interphase may be due to sample dehydration and it is mostly cohesive Mustafa et al., (2020). Due to the low cohesive strength of GIC, cohesive failure may be present, suggesting that GIC-tooth flexural strength is higher and GIC cracks in the bulk. Several factors can contribute to cohesive failure, such as errors in aligning the specimen along the long axis of the testing device, the generation of microcracks in the specimen during cutting or trimming, and the inherent brittleness of the involved material. (Jiang et al., 2020)

Adhesive failure was reported as a complete detachment of GIC with the dentine. Mixed mode of failure was reported when having partially attached GIC remnants to the tooth substrate. Study done by Grossman and Mickenautsch (2002), showed that 16% of the interphase layer was detached even before mechanical testing was performed. Braga et al. (2010) showed that the maximum tensile stresses was presented along the surface of fillet, which is located at the edges of the substrate and adherent. This showed that maximum stresses was not located at the interface between substrate and adherent. The study done by Braga et al. (2010) showed that the nominal bond strength, measured as the applied load at failure divided by the cross-sectional area, is not representative of the true stresses generated at the interface.

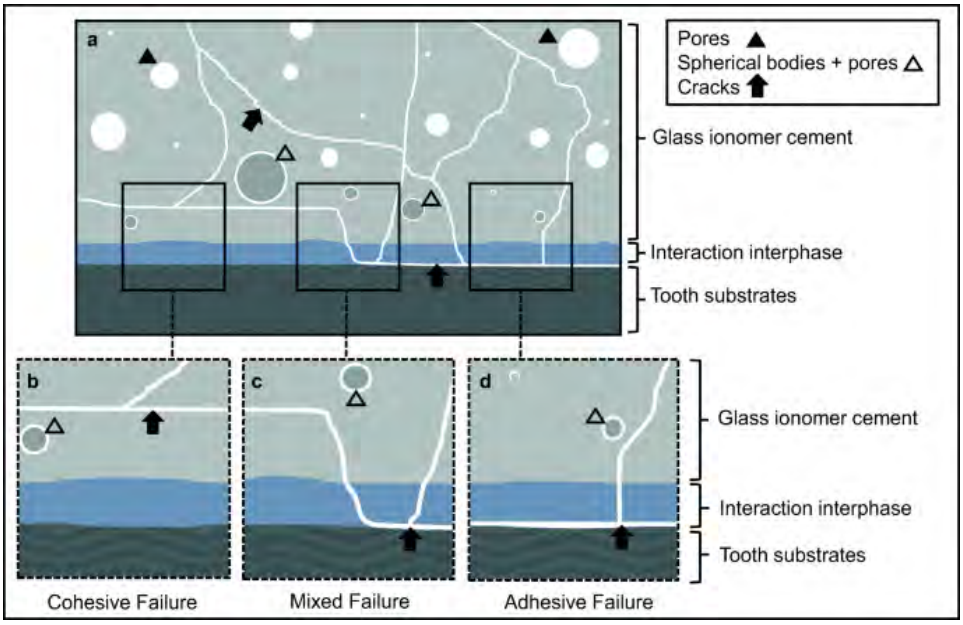


Figure 2.8: A schematic illustration of the GIC interfacial characteristics by Mustafa et al., (2020)

If the failure is adhesive in nature (located at the adhesive interface), wetting properties or chemical reactions with the substrate will most likely be required to improve bond strength. Structural cracking on GIC surfaces might form due to dehydration and stress during mechanical tests. Cracking observed on GIC surfaces typically did not result in the complete fracture of the GIC restoration at the base of the cavity. However, this differs from mechanical tests where force was applied until the entire bulk of the GIC experienced complete failure.

2.9 Research gap

In our pilot study, sound dentine exhibits higher bond strength in both LC and SC HVGIC groups as compared to demineralised dentine when the control group (no surface treatment) in SDF-treated dentine. When 25-30% polyacrylic acid was placed, the bond strength has slight increase compared to no surface treatment groups. However, the large variation in co-efficient was present. Pilot study was performed on sound dentine and superficial dentine. On polyarcylic acid, before or after the SDF were placed. In the pilot

test, the SC reading was higher when sound dentine was used when compared to simulated carious dentine (SCD). The reduced adhesion was noted, but it may be due to application of SDF, which was left unknown if there's no control group experiment were performed in our study for all the treatment groups.

Surface treatments for optimizing the bonding of self-cure (SC) and light-cure (LC) high-viscosity GICs (HVGICs) in SDF- treated dentine are still limited. This poses a significant gap in understanding the effectiveness of surface treatments in enhancing the bond between these materials. With their finer glass particle sizes and higher powder-to-liquid ratios, HVGIC materials offer shorter setting times, better handling, and higher strengths (Scholtanus & Huysmans, 2007). The effective marginal seal between GIC and the tooth surface is important after the placement of SDF. The usage of different surface treatment system such as universal adhesive, phosphoric acid and RMGIC-based adhesive may be considered as to enhance the bond strength of GICs to the SDF-treated carious teeth.

Chapter Three: Materials and Methods

3.1 Materials

This study was approved by the Ethical Committee of the Faculty of Dentistry, University of Malaya, Kuala Lumpur, Malaysia (ID: DF RD1933/0096(P). The materials evaluated in this study and their compositions are displayed in Table 3.1.

Table 3.1: Glass ionomer materials, surface treatment materials and their composition used in this study.

Material	Manufacturer	Abbreviation	Matrix Composition	Batch Number
Riva Star	SDI, Bayswater, Australia	SDF	35– 40% silver fluoride, 15-20% ammonia solution and deionised water	8800535
Riva Self Cure HV	SDI, Bayswater, Australia	SC	Polyacrylic acid, Tartaric acid, Fluoroaluminosilicate glass	K2106034EA
Riva Light Cure HV	SDI, Bayswater, Australia	LC	HEMA, acrylic acid homopolymer, dimethacylate cross-linker, acidic monomer, tartaric acid glass powder	8630002
Riva Conditioner	SDI, Bayswater, Australia	PAA	25-30% PAA	191214
Super Etch	SDI, Bayswater, Australia	PPA	37% phosphoric acid	181104
ZipBond	SDI, Bayswater, Australia	ZB	10-MDP, fluoride, photoinitiator	1170158
Riva Bond LC	SDI, Bayswater, Australia	RB	HEMA, polyacrylic acid, acidic monomer, dimethacylate cross-linker, tartaric acid.	8800600

Abbreviations: PAA: polyacrylic acid; HEMA: 2-hydroxyethyl methacrylate; 10-MDP: 10-Methacryloyloxydecyl dihydrogen phosphate

3.2 Methods

This research was divided into two: Part A and Part B. Total of one hundred and fifty extracted maxillary and mandibular premolar teeth were collected from patients aged from 18 years old to 45 years old for orthodontic treatment purpose which were collected from private dental clinics in Cheras, Selangor region. For Part A, 140 teeth were collected for the evaluation of SBS and failure modes. For Part B, 10 teeth were collected for interfacial analysis of the bonded specimens by using SEM/EDX.

The inclusion criteria consisted of:

- The storage period no longer than 6 months in distilled water at 4°C until further use
- Age of the subjects ranged from 18-45 years old
- Occlusal surface of the enamel is intact.

The exclusion criteria were as follows:

- Teeth with developmental defects
- Teeth with cracks caused by the extraction forceps
- Dental caries
- Teeth undergoing attrition
- Teeth which had subjected to any pre-treatment chemical agent.

The teeth were kept in a container containing 0.5% chloramine-T trihydrate solution for a week for disinfection purpose. An ultrasonic scaler was used to clean these teeth (Piezon® Master 400, Switzerland) to remove hard and soft debris from the teeth specimen. Following that, the teeth were stored no longer than 6 months in distilled water at 4°C until further use.

3.2.1 Part A Shear Bond Strength

3.2.1.1 Tooth preparation

Each specimen was sectioned 2.0mm from the central fissure by using a high-speed sectioning machine (Micracut 176, Metkon®, Bursa, Turkey) (Figure 3.1) to expose the flat occlusal dentine surface. Sectioning of premolars was performed perpendicularly to the long axis of the tooth. The flat surfaces were inspected under a stereomicroscope (Olympus, Japan) at 10x magnification (Figure 3.3) to ensure no remnants of enamel or exposed pulp were in the specimen. The tooth surfaces were wet all times.

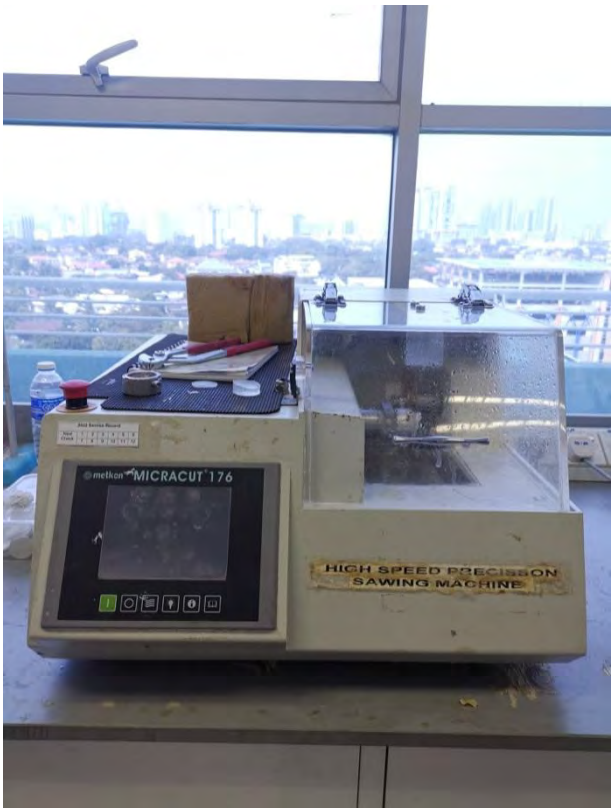


Figure 3.1 High speed sectioning machine (Micracut 176, Metkon®, Bursa, Turkey).

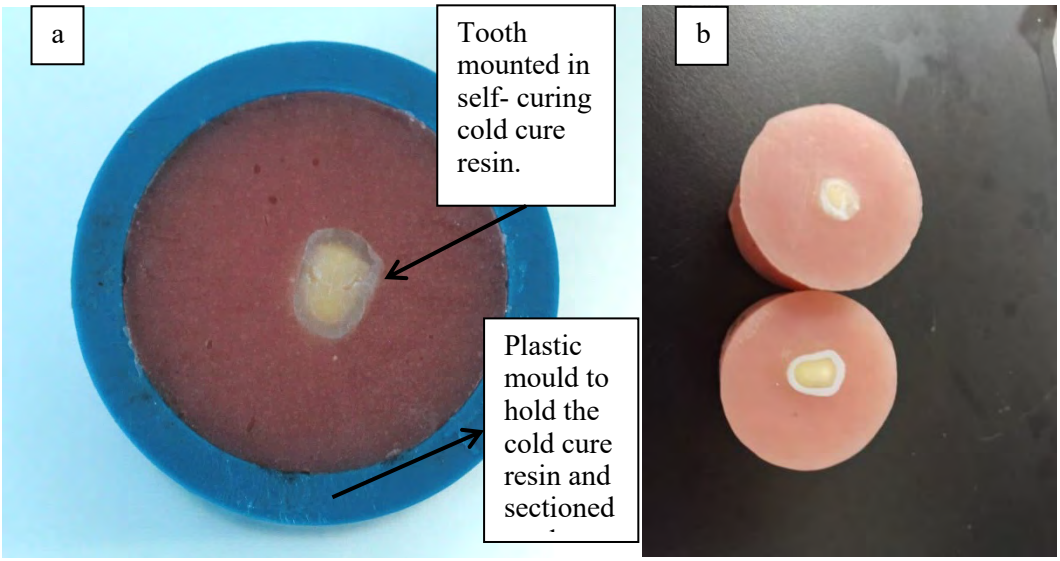


Figure 3.2 (a) Mounting of premolar tooth specimen in self-curing cold cure resin. (b) Samples removed from the plastic mount and demineralised using pH-cycling method.

Figure 3.3: Stereomicroscope (OLYMPUS szx7, Olympus Corp., Tokyo, Japan)

Teeth were mounted using plastic rings with about 25mm internal diameter and a height of about 25mm. Each specimen was embedded vertically in clear viscous self - curing resin (Kemdent, Wiltshire, UK) (Figure 3.2)**.** Removal of the mounted tooth was done immediately after they set, then stored immediately in water (ISO3696, grade 3) at 4ºC.

The dentine surface of each tooth was further polished for 60 seconds with 400 grit, 600 grit, and 1200 grit silicon carbide papers (Metkon® Instrument Ltd, Turkey) using a water-cooled polishing machine (Isomet, Buehler; Lake Bluff, IL, USA) (Figure 3.4). The ground surface was perpendicular to the mounted tooth, with top and bottom surfaces of the specimens made parallel to ensure the bonded substrate was well-aligned during bonding.

Figure 3.4: Grinding and polishing machine (Isomet, Buehler; Lake Bluff, IL, USA).

3.2.1.2 Surface preparation by pH cycling/ Demineralisation method

The polished teeth were immersed in different solution for pH cycling. The demineralising solution comprised 2.2 mM $CaCl_2$, 2.2 mM NaH_2PO_4, and 50 mM acetic acid with a pH of 4.6, while the remineralising solution comprised 1.5 mM $CaCl_2$, 0.9 mM NaH_2PO_4, and 0.15 M KCl adjusted to a pH of 7.0 (Table 3.2). Each specimen was immersed (Figure 3.2b) for 8 hours in the 10 mL demineralising solution and 16 hours in the 10 mL remineralising at room temperature (24 ± 0.1 ºC) for 10 days as shown in Table 3.2 (Dias et al., 2016; Buzalaf et al., 2010; Schüpbach et al., 1989). Fresh de- and remineralisation solutions were used every time, due to the pH solution needing to be standardised because frequent titration between high and low pH would either cause an excessive increase in ionic strength or dilute the solution (Buzalaf et al., 2010). This also prevented the solution from becoming exhausted or saturated, as well as preventing the accumulation of dentine dissolution products. It is necessary to affirm the model developed in this study by pH- cycling (Bassi et al, 2020).

A total of 4 demineralised samples from part A were being selected and were viewed under polarisation sensitive optical coherence tomography (PS-OCT) by using ThorLabs OCT system (OCM1300SS, Thorlabs, Newton, USA) for acquisition of experimental images and verification of demineralisation procedure (Figure 3.5). A 700±14μm deep, partially demineralised baseline lesion was confirmed with PS-OCT, simulating caries-affected dentine.

Table 3.2: Remineralising solution, demineralising solution and artificial saliva used in the study

Code	Material	Composition
RS	Remineralising solution (pH 7.0)	KCL, 8.83mM $MgCl_2.6H_2O$, 0.29mM $CaCl_2$. 2 H_2O, 1.13mM K_2HPO_4, 4.62mM Fluoride, 0.022ppm
DS	Demineralising solution (Adjusted to pH 4.6)	2.2mM NaH_2PO_4, 2.2mM $CaCl_2$ 50mM acetic acid 1.0M NaOH, Distilled Water
Artificial saliva	SAGF medium	NaCl,125.6mg/L KCl, 963.9 mg/L KSCN, 189.2 mg/L $CaCl_2.2H_2O$, 227.8 mg/L KH_2PO_4, 654.5 mg/L Urea, 200 mg/L NH_4Cl, 178 mg/L $NaHCO_3$, 630.8 mg/L $Na_2SO_4.10H_2O$,763.2mg/L

Figure 3.5 ThorLabs OCT system (OCM1300SS, Thorlabs, USA).

3.2.1.3 Groupings

One hundred forty premolar teeth were divided randomly into ten groups of fourteen specimens for each adhesive group (Table 3.3).

3.2.1.4 Restorative procedure

Custom-made SDI jig mould (Figure 3.6) were used. The moulds were 5mm in height with a 3.5mm inner diameter. The SDI bonding jig was placed perpendicularly on the centre of each tooth mounted after the SDF and surface treatment were placed.

The SDF-treated dentine surfaces were cleaned with pumice-water slurry and a layer of 38% SDF (Figure 3.7) (Riva Star, SDI, Bayswater, Australia) was applied by continuous scrubbing the silver capsule for 2 minutes, followed by washing, drying, and placing in deionised distilled water for 2 weeks. Potassium iodide (KI) was not used in the study.

Before the application of the tested restorative material, the surfaces were again cleaned with pumice prior to surface treatment (shown in Table 3.3). The prepared teeth were randomly divided into 10 groups of 14 and surface treated as follows:

Group 1 (T1) - no surface treatment was applied (control).

Group 2 (T2) – conditioned with 25-30% polyacrylic acid (PAA) (Riva Conditioner, SDI, Bayswater, Australia) (Figure 3.8) for 10 seconds, washed, and gently air dried.

Group 3 (T3) - etched with 37% phosphoric acid (Super Etch, SDI, Bayswater, Australia) (Figure 3.9) for 5 seconds, washed, and gently air dried.

Group 4 (T4) – etched with 37% phosphoric acid for 5 seconds, washed, and gently air dried, followed by application of a layer of universal bonding agent (Zipbond,

SDI, Bayswater, Australia) (Figure 3.10) which was thinned with air and light cured for 20 seconds. The bonding area was delineated by observing the dentine surface only.

Group 5 (T5) - etched with 37% phosphoric acid for 5 seconds, washed, and gently air dried, followed by application of a layer of RMGIC-based adhesive (Riva Bond LC, SDI, Bayswater, Australia) (Figure 3.11) was which was thinned with air and light cured for 20 seconds.

