

A study of various factors that influence the success of posterior resin-based restorations

Negin Alvanforoush

ORCID: 0000-0002-5352-2625

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Abstract

This thesis is centred on the investigation of several physical properties related to bulk-fill resin composites that may influence the success of posterior resin-based restorations. In addition, a test methodology to investigate the fracture toughness of brittle restorative materials was evaluated, including the potential influence of the storage medium.

The thesis work begins with a systematic review comparing the clinical success rates of resin composite restorations in posterior teeth, over two time periods, namely 1995-2005 and 2006-2016. It was shown that the main failure reasons for posterior resin composite restorations changed slightly over these periods, with factors related to the material and tooth becoming more significant in the later period, although recurrent caries remained an important reason for failure. Based on the broad findings of the review, three laboratory-based projects were designed to examine the physical properties of resin-based composites, focusing on the recent bulk-fill restorative materials. The experiments also included the effects of polymerising light-curing units, since the new high irradiance (light intensity) units recommend shorter curing times. In addition, a study on fracture toughness of glass-ionomer cements were conducted to determine whether a simple fracture toughness test would provide useful outcomes for a brittle material such as GIC and whether an ion-containing storage medium such as artificial saliva would influence outcomes.

The results of these experiments indicated that variations in material composition and curing light devices had a significant influence on the physical properties such as curing stress shrinkage, the combined temperature of polymerisation and curing light radiant exposure (energy), and wear after thermal ageing. A final experiment investigated the effects of the polymerisation shrinkage on the movement of tooth cusps when the resin composite was exposed to two different irradiances curing light devices. This experiment showed that the high irradiance produced greater and more rapid cuspal movement and a greater rise in temperature

within the pulp chamber. The results probably cannot provide a direct translation to the clinical situation. However, it would seem that the use of bulk-fill resin composite materials needs to be managed more carefully than perhaps what has been recommended by manufacturers of such materials.

Declaration

This is to certify that:

1. This thesis contains only my original work towards the PhD;
2. Due acknowledgment has been made to all other material used;
3. the thesis is fewer than the maximum word limit in length, exclusive of tables, bibliographies and appendices.

Negin Alvanforoush

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Preface

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Dedication

To my parents for raising me to believe that anything is possible, and to my husband for making everything possible with his support.

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List of abbreviations

GIC	Glass Ionomer Cement
RMGIC	Resin-modified Glass Ionomer Cement
HEMA	2-hydroxyethyl Methacrylate
PRG	Prereacted Glass
UDMA	Urethane Dimethacrylate
S-PRG	Surface Pre-reacted Glass
Bis-GMA	Bisphenol-A Glycidylmethacrylate
UDMA	Urethane Dimethacrylate
TEGDMA	Triethyleneglycol Dimethacrylate
CT	Compact Tension
SEVNB	Single Edge V-Notch Beam
CEJ	Cementoenamel Junction
QTH	Quartz-Tungsten-Halogen
LED	Light-Emitting Diode
ACTA	Academisch Centrum for Tandheelkunde Amsterdam
MOD	Mesio occluso distal

Chapter 1: Introduction

The research projects in this thesis were developed to investigate various factors that influence the success of posterior resin-based restorations with a focus on the more recently introduced so-called bulk restorative materials. Every time a restoration fails, and replacement is necessary, a cavity often needs to be prepared or at least modified, leading to further removal of tooth structure. Consequently, each restoration replacement is typically larger; the residual tooth structure becomes weaker, which increases the risk of restoration failure (1-3). Therefore, improving the success rate of restorations aims to break this cycle, ultimately extending the life of a restored tooth and the subsequent life quality benefits this will have.

Tooth-coloured restorative materials are now the most popular restorative materials used by dentists and desired by patients. This popularity is mainly due to aesthetic reasons (1, 2). For smaller direct restorations, the most commonly used restorative material is resin composite. Resin composites and resin-based adhesive systems have many advantages with respect to physical properties, such as the capacity to bond to the tooth structure, minimal tooth removal during cavity preparation, resistance to occlusal loading and wear, as well as excellent aesthetic qualities (4, 5). However, resin-based composites still have a number of shortcomings, such as polymerisation shrinkage(6, 7) and the stresses the shrinkage generates at the adhesive interface and within the remaining tooth structure. To improve the long-term success of resin composite restorations, there is a need to more fully understand the effects of physical and mechanical factors, particularly with respect to the new bulk-fill materials that have been marketed to reduce the time needed to place a restorative material into a cavity. It is known that a bulk of resin composite will potentially cause greater shrinkage stresses, increased exotherm from polymerisation and may also have a limited depth of cure. The question posed is, do the newer 'bulk-fill' materials minimise these potential problems?

Another material that has been recently promoted for used in load-bearing areas is glass ionomer cement. Compared to resin composite, glass ionomer cements exhibit several advantages, such as

their ability to adhere to enamel and moist dentine and the associated potential for anti-cariogenic properties such as the long-term fluoride and ion release (8, 9). Up until now, when physical tests are performed in restorative materials, the materials are typically stored in distilled or de-ionised water. Water is a poor solution for replicating the complexity of the saliva. Therefore, one of the projects of this thesis was designed to investigate how an artificial saliva solution that has a similar ionic content as human saliva may affect the physical properties of high viscosity glass ionomer cements. In addition, two fracture toughness methods were employed to determine which may be the simplest and most reliable to evaluate glass ionomer cements recommended for load-bearing restorations.

The success of a restoration is dependent on multiple factors, such as the operator, material, and also the patient (1, 10). In this thesis, the focus was set on material factors. Chapter two reviews the literature with respect to the composition of resin composites and glass ionomer cements. It also outlines the tests that can be used for evaluating materials in a laboratory setting. Chapter 3 is a systematic review investigating the failure reasons for posterior resin composite restorations clinically over a period of 20 years. The review was divided into 2 periods of 10 years to discover whether restoration failure patterns changed as materials changed but also use of the resin restorations changed. Then, four projects were designed to investigate these factors. The methods and results of these project are presented in the following chapters. Chapter 5 is an experiment to measure the fracture toughness of glass ionomers. In this experiment, two common laboratory methods used for testing fracture toughness of dental materials were chosen. Another issue that was investigated in this chapter was the effect of artificial saliva on fracture toughness of glass ionomers. Chapter 6 explains a project that investigates the effects of different intensity light curing units on core temperature and polymerisation shrinkage stress of resin composites. The following chapter (chapter 7) is a study on the impact of thermal ageing on flexural strength, wear, and hardness of resin composites. In this chapter mentioned physical properties are compared between bulk-fill and conventional resin composites. The last experiment is explained in chapter 8, which was an investigation of the influence of different intensity of light curing units on cuspal strains of teeth

restored with different bulk-fill resin composites. And the last chapter discusses the results and findings of the conducted experiments.

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Chapter 2: Literature review

2.1: Teeth

Knowledge about tooth structure is critical and crucial to understanding how it might be affected by dental diseases as well as how a tooth will be altered when a restoration may be inserted into a prepared cavity (1). Each tooth consists of two broad parts, the crown and root. The crown is that part

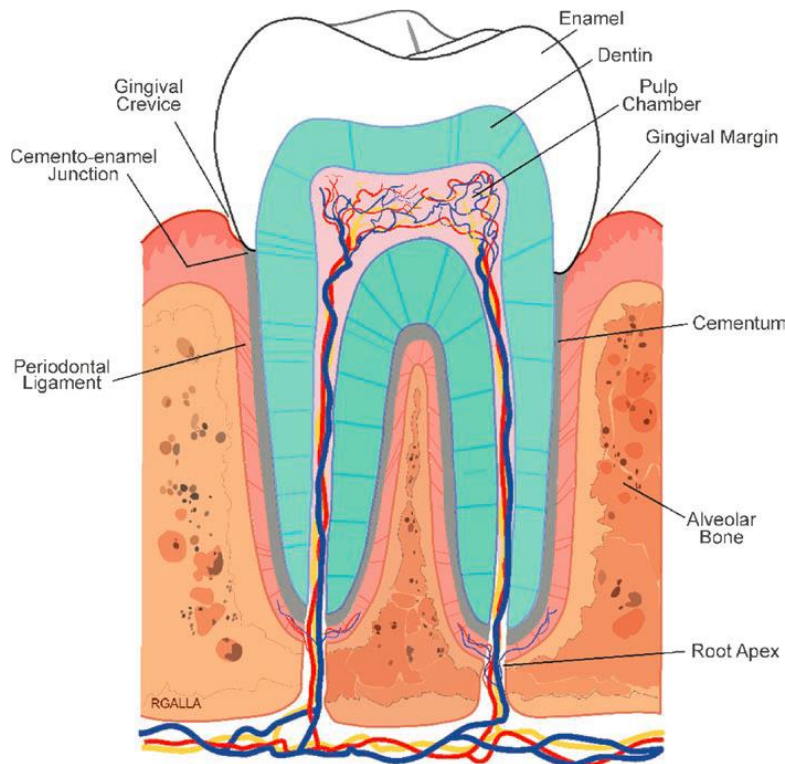


Figure-1 Tooth structure (2)

which is visibly present in oral cavity and root is that part beneath the gingival tissues and surrounded by the supporting alveolar bone. The linking tissue between the tooth and bone is called periodontium or periodontal ligament (Fig-1) The major portion of tooth structure is comprised of the tissue called dentine. The dentine is covered by a layer of enamel in crown and cementum in the root. The junction between these two outer layers is called the Cemento-Enamel Junction (CEJ). The inner portion of the tooth is a soft tissue containing blood vessels and nerve endings, which is called the dental pulp. This soft tissue is surrounded by dentine to form a bounded chamber, called the pulp chamber. The dental pulp is connected too nerve fibres and blood vessels via a small opening at the end, or apex, of the root (2).