Two types of GICs were included in this study: Self-cured (Riva Self Cure HV [SC]) (Figure 3.12) and light-cured (Riva Light Cure HV [LC]) high-viscosity glass ionomer cements (HVGICs)(Figure 3.13). The 38% SDF used is Riva Star. The technical profiles of the two materials are described in Table 3.1.

Table 3.3: Surface treatment groups involved in the study.

Material	Code	Number of samples (n) Per group
GROUP 1 No treatment	T1	14
GROUP 2 Riva conditioner: 25-30% polyacrylic acid (PAA)	T2	14
GROUP 3 Super etch: 37% phosphoric acid (PPA)	T3	14
GROUP 4 Super etch: 37% phosphoric acid (PPA) + Zipbond : Universal adhesive	T4	14
GROUP 5 Super etch: 37% phosphoric acid (PPA) + Riva bond LC: RMGIC-based adhesive	T5	14

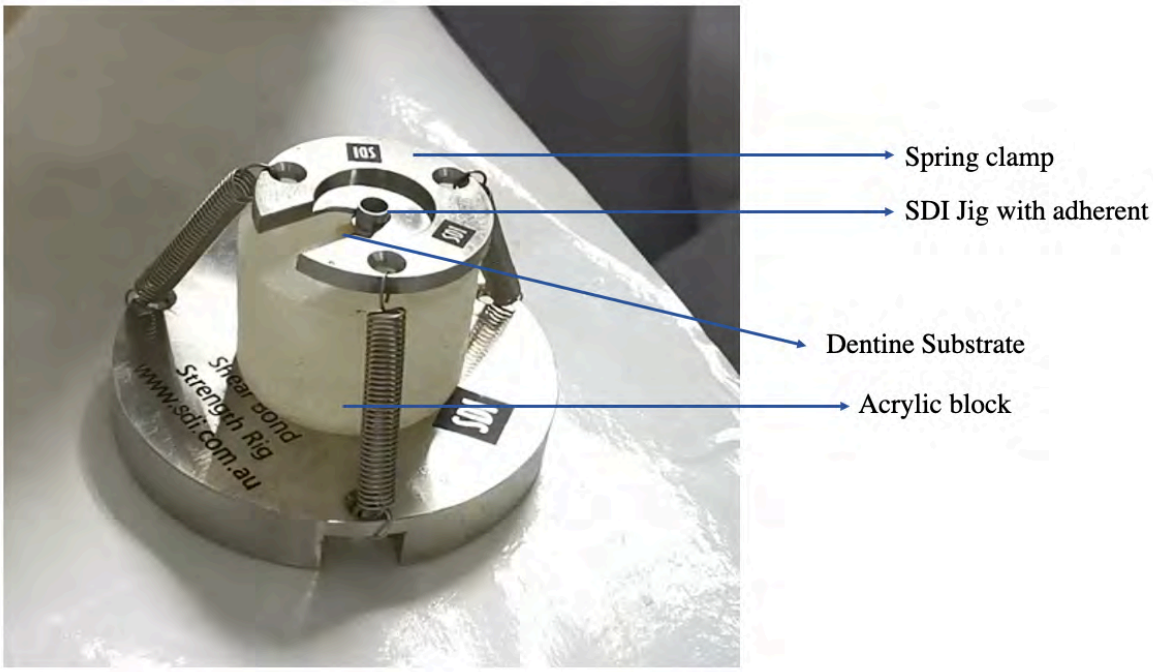


Figure 3.6: SDI Jig Mould (SDI Shear-bond Strength Rig, SDI Limited, Bayswater, Australia)

Figure 3.7 Riva Star, 38% SDF/KI (SDI, Bayswater, Australia)

Figure 3.8: Riva conditioner:

25-30% polyacrylic acid(PAA)

(SDI, Bayswater, Australia)

Figure 3.9: Super etch:

37% phosphoric acid (PPA)

(SDI, Bayswater, Australia)

Figure 3.10: Zipbond :

Universal adhesive

(SDI, Bayswater, Australia)

Figure 3.11: Riva bond LC :

RMGIC based adhesive

(SDI, Bayswater, Australia)

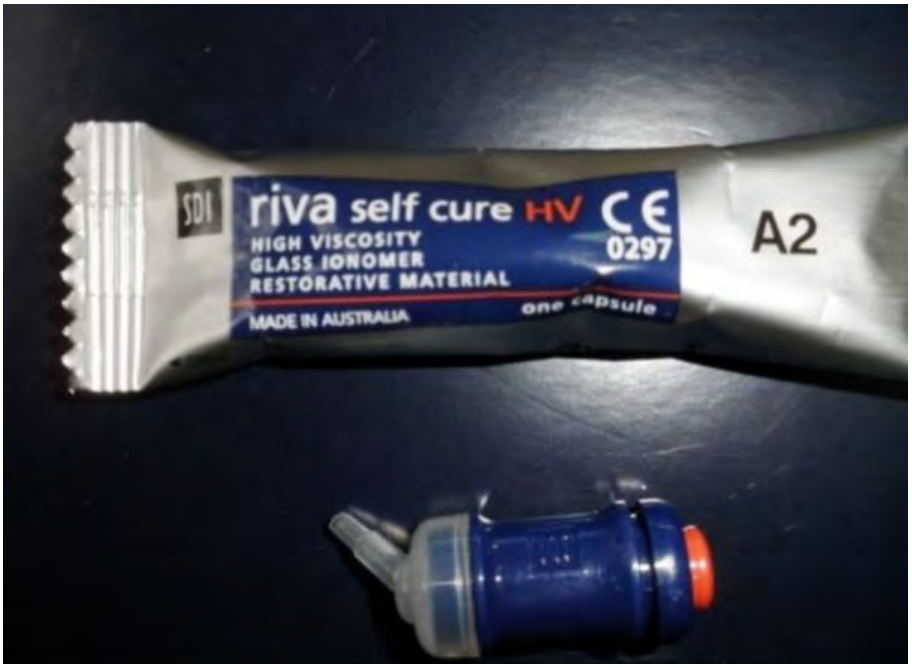


Figure 3.12 Riva self-cure HVGIC (SDI, Bayswater, Australia)

Figure 3.13 Riva light-cure HVGIC (SDI, Bayswater, Australia)

Figure 3.14 Capsule Mixer (CMII, GC, America)

Figure 3.15: Placement of GIC on to the SDI Jig Mould.

The SC and LC capsules were activated using capsule mixer (Figure 3.14) for 10s before placing them into the SDI mould (Figure 3.15). For SC, the GIC was then compressed with a mylar strip to ensure good adaption. Each layer of LC was placed 2mm at a time and light cured for 20 seconds using LED light curing unit (Demi Ultra, Kerr, Orange, USA) (Figure 3.16). The metal mould was left, unremoved at the dentine surface. A total of 14 GIC-dentine samples were fabricated for each type of the restoration material. The dentine surface of each specimen group received different surface treatments prior to placement of the GIC as outlined in the previous section (Table 3.3). The HVGICs were left to set for 5 minutes in the moulds and metal jig before removing the spring clamp from the assembly. The bonded specimens were stored with the metal moulds in-situ in artificial saliva (SAGF medium) (Table 3.2) at 37◦C and 100% relative humidity for a week.

Figure 3.16: Light curing machine (Demi Ultra, Kerr, Orange, USA)

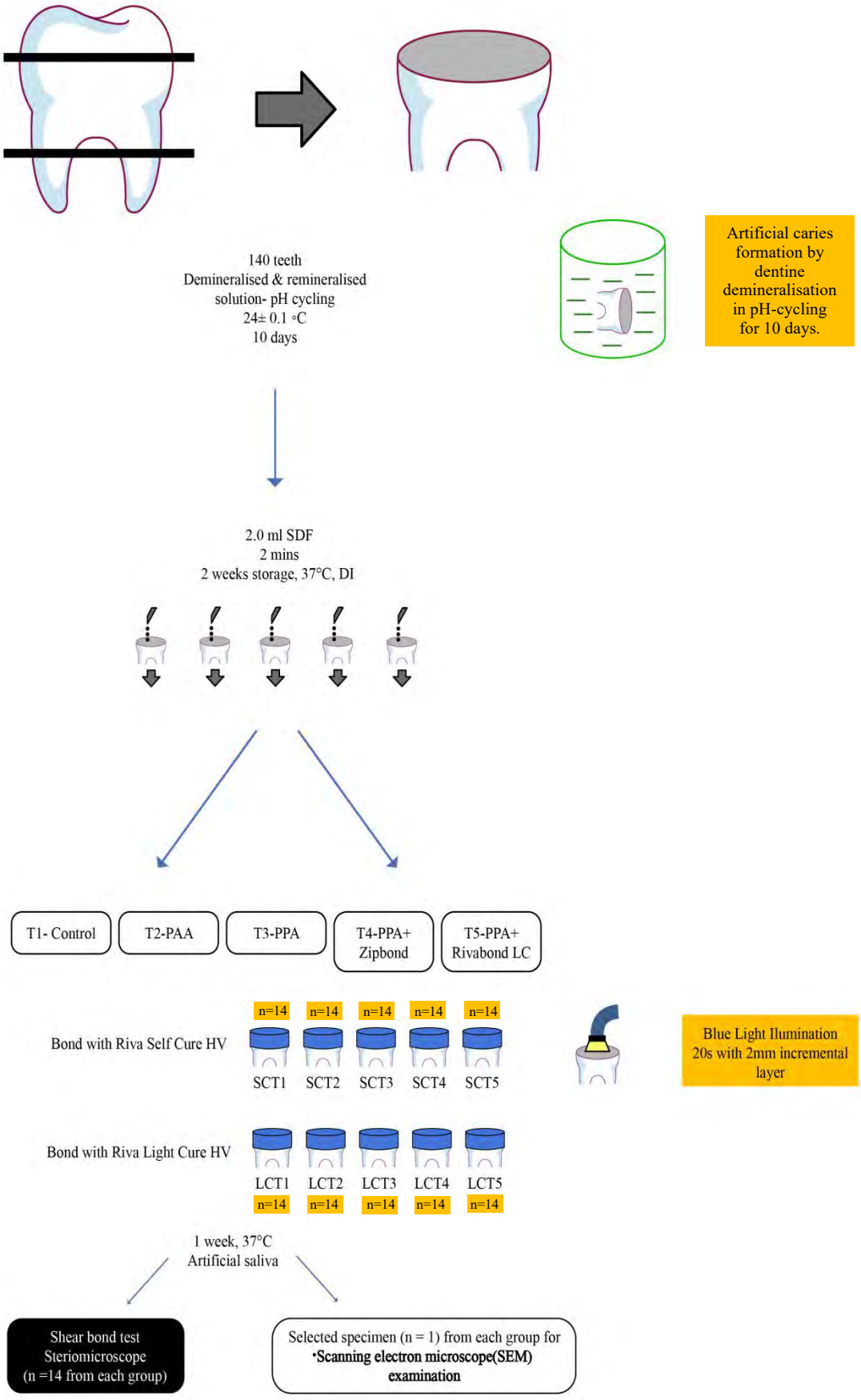


Figure 3.17: The schematic diagram of sample preparation.

3.2.1.5 Testing Procedure

The flow of the experiment was summarised in Figure 3.17. The specimens were positioned in the universal testing machine (UTM) (Shimadzu, Japan) fitted with a custom-made notched-edge blade (Figure 3.18). The long axis of each specimen was placed perpendicular to the direction of the applied force (Figure 3.19a). The experiment was carried out at a temperature of 24°C, and the specimens were removed from the deionised water immediately before the experiment were carried out. The shear loading test was performed at a crosshead speed of 1.0 mm per minute (Figure 3.19b-c) using UTM with 500N load force. The shear-bond strength was calculated as below:

F = N/A, where F is the shear-bond strength (MPa), N is the maximum force exerted on the specimen (in Newtons), and A is the size of the bonding area (mm^2).

After testing, all debonded surfaces were being examined under stereomicroscope (Olympus, Japan) (Figure 3.3) using 25x magnification to inspect the amount of GIC that are left on the dentine and to determine the mode of failure. The mode of failure was classified as below:

a) Adhesive failure is defined as more than 75% of the bonding area is exposed dentine.
b) Cohesive failure in GIC is defined as more than 75% of the bonding area is covered with remnants of GIC.
c) Mixed failure would be the combination of both adhesive and cohesive failures between 25% and 75% of the bonding area.

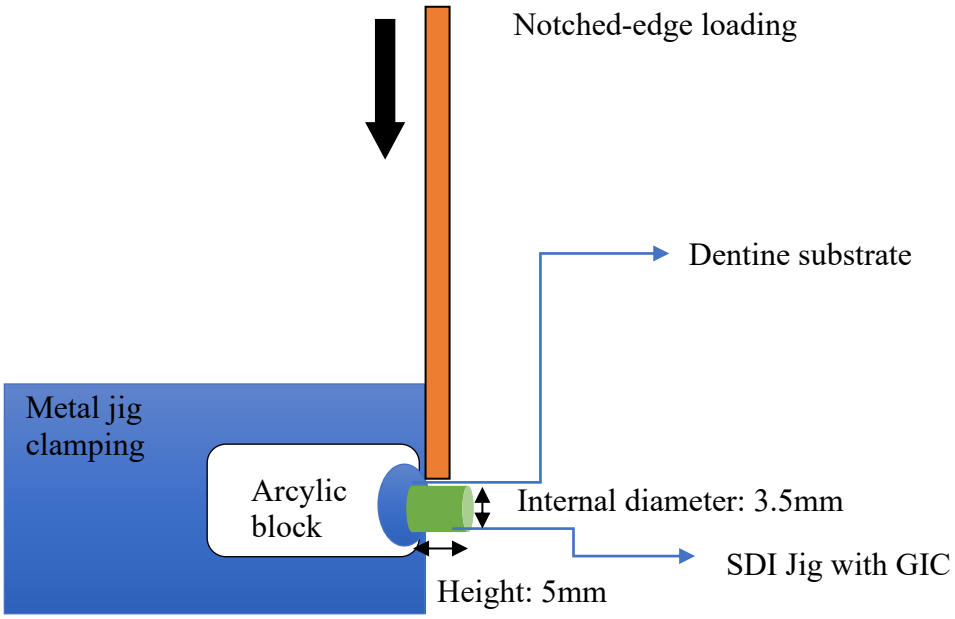


Figure 3.18: Schematic diagram of specimen with GIC build up occlusally. The cylindrical stainless-steel mould was placed with an internal diameter of 3.5mm and height of 5.00mm.

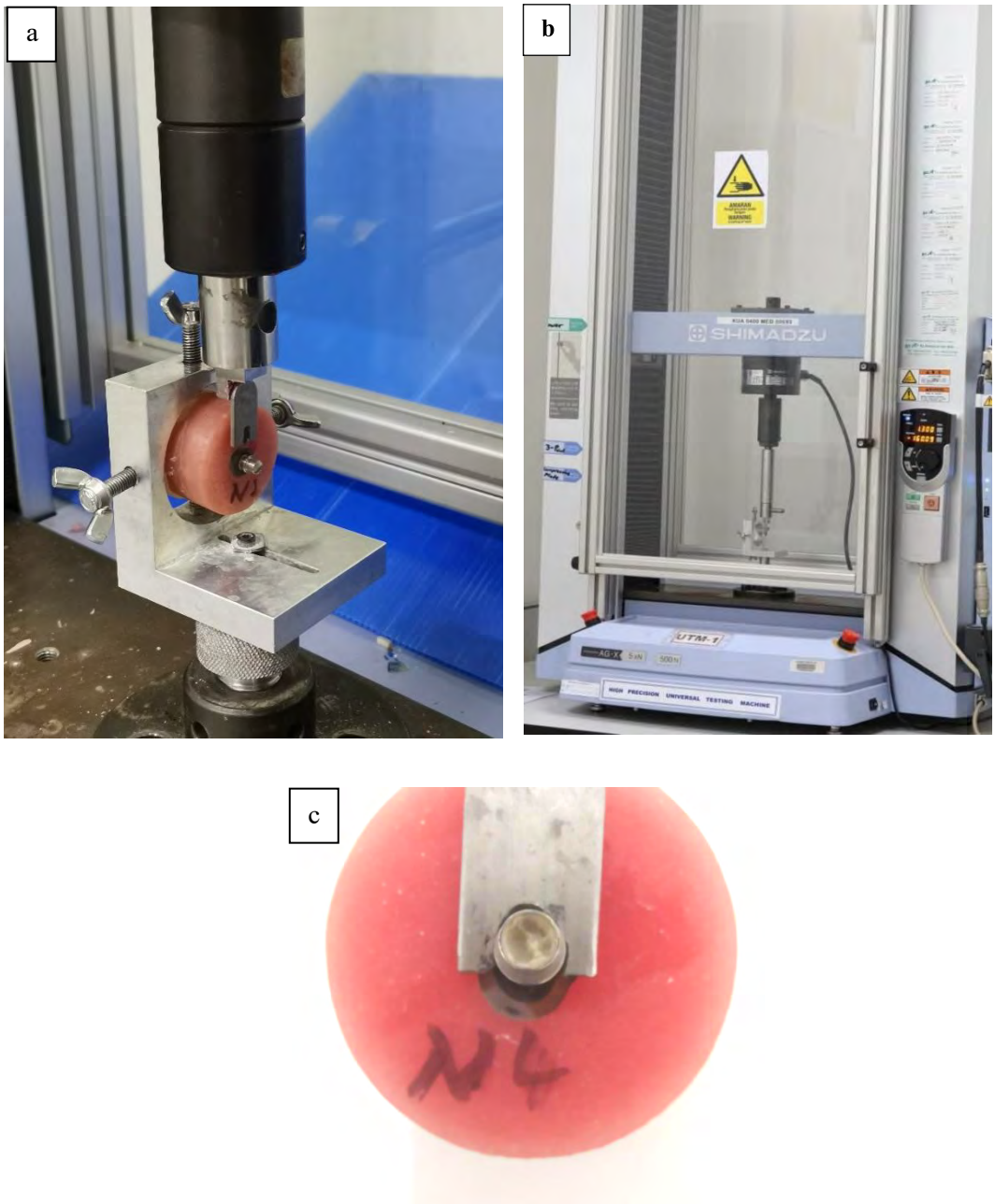


Figure 3.19: a) Universal testing machine (UTM) (Shimadzu, Japan). b) Specimen alignment with the jig in UTM. c) Close-up view of notched-edge jig with metal enclosed mould.

3.2.1.6 Data analysis

Normality of data was assessed using the Shapiro–Wilk test. Shapiro-Wilk test which is one of the most widely used methods to test the normality of the data besides Kolmogorov–Smirnov test. Shapiro-Wilk test was done because it is more appropriate for small sample size less than 50 samples). Based on Shapiro-Wilk test analysis, the data were not normally distributed. The data comparison among treatment groups SC and LC was non-parametric, thus the statistical analysis was done with Kruskal-Wallis test and post hoc pairwise comparisons (α=.05). Data was analysed using SPSS version 26.

3.2.2 Part B SEM/EDX Analysis

3.2.2.1 Tooth selection

Ten extracted sound premolar tooth were selected for Part B. They were randomly assigned to one of the five groups.

3.2.2.2 Tooth preparation

Each specimen was sectioned 2.0mm from the central fissure by using a high-speed sectioning machine (Metkon®, Turkey) (Figure 3.1) to expose the flat occlusal dentine surface (Figure 3.2). The flat surfaces were inspected under a stereomicroscope (Olympus, Japan) at 10x magnification (Figure 3.3) to ensure no remnants of enamel or exposed pulp were in the specimen. The tooth surfaces were wet at all times.

The dentine surface of each tooth was further polished for 60 seconds with 400 grit, 600 grit, and 1200 grit silicon carbide papers (Metkon® Instrument Ltd, Turkey)

which were placed on the water-cooled polishing machine (Isomet, Buehler; Lake Bluff, IL, USA) (Figure 3.4).

3.2.2.3 Surface preparation by pH cycling/ Demineralisation method

The demineralised dentine was created as mentioned in section 3.2.1.2.

3.2.2.4 Grouping

The grouping of adhesive systems in part B was the same as in part A (Table 3.3).

3.2.2.5 Restorative Procedures

The restorative procedures were carried out as mentioned in section 3.2.1.4.

3.2.2.6 Preparation of samples

Each specimen was embedded vertically in clear viscous self-curing cold cure resin in a rectangular block**.** Removal of the mounted tooth was done immediately after the setting of the resin, and stored immediately in water (ISO3696, grade 3) at 4ºC.

The specimens were sectioned through the restoration sagittally (Figure 3.17) under continuous water agitation with a high-speed sectioning machine (Metkon®, Turkey) (Figure 3.1). Each tooth was sliced into two specimens, each with a flat surface on both sides (Figure 3.20). The specimens (Figure 3.21) were then polished with a grinding and polishing machine (Figure 3.4) for 60 seconds under running water with an increasing grit of silicon carbide paper (600 grit, 800 grit, 1200 grit).

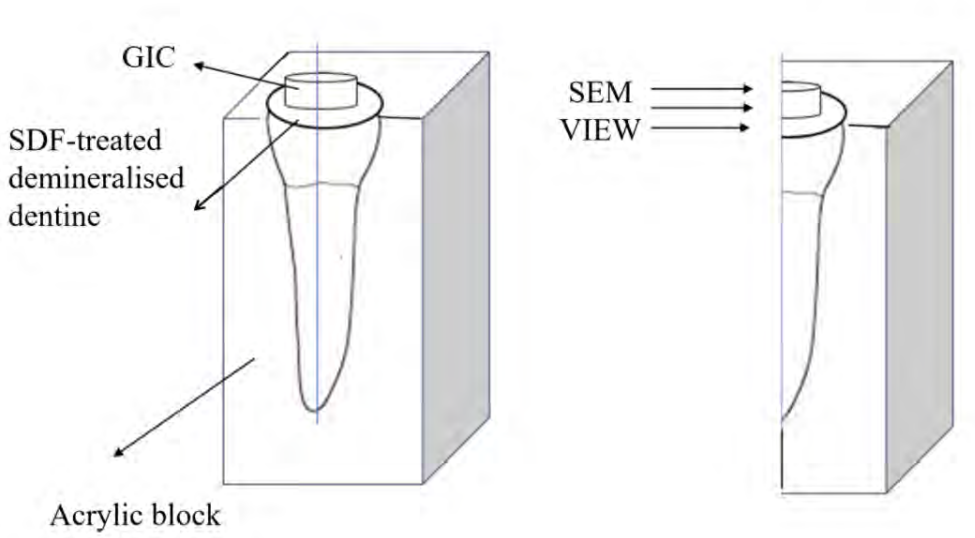


Figure 3.20: Schematic diagram of SEM/EDX samples preparation.

Figure 3.21: Specimen preparation for SEM/EDX analysis.

3.2.2.7 Examination procedure

Scanning electron microscopy (SEM) and elemental analysis were done using vacuum scanning electron microscope (SEM) (Quanta-FEG 50, FEI,

Germany) (Figure 3.22). At each time point, an image (n=1) was taken of the surface of the dentine and GIC interface at two magnifications of 500× and 2000x. SEM micrographs were produced in a SEM machine at various magnifications using an accelerating voltage of 10 kV.

Figure 3.22 Scanning Electron Microscope (SEM)

(Quanta-FEG 250, FEI, Germany)

The energy-dispersive X-ray analysis (EDX) (n=1) is ultilised to analyse the superficial dentine presented with elements including oxygen, calcium, phosphorus, silver, aluminum, fluoride, silicon and strontium.

3.2.2.8 Evaluation of adaptation failure

ImageJ software was used to analyse and calculate the amount of GIC remnants left on the dentine surface (Figure 3.23). A red photo contrast was placed on the area with remnant GIC.

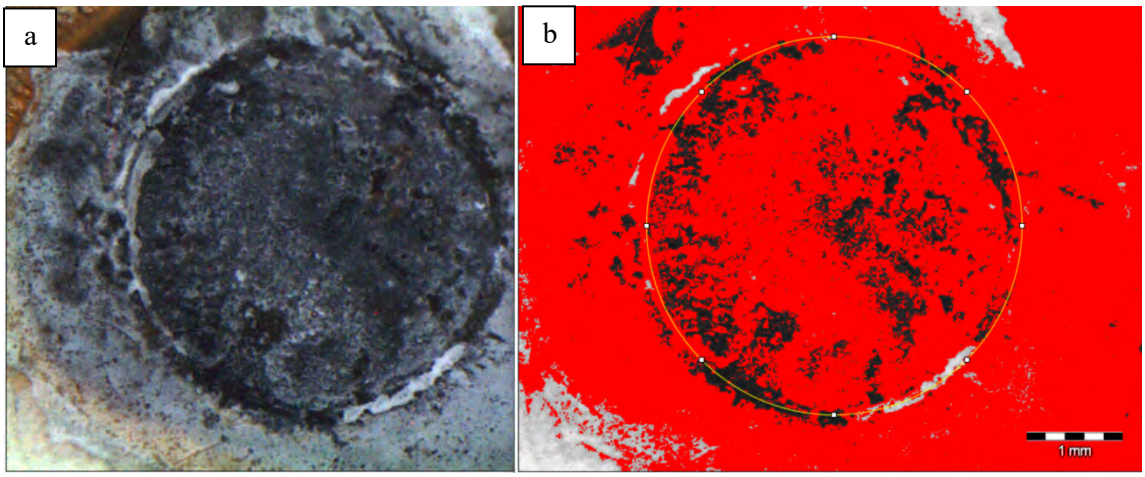


Figure 3.23: Evaluation of failure mode (a) Sample image (b) Example of calculation % using NIH ImageJ image analysis software (red colored areas) for adaptation failure on stereomicroscope image. The red colored area was showed remnant GIC failure areas in the stereomicroscopy image with failure was recorded and classified according to adhesive (less than 25%), cohesive in material (more than 75%) or mixed failure (between 25%-75%).

3.2.2.9 Data analysis

Data was analysed using SPSS version 26. The data was subjected to Pearson Chi-Square with Bonferroni Adjustment ($p<0.05$).

Chapter Four: Results

4.1 Part A

4.1.1 Shear-bond strength of different surface treatment groups

Table 4.1: Mean ± Standard Deviation of Shear-bond Strength (SBS) for the two glass ionomer cements and various surface conditioning treatment groups

Pre-treatment materials		T1	T2	T3	T4	T5	p-value	Post hoc
Riva HV self-cure (SC)	Mean	6.12 ± 3.01	6.40 ± 3.06	2.98 ± 2.50	0.00	3.44 ± 1.03	0.003	T2, T1 >T5, T3
	Median	6.50 (6.02)	6.57 (4.08)	2.15 (3.99)	0.00	3.57 (1.13)		
Riva HV light-cure (LC)	Mean	0.00	4.75 ± 3.73	7.09 ± 3.01	7.57 ± 3.23	9.23 ± 4.32	0.014	T5, T4, T3> T2
	Median	0.00	3.71 (4.60)	6.34 (4.36)	6.90 (3.20)	10.52 (5.78)		
p-value		-	0.129	0.001*	-	0.001*		

*Abbreviations: **T1**, control; **T2**, Riva Conditioner; **T3**, Super Etch; **T4**: Super Etch + Zipbond; **T5**: Super Etch + Riva Bond LC.*

Result of Kruskal Wallis test, with post hoc pairwise comparisons (α=.05).

*indicates statistically significant differences between groups *(P< 0.05)*.

The p-value of the normality test (Shapiro-Wilk) for SBS was less than 0.05 in all adhesive groups. Hence, the data was not normally distributed. Non-parametric univariate statistical analysis was performed using Median and Interquartile Range. Kruskal-Wallis

test was used to compare the median rank within the treatment groups. Overall, there was a significant difference in SBS between the groups ($p < 0.001$). Thus, post hoc test multiple comparison ($p<0.05$) was used. Table 4.1 shows the median and mean values of the SC and LC to simulated carious dentine after 1-week storage in artificial saliva.

Within the SC groups, the SC-T2 group showed the highest median (IQR) SBS followed by the SC-T1 and SC-T5 respectively. The lowest median (IQR) SBS was observed in the SC-T3. The difference between them was significant ($p<0.05$). The universal adhesive surface treatment group resulted in no specimens surviving.

In the LC groups, the differences between groups were significantly different ($p<0.05$), in which the LC-T5 group showed the highest median (IQR) SBS followed by the LC-T4 and LC-T3 group respectively. The lowest SBS was obtained in LC-T2 group. The "none" surface treatment group resulted in no specimens surviving after the 1-week storage.

The result of the Kruskal-Wallis post hoc test at 95% confidence level (Table 4.1) showed significant mean difference in SBS between SC and LC group in treatment material T3 ($p<0.001$). In addition, there was significant mean difference in SBS between SC and LC group with T5 ($p <0.0001$).

4.2 Part B

4.2.1 Mode of failure

According to Figure 4.1 and Table 4.2, data of failure modes for all adhesive groups were analysed using Pearson Chi-Square test with Bonferroni Adjustment. The

example of stereomicroscope images were displayed in Figure 4.2. SC generally exhibited adhesive failures, when compared to mixed failure groups. Only group SC-T5 presented with one cohesive failure. Conversely, LC exhibited mostly adhesive and mixed failure in the material for most surface treatments. Only LC-T2 presented with one cohesive failure. When comparing the failure modes between groups, the result of failure mode was not significant. SC groups presented with higher adhesive failure when compared with LC, as LC has more mixed failure.

There are no data for SC-T4 and LC-T1 as the specimens failed prematurely. The minimum expected count is less than 5, the results are inconclusive, which might be influenced by the small sample size.

Table 4.2: Distribution of failure modes for the various surface treatment, Number (%)

Treatment	**Riva HV self-cure (SC)**			**Riva HV light-cure (LC)**		
	Adhesive (%)	**Cohesive in materials (%)**	**Mixed (%)**	**Adhesive (%)**	**Cohesive in materials (%)**	**Mixed (%)**
T1	10 (71.4)	-	4 (28.6)	0(0.00)	0(0.00)	0(0.00)
T2	9 (64.3)	-	5 (35.7)	4 (28.6)	1 (7.1)	9 (64.3)
T3	13 (92.9)	-	1 (7.1)	7 (50.0)	-	7 (50.0)
T4	0(0.00)	0(0.00)	0(0.00)	7 (50.0)	-	7 (50.0)
T5	11 (78.6)	1 (7.1)	2 (14.3)	7 (50.0)	-	7 (50.0)

n=14; Abbreviations: ***T1****, control;* ***T2****, Riva Conditioner;* ***T3****, Super Etch;* ***T4****: Super Etch + Zipbond;* ***T5****: Super Etch + Riva Bond LC;* **Adhesive failure %,** more than 75% of the bonding area is exposed dentine; **Cohesive failure %** in GIC, more than 75% of the bonding area is covered with remnants of GIC; **Mixed failure %**. the combination of both adhesive and cohesive failures between 25% and 75% of the bonding area.

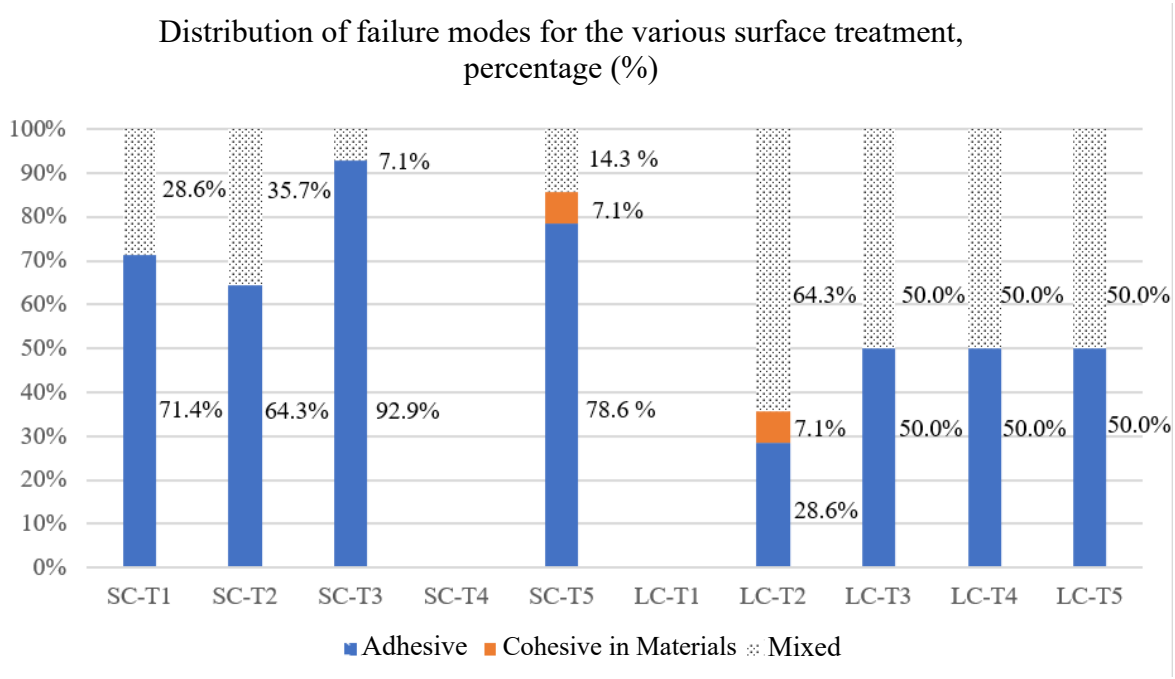


Figure 4.1: Bar graph of distribution of failure modes for the various surface treatment, percentage (%)

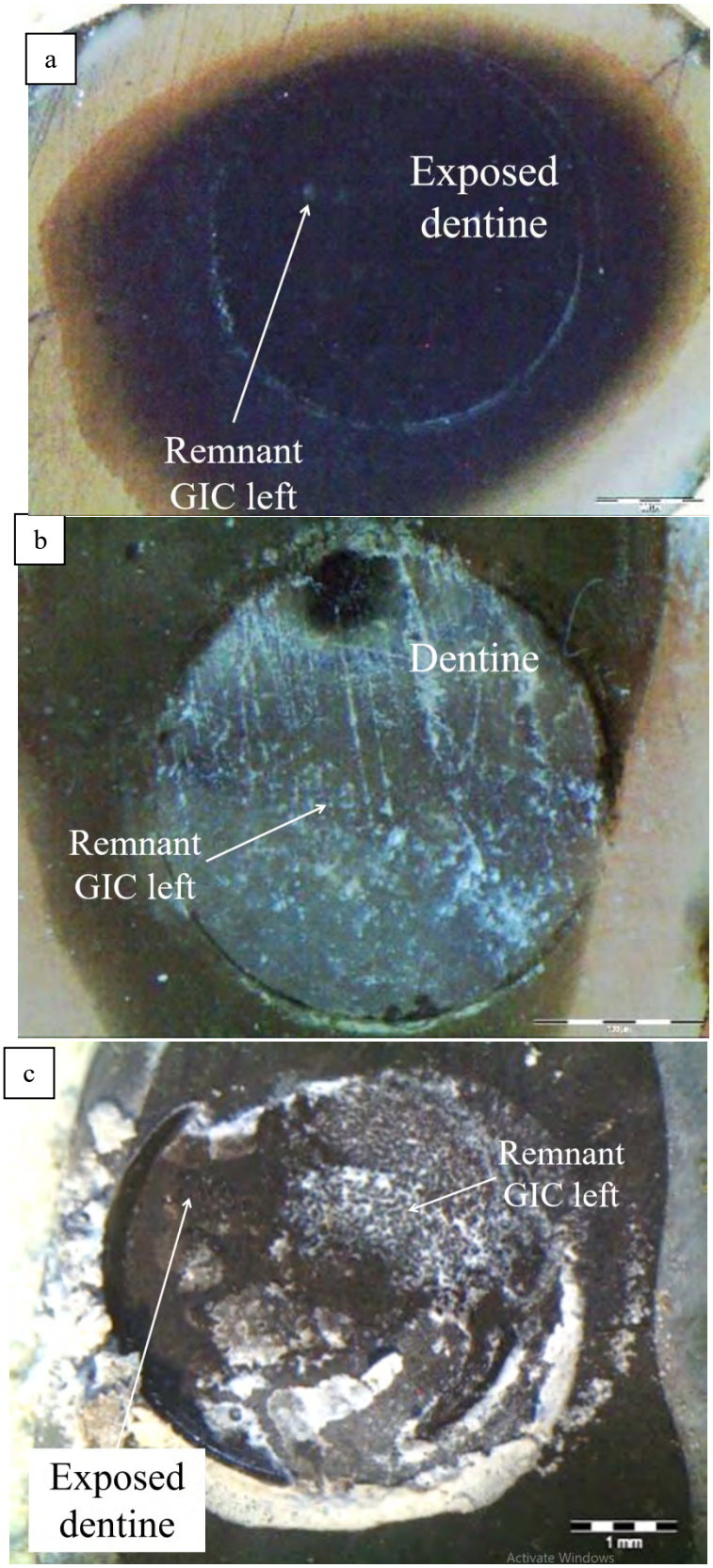


Figure 4.2: Stereomicroscope images of a) adhesive failure b) cohesive failure c) mixed failure. D = Dentine surface; GIC = Glass ionomer cement

4.2.2 SEM Qualitative Evaluation

In Figure 4.3a, the SEM image showed 2000x magnification SC specimen without any surface conditioning. The ion exchange layer seems to be well adapted between both dentine and GICs compared to the others HVGIC group. SEM analysis showed a gap in between SC, LC and dentine in Figure 4.3 (b-g). These surfaces were coated with formation of precipitates on tooth surface.