2.1.1: Enamel

Enamel is a hard protective covering on the crown. Enamel is the hardest tissue in the human body, due to its mineral content (96 wt %)(3). The inorganic part of enamel is made of a slightly carbonated hydroxyapatite (4). The physical structure of enamel is a grouped into rods (prisms), which are cylindrical in longitude section(3, 5).(Fig-2) The pattern and arrangement of these rods differs depending on the region of the crown of the tooth. Those rods located in cusps and the occlusal part

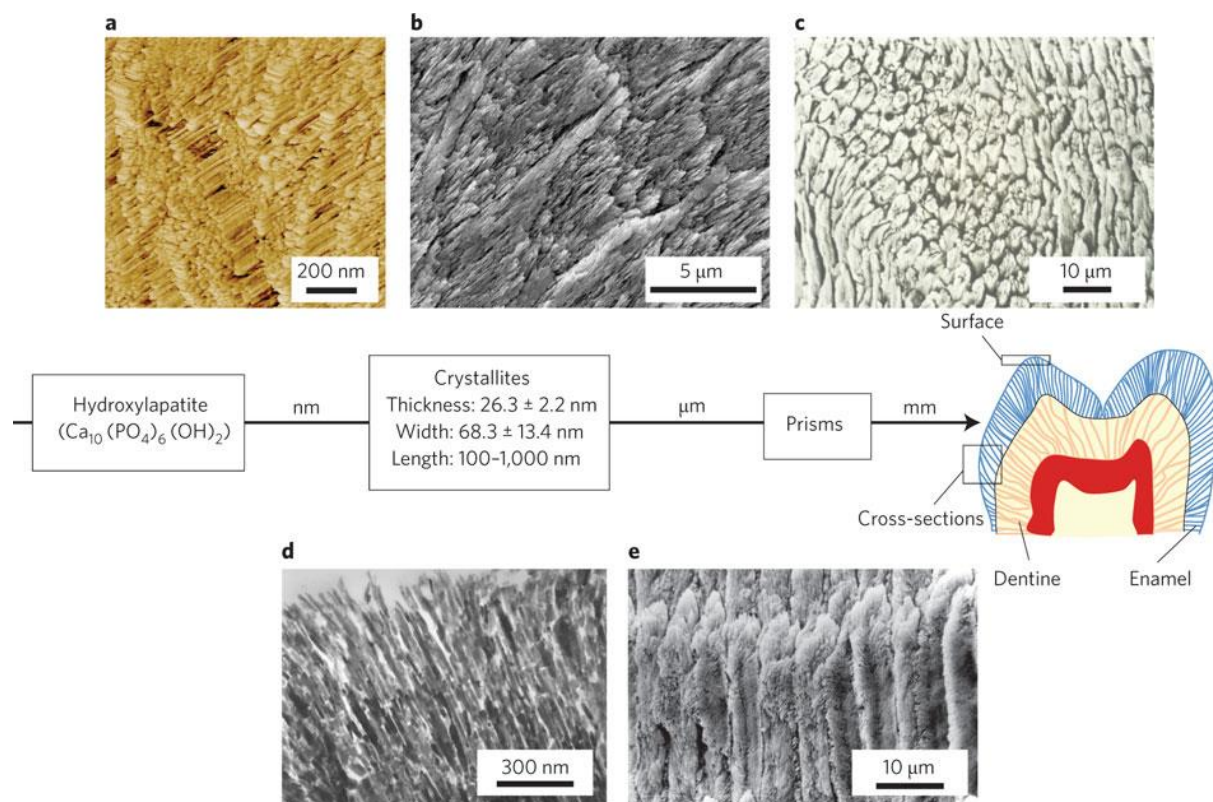


Figure-2 Enamel structure(6)

are longer those at the CEJ. Generally, prisms are located at right angles to underlying dentine. However, in permanent teeth in the cervical area of the crown, the direction of the rods is apically positioned (3, 5).

After tooth eruption into the oral cavity, this highly mineralised part of tooth continues a process of dynamic equilibrium of remineralisation and continues to take up further mineral on the tooth surface. Thus, over time carbonate is replaced with phosphate and hydroxyl groups with fluoride. This process is greatly affected by environmental conditions such as surrounding pH (1).

2.1.2: Dentine

Dentine is a calcified tissue which is covered by enamel in crown section of tooth. This material builds the bulk of the tooth. In volume 45% of dentine consist of mineral hydroxylapatite, 33% organic material and 22% water (7). And by weight 70% of dentine is minerals, 20% organic matter and 10% water (8). The organic matrix contains collagen (90%), non-collagenous protein (NCPs), phospholipids and growth factors. On the other hand, the inorganic part includes crystalline hydroxyapatite, sodium and magnesium (9).

Dentine is a complex tubular structure. Dentinal tubules are pathways for bacteria chemicals and dentine fluid. (Fig-3) The tubules are compound two different parts: 1- peritubular dentine, which forms the walls of the tubule and 2- intertubular dentine, which is located between peritubular dentin. The intertubular dentine is the main part of dentine structure, and it is rich in collagen fibres (3). Peritubular dentine is a hypermineralized tissue with a mineral content of more than 90%. In contrast

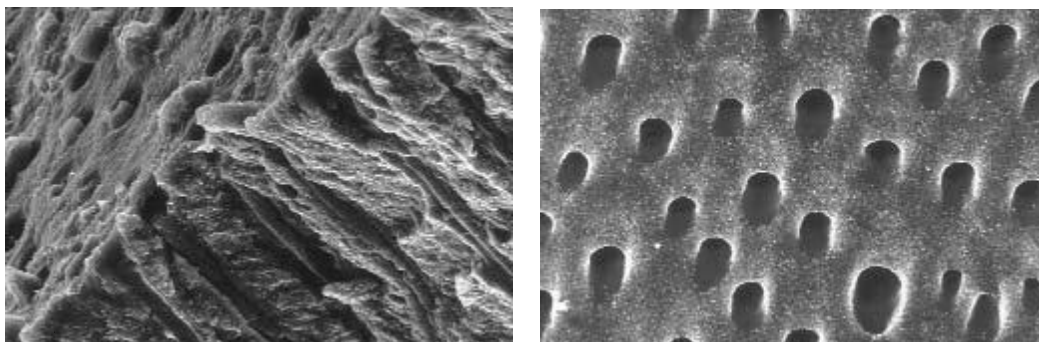


Figure-3 Dentine Structure A-vertical split x4200 B-Cavity floor after acid etch x4000 (1)

to the mineral content of intratubular dentin which is 70% (10, 11).

The cells responsible for the maintenance of this layer are called odontoblasts. They are located at the inner surface of the dentine layer. Modification of the structure is achieved by secretion of protein matrix and mineral to create dentine in response to some insult eg., disease, or aging (1).

2.1.3: Physical and mechanical properties

(Table-1)

TABLE-1 PHYSICAL PROPERTIES OF ENAMEL AND DENTINE (5,12)

Property	Enamel	Dentine
COMPRESSIVE MODULUS OF ELASTICITY (GPa)	60-120	18-24
HARDNESS (GPa)	3-6	0.13-0.51
FLEXURAL STRENGTH (MPa)	60-90	245-280
TOUGHNESS (MPam^{0.5})	0.8	3.1

2.2: Adhesive restorative materials

The popularity of adhesive restorative materials is growing rapidly and replacing silver amalgam. In conjunction with the 2013 FDI directive, which adverted to phase down the usage of amalgam, questionable biocompatibility of mercury in this material and aesthetic reasons, usage of these materials is considerably increasing (13-15). These materials contain different groups, Including Resin composites, Glass ionomer cements, Polyacid-modified resin composites (Compomers) (16) and Pre-reacted glass filled resin composites (Giomers). Some of these groups have subgroups (17).

Resin composite subgroups are macro-fill, micro-fill, small particle hybrid/midi-fill, micro-hybrid/mini-fill, nano-fill and nano-hybrid (18).

Glass ionomers group contain Type 1 luting and bonding (Resin-modified Glass Ionomer adhesives, Type 2 restorative material and Type 3 lining or base cement (16, 19, 20).

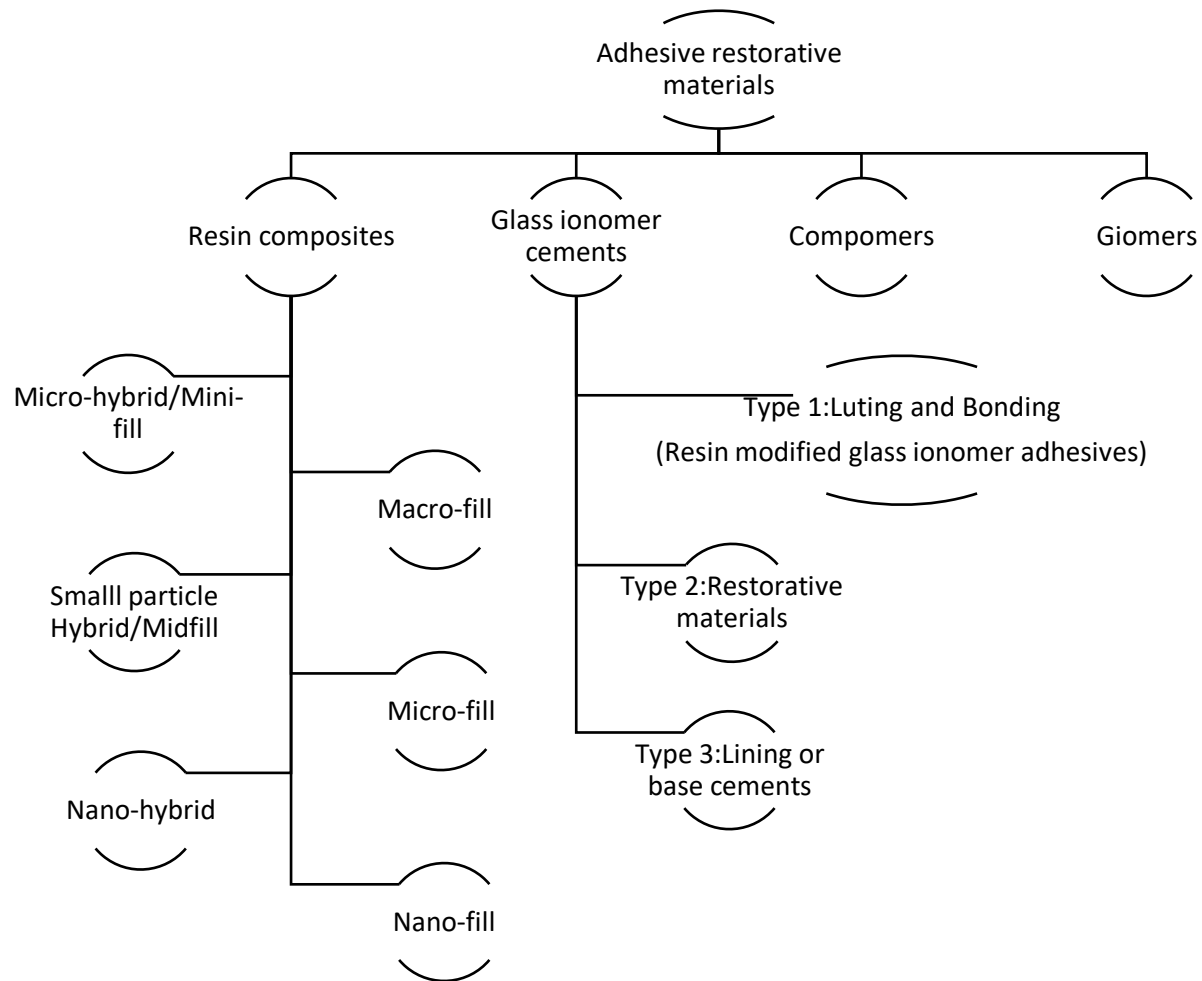


Figure-4 Adhesive restorative materials

2.2.1: Resin composites

Composite is a material made from several components. Resin composites (as the name indicates) are a combination of resin and other materials. They were introduced to dentistry in the mid-1960s by Bowen (21, 22). The first resin composites were not particularly strong and hence were limited to the use in anterior teeth. The resin matrix introduced by Bowen contained Bisphenol-A GlycidylMethAcrylate (bis-GMA) (21, 22) which is still used as the main resinous matrix material today. There have since been many alterations and improvements, but these have tended to be more closely related to the filler particles. (Table-2 (18,19)) Nowadays the resin composite restorative materials are available for use in almost every part of the oral cavity. They are very suitable restorative materials as a result of their improved physical properties and aesthetic appearance (19).