Figure 4.3e showed dense spherical nodular structure, suggestive of filler present in the layer between dentine and GIC. Figure 4.3f showed thin cylindrical configuration of resin tags could be observed after conditioning the dentine surface. Noted dense spherical nodular structure presented in the layer between dentine and GIC. All LC groups exhibited hybrid layer (Figure 4.3 (f-g), except the one treated with polyacrylic acid. Figure 4.3(f) showed short and discrete resin tag were seen with thin hybrid layer in phosphoric acid-treated LC groups. Figure 4.3(a) and (h) presented with reduce gap in the interfacial region.

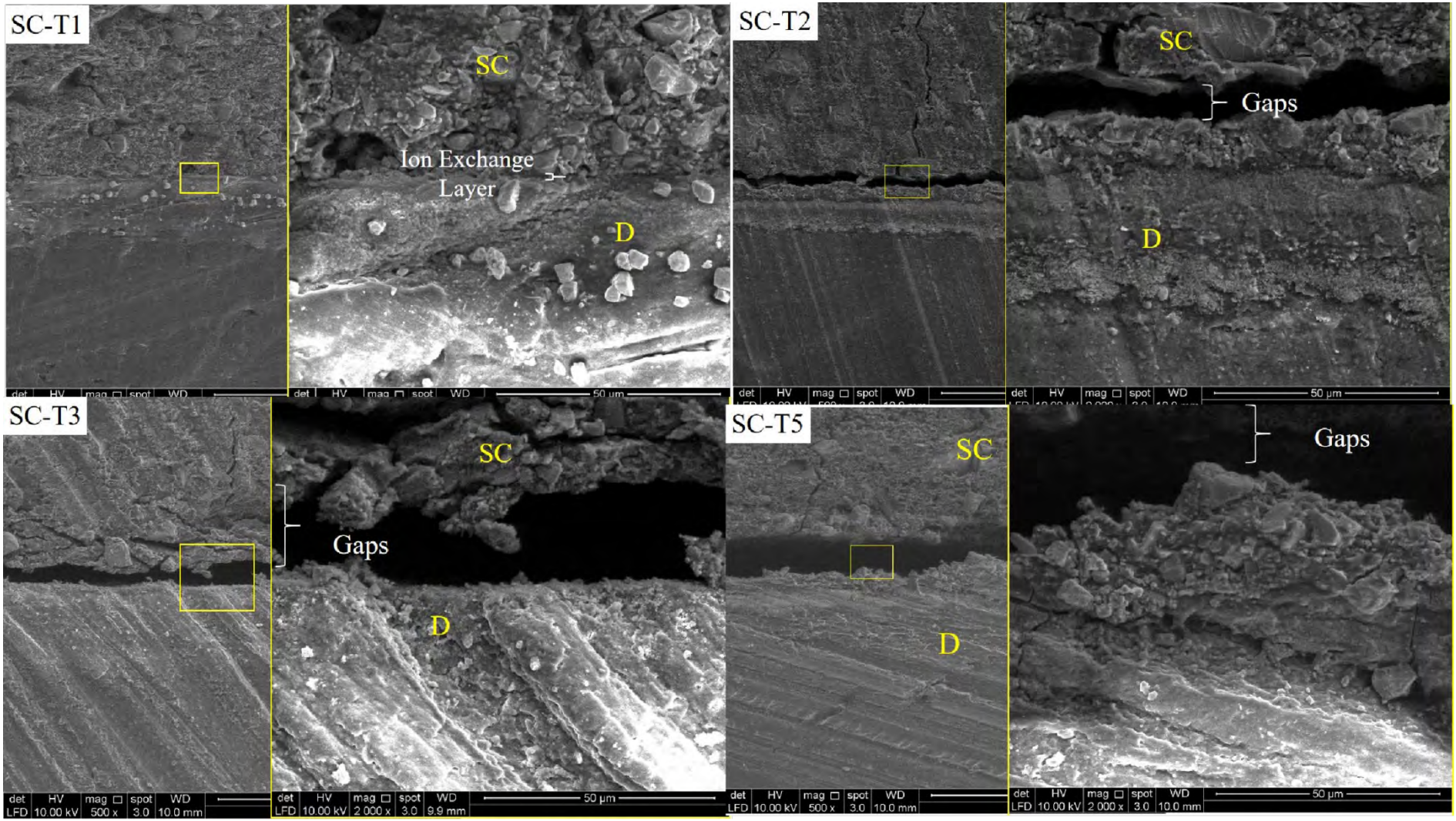


Figure 4.3 SEM photomicrographs of 2000x magnification of the dentine specimen interface after treatment with surface treatment.

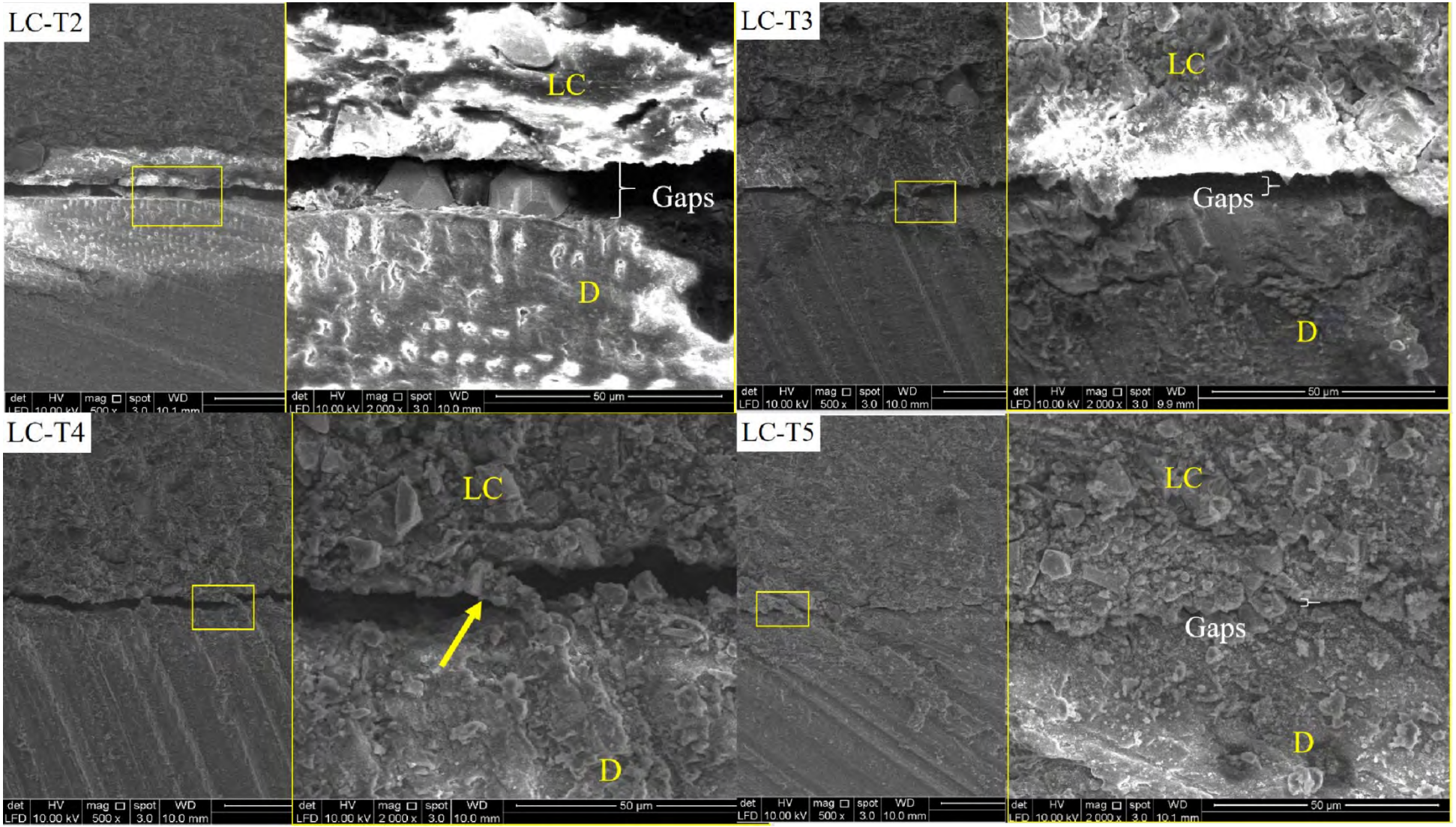


Figure 4.3, continued. SEM photomicrographs of 2000x magnification of the dentine specimen interface after treatment with surface treatment.

Figure 4.3, continued.

SEM photomicrographs of 2000x magnification of the dentine specimen interface after treatment with surface treatment.

(SCT1) SC with dentine treated without any surface treatment;

(SCT2) SC with dentine treated with PAA;

(SCT3) SC with dentine treated with PPA;

(SCT5) SC with dentine treated with PPA and Riva bond LC;

(LCT2) LC with dentine treated with PAA;

(LCT3) LC with dentine treated with PPA;

(LCT4) LC with dentine treated with PPA and Zipbond;

(LCT5) LC with dentine treated with PPA and Riva bond LC.

SC: high viscosity glass ionomer cement; LC: resin-modified glass ionomer cement;

D: dentine. *Yellow arrow shows presence of resin tags.

EDX Qualitative Evaluation

Table 4.3 Representative EDX analysis of dentine surface treated with SDF with SC.

	SC-T1		SC-T2		SC-T3		SC-T5	
Element	**Weight %**	**Atomic %**	**Weight %**	**Atomic %**	**Weight %**	**Atomic %**	**Weight %**	**Atomic %**
C	39.50	2.60	28.80	2.00	14.80	1.90	17.97	30.30
O	33.50	1.80	41.40	1.40	36.20	1.00	37.13	46.99
F	4.60	0.80	5.40	0.60	3.60	0.30	7.06	7.52
Al	3.30	0.40	2.20	0.20	7.50	0.30	1.99	1.49
Si	3.10	0.40	0.60	0.20	9.20	0.40	7.34	4.78
P	2.50	0.50	7.70	0.50	9.60	0.40	1.57	0.93
Ca	0.70	0.40	7.40	0.50	5.50	0.30	1.54	0.70
Sr	5.80	1.00	3.40	0.60	12.40	0.70	9.75	2.04
Ag	6.90	1.30	3.10	0.80	1.20	0.50	0.37	0.06

Table 4.4: Representative EDX analysis of dentine surface treated with SDF with LC.

	LC-T2		LC-T3		LC-T4		LC-T5	
Element	**Weight %**	**Atomic %**	**Weight%**	**Atomic %**	**Weight %**	**Atomic %**	**Weight %**	**Atomic %**
C	24.02	35.39	15.99	26.14	29.00	40.80	16.08	28.10
O	40.57	44.89	40.51	49.71	40.93	43.23	36.55	47.95
F	7.60	7.08	2.86	2.96	6.36	5.66	4.68	5.17
Al	0.60	0.39	0.69	0.50	2.81	1.76	1.29	1.00
Na	0.37	0.29	0.45	0.39	0.41	0.30	0.40	0.36
P	7.65	4.37	11.23	7.12	3.33	1.81	6.89	4.67
Ca	15.13	6.68	24.15	11.83	6.10	2.57	13.41	7.02
Ag	3.4	0.56	2.75	0.50	2.24	0.35	16.97	3.30
S	0.26	0.14	0.47	0.29	-	-	-	-
Si	-	-	-	-	4.84	2.91	0.94	0.70

Results from Table 4.3 and Table 4.4 shows qualitatively recorded presence of fluoride (F) and silver (Ag) in all the dentine surfaces. The SEM-EDX study was performed without any dehydrating and coating. The treated dentine surface was mainly composed of oxygen, carbon, fluoride, calcium, phosphorus and silver. Fluoride levels were present in all dentine surface. Silver ions were also found in the dentinal tubules. In our study the atomic percentage (at %) does not have a correlation with the SBS involved. However, in LC-T5, it was reported that the content of Ag is highly detected (3.30 at%) on the dentinal surface.

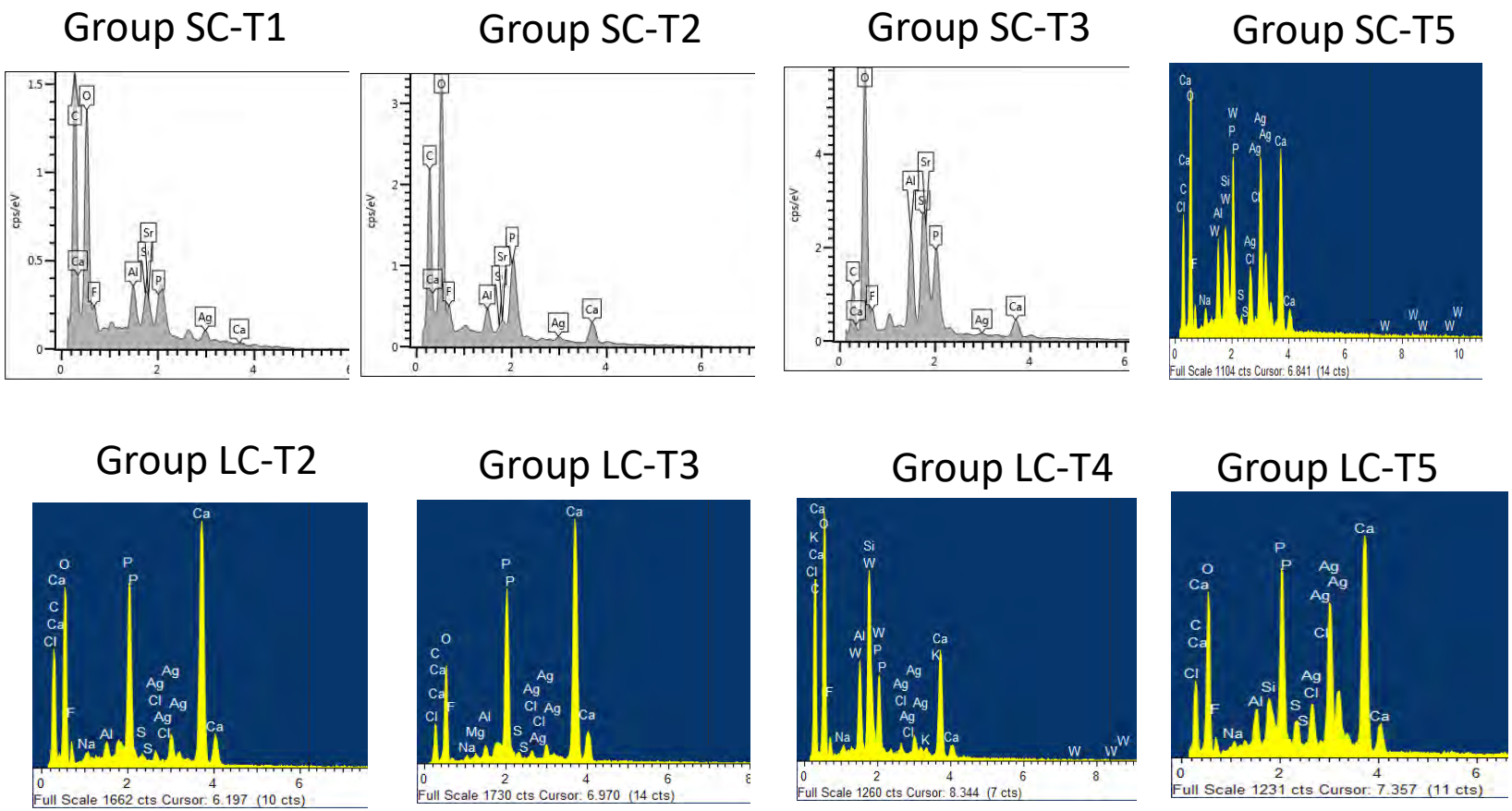


Figure 4.5: EDX Spectroscopy from all 8 different groups. Note that Ag was detected in all the treatment groups.

Chapter 5: Discussion

5.1 Methodology

The main objective of the present study was to investigate the effects of different surface treatments on the SBS of SC and LC on SDF-treated carious dentine. Furthermore, we wanted to analyse the associated failure mode using a stereomicroscope and digital imaging software. Although several methods of placement of GIC after the application of SDF have been researched, very few have evaluated whether surface treatment on demineralised dentine could have any effect on the SBS between the GIC and dentine. In our study, the first null hypothesis was rejected, because all surface treatments affected on the bond strength of SC and LC to SDF-treated simulated carious dentine (SCD), except SC-T4 and LC-T1 because they failed to bond on the tooth surface. The second and third null hypotheses are accepted, as the surface treatments on SDF-treated SCD did not affect the failure type. Furthermore, the presence or absence of the ion exchange layer did not relate to the bond strength changes for GICs adhering to SDF-treated SCD.

When evaluated, the decrease in mean SBS was not similar for all surface treatment materials. The bond strength of LC was comparable with SC, which is not similar to a study that showed LC to simulated carious dentine was reported to be higher than SC (Choi et al., 2006). This may be because, in LC, there was a lack of formation of tags at the dentine-cement interface, due to the lack of penetration of RMGIC polymer into the dentinal tubules as the SDF layer was shown present on the dentine surface. This was observed in the present study in the elemental analysis where Ag was present in all surfaces.

No surface treatment was chosen as the control group because GIC can adhere to the tooth surface with chemical bonding even without surface treatment (van Meerbeek

et al., 2003). In our study, there is little Ca in this region due to the demineralisation, and the bond strength should be comparable to the self-cure GIC. This may be due to the outer surface layer of the SDF- treated dentine has an elevated level of calcium and phosphorus and a corresponding increase in microhardness (Chu et al., 2008). However, in our study, SC could bond with SDF-treated dentine, while LC material failed to bond to the dentine surface in the group without surface treatment. It differs from a study by Tanumiharja et al. (2000) which LC can adhere to the dentine interface without dentine conditioner. In our study, this may have been difficult as the silver-incorporated precipitate layer was thick, HEMA and resin from LC could not diffuse into the calcium-rich hydroxyapatite layer. Therefore, without suitable surface conditioning, there is no bonding between the dentine and the LC, thus the LC specimens failed. Glass ionomer products that contain resin monomer may require dentine treatments that differ from traditional glass ionomer cements. This was anticipated before the experiment was carried out.

In our study, SBS in SC wasn't statistically significant when no conditioner or when PAA was placed. This result concurs with the result of Tanumiharja et al. (2000), which mentioned that there were no significant differences between conditioned and non-conditioned GIC. The reported findings confirm previous research that showed no advantage of surface pre-treatment with 10-25% polyacrylic acid in terms of GIC bond strength to the dentine (Bassi et al., 2020; Ng et al., 2020; Francois et al., 2020). However, other reports refute this observation by showing polyacrylic acid had several benefits which can enhance the bonding of GIC to caries-affected dentine (Saad et al., 2017; Ugurlu et al., 2002). Conditioning with a weak acid such as 26% polyacrylic acid is known as the gold standard (Hoshika et al., 2015). It is believed that in the current study, PAA had to diffuse downward through a bed of demineralised collagen fibrils to infiltrate the interfibrillar spaces with either chemical adhesion or micromechanical retention. It increases the wettability of the dentine surface and improves ion exchange with the

cement (Powis et al., 1982). However, it might be due to the thickness of the SDF layer, the resin component from LC could be difficult to penetrate into the demineralised interfibrillar spaces.

Application of phosphoric acid as pre-treatment yields the lowest bond strength in the SC group, while in the LC groups, it had significantly higher median SBS reported compared to the polyacrylic conditioner and control groups. Our former result was similar to studies that found a drop in SBS of SC to phosphoric acid-etched teeth, both in caries-affected and normal dentine (Bassi et al., 2020; Kokmaz et al., 2010). It could be due to during SC placement to phosphoric acid-conditioned SDF dentine, there is a diffusion gradient. The demineralised dentinal collagen in the phosphoric acid group relies on the infiltration of PAA from the setting SC cement, unlike the dentine pre-treatment with PAA where some of the PAA may be retained by chemical bonding to the partially demineralised collagen. The high viscocity self- curing and light-curing GIC is used in this study, the viscosity might be too thick for the GIC to wet the demineralised dentine surface, thus failed to infiltrate the dentine.

On phosphoric-acid-etched dentine, there is a non-continuous thin intermediate layer as noted in our SEM observation, that contains SDF and banded collagen, as the apatite crystallites are partially dissolved in the demineralised collagen network (Van Meerbeek, 1996). Due to the aggressiveness of pre-treatment protocols, this can cause excessive demineralisation and consequent lack of minerals for SC chemical bonding. The SC bonding mechanism is mainly produced by ionic exchange with calcium-free radicals. When the smear layer and smear plug is removed by phosphoric acids, the calcium radicals and the capability to form a bonding bridge between the cement and dentine walls are also reduced (Van Meerbeek et al., 2006; Powis et al., 1982). However, there might be a collapse of the demineralised collagen network if the acid-etched dentine

is desiccated before GIC placement. This aggressive pre-treatment protocol can leave a bed of denuded collagen within the subsurface of demineralised dentine (Gwinnett A. J., 1994). When acid etching, in contrast to conditioning, the smear layer and smear plug are removed, which may increase the dentine permeability and outward flux of dentinal fluids during bonding. The fluids might dilute the chemically cured GICs, producing weak bonds to dentine. However, the removal of the smear layer will clean and create a uniform surface for bonding and prevent any blocking of dentinal tubules. Therefore, our study uses the 5s-etching method as Scheffel et al. (2012) reported that phosphoric acid etching for 5s showed the lowest dentine calcium loss compared to 10s and 15s etching. Short etching is thought to limit the loss of calcium ions from the superficial dentine. In the later result, the use of phosphoric acid before LC had improved the bond strength to SDF-treated dentine, which corroborates with some recent studies (Valente et al., 2002; Coutinho et al., 2006; Nicoló et al., 2007; Pereira et al., 2002). Surface treatment with phosphoric acid can promote the formation of an acid-base resistant layer (ABR) by the interaction of adhesives to the hydroxyapatite crystals to form less soluble calcium salts that can resist the acid-base challenge (Waidyasekera et al., 2009). In our study, thin cylindrical resin tags were observed in all phosphoric acid-treated- LC groups, which could be the ABR layer.

In this study, the group tested with universal adhesive failed to bond SC on the SDF-treated dentine surface. Failure of SC to bond in SC-T4 was anticipated, mainly due to the thickness of the universal adhesive which acted as a barrier preventing the cement from chemically curing directly to the dentine. However, there's an increase in SBS of LC to SDF-treated SCDs using universal adhesive, which is similar to a few studies conducted by Besnault et al. (2004), Imbery et al. (2009), El-Askary and Nassif (2011), and Ugurlu (2020). However, the SDF-treated surfaces were found to have significantly lower tensile bond strengths to dentine for two dental adhesive resin cements, which

contained MDP or 4-META (Soeno et al., 2001). In our study, dentine bonding agents can form a chemical union with LCs due to the HEMA and other resins in LCs. As Zipbond contains 10-MDP as the acidic functional monomer, this might positively improve the bond strength of LC (El-Askary et al., 2011; Saad et al., 2019). In our study, the fluoride ions level after the application of universal adhesive was about the same level with other treatment groups. This contrasts with a study by Mazzaoui et al. (2000) found that dentine bonding agents significantly reduced the fluoride released by LC; however, they do not prevent the fluoride passing through. In our study, the consistent level of fluoride might be due to the fluoride released by SDF, not by the GICs.

Another very interesting point is, this study has demonstrated that RMGIC-based adhesive, had the highest bond strength of LC to dentine, but low bond strength of SC to dentine. This is in contrast with the study carried out by Talip et al. (2017) which shows that RMGIC-based adhesive increased bond strength for SC groups which was similar to our GIC used in the study. This finding confirms previous research that showed SDF-treated dentine was less affected when RMGIC adhesive was placed before resin composite (Koizumi et al., 2016). RMGIC adhesive, which adheres chemically and mechanically to dentine and adheres chemically to the SC (Cho and Cheng, 1999), did not work in our current study. It may be due to the light-cure set RMGIC adhesive being unable to reach its maturation stage to bond with HVGIC with additional calcium ion exchange. The elevated HEMA resin content (25-40%) within the LC material results in less predisposed to adhere to HVGIC unless the surface is appropriately roughened. During LC development, the acid-base reaction and light-polymerisation reactions compete with and inhibit one another. The essential component of the acid-base reaction requires water; however, in LC, the content of water is reduced, which may retard the acid-base reaction. In conventional GIC, it could take 24 hours to 1 week to reach maturation, whereas in light-cure GIC, the resinous portion might have the immediate

setting but not the GI portion (Berzins et al., 2010). According to manufacturers' data, the P/L ratio for Riva Self Cure HV is 3.85 (0.5/0.13), which is high. GIC with a low P/L ratio will usually produce lower bond strength because of decreased ion released to form an ion exchange layer (Shebl et al., 2015). However, our observation is when the P/L ratio is high, the GIC has more unreacted particles, which act as stress concentration points to reduce the SBS value recorded for SC, as compared to LC (Yap et al., 2001). In the meantime, LC shows increased bond strength with RMGIC adhesive due to their high wettability and their resin content, thus an increase in surface energy and shear-bond strength. This may be due to the reduction in the consistency of the LC; the powder/liquid ratio in Riva Bond LC is 0.7 (0.7:1) compared to Riva Light Cure GIC which is 3 (3:1). Pereira et al. (1997) noticed that RMGIC-based adhesive contains HEMA, which has excellent wetting ability on the tooth surface, as it can improve the infiltration of adhesive monomer into demineralised dentine, thus improve the bond strength.

The reported failure modes varied in this study. In SC groups, generally, adhesive failure was exhibited, while in LC groups, generally adhesive and mixed failures were exhibited. Failure is not purely random; instead, it occurs wherever a crack in the material propagates (Wang & Darvell, 2007). This type of failure shows that the adhesive strength of SC and LC to dentine was weaker than its cohesive strength. This demonstrates a higher bond strength between the dentine and GICs as compared to between the GICs and the surface treatment materials. In both groups, if adhesive failure and mixed failure were observed, it may have been due to the gap between GIC and teeth specimens noted in the SEM analysis. In our study, cohesive failure specimens occurs once in each specimens. It was believed that in terms of cohesive failure mode, it can represent a combination of the mechanical characteristics of the many materials involved rather than a strong interface bonding. Several factors can lead to cohesive failure, such as microcracks in the specimen created during cutting or trimming, faults in specimen alignment along the long

axis of the testing apparatus, and the brittleness of the material involved. Cohesive failure specimens should be discarded, and only data from adhesive failure or mixed failure specimens with a limited region (< 10%) involved should be used for the computation of bond strength. (Jiang et al., 2020)

In the failure mode, we used ImageJ software to analyze and calculate the amount of GIC remnants left on the dentine surface. A red photo contrast was used to contrast the remnant GIC seen in the dentine. According to Jensen (2013), it was important to use image analysis to compare the intensity of the marker in the image, compared to "eyeballing" an image to state a particular area. In order to calculate the selected area, the software can choose the selection area and shape to view the percentage of remnant GICs left on the confined mould area rather than the whole dentine surface. HVGIC is full of porosities and focal points for crack initiation and propagation. The adhesive used is likely to have fewer cracks, leading to an increase in bond strength. This could possibly result in a change in the common mode of failure.

Air-dried and uncoated samples were examined by SEM using low vacuum imaging to allow better analysis of the interface, with EDX analysis. Both GICs contain water, therefore they are difficult to be observed at the bonding interface due to dehydration and metallisation. The vacuum required to perform the SEM and EDX analysis would cause cracks in the glass ionomer. In ionomeric materials, the presence of cracks affects the mineral measurements, therefore only continuous regions were assessed and the cracked areas were excluded.

When the SEM/EDX was carried out at each time point, an image was taken of the surface of the specimen at two magnifications of 500× and 2000× (Ahmad et al., 2009). This enabled detailed views of the focused area. SEM analysis showed a gap between SC,

LC, and dentine in SC-T2 to SC-T5 and LCT2- LCT4, this was coated with the formation of precipitates on the tooth surface. The presence of gaps in this layer might indicate high filler content of GIC and lack of ion exchange layer formation. The lack of ion exchange could mean the inability of the polyacrylic acid and phosphoric acid to decalcify the underlying dentine. The noted gap could be attributed to the contraction of dentine and GIC which causes separation of the materials at its weaker point (El-Askary et al., 2011). In this study, a slight improvement in the SBS of SC with the polyacrylic acid step could be explained by the SEM result. This is due to the application of polyacrylic acid can preserve the silver precipitate layer. The continuous layer of silver precipitate was found in HVGIC treatment by none surface pre-treatment and polyacrylic acid. All LC groups exhibited a hybrid layer, except the one treated with polyacrylic acid. Short and discrete resin tags were seen with thin hybrid layers in phosphoric acid-treated LC groups. SEM in the phosphoric acid treated group in SC and LC decalcified the underlying dentine, aiding the GIC to form an ion exchange layer. In the LC group, phosphoric acid with universal adhesive and RMGIC adhesive resulted in the formation of numerous long, funnel-shaped resin tag extensions. In the SEM view for the LC group of RMGIC-adhesive-treated-dentine, the bud-like configuration of tags was observed (Figure 4.3h), which might be due to the high glass-filler content. The formation of a hybrid layer is important to resist abrupt debonding stress (De Munck et al., 2005). SCT1 and LC-T5 presented with reduced gaps in the interfacial region. The reduced width in gaps in SCT1 and LC-T5 revealed a thin continuous adhesive interface, this could indicate high wettability of the substrate to dentine, and it could be due to the type of conditioning which allowed better monomer penetration into the dentine.

Chemical analysis for the assessment of the relative mineral content of SDF-treated dentine after GIC was placed was carried out by SEM/EDX spectroscopy. EDX analysis of superficial dentine presented with elements including oxygen, calcium, phosphorus,

silver, aluminum, fluoride, silicon, and strontium. In groups SC-T2 to SC-T5 and LC-T2 to LC-T5, EDX confirmed the precipitation of silver precipitates and fluoride ions in the superficial dentinal tubules. This shows inorganic exchanges between the SDF and GIC to superficial dentine. The silver chemicals can effectively increase the interfacial hardness and roughness at the GIC-dentine interface. Silver and silver oxide created might be important to improve the SBS between glass ionomer to stainless steel metal orthodontic bracket (Fricker, 1998). The application of surface-conditioning did not induce any increase in the concentration of the F and Ag, except in the group with RMGIC adhesive with LC, there's a peak of Ag precipitates observed. This is related to a significantly high SBS reading reported in LC with the surface pre-treatment with RMGIC adhesive. Conversely, SEM/EDX showed that when pure phosphoric acid was placed, the Ag level drops. This may be due to etching removed some silver precipitates on the dentinal surface. Fluoride (F) is an important ion involved in caries prevention. In both groups, the fluoride level did not show significant differences. Strontium-apatite was found in SC as compared to LC. The formation of more insoluble apatites, such as strontium-apatite and fluorapatite will protect against the formation of caries (Paiva et al., 2014). In a study by Ngo et al. (2006), the ion exchange happens when fluoride and strontium ions are available in GIC. There will be both fluoride and strontium ions to undergo apatitic activity for areas in dentine where calcium ion levels are low with strontium ions replacing the missing calcium. The presence of calcium (Ca) in SC is lower as compared to the LC group. This could be due to the apparent exchange of Ca from the dentine with Sr at the restoration interface, which was higher in the SC group. The presence of aluminium (Al) in the dentine surface of GIC groups may be due to the AlF^{2-} and AlF_2^- ions leached from glass ionomers. The aluminium chloride could stabilise the collagen matrix during future demineralisation.

In this experiment, extracted sound human premolars were used; the exclusion criteria for selection were teeth with cracks and caries. There are several studies that use extracted premolars as specimens; as written in one study by Pushpalatha et al. (2014), this tooth is usually removed for orthodontic reasons. Premolars were easier to obtain when compared to third molars as not all patients require third molar removal, and the tooth tends to fracture during minor oral surgery. Age is an important factor as the deposition of caicified tissue which cause the secondary dentine to become highly mineralised and brittle. Dentine sclerosis increases mineralisation of peritubular dentine, resulting in the complete obliteration of the tubules (Schüpbach et al., 1989). In a study by Lee et al. (2007), it was reported that there was no significant effect on bond strength in teeth disinfected with 0.5% chloramine-T. The storage time in the study was about 60 days. However, the samples used in that study were bovine incisor teeth. The teeth were stored in distilled water at 4°C until further use for no longer than 6 months, as is recommended by ISO/TS 11405:2003. The teeth were thoroughly rinsed under running water to remove the chloramine solution before surface preparation. In our study, the dentine surfaces were ground only up to superficial dentine to reduce variations. Superficial dentine has few tubules and is composed mainly of intertubular dentine. Deeper dentine nearer to the pulp is composed mainly of larger, funnel-shaped dentinal tubules. Type 1 collagen forms the major protein of intertubular dentine (Carberoglio et al., 1976). Smear layers are created when the teeth are cut by hand or with rotary instruments. It is a zone of tooth preparation debris that has been spread on the surface following tooth preparation. Smear layers can form smear plugs that can decrease dentine permeability. The dentine surface of each tooth was polished for 60 seconds with 400-grit, 600-grit, and 1200-grit silicon carbide papers (Metkon® Instrument Ltd, Turkey) which were placed on the water-cooled polishing machine (Isomet, Buehler; Lake Bluff, IL, USA). McLeod et al. (2010) and Yazici et al.(2007) used standardised grit silicon

carbide paper to polish the surfaces of samples to produce a uniform surface and smear layer.