2.2.1.1: Composition:

Resin matrix:

This component includes a monomer system, initiator system and stabilisers (23).

Monomer system: The first introduced resin was bis-GMA (21). Following that introduction, other resins such as Urethane Dimethacrylate (UDMA), Triethyleneglycol Dimethacrylate (TEGDMA) and bisphenol-A-polyethylene glycol diether dimethacrylate (19) have also been used. However, bis-GMA remains the main resin used by manufacturers (Fig-5) (16, 23).

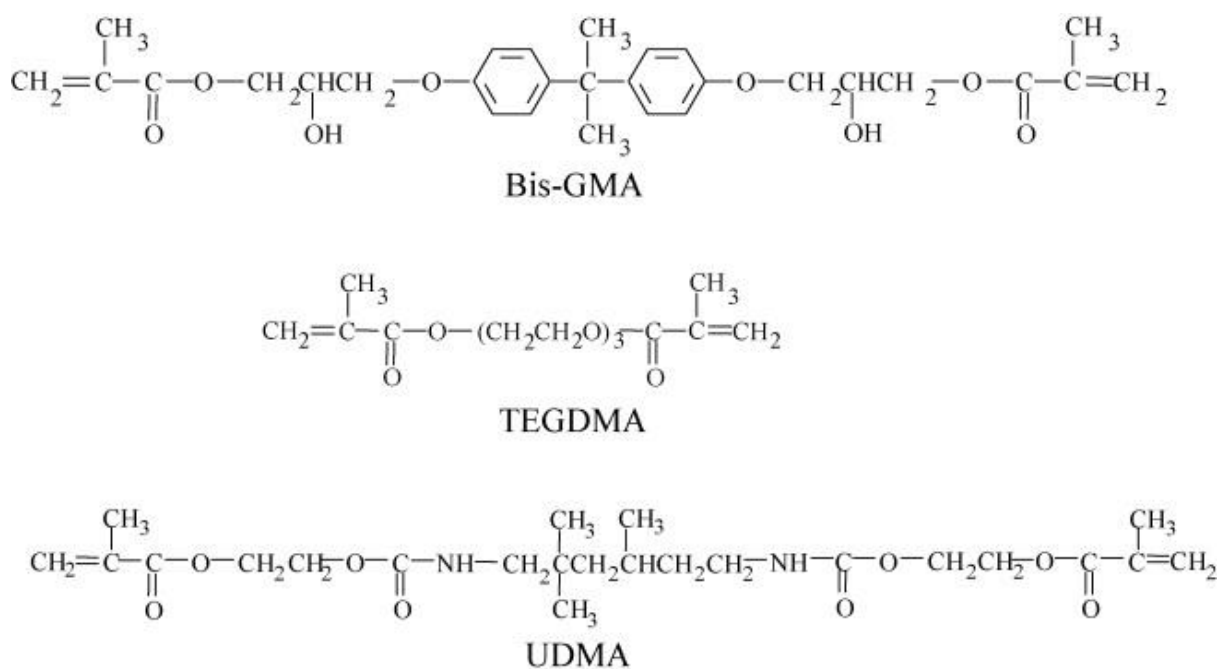


Figure-5 structures of monomers (24)

Initiators: resin composites are mostly light cured. A light cured material is a material where the polymerisation will be initiated by exposure to blue light (470nm wavelength) (19). The photo-activators such as camphorquinone and an aromatic amine are responsible for this initiation. In some of the resin composites, this reaction is between an organic amine and organic peroxide, which produces free radicals. These are dual cure resin composites, in which polymerisation starts with light exposure and continues even in the absence of the light (19). Some resin composites are self-cured or auto-cured, this groups polymerisation will be initiated by mixing two component pastes. One paste contains a chemical activator and the other chemical initiator (25).

Stabilisers: This component maximises the storage period and the stability of unreacted resin composite in addition to chemical stability of polymerised resin composite (23).

Glass filler particles:

This is an influential component on aesthetic, physical and mechanical qualities of resin composites, which has seen many changes during the life of resin composites (19, 23). Several materials are used as fillers including silicon dioxide, aluminium oxide, barium, zirconium oxide, borosilicate and barium aluminium silicate glasses (Table-2) (19).

Coupling agents:

As mentioned by Bowen (26) more desirable properties are achieved when a strong bond between filler particles and the matrix is present. By coating, the filler particles with a coupling agent having characteristics of both filler particles and matrix, this bond is strengthened. Silanes are common coupling agents. They are used in the form of a molecule with silanol on one end and methacrylate on the other. Thus, they can produce covalent bonds to silica base filler and the methacrylate group (25).

TABLE-2 CLASSIFICATION OF RESIN COMPOSITES (18,19)

CLASSIFICATION	Range of particle sizes (µm)	Filler (% by volume)
HYBRID	0.04–3.0	60–70
MICROFILL	0.04–0.2	32–50
CONDENSABLE (PACKABLE)	0.04, 0.2–20	59–80
FLOWABLE	0.04, 0.2–3.0	42–62
NANOCOMPOSITE	0.002–0.075	68–78.5

[2.2.1.2: Setting reaction and polymerisation shrinkage:](#)

The process of producing of a composite material starts with the initiator element. As mentioned before resin composite can be self-cure, light cure or both. The reaction results in a covalent bond between two monomers, which are in proximity of each other. This reaction results in a reduction of the distance between the initial two atoms and a reduction in free volume. These factors both lead to a volumetric shrinkage. This shrinkage is dependent on the filler volume, composition of resin matrix and the degree of conversion of resin (27). Shrinkage volume varies between monomers. Previous studies have indicated a higher value for monomers such as BisGMA and TEGDMA (respectively: 5.2%

and 12.5%)(27-30). So, in recently developed resin composites these monomers are diluted with other monomers with lower polymerisation shrinkage such as UDMA. Also, new monomers have been introduced in hope to overcome the shortcomings of previous monomers and add new positive qualities to the resin composite. One of these monomers is Methacrylate modified polysiloxane (organically modified ceramic) (ORMOCER) which was introduced by Dentsply in Ceram X resin composite. Investigations on these materials are limited and further studies on important physical properties such as polymerisation shrinkage are necessary.

2.2.1.3: Classification:

Classification of resin composites can be built on consistency, filler size or volume (18, 19). Based on consistency they are divided into three groups: Universal, flowable (low viscosity) and packable (high viscosity). Each group has several subgroups based on their characteristics of filler. In another classification based on filler size they are categorised to following groups, macro-fill (10-50 μ m), micro-fill(40-50nm), small particle hybrid/midi-fill (10-50 μ m + 40nm), micro-hybrid/mini-fill (0.4-1.0 μ m + 40nm), nano-fill (5-100nm) and nano-hybrid (include more nanoparticles (0.4-1.0 μ m + 40nm)) (18).

Macro-fill:

Macro-fill resin composites were hard to polish and unable to retain the polished and smooth surface although they were very strong (18).

Micro-fill:

This group have low filler content and is weak, however, polishable (18, 19).

Hybrid:

Hybrid composites were introduced as universal restorative materials (micro-hybrids)(18). They have good aesthetics for anterior restorations and enough strength for posterior. Some of these materials are specially labelled as posterior restorative materials (19). This category of composites includes materials with fillers with different size. Various combinations are possible, like micro-hybrid and nano-hybrid. Presently, this category is marked as nano-hybrids, which indicates that they contain

nanoparticles (<100nm) and sub-micrometre particles ($\leq 1\mu\text{m}$, Average: 0.5-1.0 μm). Micro-hybrids are expected to have larger filler particles. However, it was difficult to determine the exact amount and size of fillers in the hybrid category with just the name (18, 31).

Nano-fill:

This group is highly polishable as a result has high aesthetic qualities. Also, they have high wear resistance, lower polymerisation shrinkage, and more desirable physical properties compared to micro-fill (18, 19).

Flowable:

The low viscosity of these resin composites is due to lower filler content or addition of modifying agents (18). They are good at wetting the surface but show high polymerisation shrinkage and wear. Because of their lower modulus of elasticity, they are suggested to be appropriate for use in cervical cavities. They are also used as lining materials to act as a flexible stress-breaker (16, 19, 32).

Packable (high viscosity):

Packable composites gain their high viscosity from modification to filler size distribution or type of particles (fibres). (18). They are specifically designed for posterior restorations and similar to amalgam need packing when used (16, 19).

Bulk-fill:

This type of composites has been recently introduced. Manufacturers have claimed that they have the curing depth of up to 5mm and lower polymerisation shrinkage (33). This quality may be achieved with improved translucency or additional photoinitiators (34). However, further studies are needed to confirm these claims.

Low shrinkage:

These are resin composites with altered monomer formulation. Concepts like ring opening system(cyclopolymerization) were used to achieve this goal. In this system, during polymerisation, van der Waals connections are converted to covalent, which will open at least two rings. However, when the mutagenicity of Spiro Orthocarbonate epoxy formulation as an essential element in these

materials was reported, these materials were failed. Following that oxybismethacrylate monomers were introduced for cyclopolymerization. Siloxane based oxirane molecules produce composite materials with adequate mechanical and physical properties, and it is claimed that they show half of the polymerisation value of posterior composite (35).

Ultrasonic activated:

This is a new resin composite introduced by Kerr Corporation, named Sonicfill. It is claimed that this material is a bulk-fill and sonically activated resin composite, which allows the clinician to lower the viscosity with a sonic activator for easy adaptation. Following this process, it is claimed that the resin will return to non-slumping state (34).

2.2.1.4: Properties:

In the beginning, resin composite had lower strength compared to amalgam however, with recent developments some resin composites even have higher strength than dental amalgam (16). However elastic modulus of amalgam is still much greater than resin composite.