In our study, demineralised dentine was formed by pH-cycling for 10 days. Each sample was cycled in the demineralising solution for 8 hours and then remineralising solution for 16 hours (Lenzi et al., 2014; Kucukyilmaz et al., 2016). The pH-cycling used in the study will replicate the dentine that can be remineralised in the carious process, which resembles caries-affected dentine. This is because of the other methods such as the microbiological method similar to caries lesions with an infected layer (Marquezan et al., 2009).

Artificial carious dentine has more clinical significance and has a significantly higher bond strength value when SDF was applied before GIC was placed compared to normal dentine (Wang et al., 2016). OCT as shown in Figure 5.1 was carried out to inspect the demineralisation lesion (Schneider et al., 2017). It is a non-invasive optical imaging technique that produces real-time, 2D cross-sectional and 3D volumetric images to measure the severity of subsurface demineralisation in enamel and dentine, track lesion progression over time, and measure remineralisation. A 700±14μm deep, partially demineralised baseline lesion was formed, to imitate caries-affected dentine as compared to a previous study in which 220±20μm depth is formed by 7 days of demineralisation (Liu et al., 2011).

There were two methods of placing GIC after SDF were placed on the dentine: (1) SDF-review-GIC placement, and (2) SDF-immediate-GIC placement. In our study, a waiting period was suggested before GICs were placed on the SDF-treated dentine. They were not placed immediately since low SBS (62% lower) was reported when GIC was placed immediately after SDF was placed. This is due to the low reaction and penetration

kinetics of SDF when immediate placement of GIC was done (Ng et al., 2020). In our study, placement of GIC was done after 14 days of SDF being applied. This is similar to a review of two to four weeks after the placement of SDF allows caries lesion arrest and allows for reapplication to ensure that all lesions are arrested (Crystal et al., 2017). Silver ion penetration can extend deeper when kept for 2 weeks, infiltrating the demineralised dentine with further penetration into the underlying dentine (Sayed et al, 2019). The reason for storing in deionised distilled water (DI) is to maintain the SDF-treated dentinal surface humidity so it exhibits a thin, smooth, regular coating with some occlusion of dentine tubules (Peng et al., 2021). With regards to the choice of storage medium after GIC bonding and before the SBS test, the use of artificial saliva solution to store GIC shows better bond strength as compared to storage in deionised water (Bali et al., 2015). Furthermore, artificial saliva simulates the oral cavity with its complex chemistry. GIC may release more fluoride in deionised water than artificial saliva (el Mallakh & Sarkar, 1990).

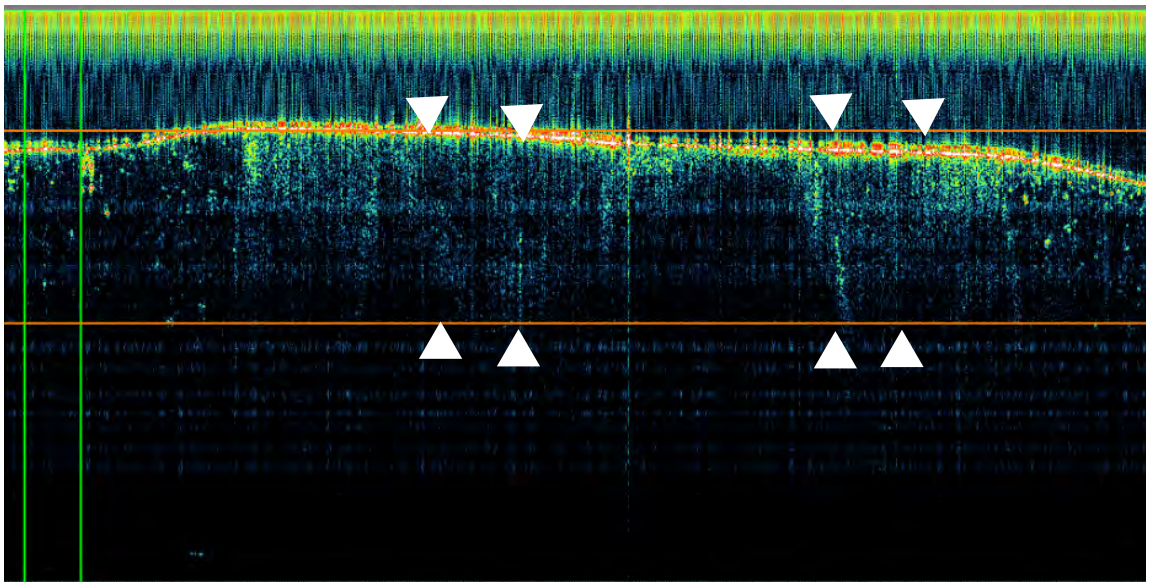


Figure 5.1: Imaging captured from OCT shows the depth of demineralised baseline lesion in the deep artificial caries (Technique adopted from Schneider et al., 2017)

Limited bonding area could be one of the important factors affecting SBS. In this study, SDI jigs were used to limit the bonding area. The benefit of using this jig is adherend could be adhered to dentine until shear-bond strength testing without the removal of the jig, as most of the experiment has the jig removed before the shear-bond strength testing. Bond strength results of specimens with limited bonding area and without bonding area were compared (Chai et al., 2015; Shimaoka et al., 2011). The comparisons performed showed that, for a non-enclosed area as compared to a confined bonding area, there was higher bond strength. The delimiting technique may help the restorative material be inserted into a defined surface. Operator skills may affect the bond strength, but in this study, only one operator prepared the SBS test (Söderholm et al., 2005). In our study, the preparation of specific jigs, such as the SDI jig (SDI, Bayswater, Australia), which was used for proper adaptation and stabilisation of the restorative material to the bonded substrated and to standardize the test protocol (Alzraikat et al., 2010).

There are large coefficients of variation, approximately 50% or more in some cases. This variability may be attributed to factors such as bonding, but it could also result from methodological errors, including the positioning of the shear blade, desiccation of specimens, or errors in GIC placement due to its viscous nature. There are large co-efficients of variation which are about 50% or more in some cases. This may be due to the bonding, but additionally it may be due to a methodological error such as positioning of the shear blade, desiccation of specimens, or errors in GIC placement as the HVGIC is viscous. In essence, it is crucial to guarantee that both the method of applying force and the way in which a material breaks align with real-world usage. This involves considering factors such as the preparation methods, the surrounding environment, and elements like the rate of strain. Ensuring that the outcomes are not only interpretable but also practical

and suitable for the intended purpose is imperative. (Darvell, 2020)

Chisel assembly and crosshead speed could affect the SBS. In this study, the conventional SBS test was employed using a notched-edge tool. The stress concentration at the bonded interface is much more severe in shear compared to tensile in a composite when a knife-edge chisel was used (Braga et al., 2010). However, both knife-edge and notched-edge chisel assemblies had shown no significant difference in bond strength, but it shows a significant difference between the mould enclosure and non-mould enclosure (Barkmeier et al., 2022). Within the limits suggested by ISO/TS 11405, the crosshead speed does not influence the bond strength value. In addition to that, ISO/TS 11405: 2003 recommends the rate of loading for a bonded specimen should be 0.75 ± 0.30mm/min. The evaluated test variables should be standardised throughout the treatment. GIC adheres to tooth structure; however, the mechanism of whether it adheres to the tooth surface after being treated by SDF is not known. Shear tests have been widely used and were selected in this study mainly as it is relatively simple to test when compared to tensile bond strength, especially during the alignment of the specimens in the universal testing machine. The specimen preparation in the shear-bond test would be easier when compared to the tensile bond strength test (McDonough et al., 2002).

Therefore, it is recommended that surface conditioning using polyacrylic acid did not have a significant difference with HVGIC, which suggests the use of a conditioner is optional. PPA five-second conditioning with RMGIC adhesive may enhance the infiltration of the resin component of the LC into the partially demineralised SDF-treated superficial carious dentine. Future studies need to be carried out to evaluate the interaction of the materials.

5.2 Limitations of the study

5.3.1 Part A

a. Due to large variations in bond strength, there might be possible that the macro shear-bond strength tests underestimate the bond strength values, which is why the most recent studies have preferred micro tests.

b. Technique sensitivity during restorative procedures, despite the manufacturer's recommendations were adhered to.

5.3.2 Part B

a. Technique sensitivity, since some samples were rejected because of a significant gap between the GIC and the teeth especially in SEM/EDX view. The quality of SEM performed in the study is not for diagnostic value.

5.3 Conclusion

Within the limitations of the study, the following conclusions were made: The first null hypothesis for the study was rejected. The second and third null hypotheses were accepted. Surface conditioning changed the bond strength of SC and LC to SDF-treated simulated carious dentine (SSCD). Surface treatments on SDF-treated SCD did not effect on the failure type. The presence or absence of the ion exchange layer did not relate to the bond strength changes for GICs adhering to SSCD. Surface treatment on SDF-treated SCD affected the bond strength of SC and LC. Except for PPA with RMGIC adhesive, SC generally exhibited adhesive or mixed failures. Conversely, LC exhibited mostly cohesive failure in the material for most surface treatments.

The preferred method for surface treating SDF-treated carious dentine before restoration application is PAA for SC and PPA plus RMGIC adhesive for LC. As treatment with etching and universal adhesive (T4) and no surface treatment (T1) resulted in unsatisfactory bonding for SC and LC correspondingly, they should be avoided clinically.

Chapter Six: Recommendations for Further Study

a) Further studies on long-term adhesion tests are needed to further test the surface treatment method that is best for simulated carious dentine.

b) The chemical and mechanical properties of ART materials with SDF- treated SCDs need to be studied further.

c) The chemical interaction between surface treatment groups with SC and LC, needs to be studied further.

d) Additional groups, involving sound dentine polished with 600-grit paper and sound dentine treated with SDF, could be added to understand the effect of demineralised dentine or the SDF effect on bonding.

e) During the SEM test, the removal of intrinsic water by using ethanol may be performed to reduce the gaps in between the interface between dental materials and dentine.

REFERENCES

A Saad, G Inoue, T Nikaido, M Ikeda, MF Burrow, J Tagami (2017). Microtensile Bond Strength of Resin-Modified Glass Ionomer Cement to Sound and Artificial Caries–Affected Root Dentin With Different Conditioning. *Oper Dent*; 42 (6): 626–635.

Ahmad, F., Dent, Mc., & Yunus, N. (2009). Shear Bond Strength of Two Chemically Different Denture Base Polymers to Reline Materials. *Journal of Prosthodontics,* 18(7), 596–602.

Almuammar MF, Schulman A, Salama F (2001). Shear bond strength of six restorative materials. *J Clin Pediatr Dent.* ;25(3):221–225.

Angelina Shuhua Wang, Michael G. Botelho, James K.H. Tsoi, Jukka P. Matinlinna, (2016). Effects of silver diammine fluoride on microtensile bond strength of GIC to dentine, *International Journal of Adhesion and Adhesives*, Volume 70, 2016, Pages 196-203, ISSN 0143-7496.

Armstrong, S., Geraldeli, S., Maia, R., Raposo, L. H., Soares, C. J., & Yamagawa, J. (2010). Adhesion to tooth structure: a critical review of "micro" bond strength test methods. *Dental materials*: official publication of the Academy of Dental Materials, 26(2), e50–e62.

Arnold, S, Warren, N, Buchanan, G D, & Lombard, R. (2022). Comparison of capsule-mixed versus hand-mixed glass ionomer cements. Part 1: compressive strength and surface hardness. *South African Dental Journal* , *77*(2), 57-64.

Bali, P., Prabhakar, A. R., & Basappa, N. (2015). An Invitro Comparative Evaluation of Compressive Strength and Antibacterial Activity of Conventional GIC and Hydroxyapatite Reinforced GIC in Different Storage Media. *Journal of clinical and diagnostic research : JCDR,* 9(7), ZC51–ZC55.

Barkmeier, W. W., Tsujimoto, A., Latta, M. A., Takamizawa, T., Radniecki, S. M., & Garcia-Godoy, F. (2022). Effect of mold enclosure and chisel design on fatigue bond strength of dental adhesive systems. *European journal of oral sciences*, *130*(3), e12864.

Bassi, Julio & Tedesco, Tamara & Raggio, Daniela & Santos, Ana & Bianchi, Renata & Sant'Anna, Giselle. (2020). Is it necessary to pre-treat Dentine before GIC Restorations? Evidence from an in Vitro Study. A*cta odontologica latinoamericana : AOL.* 33. 27-32. 10.54589/aol.33/1/027.

Besnault, C., Attal, J.P., Degrange, M. and Ruse, D. (2004). Self-etching adhesives improve the shear bond strength of a resin-modified glass-ionomer cement to dentin, *Journal of Adhesive Dentistry*, Vol. 6 No. 1, pp. 55–59

Bonifácio, C. C., Hesse, D., Bönecker, M., Van Loveren, C., Van Amerongen, W. E., & Raggio, D. P. (2013). A preliminary clinical trial using flowable glass-ionomer cement as a liner in proximal-ART restorations: the operator effect. *Medicina oral, patologia oral y cirugia bucal,* 18(3), e529–e532.

Braga MM, Mendes FM, De Benedetto MS, Imparato JC. (2009). Effect of silver diammine fluoride on incipient caries lesions in erupting permanent first molars: a pilot study. *J Dent Child (Chic).* 76(1):28-33.

Braga, R. R., Meira, J. B., Boaro, L. C., & Xavier, T. A. (2010). Adhesion to tooth structure: a critical review of "macro" test methods. *Dental Materials*, 26(2), e38-e49.

Brännström, M., Glantz, P. O., & Nordenvall, K. J. (1979). The effect of some cleaning solutions on the morphology of dentin prepared in different ways: an in-vivo study. *ASDC journal of dentistry for children,* 46(4), 291–295.

Braz, P. V. F., Dos Santos, A. F., Pereira, P. N., & Ribeiro, A. P. D. (2020). Silver diamine fluoride and cleaning methods effects on dentin bond strength. *American journal of dentistry*, *33*(6), 315–319.

Burgess, J., & Vaghela, P. (2018). Silver Diamine Fluoride: A Successful Anticarious Solution with Limits. *Advances in Dental Research*, 29(1), 131-134. doi:10.1177/0022034517740123

Burrow, M.F., Satoh, M. & Tagami, J. (1996) Dentin bond durability after three years using a dentin bonding agent with and without priming. *Dental Materials*, 12, 302.

Buzalaf, M. A., Hannas, A. R., Magalhães, A. C., Rios, D., Honório, H. M., & Delbem, A. C. (2010). pH-cycling models for in vitro evaluation of the efficacy of fluoridated dentifrices for caries control: strengths and limitations. *Journal of applied oral science* : revista FOB, 18(4), 316–334.

C. Francci, T. G. Deaton, R. R. Arnold, E. J. Swift, Jr, J. Perdigão, J. W. Bawden (1999). Fluoride release from restorative materials and its effects on dentin demineralisation. *J Dent Res*. 1999 Oct; 78(10): 1647–1654

Chai, Y., Lin, H., Zheng, G., Zhang, X., Niu, G., & Du, Q. (2015). Evaluation of the micro-shear bond strength of four adhesive systems to dentin with and without adhesive area limitation. *Bio-Medical Materials and Engineering, 26*(s1), 63-72.

Cheetham, J. J., Palamara, J. E., Tyas, M. J., & Burrow, M. F. (2013). A comparison of the micro-shear bond strength and failure mode of non-enclosed and mold-enclosed luting cements bonded to metal. *Dental materials journal*, *32*(6), 896–905. https://doi.org/10.4012/dmj.2013-065

Cho, S. Y., & Cheng, A. C. (1999). A review of glass ionomer restorations in the primary dentition. *Journal (Canadian Dental Association)*, *65*(9), 491–495.

Choi K, Oshida Y, Platt J.A., Cochran M.A., Matis B.A., Yi K.; Microtensile Bond Strength of Glass Ionomer Cements to Artificially Created Carious Dentin. *Oper Dent* 1 September 2006; 31 (5): 590–597.

Chu, C. H., Lo, E. C., & Lin, H. C. (2002). Effectiveness of silver diamine fluoride and sodium fluoride varnish in arresting dentin caries in Chinese pre-school children. *Journal of dental research*, *81*(11), 767–770.

Chu, C. H., & Lo, E. C. (2008). Microhardness of dentine in primary teeth after topical fluoride applications. *Journal of dentistry*, *36*(6), 387–391.

Chu, C. H., & Lo, E. C. (2008). Promoting caries arrest in children with silver diamine fluoride: a review. *Oral Health Prev Dent*., 6(4):315-321.

Chu, C. H., Mei, L. E. I., Seneviratne, C. J., & Lo, E. C. M. (2012). Effects of silver diamine fluoride on dentine carious lesions induced by Streptococcus mutans and

Actinomyces naeslundii biofilms. *International journal of paediatric dentistry*, *22*(1), 2-10.

Chu, Chun-Hung & Lo, Edward. (2008). Promoting caries arrest in children with silver diamine fluoride. *Oral health & preventive dentistry*. 6. 315-21.

Contreras, V., Toro, M. J., Elías-Boneta, A. R., & Encarnación-Burgos, A. (2017). Effectiveness of silver diamine fluoride in caries prevention and arrest: a systematic literature review. *General Dentistry*, 65(3), 22–29.