Polymerisation shrinkage:

This is a critical and influential physical property of resin composite, which has often been studied (16, 36, 37). This shrinkage is a result of the polymerisation process and as this process advances the rigidity of material is increased, and the evidence of this stress is more evident and less compensated with the plastic deformation of resin composite. This stress can cause deformation in cusps, especially those which are weakened, even can be a result of a failure in adhesive bond or fracture tooth syndrome (19, 36, 37).

At first, it was suspected that this shrinkage is toward the light curing device. However now is believed that the polymerisation shrinkage is toward the bonded walls thus the smaller the number of the bonded surfaces, the lower the stress. This concept is defined as C-factor which is the value of the number of bonded surfaces to non-bonded (38). (Fig-6)

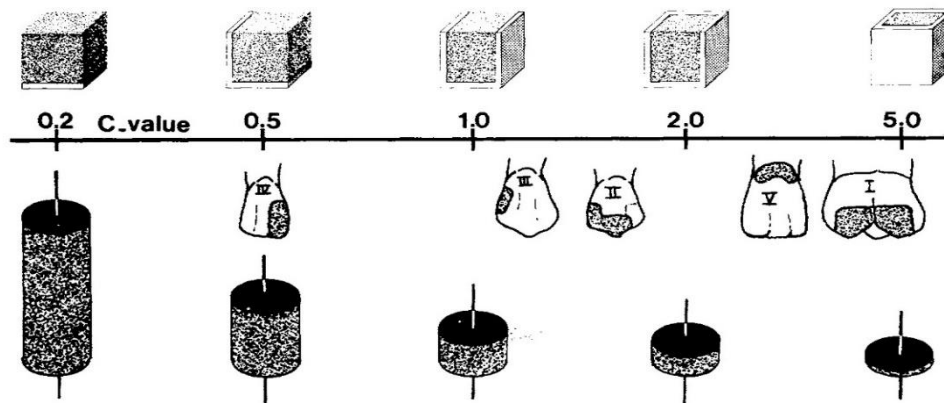


Figure-6 C-factor and C-value (38)

Another factor that is found to have an effect on stress shrinkage is the speed of polymerisation. This belief introduced the “soft-start” light curing concept, which slows the polymerisation process to reduce the resulted shrinkage. It has been suggested that even the type of light curing device it is influential on this stress(39-41).

Water sorption:

Water absorption of resin composites can be enhanced with incomplete polymerisation and lower filler content (19). It has been reported that this characteristic can cause hygroscopic expansion that might match the polymerisation shrinkage. However, it will take several days to overcome this shrinkage (42-44). Since the possible damages to tooth structure resulting from the shrinkage is during the polymerisation, and it is almost immediate so several days after compensation might not be a good solution(19). Nevertheless, it has been suggested that this expansion can fill the gaps formed after shrinkage between the restorative material and tooth structure(42, 44, 45).

2.2.1.4: Bulk-fill resin composites:

This type of composites has been recently introduced. Manufacturers have claimed that they have the curing depth of up to 5mm and lower polymerisation shrinkage (33). This quality may be achieved with improved translucency or additional photo initiators (34). Several studies have been conducted on the properties of these materials such as depth of cure, interface integrity, shear bond strength, volumetric shrinkage, colour stability and degree of conversion (46-50). Also, few review papers have been published regarding these properties (51, 52), which indicate that further investigations are required to better understand these materials. Taking the bulk volume of the resin composite in a restoration with 5mm depth into consideration, a greater stress or potential interface gap due to polymerisation shrinkage is expected. So, with the limited studies on this type of resin composite further investigations are necessary.

2.2.2: Glass Ionomer Cements

Glass Ionomer Cements (GIC) have been valuable materials in dentistry since they were introduced in the 1970s (53-56). These materials result from a reaction between weak polymeric acids and a glass powder (57). At first, these materials had many undesirable properties such as long setting time, poor aesthetics and work sensitivity (19). Many changes have been applied to them to improve these undesirable qualities. Some of these changes resulted in modifications, such as resin reinforced and high viscosity GICs (16, 19).

2.3.2.1: Composition:

GICs have three fundamental elements: Polymeric acid, glass powder, and water (57). Also, Tartaric acid has been added to them to reduce the time required for the initial setting time (19).

Polymeric acid:

This element is in the shape of liquid and mainly contains polyalkenoate acids (20). These acids can be homopolymer or copolymer of acrylic acids.(57) The polymer can have a substantial effect on the physical properties of GICs. An increase in molecular weight of them can enhance the strength of the

final material. However, higher molecular weight means higher viscosity and difficulty in mixing. As a result, an average molecular weight of 11,000 is suggested for the best outcome (57, 58).

Glass powder:

This element basically is fluoroaluminosilicate glass, which is made of alumina, silicon dioxide, metal oxides, metal fluorides and phosphates (16). Manufacturers have introduced different types of glasses. However, they follow the basic formula of $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-CaF}_2$ or $\text{SiO}_2\text{-Al}_2\text{O}_3\text{-CaO}$. They have been modified with various elements including strontium, zinc, sodium and silver (16, 57, 59). The characteristic property of GICs, fluoride release, is a result of setting reaction, which is in the shape of CaF^+ and AlF^{2+} . These cations slow the initial setting process by gelation, consequently lengthen the working time (19).

Water:

A set GIC contains 11-24% water, which is in the form of loosely bonded and tightly bounded. The loosely bonded portion is easily lost and can cause dehydration, shrinkage followed by cracking. This reduction will result in reduced aesthetic and physical properties. GICs have a vulnerable period regarding dehydration varying between two weeks to six months (19, 56). The percentage of tightly bound water increases in the first month. This can be on the surface resulting in an increase of -Si-OH or by coordination to metal ions or by hydration of the polyanion molecules (57, 60). To prevent the water loss, several techniques have been suggested such as resin coating

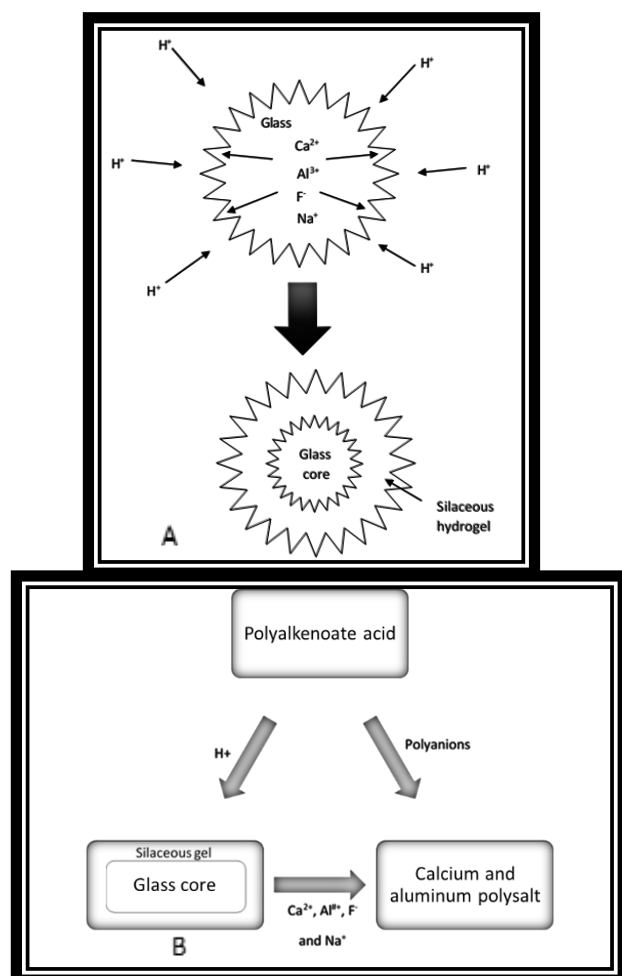


Figure-7 Setting reaction

agents, petroleum jelly, varnish. All of these methods are useful solutions, however, compare to others Petroleum jelly is less efficient, solvent base varnish has an unpleasant taste and resin base light cure coating is expensive (61, 62)

Chelating additives:

Several materials have been suggested like, tartaric acid or citric acid. Although, the most appropriate was tartaric acid, which is now widely used. This element contains 5-10 % of the cement and has the role of delaying the setting reaction so the mixing process will be easier and the clinician will have enough time to manipulate the material in the prepared cavity (19, 56, 57). Also, it has been mentioned by Nicholson et al. (60) that this component can have a positive influence on the compressive strength of the final material.

2.3.2.2: Setting reaction:

In a conventional GIC setting reaction begins when glass powder and acid are mixed. Acid releases H^+ cations, which attack the fluoro aluminosilicate glass resulting in the release of Ca^{2+} , Al^{3+} , F^- and Na^+ (Acid attack phase). As a consequence of this release, the outer surface of the glass becomes a siliceous gel. Released ions react with a polyanion, which remains of acid after releasing H^+ . This reaction produces calcium and aluminium polysalts. Resulting initiating the setting reaction (gelation phase). Following these reactions, a matrix will be generated with ionic cross-link between aluminium complexes and acid (hardening phase), which takes several days to complete. The setting reaction has been reported to be continued up to a year (16, 17, 19, 56, 57, 60, 63). (Fig-7)

Reinforced glass ionomer cements:

These type of GICs contains a conventional GIC, which is reinforced with silver, zinc or other metal elements (64).

High viscosity glass ionomer cements:

This group is also known as “packable” glass ionomers. The higher viscosity results from a higher powder to liquid ratio and smaller particle size (16, 65)

2.3.2.3: Properties:

GICs have many valuable properties such as fluoride release, which is one of the famous and advantageous characters of these materials(19, 57). It has been claimed that this quality reduces the occurrence of secondary caries (66). This release has two phases: early burst and sustained (19, 67). It has been reported that the first phase “early burst” is only seen in conventional GICs (68).

These materials have different clinical indications, such as luting and bonding, restorative particularly cervical area, lining and base.

TABLE-3 ISO REQUIREMENTS FOR CLINICALLY ACCEPTABLE GICS (70)

PROPERTY	Luting Cement	Restorative Cement	Base/Lining
FILM THICKNESS (MAXIMUM)(μM)	25	-	-
SETTING TIME (MIN-MAX)	1.5-8	1.5-6	1.5-6
COMPRESSIVE STRENGTH (MPA)	50 (minimum)	100(minimum)	50(minimum)
ACID EROSION (MAXIMUM)(MMH^{-1})	0.40	0.17	0.40
OPACITY $C_{0,70}$ (MIN-MAX)	-	0.35-0.90	-
ACID-SOLUBLE AS (MG KG^{-1})	2	-	2
ACID-SOLUBLE PB (MG KG^{-1})	100	100	100

Some physical properties of GICs have standard minimum value dictated by ISO standard, to be a suitable choice for clinical use (57, 69, 70), which are arranged in Table-3.

Several properties of these materials will be briefly explained in this section, which are related to the context of this thesis. Including Strength, Hardness, Adhesion, and Dimensional stability.

1)Strength and hardness

These materials are brittle in nature. Consequently, they have lower tensile strength compared to their compressive strength. The method commonly used to measure their strength are those designed for flexural strength. However, new approaches are also introduced, such as shear punch, which measures shear strength (16, 17, 71, 72). Some of these tests will be discussed later in this chapter.

2)Adhesion:

In contrast to resin composites these materials do not need any adhesive agent, and they easily bond to tooth structure (17, 19, 20, 73, 74). They even bond easily to stainless steel and non-precious metals. The quality of this bond can be reduced with a contaminated tooth surface. Thus 10% polyacrylic acid is applied and washed before applying the GIC material. This process will expose the collagen fibrils and enable a micromechanical retention in addition to the chemical bond, which enhances the bonding strength (17, 19).

3)Dimensional stability:

A conventional GIC exhibit a 2-3% contraction in a short time after setting, which has been reported to cause a spontaneous cohesive fracture in the cement(75). Since they also uptake water, this contraction can be compensated (19).

2.2.3: Resin-Modified Glass Ionomer cements

Resin-Modified Glass Ionomer Cements (RMGICs) were introduced in the 1980s (19). The purpose of this modification was to improve their physical properties such as flexural strength, fracture toughness, and compressive strength. This was achieved by adding 2-Hydroxyethyl Methacrylate (HEMA), a water-soluble resin and camphorquinone, a photo-initiator expanding the subtypes of these group of materials. Subsequently, their problem of long setting time and short working time was solved (16, 19, 57, 73) by light curing.

These materials contain the GIC part including a water-soluble polymeric acid, ion-leachable glass, and water. Also, they have a resin portion including organic photopolymerizable monomers and their

initiation system (60). HEMA plays a linking role between polyacrylate salt matrix or between photopolymerizable groups and polyacid chains (19). Some of these materials have unsaturated vinyl groups as side chains in the polymeric acid, which result in additional polymerisation reaction in the form of a covalent crosslink between polymer chains.(57)

2.3.3.1: Setting reaction:

Setting reaction of these materials is similar to GICs with one more step. First HEMA is polymerised, and camphorquinone produce a methacrylate matrix around the glass particles during the light cure, which results in the shorter setting time and then the same acid-based reaction as GICs takes place between the glass particles and polyalkenoate acid. The difference in the outcome is shown in Figure 8.



Figure-8 Structure after setting reaction A- RMGIC B-GIC

2.3.3.2: Properties:

RMGICs with the benefit of resin element have better physical properties compared to conventional GICs, such as higher strength and on “command-set “. The minimum requirement of some of the key physical properties is determined by ISO standard (19, 69).(Table-4)

Fluoride release is one of the beneficial characteristics that is preserved in this modification of GICs.

1)Strength and hardness

Strength is the main advantage of RMGICs to GICs. However, their microhardness has been reported to be lower than GICs (71, 76).

TABLE-4 ISO REQUIREMENT FOR CLINICALLY ACCEPTABLE RMGICS(69)

PROPERTY	Luting Cement	Restorative Cement	Base/Lining
FILM THICKNESS (MAXIMUM)(μM)	25	-	-
WORKING TIME (MIN-MAX) (SEC)	1.5	1.5	1.5
SETTING TIME (MIN-MAX) (SEC)	8	6	6
FLEXURAL STRENGTH (MINIMUM)(MPA)	10	25	10

2)Adhesion:

This property in RMGICs is similar to GICs and is easily achieved without any adhesive agent (17, 19).

3)Dimensional stability:

Since HEMA is hydrophilic, RMGICs have a higher water absorption than GICs. The earlier formulation of RMGICs even shows higher water expansion than their after setting shrinkage, which results in an overall expansion. One of the problems reported as a consequence of this quality was crown fracture after cementing in the case of a non-reinforced all ceramic crowns with RMGIC luting agents (19). However, recently it has been claimed that the hygroscopic expansion does not compensate the polymerisation shrinkage (77). However, the hygroscopic expansion just needs to make up for the post-gel component of shrinkage to eliminate the shrinkage stress; hygroscopic expansion seems even to overcompensate this shrinkage stress, which explains the expansion (78).

2.2.4: Giomers

Shofu Inc introduced Giomers. They are Pre-reacted glass filled resin composites. They have better aesthetic qualities and less tendency to dehydrate compare to GICs. These materials are resin based, and they contain Urethane Dimethacrylate (UDMA), hydroxyethyl methacrylate and Prereacted Glass particles (PRG). PRG is a result of the reaction between a polyacrylic acid and fluorosilicate glass. This reaction can involve just the surface (S-PRG) (Beautifil) or almost entire (Reactmer) particle. These materials need adhesive agents for bonding to tooth structure (19).

They have higher strength compared to GICs. Also, there have been some reports on their ability to release fluoride. However, they lack the “early burst” phase (68, 79). They have a higher water absorption than resin composites and compomers but lower than GICs (45). Also, they have smoother surface after finishing and polishing than GICs, which is comparable with resin composites and compomers (19).

2.3: Adhesives

Adhesives are materials that bond the restorative material to the remaining tooth structure. The strength of this bond should be enough to prevent the formation of a gap between material and tooth which can cause microleakage and penetration of bacteria. Moreover, microleakage can result in secondary carries or postoperative sensitivity. (80, 81) Adhesives reduce the necessity of preparation of retentive areas in cavity design, and they may even strengthen the remaining tooth structure.(81)

Powers et al. (81) describe the term adhesion as an interaction between adhered and adhesive in close contact, creating an adhesive Joint. The presence of water and the complex dental structure are obstacles that make a true chemical bonding between the tooth and restorative material challenging. GICs and RM-GICs are capable of establishing a chemical bond with hydroxyapatite, but this bond is more difficult in resin composites. As a result, this adhesion is more based on micromechanical interlocks. (81)

The process of applying the adhesive has three steps that may be done in sequentially or all together depending on the adhesive system. Adhesive systems are categorised base on etching system. This classification includes *etch and rinse and self-etch*, which each have several subgroups.

Three steps of applying the adhesive include:

1)Acid etching:

The etching process is conducted using an acid such as phosphoric. This acid conditions the tooth structure in order to increase the surface area and enhance the micromechanical locks.

2)Priming:

Primers are hydrophilic materials in shape of monomer or polymer in a solvent. They have bifunctional molecules containing hydrophilic and hydrophobic ends. During the priming process, the solvent component evaporates, and the hydrophilic layer will remain.

3)Bonding:

Bonding agents are resin-based materials, which are light or dual cured. They may contain fillers, fluoride, antimicrobial agents or desensitisers. (81)

Due to the variation in the structure of dentine and enamel, the bond of adhesive materials is different in these two areas of tooth structure each process will be explained separately.

2.3.1: Bonding to enamel

This bond is achieved via micromechanical interlocks with the infiltrated resin, after acid etching the surface of enamel. (81)

The acid used for this process is mostly 30% to 40% phosphoric acid, which removes the smear layer and conditions enamel surface. After contact (about 15 seconds) with enamel structure etch patterns are produced, by reduction of enamel prism periphery, enamel prism core or even no specific pattern. This acid etched areas have an increase in the contact surface due to these irregularities. (19)

2.3.2: Bonding to dentine

The first step in this layer is to remove the smear layer similar to enamel; this can be achieved with a weak acid. Dentine bond is more challenging than enamel bond since dentine has a high water content. When the outer layer is dehydrated the collagen scaffold collapses onto itself. This will result in an unsuccessful flow of primer and bonding agent to inter collagen space and will cause a weak bond and failure in bonding. Self-etch systems by skipping the drying step seem more suitable for this layer. (19, 81)

2.4: Bonding to tooth structure

One of the vital qualities of resin-based restorative materials is their adhesion to remaining tooth structure. An efficient and good marginal adaptation between the restorative material and tooth structure can prevent leakage of oral fluids and as a consequence gap formation and reoccurring caries. Therefore it can be said that the success of a resin-based restoration depends on an excellent adhesion between material and remaining tooth structure. (82)

Resin-based restorative materials differ in bonding and adhesion qualities. Some such as resin composites need another bonding agent and some such as Glass ionomer cements just need a clean surface. This quality will be explained for each group of materials. (17, 19, 20, 73, 74).

2.5: The oral environment

2.5.1: Saliva and artificial saliva

Saliva is a valuable element in the oral environment. It plays a critical role in oral health due to its composition and flow. (83) This fluid is a clear and is a slightly acidic mucoserous exocrine secretion. The average daily flow of saliva in health varies between 1 to 1.5 L in an individual. (84) The composition of saliva includes numerous electrolytes such as sodium, potassium, calcium, magnesium, bicarbonate and phosphates, immunoglobulins, proteins, enzymes, mucins and nitrogenous

products.(83) All these elements may have a major effect on materials used to restore damaged tooth structure.

Many researchers have studied the effect of various storage media on dental materials in laboratory studies. The most commonly used medium is distilled water which has been used in the majority of laboratory-based studies. However, some concerns have remained about the dissimilarity of distilled water and saliva in the oral environment. As a result, many studies have been conducted on the comparison of artificial saliva and distilled water (85-88). Several formulas have been used for artificial saliva with acidic or neutral pH to replicate the ionic environment of the mouth. One of the commonly used formulas is: 50mM NaCl+ 0.5 mM CaCl₂+ 0.5 mM Na₂HPO₄. with pH 7.0. Which has been used with or without mucin.(89-93) This formula with a high ion saturation can easily replicate the interaction of the dental materials and saliva in the oral environment.