Coutinho, E., Van Landuyt, K., De Munck, J., Poitevin, A., Yoshida, Y., Inoue, S., Peumans, M., Suzuki, K., Lambrechts, P., & Van Meerbeek, B. (2006). Development of a self-etch adhesive for resin-modified glass ionomers. *Journal of dental research*, *85*(4), 349–353.

Crystal, Y. O., Marghalani, A. A., Ureles, S. D., Wright, J. T., Sulyanto, R., Divaris, K., Fontana, M., & Graham, L. (2017). Use of Silver Diamine Fluoride for Dental Caries Management in Children and Adolescents, Including Those with Special Health Care Needs. *Pediatric dentistry*, *39*(5), 135–145.

D. W. Berzins, S. Abey, M. C. Costache, C. A. Wilkie, and H. W. Roberts. (2010). Resin-modified glass-ionomer setting reaction competition, *Journal of Dental Research*, vol. 89, no. 1, pp. 82–86, 2010.

Darvell B. W. (2020). Misuse of ISO standards in dental materials research. *Dental materials : official publication of the Academy of Dental Materials*, *36*(12), 1493–1494. https://doi.org/10.1016/j.dental.2020.10.014

De Munck, J., Van Landuyt, K., Peumans, M., Poitevin, A., Lambrechts, P., Braem, M., & Van Meerbeek, B. (2005). A critical review of the durability of adhesion to tooth tissue: methods and results. *Journal of dental research*, *84*(2), 118–132. https://doi.org/10.1177/154405910508400204

Deepa, V. L., Dhamaraju, B., Bollu, I. P., & Balaji, T. S. (2016). Shear bond strength evaluation of resin composite bonded to three different liners: TheraCal LC, Biodentine, and resin-modified glass ionomer cement using universal adhesive: An in vitro study. *Journal of conservative dentistry: JCD*, *19*(2), 166–170.

Delbem AC, Bergamaschi M, Sassaki KT, Cunha RF. (2006). Effect of fluoridated varnish and silver diammine fluoride solution on enamel demineralisation: pH-cycling study. *Journal of Applied Oral Science*, 14:88–92.

Di Nicoló, R., Shintome, L. K., Myaki, S. I., & Nagayassu, M. P. (2007). Bond strength of resin modified glass ionomer cement to primary dentin after cutting with different bur types and dentin conditioning. *Journal of Applied Oral Science*, 15(J. Appl. Oral Sci., 2007 15(5)), 459–464.

Dias, G. F., Chibinski, A. C. R., Santos, F. A. Dos., Hass, V., Alves, F. B. T., & Wambier, D. S.. (2016). The hardness and chemical changes in demineralized primary dentin treated by fluoride and glass ionomer cement. *Revista De Odontologia Da UNESP*, 45(1), 33–40.

Dos Santos VE Jr, de Vasconcelos FM, Ribeiro AG, Rosenblatt A. (2012). Paradigm shift in the effective treatment of caries in schoolchildren at risk. *Int Dent J*., 62(1):47-51.

Duangthip D, Chu CH, Lo EC. (2016). A randomized clinical trial on arresting dentine caries in pre- school children by topical fluorides—18 month results. *J Dent.*44:57-63.

Eick, J. D., Wilko, R. A., Anderson, C. H., & Sorensen, S. E. (1970). Scanning electron microscopy of cut tooth surfaces and identification of debris by use of the electron microprobe. *Journal of dental research*, 49(6).

el Mallakh BF, Sarkar NK. Fluoride release from glass-ionomer cements in de-ionized water and artificial saliva. *Dent Mater*. 1990 Apr;6(2):118-22.

El-Askary, F. M. S., Nassif, M. S. A., Andrade, A. M., Reis, A., & Loguercio, A. D. (2012). Effect of ssurface area and air-drying distance on microstrength of etch-and-rinse adhesive. *Brazilian oral research*, *26*, 418-423.

El-Askary, F. S., Nassif, M. S., & Fawzy, A. S. (2008). Shear bond strength of glass-ionomer adhesive to dentin: effect of smear layer thickness and different dentin conditioners. *The journal of adhesive dentistry*, *10*(6), 471–479.

El-Askary, F., & Nassif, M. (2011). Bonding nano-filled resin-modified glass ionomer to dentin using different self-etch adhesives. *Operative dentistry*, *36*(4), 413–421.

Erickson RL, Glasspoole EA. (1994) Bonding to tooth structure : A comparison of glass-ionomer and composite resin systems. J Esthet Dent , 6:227–244.

Farah, C.S., Orton, V.G. and Collard, S.M. (1998), Shear bond strength of chemical and light-cured glass ionomer cements bonded to resin composites. Australian Dental Journal, 43: 81-86.

Featherstone, J. D. B., ten Cate, J. M., Shariati, M., & Arends, J. (1983). Comparison of Artificial Caries-Like Lesions by Quantitative Microradiography and Microhardness Profiles. *Caries Research,* 17(5), 385–391.

François, P., Greenwall-Cohen, J., Le Goff, S., Ruscassier, N., Attal, J. P., & Dursun, E. (2020). Shear bond strength and interfacial analysis of high-viscosity glass ionomer cement bonded to dentin with protocols including silver diammine fluoride. *Journal of oral science*, *62*(4), 444–448.

Fricker JP (1998). A new self-curing resin-modified glass-ionomer cement for the direct bonding of orthodontic brackets in vivo. American journal of orthodontics and dentofacial orthopedics: official publication of the American Association of Orthodontists, its constituent societies, and the American Board of Orthodontics. 113; 1998. p. 384–386.

G. J. Mount, C. Patel, and O. F. Makinson, “Resin modified glass-ionomers: strength, cure depth and translucency,” *Australian Dental Journal*, vol. 47, no. 4, pp. 339–343, 2002.

Garberoglio, R., & Brännström, M. (1976). Scanning electron microscopic investigation of human dentinal tubules. *Archives of oral biology*, *21*(6), 355–362.

Grossman, E. S., & Mickenautsch, S. (2002). Microscope observations of ART excavated cavities and restorations. *SADJ : journal of the South African Dental Association = tydskrif van die Suid-Afrikaanse Tandheelkundige Vereniging*, *57*(9), 359–363

Gupta, J., Thomas, M. S., Radhakrishna, M., Srikant, N., & Ginjupalli, K. (2019). Effect

of silver diamine fluoride-potassium iodide and 2% chlorhexidine gluconate cavity cleansers on the bond strength and microleakage of resin-modified glass ionomer cement. *Journal of conservative dentistry : JCD*, *22*(2), 201–206. https://doi.org/10.4103/JCD.JCD_485_18

Gurgan, S., Kutuk, Z. B., Ergin, E., Oztas, S. S., & Cakir, F. Y. (2015). Four-year randomized clinical trial to evaluate the clinical performance of a glass ionomer restorative system. *Operative dentistry*, *40*(2), 134–143.

Gwinnett A. J. (1994). Chemically conditioned dentin: a comparison of conventional and environmental scanning electron microscopy findings. *Dental materials : official publication of the Academy of Dental Materials*, *10*(3), 150–155.

Hamama, H. H., Burrow, M. F., & Yiu, C. (2014). Effect of dentine conditioning on adhesion of resin-modified glass ionomer adhesives. *Australian dental journal*, 59(2), 193–200.

Hamama, H., Yiu, C., & Burrow, M. (2015). Effect of silver diamine fluoride and potassium iodide on residual bacteria in dentinal tubules. *Australian Dental Journal*, 60(1), 80–87.

Horst JA, Ellenikiotis H, Milgrom PL.(2016). UCSF protocol for caries arrest using silver diamine fluo- ride: rationale, indications, and consent. *J Calif Dent Assoc.* 2016;44(1):16-28.

Hoshika, S., De Munck, J., Sano, H., Sidhu, S. K., & Van Meerbeek, B. (2015). Effect of Conditioning and Aging on the Bond Strength and Interfacial Morphology of Glass-ionomer Cement Bonded to Dentin. *The journal of adhesive dentistry*, *17*(2), 141–146.

Mustafa, H., Soares, A., Paris, S. *et al.* The forgotten merits of GIC restorations: a systematic review. *Clin Oral Invest* **24**, 2189–2201 (2020).

Imbery, Terence & Swigert, Ryan & Charles, Janus & Moon, pc. (2009). An evaluation of dentin conditioners for resinmodified glass ionomer cements. *General Dentistry.* 57. 356-62.

Inoue, S., Abe, Y., Yoshida, Y., De Munck, J., Sano, H., Suzuki, K., Lambrechts, P., & Van Meerbeek, B. (2004). Effect of conditioner on bond strength of glass-ionomer adhesive to dentin/enamel with and without smear layer interposition. *Operative Dentistry*, 29(6), 685–692.

International Organisation for Standardisation. ISO/TS 11405 Dentistry -- Testing of adhesion to tooth structure. 3rd ed. Geneva: International Organisation for Standardisation; 2015.

Ismail, A.M.; Bourauel, C.; ElBanna, A.; Salah Eldin, T. Micro versus Macro Shear Bond Strength Testing of Dentin-Composite Interface Using Chisel and Wireloop Loading Techniques. *Dent. J.* 2021, 9, 140.

Jennings, M. C., Minbiole, K. P., & Wuest, W. M. (2015). Quaternary Ammonium Compounds: An Antimicrobial Mainstay and Platform for Innovation to Address Bacterial Resistance. *ACS infectious diseases*, *1*(7), 288–303.

Jensen, E.C. (2013), Quantitative Analysis of Histological Staining and Fluorescence Using ImageJ. Anat. Rec., 296: 378-381.

Jiang, M., Mei, M. L., Wong, M. C. M., Chu, C. H., & Lo, E. C. M. (2020). Effect of silver diamine fluoride solution application on the bond strength of dentine to adhesives and to glass ionomer cements: a systematic review. *BMC oral health*, *20*(1), 40. https://doi.org/10.1186/s12903-020-1030-z

Jin, X. Z., Homaei, E., Matinlinna, J. P., & Tsoi, J. (2016). A new concept and finite-element study on dental bond strength tests. *Dental materials: official publication of the Academy of Dental Materials,* 32(10), e238–e250.

Judy Bendit, Douglas A. Young. (2017) Dental Academy of Continuing Education. Supplement to *PennWell Publications*. Silver diamine fluoride: The newest tool in your caries management tootkit, Published online Jul 2017

Kassebaum, N. J., Bernabé, E., Dahiya, M., Bhandari, B., Murray, C. J., & Marcenes, W. (2015). Global burden of untreated caries: a systematic review and metaregression. *Journal of dental research*, *94*(5), 650–658.

Khoroushi M, Keshani F (2013) A review of glass-ionomers: from conventional glassionomer to bioactive glass-ionomer. Dent Res J (Isfahan) 10, 411-420.

Knight, G., McIntyre, J. and Mulyani, (2006), The effect of silver fluoride and potassium iodide on the bond strength of auto cure glass ionomer cement to dentine. Australian Dental Journal, 51: 42-45.

Knight, Geoffrey & Mclntyre, JM & G. Craig, G & , Mulyani. (2006). Ion uptake into demineralized dentine from glass ionomer cement following pretreatment with silver fluoride and potassium iodide. *Australian Dental Journal*. 51. 237 - 241.

Knight, G., Mcintyre, J., Craig, G., Zilm, P., & Gully, N. (2007). Differences between normal and demineralized dentine pretreated with silver fluoride and potassium iodide after an in vitro challenge by Streptococcus mutans. *Australian Dental Journal*,52(1), 16-21.

Knight GM, McIntyre JM, Craig GG, Zilm, P.S, Gully, N.J. (2009). An in vitro model to measure the effect of a silver fluoride and potassium iodide treatment on the permeability of demineralized dentine to Streptococcus mutans. *Aust Dent J.* 2005;50(4):242-245.

Knight GM, McIntyre JM, Craig GG, Mulyani (2010). "Leaving Decay in My Cavity? You Must Be Kidding!" *Dentistry Today*, February 2010

Knight GM, J. M. McIntyre, Mulyani (2016). The effect of silver fluoride and potassium iodide on the bond strength of auto cure glass ionomer cement to dentine.

Koibuchi, H, Yasuda N, Nakabayashi N, Bonding to dentin with a self-etch primer: the effect of smear layers, *Dent. Mater.,* 2001;17:122-126.

Koizumi, Hiroyasu & Hamama, Hamdi & Burrow, Michael. (2016). Effect of a silver diamine fluoride and potassium iodide-based desensitizing and cavity cleaning agent on bond strength to dentine. *International Journal of Adhesion and Adhesives*. 68. 10.1016/j.ijadhadh.2016.02.008.

Kokmaz Y, Ozel E, Attar N, Bicer CO. Influence of different conditioning methods on the shear bond strength of novel lightcuring nanoionomer restorative to enamel and dentin. *Lasers Med Sci* 2010; 25:861866.

Koutsi, V., Noonan, R. G., Horner, J. A., Simpson, M. D., Matthews, W. G., & Pashley, D. H. (1994). The effect of dentin depth on the permeability and ultrastructure of primary molars. *Pediatric dentistry*, 16(1), 29–35.

Kucukyilmaz E, Savas S, Akcay M, Bolukbasi B. (2016). Effect of silver diamine fluoride and ammonium hexafluorosilicate applications with and without Er:YAG laser irradiation on the microtensile bond strength in sound and caries-affected dentin. *Lasers Surg Med.*, 48:62-69.

Liebermann A, Ilie N, Roos M, Stawarczyk B. (2017). Effect of Storage Medium and Aging Duration on Mechanical Properties of Self-Adhesive Resin-Based Cements. Journal of Applied Biomaterials & Functional Materials. 2017;15(3):206-214.

Lin A, McIntyre NS, Davidson RD. (1992). Studies on the adhesion of glass-ionomer cements to dentin, *J. Dent. Res.,* 7:1836-1841.

Liu, Y.; Li, N.; Qi, Y.; Niu, L.N.; Elshafiy, S.; Mao, J.; Breschi, L.; Pashley, D.H.; Tay, F.R.(2011) The use of sodium trimetaphosphate as a biomimetic analog of matrix phosphoproteins for remineralisation of artificial caries-like dentin. *Dent. Mater.* 27, 465–477

Llodra JC, Rodriguez A, Ferrer B, Menardia D, Ramos T, & Morato M. (2005). Efficacy of silver diamine fluoride for caries reduction in primary teeth and first permanent molars of schoolchildren: A 36-month clinical trial *Journal of Dental Research* 84(8) 721-724.

Lohbauer U. (2010) Dental glass ionomer cements as permanent filling materials? – Properties, limitations and future trends, Materials, 3:76-96.

Lou, Y.L. & Botelho, Michael & Darvell, Brian. (2011). Reaction of silver diamine fluoride with hydroxyapatite and protein. *Journal of dentistry.* 39. 612-8. 10.1016/j.jdent.2011.06.008.

Mahendra Rai, Alka Yadav, Aniket Gade. (2009). Silver nanoparticles as a new generation of antimicrobials, Biotechnology Advances, Volume 27, Issue 1, 2009, Pages 76-83, ISSN 0734-9750, https://doi.org/10.1016/j.biotechadv.2008.09.002.

Marquezan, M., Corrêa, F. N., Sanabe, M. E., Rodrigues Filho, L. E., Hebling, J., Guedes-Pinto, A. C., & Mendes, F. M. (2009). Artificial methods of dentine caries induction: A hardness and morphological comparative study. *Archives of oral biology*, *54*(12), 1111–1117. https://doi.org/10.1016/j.archoralbio.2009.09.007

Mei ML, Ito L, Chu CH, Lo EC, Zhang CF. (2013) Prevention of dentine caries using silver diamine fluoride application followed by Er:YAG laser irradiation: an in vitro study. *Lasers Med Sci*

Mei, M. L., Li, Q. L., Chu, C. H., Yiu, C. K., & Lo, E. C. (2012). The inhibitory effects of silver diamine fluoride at different concentrations on matrix metalloproteinases. *Dental materials : official publication of the Academy of Dental Materials*, *28*(8), 903–908.

Mei, M. L., Li, Q., Chu, C.-H., Lo, E. C.-M., & Samaranayake, L. P. (2013). Antibacterial effects of silver diamine fluoride on multi-species cariogenic biofilm on caries. *Annals of Clinical Microbiology and Antimicrobials*, 12, 4.

Mei, M. L., Ito, L., Cao, Y., Li, Q. L., Lo, E. C., & Chu, C. H. (2013). Inhibitory effect of silver diamine fluoride on dentine demineralisation and collagen degradation. *Journal of dentistry*, *41*(9), 809–817.

Mei, ML, Chu CH, Low KH, Che CM, Lo EC. (2013) Caries arresting effect of silver diamine fluoride on dentine carious lesion with S. mutans and L. acidophilus dual-species cariogenic biofilm. *Med Oral Patol Oral Cir Bucal*,18:824–831.

Mei, M. L., Ito, L., Cao, Y., Lo, E. C., Li, Q. L., & Chu, C. H. (2014). An ex vivo study of arrested primary teeth caries with silver diamine fluoride therapy. *Journal of dentistry*, *42*(4), 395–402.

Meredith, N., Sherriff, M., Setchell, D. J., & Swanson, S. A. (1996). Measurement of the microhardness and Young's modulus of human enamel and dentine using an indentation technique. *Archives of oral biology*, *41*(6), 539–545.

Mount GJ, Hume WR, Preservation and restoration of teeth, 2nd ed, Knowledge Books and Software, Middlesbrough, 2005

Nakanuma K., Hayakawa T., Tomita T., Yamazaki M., Effect of the application of dentin primers and a dentin bonding agent on the adhesion between the resin-modified glass-ionomer cement and dentin, *Dent Mater*, 14 (1998) 281-6.