2.5.2: Thermal conductivity of tooth structure

The temperature of the oral cavity can be affected by hot and cold foods and drinks.(94) It has been reported that this change of temperature can vary between 0°C to 67°C in tooth surface. (95) The sensitivity of pulp tissue to heat is dependent on the duration and the intensity of the thermal stimuli. Even small changes in the pulp temperature can cause an irreversible pulpitis and eventually death of pulp tissue. (94, 96, 97) A study which was conducted on the effect of the thermal change on pulp tissue reported that a 5.5°C increase in pulp temperature would result in irreversible pulpitis due to change of 135°C on the surface. (98)

TABLE-5 THERMAL DIFFUSIVITY AND THERMAL CONDUCTIVITY VALUES OF HUMAN TEETH (109)

	ENAMEL	DENTIN	METHOD	AUTHOR	YEAR
Thermal diffusivity ($\times 10^{-7} \text{m}^2/\text{s}$)	4.2	2.6	Thermocouple	Braden	1962
	4.69	1.87-1.83	Thermometer	Brown, Dewey, Jacobs	1989
	2.27	1.92	Pulse Laser and Thermocouple	Panas, Zmuda, Terpilowski, Preiskorn	2003
	4.08	2.01	IR Thermography	Lin, Liu, Kim, Xu, Bai, Lu	2010
Thermal conductivity (W/mK)		0.96	Thermocouple	Lisanti, Zander	1950
		0.11	Thermocouple	Phillips, Johnson, Phillips	1956
		0.45	Thermistor	Soyenkoff, Okun	1958
	0.93	0.58	Thermocouple	Craig, Peyton	1961
	0.92	0.63	Thermocouple	Braden	1962
	0.81	0.48	IR Thermography	Lin, Liu, Kim, Xu, Bai, Lu	2010

The conductivity of heat and cold via the enamel and dentine is relatively low, and are considered good thermal barriers or insulators for pulp tissue.(99) However, the disrupted structure in restored teeth cannot perform this task optimally. This disruption is due to the difference in thermal properties of restorative materials and tooth structure.(100) Many researchers have measured and reported the thermal conductivity and thermal diffusivity of enamel and dentin of human tooth. They have used several methods such as thermocouple, thermistor and IR thermography. (101-108) Reported results are arranged in Table-5. (109)

Resin-based restorative materials depending on their components have different thermal conductivity and thermal diffusivity. However, most of these materials are good insulators. (110-112)

Additionally, the heat produced by polymerization and curing devices in restorative materials can be harmful to the pulp tissue due to the proximity and lack of enough protective structure of enamel and dentin.(97, 113, 114) The most high-risk situations are applying thin layer of adhesives before restoration and absence of a liner layer in deep cavities. (110-112)

These facts all highlight the importance of keeping thermal changes during restoration and rehabilitation of the oral structure to a minimum. Since polymerisation of many dental restoratives is exothermic, an increase in temperature during the restoration process is inevitable. Therefore, more investigations are necessary with recently released high power curing devices which can add to the temperature increase and new materials with new compositions, some that are bulk filled, which can have detrimental thermal consequences for the pulp.

2.5.3: Occlusal loading

All restorative materials are not indicated to use in load-bearing cavities. Some are more suitable for anterior teeth and non-load bearing areas. Maximum load- bearing areas are restorations in posterior teeth, which are under a full masticatory force.(115) As a result, being familiar with the bite force is essential for a better understanding of dental restorative materials required.

The bite force is related to the muscle activity and dentition of individuals. Measurement of this force is notoriously tricky. (116, 117) The average maximum force varies in different areas of the mouth. In the western population in molar area range of 600 – 750 N (118), in canine area 120-350 N(119) and between incisors 140-200 N(120). Consequently, the strength of a restorative material to bear these forces without deformation and fracture is critical for the restoration's longevity. This strength is measured by fracture toughness, flexural strength and hardness tests.

2.6: The environment effect on resin-based materials

The oral cavity is a dynamic environment, which has diverse effects on the restorations. This effects can result from a change in temperature, sliding abrasion, chemical degradation, fatigue, consumed food and beverages and saliva content. (121)

The chemical factors such as plaque acids, food and enzymes can soften the resin-based restorative materials and reduce their physical qualities. (122, 123) The combination of these factors and mechanical forces of oral cavity can increase the damage and deterioration of restorative materials.(124-126)

As a result, one of the aims of laboratory-based studies in literature, is to mimic this environment. Examples of influential environmental factors are storage media, temperature, food particles and chewing forces. Many of these factors and their effects have not been fully studied before, such as, artificial saliva as storage media. Some of the projects in this study were planned to better understand the effect of these factors and how to imitate a better replica of oral environment in the laboratory.

2.7: light curing units

As the use of resin-based restorative materials increase, the importance of light curing units also becomes more significant. These devices have an important role in the “on demand” setting of resin-based materials (127, 128). Light curing Units have several implications in dentistry such as identifying residual resin composite, diagnosis of cracks and caries (129). It has been known that the degree of cure for resin composite depends on irradiance, exposure time, resin's shade, filler size and loading level (130, 131). Due to the decrease in transmission of the light throughout the materials thickness, depth of cure is limited (132). To achieve higher physical properties an adequate curing is necessary. Irradiance as a valuable and important factor in choosing light cure units, it is function of the type of unit and varies with the distance between unit's tip and a resin material (132). Various curing protocols have been suggested to overcome and reduce the polymerization shrinkage such as soft start programs with two step or pulse delay curing (131, 133). However, there is a very little information regarding the effect of these protocols on physical properties of bulk-fill composites.

2.7.1: Quartz-Tungsten-Halogen (QTH)

QTH units have been very popular with dentists.(Fig-9) This group of light curing units uses a low voltage, high current tungsten-halogen reflector lamp to produce a spectrum of 300-1000nm, then this light is filtered to 400-500nm (127, 132). One of the disadvantages of these units is heat generation, which increases with increase in radiation time, however, it has been claimed that this heat does not affect the pulp tissue (132). Another disadvantage is a limited lifetime of the bulb and filter(134).

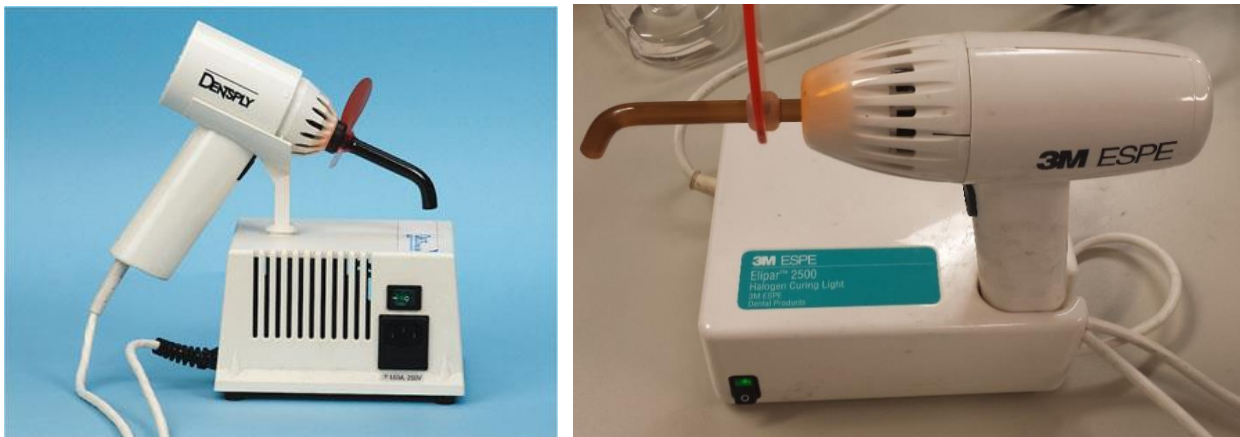


Figure-9 QTH light cure units

2.7.2: Light Emitting Diode (LED)

LED units (Fig-10) have been introduced as an answer to shortcomings of QTH units (135). It has been claimed that they cause a lower tension in the material (133) and produce less heat during



Figure-10 A range of LED light cure units (136)

radiation (135). However, at first, they had very short curing depth. This problem was solved by using small chips that contain large surfaced emitting LED chips (137).

LED units produce a spectral output of 450-490nm, which is the maximum absorption peak of camphoroquinone (Photoinitiator) (137).

The average energy intensity for LED curing lights is about 1000 mW/cm². The new revolution in this type of curing light devices is the introduction of the high intensity lights. One of these devices with a maximum 3200 mW/cm² intensity is among the highest intensities that now is available. The high intensity may cause a rapid polymerisation reaction that leads to higher polymerisation shrinkage rate. Also, higher intensity results in higher thermal energy output that may increase the temperature of resin composite. Depending on the amount of this change pulp tissue may be affected in deep restorations. So, further investigations on this topic is necessary.

2.7.3: Curing protocols

As reported by Feitosa et al. (138) different curing protocols can affect the physical properties of resin materials. Including, polymerisation shrinkage, microhardness and tensile strength. However, this effect can vary for different materials. Feitosa et al.(138) reported that soft start mode can improve the micro-tensile bond between dentine and resin, tensile strength and microhardness but it may reduce the crosslink density of the polymeric network of the composite. Similar results were reported by other researchers an improvement in bond to tooth structure but lower degree of conversion (139).Also, It has been suggested that soft-start methods can reduce the rate of shrinkage and temperature increase of the restorative material during polymerisation. (41, 140, 141) Since the light intensity only increases gradually to the maximum output, the result is a slower activation of the radicals associated with the conversion of the monomer to polymer thus prolonging the 'gel stage' of polymerisation. (142-145) However, this ramp mode extends the chairside time, which in some circumstances may not be feasible. Also, in pulse delay protocol it has been reported that high

irradiation is necessary to achieve appropriate polymer network structure and degree of conversion (146).

2.8: Laboratory testing of dental materials

2.8.1: Fracture toughness tests

In materials science, fracture toughness has been described as the ability of a material containing a crack to resist fracture (147). There have been several methods for studying this physical property.

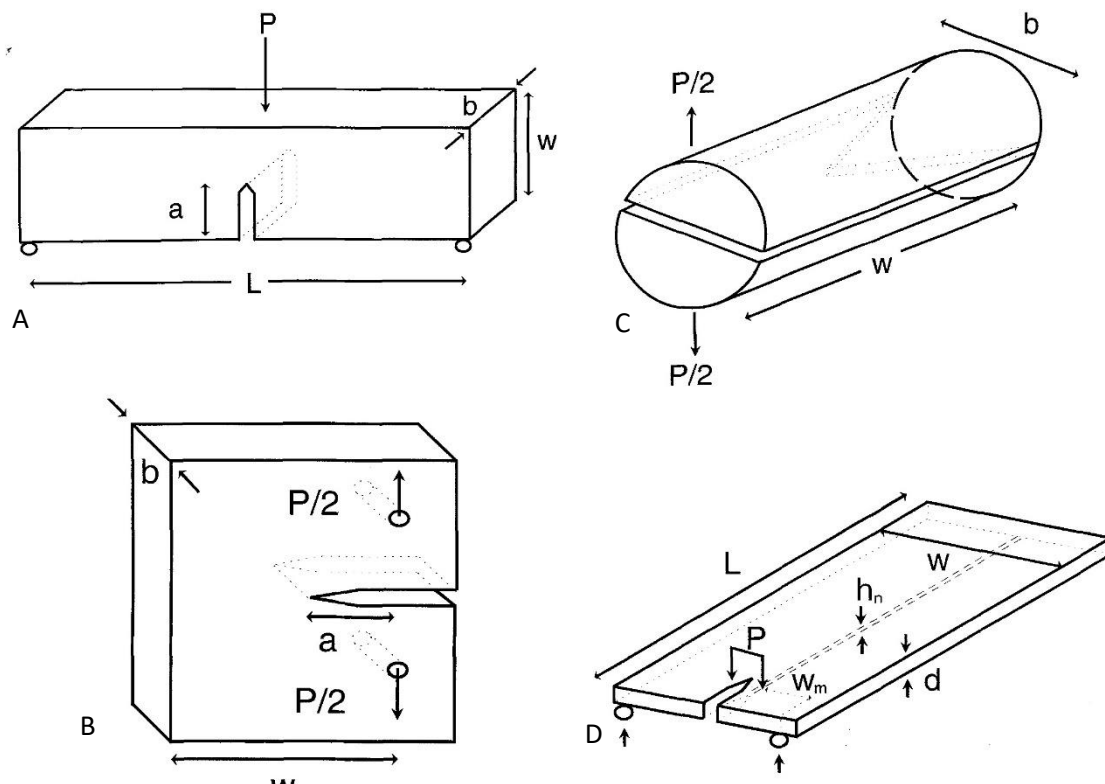


Figure-11 Four different methods of fracture toughness test A-SEVNB B-CT C-short rod D-double torsion (148)

Fujishima et al.(148) have studied four different methods for testing fracture toughness including, Single Edge V-Notch Beam (SEVNB), Compact Tension (CT), short rod and double torsion. (Fig-11)

This study revealed that double torsion is the most reliable method to deliver the information about crack propagation pattern and true value for fracture toughness. However, this method is the most technique sensitive. On the other hand, short rod test resulted in higher values and evaluation

of load-deflection curves and fracture surface revealed that the data might not be valid since there was a lack of a stable crack growth. Consequently, SEVNB and CT test data were reported to be valid in conjunction with a higher success rate for related specimens.

Fujishima et al. (148) study was a conclusion of several studies that have been conducted on fracture toughness of dental restorative materials with different methods. The value of reported fracture toughness for same material varied with different methods and since this is a characteristic property of materials, so the resulted values are influenced by the method (149). The test protocol for SEVNB and CT test is ASTM E399(150), which is initially designed for metallic materials and requires a precrack in the V-shape notch produced by fatigue. This process is not practical in dental restorative materials, as result of their brittleness (148). So several procedures were suggested for different methods to produce this requirement(149). Such as, under hand pressure with a blade, placing a razor blade in the mould before polymerisation and specimen loading for a short time.(Fig-12)

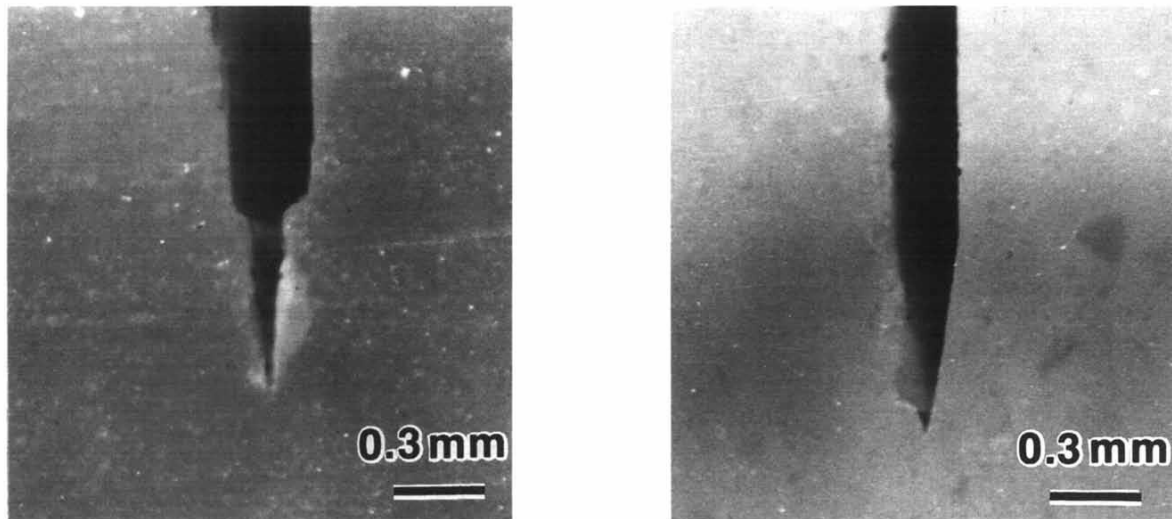


Figure-12 Precrack image A- Blade under hand pressure B- Blade in mould (149)

Comparing these methods Kovarik et al.(149) reported a lower linear-elastic fracture toughness value for groups with blades in the mould. Accordingly, different fracture toughness values are expected of the same material when the test and precrack production methods are varied. However, it has been reported that although the values are different, the patterns are similar in different test methods (151).

2.8.2: Flexural strength tests

Strength is one of the crucial properties of restorative materials, which is dictated by materials microstructure, composition, test method, environment and failure mechanism (152, 153). Also, Internal defects can have an effect on this property (154, 155)

As a standard in dental materials, International Organization for Standardization (ISO) indicates uniaxial flexural test: 3-point bending or 4-point bending, and biaxial flexural test for ceramics (156) and 3-point bending test for dental composite (157). Several studies have tested this physical property of composites with 4-point bending and biaxial test and compared these methods to 3-point bending test (152, 158-161). Such as, Miura et al., who studied and examined the results of all these three tests on dental hard resins, which indicated that biaxial flexural test has more reliable results compared to 3-point bending test in these materials (162).

Uniaxial flexural test (3-point and 4-point bending test):

Bar shape samples are prepared for these tests, and they are loaded with compressive force at the top, which produces tensile stress in the bottom surface, until fracture.

The difference between these two test methods is the loading point. In 3-point bending test, the force will be pointed at one point which has equal distance from both right and left side of the sample. However, in 4-point bending test force will be on two points, which exposes more area to stress and increases the chance of failure due to the internal flaw. As a result, a lower strength value is expected in 4-point bending test (152, 163). (Fig-13)

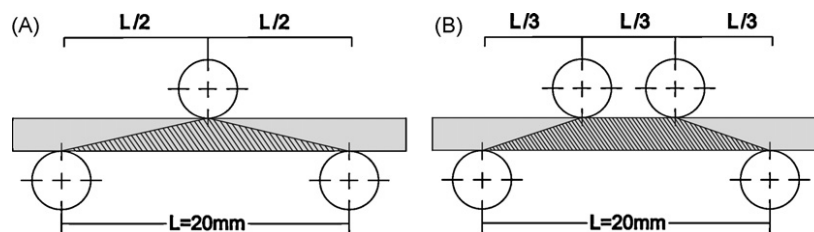


Figure-13 Schematic illustration of A) 3-point bending test B) 4-point bending test (152)

Biaxial flexural test:

This test has three categories: ring on ring, ball on ring and piston on 3-ball (160, 164, 165). (Fig-14) Each test follows a protocol indicated by the related standard (156, 166). This type of test is considered more accurate due to concerns involving the wedging stress and counter moments produced by friction, at the loading points in 3-point bending test and also replicates a more similar situation to occlusal forces(160).

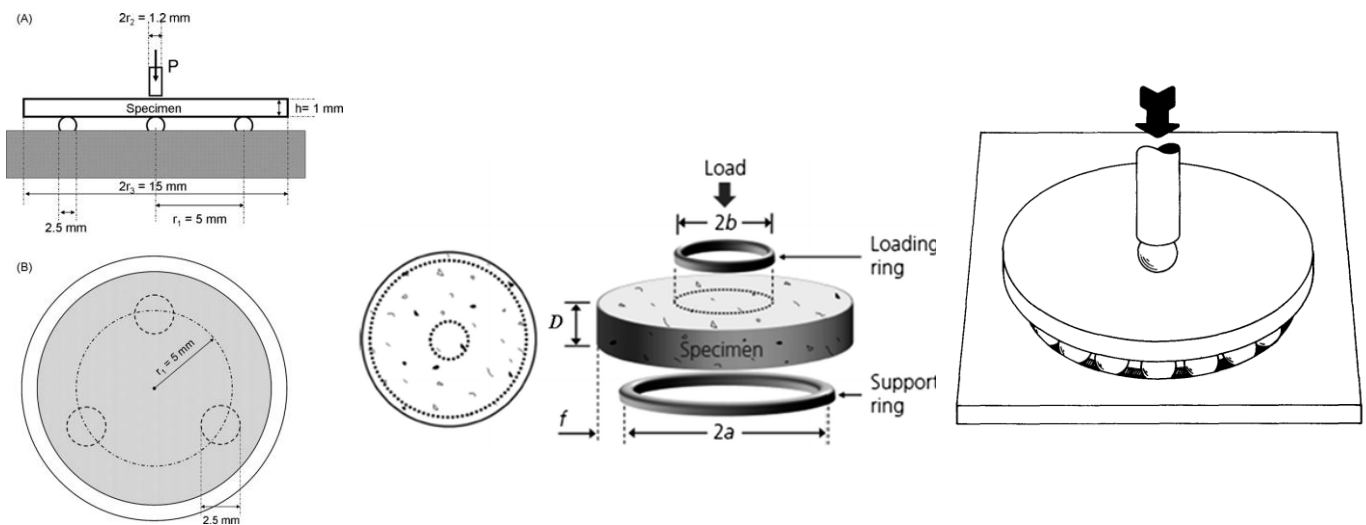


Figure-14 Schematic view of A)"piston on 3 ball" B)" Ring on ring" C) "ball on ring" biaxial test (160,164,165)

ISO also indicates a minimum value for the flexural strength of each dental material category (69, 157) (Table-6). As a result, The Biaxial flexural test is able to provide a more precise result, and this was the reason for choosing this test method as the preferred method in these projects.