Naves, L. Z., Silva, G. R., Correr-Sobrinho, L., Costa, A. R., Valdivia, A. D., & Soares, C. J. (2016). Influence of crosshead speed on failure load and failure mode of restored maxillary premolars. *Brazilian oral research*, *30*, S1806-83242016000100206.

Ng, E., Saini, S., Schulze, K. A., Horst, J., Le, T., & Habelitz, S. (2020). Shear Bond Strength of Glass Ionomer Cement to Silver Diamine Fluoride-Treated Artificial Dentinal Caries. *Pediatric dentistry*, *42*(3), 221–225.

Ngo HC, Mount G, McIntyre K, Tuisuva J, Von Doussa RJ (2006). Chemical exchange between glass-ionomer restorations and residual carious dentine in permanent molars: an in vivo study. *J Dent* 2006; 34(8):608-13

Ngo, H., Mount, G. J., & Peters, M. C. (1997). A study of glass-ionomer cement and its interface with enamel and dentin using a low-temperature, high-resolution scanning electron microscopic technique. *Quintessence international (Berlin, Germany : 1985)*, *28*(1), 63–69.

Ogata M, Harada N, Yamaguchi S, Nakajima M, Periera PN, Tagami J, Effect of different burs on bond strengths of self-etching primer systems, *Oper. Dent.,* 2001;26:375-382.

Oilo G. (1993). Bond strength testing--what does it mean?. *International dental journal*, 43(5), 492–498.

Paiva, Lilian & Fidalgo, Tatiana & Maia, Lucianne. (2014). Mineral content of ionomer cements and preventive effect of these cements against white spot lesions around restorations. Brazilian oral research. 28. 1-9. 10.1590/1807-3107BOR-2014.vol28.0034.

Peng JY, Botelho M, Matinlinna J..(2009). Silver compounds used in dentistry for caries management: a review. *J Dent* 2012 40: 531–541.Rosenblatt A, Stamford TC, Niederman R. Silver diamine fluoride: a caries ‘‘silver-fluoride bullet’’. *Journal of Dental Research*, 88:116–25

Peng, J.-Y., Botelho, M. G., Matinlinna, J. P., Pan, H.-B., Kukk, E., & Low, K.-J. (2021). Interaction of storage medium and silver diamine fluoride on demineralized dentin. *Journal of International Medical* Research.

Pereira LC, Nunes MC, Dibb RG, Powers JM, Roulet JF, Navarro MF. (2002) Mechanical properties and bond strengths of glass-ionomer cements, *J. Adhes. Dent.,* 2002;4:73-80.

Phillips' Science of Dental Materials 12th edition, pg 327.

Pitts, N., Zero, D., Marsh, P. *et al.* Dental caries. *Nat Rev Dis Primers* **3**, 17030 (2017).

Powis DR, Folleras T, Merson SA, Wilson AD. (1982) Improved adhesion of a glass ionomer cement to dentin and enamel, *J. Dent. Res.,* 1982;61:1416-1422.

Primus, Carolyn & Gutmann, James & Tay, Franklin & Fuks, Anna. (2021). Calcium Silicate and Calcium Aluminate Cements for Dentistry Reviewed. Journal of the American Ceramic Society. 105. 10.1111/jace.18051.

Puwanawiroj, A., Trairatvorakul, C., Dasanayake, A. P., & Auychai, P. (2018). Microtensile Bond Strength Between Glass Ionomer Cement and Silver Diamine Fluoride-Treated Carious Primary Dentin. *Pediatric dentistry*, *40*(4), 291–295.

Quock RL, Barros JA, Yang SW, Patel SA (2012) Effect of silver diamine fluoride on microtensile bond strength to dentin. *Oper Dent*. 2012 Nov-Dec; 37(6): 610–616.

Ramachandran, Aparna & Jyothi Lekshmi, B & Pillai, Rajesh & O Varghese, Nettiyat & A Salim, Afzal & Sujathan, U-Nu. (2018). Effect of Conditioning on Adhesion of Glass Ionomer Cements to Dentin. *Conservative Dentistry and Endodontic Journal*. 3. 6-10. 10.5005/jp-journals-10048-0031.

Rolla G, Saxegaard E. Critical evaluation of the composition and use of topical fluorides, with emphasis on the role of calcium fluoride in caries inhibition. *Journal of Dental Research* 1990;69(Spec Iss):780–5.

Rolland, S. L., Carrick, T. E., Walls, A. W., & McCabe, J. F. (2007). Dentin decontamination using chloramine T prior to experiments involving bacteria. *Dental materials : official publication of the Academy of Dental Materials*, *23*(12), 1468–1472.

Rosenblatt, A., Stamford, T. C., & Niederman, R. (2009). Silver diamine fluoride: a caries "silver-fluoride bullet". *Journal of dental research*, *88*(2), 116–125.

Sano H, Kanemura N, Burrow MF, Inai N, Yamada T, Tagami J. Effect of operator variability on dentin adhesion: Students vs. dentists. Dent Mater J. 1998;17:51–8.

Sano, H., Shono, T., Sonoda, H., Takatsu, T., Ciucchi, B., Carvalho, R., & Pashley, D. H. (1994). Relationship between surface area for adhesion and tensile bond strength--evaluation of a micro-tensile bond test. *Dental materials : official publication of the Academy of Dental Materials*, *10*(4), 236–240. https://doi.org/10.1016/0109-5641(94)90067-1

Savas, Selcuk & Küçükyılmaz, Ebru & U. Celik, Esra & Ates, Mustafa. (2015). Effects of different antibacterial agents on enamel in a biofilm caries model. *Journal of Oral Science*. 57. 367-372. 10.2334/josnusd.57.367.

Sayed, M., Matsui, N., Uo, M., Nikaido, T., Oikawa, M., Burrow, M. F., & Tagami, J. (2019). Morphological and elemental analysis of silver penetration into sound/demineralized dentin after SDF application. *Dental materials : official publication of the Academy of Dental Materials*, *35*(12), 1718–1727.

Sayed, Mahmoud & MATSUI, Naoko & Hiraishi, Noriko & Inoue, Go & NIKAIDO, Toru & Burrow, Michael & Tagami, Junji. (2018). Evaluation of discoloration of sound/demineralized root dentin with silver diamine fluoride: In-vitro study. *Dental Materials Journal.* 10.4012/dmj.2018-008.

Scheffel DL, Tenuta LM, Cury JA, Hebling J. (2012). Effect of acid etching time on demineralization of primary and permanent coronal dentin. American Journal of Dentistry 2012;25:235-238.

Scherrer, S. S., Cesar, P. F., & Swain, M. V. (2010). Direct comparison of the bond strength results of the different test methods: A critical literature review. *Dental Materials,* 26(2), e78–e93.

Schneider, H., Park, K., Häfer, M., Rüger, C., Schmalz, G., Krause, F., Schmidt, J., Ziebolz, D., & Haak, R. (2017). Dental Applications of Optical Coherence Tomography (OCT) in Cariology. *Applied Sciences, 7*, 472.

Schüpbach P, Guggenheim B, Lutz F. Human root caries: histopathology of initial lesions in cementum and dentin. *J Oral Pathol Med.* 1989;18:146-56

Schwendicke, F., Eggers, K., Meyer-Lueckel, H., D**?**rfer, C., Kovalev, A., Gorb, S., & Paris, S. (2015). In vitro Induction of Residual Caries Lesions in Dentin: Comparative Mineral Loss and Nano-Hardness Analysis. *Caries Research*, 49(3), 259–265. doi:10.1159/000371897

Selvaraj, K., Sampath, V., Sujatha, V., & Mahalaxmi, S. (2016). Evaluation of microshear bond strength and nanoleakage of etch-and-rinse and self-etch adhesives to dentin pretreated with silver diamine fluoride/potassium iodide: An in vitro study. *Indian Journal of Dental Research*, 27(4), 421. doi:10.4103/0970-9290.191893

Seto, J., Horst, J. A., Parkinson, D. Y., Frachella, J. C., & DeRisi, J. L. (2020). Enhanced Tooth Structure Via Silver Microwires Following Treatment with 38 Percent Silver Diamine Fluoride. *Pediatric dentistry*, *42*(3), 226–231.

Shah S, Bhaskar V, Venkatraghavan K, Choudhary P, Ganesh M, Trivedi K. Silver diamine fluoride: A review and current applications. *J Adv Or Res* 2014;5:25–35.

Sharafeddin, F., Alavi, A. A., Siabani, S., & Safari, M. (2020). Comparison of Shear Bond Strength of Three Types of Glass Ionomer Cements Containing Hydroxyapatite Nanoparticles to Deep and Superficial Dentin. *Journal of dentistry* (Shiraz, Iran), 21(2), 132–140.

Shebl EA, Etman WM, Genaid TM, Shalaby ME (2015). Durability of bond strength of glassionomers to enamel. Tanta Dent J, 12(1): 16- 27

Shimizu A. Effect of diammine silver fluoride on recurrent caries. *Jap J Conserv Dent* 1974;17:183–201.

Shono, Y., Terashital, M., Pashley, E. L., Brewer, P. D., & Pashley, D. H. (1997). Effects of cross-sectional area on resin-enamel tensile bond strength. *Dental Materials*, 13(5), 290-296

Simasetha, Siripan & Klaisiri, Awiruth & Sriamporn, Tool & Sappayatosok, Kraisorn & Thamronganankskul, Niyom. (2022). Surface Treatment Effect on Shear Bond Strength between Lithium Disilicate Glass-Ceramic and Resin Cement. European Journal of Dentistry. 16. 10.1055/s-0041-1735908.

Sirisha, K., Rambabu, T., Shankar, Y. R., & Ravikumar, P. (2014). Validity of bond strength tests: A critical review: Part I. *Journal of conservative dentistry* : JCD, 17(4), 305–311.

Söderholm K-JM. Journal of Adhesion Science and Technology. 2009;23:973

Soeno K, Taira Y, Matsumura H, Atsuta M. Effect of desensitizers on bond strength of adhesive luting agents to dentin. Journal of Oral Rehabilitation 2001;28:1122–8.

Suzuki T, Nishida M, Sobue S, Moriwaki Y. (1974). Effects of diammine silver fluoride on tooth enamel. *J Osaka Univ Dent Sch*. 14:61–72.

Talip, M.S. & Zakaria, Ahmad & Sockalingam, Nagarajan. (2017). Comparative evaluation of the effect of a resin modified glass ionomer cement universal adhesive on the shear bond strength of glass ionomer cements. Archives of Orofacial Sciences. 12. 95-104.

Tanumiharja M, M.F Burrow, M.J Tyas, Microtensile bond strengths of glass ionomer (polyalkenoate) cements to dentine using four conditioners, Journal of Dentistry,Volume 28, Issue 5, 2000,Pages 361-366, ISSN 0300-5712.

Tay, F. R., Smales, R. J., Ngo, H., Wei, S. H., & Pashley, D. H. (2001). Effect of different conditioning protocols on adhesion of a GIC to dentin. *The journal of adhesive dentistry*, *3*(2), 153–167.

Tyas, Martin. (2003). Milestones in Adhesion: Glass-ionomer Cements. *The journal of adhesive dentistry*. 5. 259-66.

Ugurlu M. (2020). Bonding of a resin-modified glass ionomer cement to dentin using universal adhesives. *Restorative dentistry & endodontics*, *45*(3), e36.

Valente, R. M., De Rijk, W. G., Drummond, J. L., & Evans, C. A. (2002). Etching conditions for resin-modified glass ionomer cement for orthodontic brackets. *American journal of orthodontics and dentofacial orthopedics : official publication of the American Association of Orthodontists, its constituent societies, and the American Board of Orthodontics*, *121*(5), 516–520.

Van Meerbeek, B., Conn, L. J., Jr, Duke, E. S., Eick, J. D., Robinson, S. J., & Guerrero, D. (1996). Correlative transmission electron microscopy examination of nondemineralized and demineralized resin-dentin interfaces formed by two dentin adhesive systems. Journal of dental research, 75(3), 879–888.

Van Meerbeek B, De Munck J, Yoshida Y, InoueS, Vargas M, Vijay P et al. (2003). BuonocoreMemorial Lecture. Adhesion to enamel anddentine: Current status and future challenges.Oper Dent, 28(3): 215-235.

Van Meerbeek, B., Yoshida, Y., Inoue, S., De Munck, J., Van Landuyt, K., & Lambrechts, P. (2006). Glass-ionomer adhesion: the mechanisms at the interface. *Journal of dentistry*, *34*(8), 615-618.

Van Meerbeek, Bart Peumans, M., Poitevin, A., Mine, A., Van Ende, A., Neves, A., & De Munck, J. (2010). Relationship between bond-strength tests and clinical outcomes. *Dental Materials*, 26(2), e100-e121.

Van Noort, R., Noroozi, S., Howard, I. C., & Cardew, G. (1989). A critique of bond strength measurements. *Journal of dentistry*, *17*(2), 61–67.)

Vinh N, Cody N, Joel F, Carolyn P. Potassium Iodide. The Solution to Silver Diamine Fluoride Discoloration?. *Adv Dent Oral Health*. 2017; 5(1): 555655.

W Skilton, Jonathan & Tyas, Martin & Woods, Michael. (2006). Effects of surface treatment on orthodontic bonding to amalgam. *Australian orthodontic journal*. 22. 59-66.

Wang, J., Song, W., Zhu, L., & Wei, X. (2019). A comparative study of the microtensile bond strength and microstructural differences between sclerotic and Normal dentine after surface pretreatment. *BMC oral health*, *19*(1), 216.

Wang, Y., & Darvell, B. W. (2007). Failure mode of dental restorative materials under Hertzian indentation. *Dental Materials*, *23*(10), 1236-1244.

Willershausen I, Schulte D, Azaripour A, et al. Penetration potential of a silver diamine fluoride solution on dentin surfaces. An ex vivo study. *Clin Lab*. 2015;61(11):1695–701.

Wilson AD. (1974). Alumino-silicate polyacrylic acid cement, Brit. Polym. *J.,* 1974;6:165-179.

Wilson AD, McLean JW. (1988). Glass ionomer cement, Chicago: Quintessence; 1988.

Xie, D., Brantley, W. A., Culbertson, B. M., & Wang, G. (2000). Mechanical properties and microstructures of glass-ionomer cements. *Dental materials : official publication of the Academy of Dental Materials*, *16*(2), 129–138.

Yamaga M, Koide T, Hieda T. Adhesiveness of glass ionomer cement containing tannin-fluoride preparation (HY agent) to dentin — an evaluation of adding various ratios of HY agent and combination with application diamine silver fluoride. *Dent Mater J* 1993;12(1):36-44.

Yamaga R, Nishino M, Yoshida S, Yokomizo I. Diammine silver fluoride and its clinical application. *J Osaka Univ Dent Sch*. 1972;12:1-20.

Yap AU, Mudambi S, Chew CL, Neo JC (2001). Mechanical properties of an improved visible light-cured resin-modified glass ionomer cement. Oper Dent, 26(3): 295-301.

Yazici, A., Celik, C., Özgünaltay, G., & Dayangaç, B. (2007). Bond strength of different adhesive systems to dental hard tissues. Operative dentistry, 32(2), 166-172.

Yee R, Holmgren C, Mulder J, Lama D, Walker D, & van Palenstein Helderman W (2009) Efficacy of silver diamine fluoride for arresting caries treatment *Journal of Dental Research* 88(7) 644-647.

Yoshida Y, Van Meerbeek B, Nakayama Y, Snauwaert J, Hellmans L, Lambrechts P, Vanherle G, Wakasa K, Evidence of chemical bonding at biomaterial-hard tissue interfaces, *J. Dent. Res.,* 2000;79:709-714.

Yu, O. Y., Zhao, I. S., Mei, M. L., Lo, E. C. M., & Chu, C. H. (2018). Caries-arresting effects of silver diamine fluoride and sodium fluoride on dentine caries lesions. *Journal of dentistry*, *78*, 65–71.

Zhang, W., McGrath, C., Lo, E. C., & Li, J. Y. (2013). Silver diamine fluoride and education to prevent and arrest root caries among community-dwelling elders. *Caries research*, *47*(4), 284–290. https://doi.org/10.1159/000346620

Zhao, I. S., Mei, M. L., Burrow, M. F., Lo, E. C.-M., & Chu, C.-H. (2017). Effect of Silver Diamine Fluoride and Potassium Iodide Treatment on Secondary Caries Prevention and Tooth Discolouration in Cervical Glass Ionomer Cement Restoration. *International Journal of Molecular Sciences,* 18(2), 340.

Zhao, I. S., Mei, M. L., Zhou, Z. L., Burrow, M. F., Lo, E. C., & Chu, C. H. (2017). Shear Bond Strength and Remineralisation Effect of a Casein Phosphopeptide-Amorphous Calcium Phosphate-Modified Glass Ionomer Cement on Artificial "Caries-Affected" Dentine. *International journal of molecular sciences*, 18(8), 1723.

Zhao, I. S., Gao, S. S., Hiraishi, N., Burrow, M. F., Duangthip, D., Mei, M. L., Lo, E. C., & Chu, C. H. (2018). Mechanisms of silver diamine fluoride on arresting caries: a literature review. *International dental journal*, *68*(2), 67–76.

Zhao IS, Chu S, Yu OY, Mei ML, Chu CH, Lo ECM. (2019) Effect of silver diamine fluoride and potassium iodide on shear bond strength of glass ionomer cements to caries-affected dentine. Int Dent J 69, 341-347

Zhi QH, Lo EC, Lin HC. Randomized clinical trial on effectiveness of silver diamine fluoride and glass ionomer in arresting dentine caries in preschool children. *J Dent.* 2012;40(11):962- 967.