TABLE-6 FLEXURAL STRENGTH STANDARDS. (TYPE1: POLYMER-BASED MATERIALS SUITABLE FOR THE OCCLUSAL SURFACE. TYPE2: ALL OTHER POLYMER-BASED MATERIAL. CLASS 1:SELF-CURE CLASS 2:EXTERNAL-ENERGY-ACTIVATED GROUP 1: INTRA-ORALLY GROUP 2: EXTRA-ORALLY CLASS 3: DUAL CURE (69, 157)

RESTORATIVE MATERIALS			Flexural strength (minimum)(MPa)
POLYMER-BASED RESTORATIVE MATERIALS	Type 1	Class 1	80
		Class 2, Group 1	80
		Class 2, Group 2	100
		Class 3	80
	Type 2 (including luting materials)	Class 1	50
		Class 2, Group 1	50
		Class 3	50
RMGIC		Luting Cement	10
		Restorative Cement	25
		Base/Lining	10

2.8.3: Hardness tests

The tests designed to measure this quality are relatively simple and reliable. Commonly used tests for evaluation of polymeric materials are Knoop and Vickers hardness test (167, 168). Several studies have been conducted comparing these two test methods, which all report a strong correlation between their results (167-170).

The protocol of these test are indicated by ASTM E380 standard (171):

Vickers hardness test:

Indenter used in this test is a 136° pyramidal diamond with square base that produces a square indent.(172)

The force is usually maintained for 10-15s. Then the size of indentation will be measured using the length of two diagonal lines in the square. Vickers Hardness Number (VHN) is calculated using this equation: $\frac{\text{Geometrical Constant} \cdot P}{(\text{Average } d)^2}$ (167, 171).

The reported VHN is usually the average of three indentations on the sample (170). (Fig-15)

Knoop hardness test:

Indenter of this test is a rhombic indenter with different size diagonals. The longer diagonal is 7.14 times, the shorter. (Fig-15) Knoop hardness is calculated using this equation: $\frac{\text{Indenter Constant} \cdot P}{(\text{length of long diagonal})^2}$ (171, 172). The reported hardness value is usually the average of 3 indentations (170).

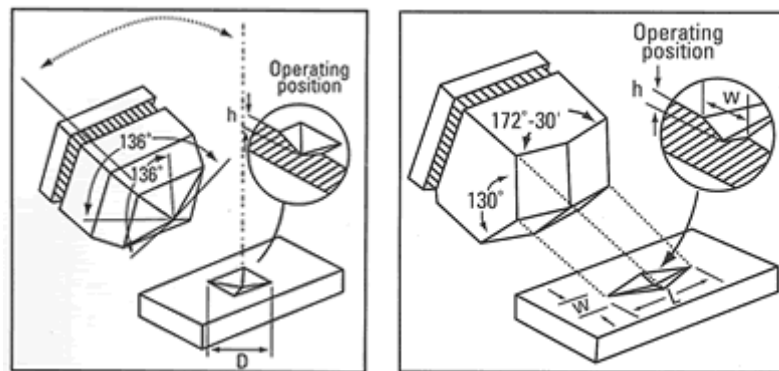


Figure-15 Hardness test indenter A) Vickers B) Knoop (173,174)

2.8.4: Wear tests

Wear tribology describes a multifactor phenomenon.(175) In simpler words, wear is a progressive loss of a surface due to mechanical interaction between two surfaces.(176) Tests for this property in dentistry are based on abrasive, fatigue and corrosive wear.(177) Abrasive wear is a result of hard

asperities plough into softer surfaces, which cause abrasion and attrition.(178) The difference

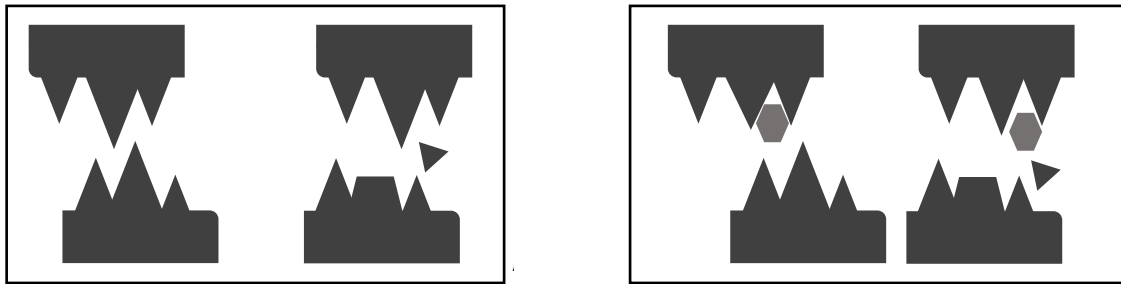


Figure-16 Schematic diagram of abrasive wear simulator A)Two-body B)Three-body

between these two is the presence of a third body between two surfaces in abrasion. (Fig-16) Propagation of subsurface cracks causes fatigue wear due to cyclic load. Corrosive wear is the effect of chemicals on the surface layer. (176, 179)

Various wear tests based on abrasive wear are commonly used, due to the difficulty of imitating the oral environment in the laboratory. Such as two-body, three-body or a combination of both. (180-182)

Two-body wear simulator:

Simulators such as SD Mechatronik are used to produce attrition wear with a sliding action of an abrasive surface against the sample.(181)

Three-body wear simulator:

Devices in this group produce abrasion. Using a sliding action of an antagonist on the sample with a replication of food in the middle as the third body, so the force is transmitted from the antagonist through the food layer to the sample.(183) Example for this group is an Academisch Centrum for Tandheelkunde Amsterdam (ACTA) wear machine.(181, 184)

Two-body and three-body wear simulator:

These tests are designed to include both abrasion and attrition. A good example of this devices is Oregon Health Sciences University (OHSU) oral wear simulator.(181, 185)

2.8.5: Scratch hardness test:

Scratch test is a more straightforward test compared to abrasion wear test.(186) Moreover, the results reported from this test have been reported to show the same trend of wear in the clinic.

(187-189) considering the simplicity of the technique of scratch test compared to wear test and the validity of results and trends between various materials makes this test a valuable technique.(190, 191) The nano scratch method follows the principle of reproductive and controlled deformation.(186) The resistance of the material to scratch can be studied by the depth and width scratch, hardness and surface damage patterns. (187)

In wear test, there several variable factors such as antagonist, force, contact area, number of cycles, the frequency of cycles, duration of contact, sliding speed, temperature, lubricant, food bolus, homogeneity of test material, chemical cycles, pH, enamel and dentine structure and wear debris. (175) Scratch hardness tester is a simpler device, which enables the operator to change all these factors as they see appropriate. As a result, this method seems more appropriate to study the effect of factors that have not been fully studied before such as sclerotic dentin as an antagonist or other conditions that have been a concern for researchers.

2.8.6: Stress rate tests

Polymerization stress rate has been a major shortcoming of adhesive restorative materials. This physical characteristic, especially in resin composites, results in a cuspal deflection and failure in bonding interface consequently microleakage and post-operative sensitivity. As it has been mentioned in earlier sections, this property is highly influenced by monomer system of the material (192-195). Many new formulas were introduced to reduce this effect (35, 196). Polymerisation shrinkage stress is the force that is measured in most of the previous researches. However, the term polymerisation residual stress has been misused in the place of polymerisation shrinkage stress before. We should keep in mind that most of methods mentioned below only measure the polymerisation shrinkage stress and not the polymerisation residual stress except Fibre optic sensors. As Koplin et al. have mentioned in their study polymerisation shrinkage stress between materials can be different but the internal stress may be the same. This variation can be due to the elasticity of resin composites.(197)

Some of the earliest work in this area was done by Bowen (198, 199) and Hegdahl et al. (200). Since then various methods have been used to measure this stress including photoelasticity,

mathematical modelling cracks analysis, finite element analysis ring slitting and mechanical (192, 197, 201-205). Mechanical techniques have been most popular (192). Some examples of these methods are two rods, single cantilever device and one of the most recent tests fibre optic sensors (192, 195, 206).

In a study, Goncalves et al.(192) compared the outcome result of four different test methods and found that the results from different test methods should be compared carefully, as a consequence of the different values and trends. This difference can be due to various sample size and system compliance.

Two-rod method:

In this method, the rods that are used are stainless steel, acrylic or glass. Since glass and stainless steel have high elastic modulus (respectively: $E=207$ GPa, $E=64$ GPa), acrylic rods are preferred, among materials used previously for this test, because they produce a high compliance system (192, 207,

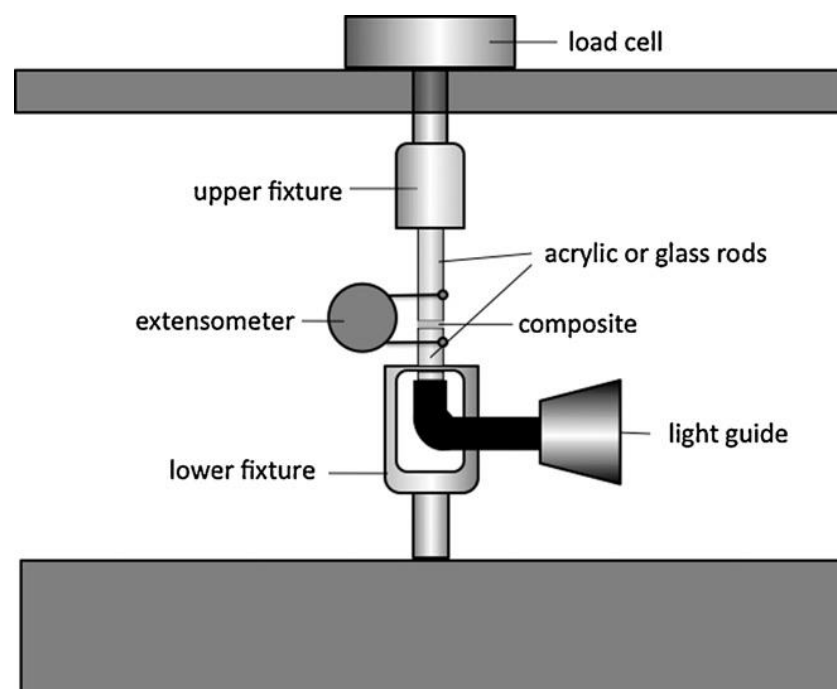


Figure-17 Two rod method using a universal machine (192)

208). In this test method, two rods will be prepared. One surface of each rod will be roughened. Then rods will be fixed to a universal testing machine. The polished surface of rods will be covered with adhesive. The material will be put on the lower rod then rods will be placed with a maintained distance with the help of linear strain gauge using an extensometer. Materials will be light cured. (Fig-17) The force necessary to maintain the space will be recorded, and polymerisation rate will be calculated for each specimen (192, 209).

The light curing method in this test can differ. Samples in this test can be exposed to light from one side or from the bottom. In the case that the light exposer is from one side it should be considered that the depth of cure will be the diameter of the sample, which in most studies is 4 to 5mm. This curing depth raises some concerns in conventional resin composites with recommended curing depth of 2-3mm. Consequently, a test which is designed using rods with less than 2mm diameter can provide more accurate and useful information.

Single cantilever device:

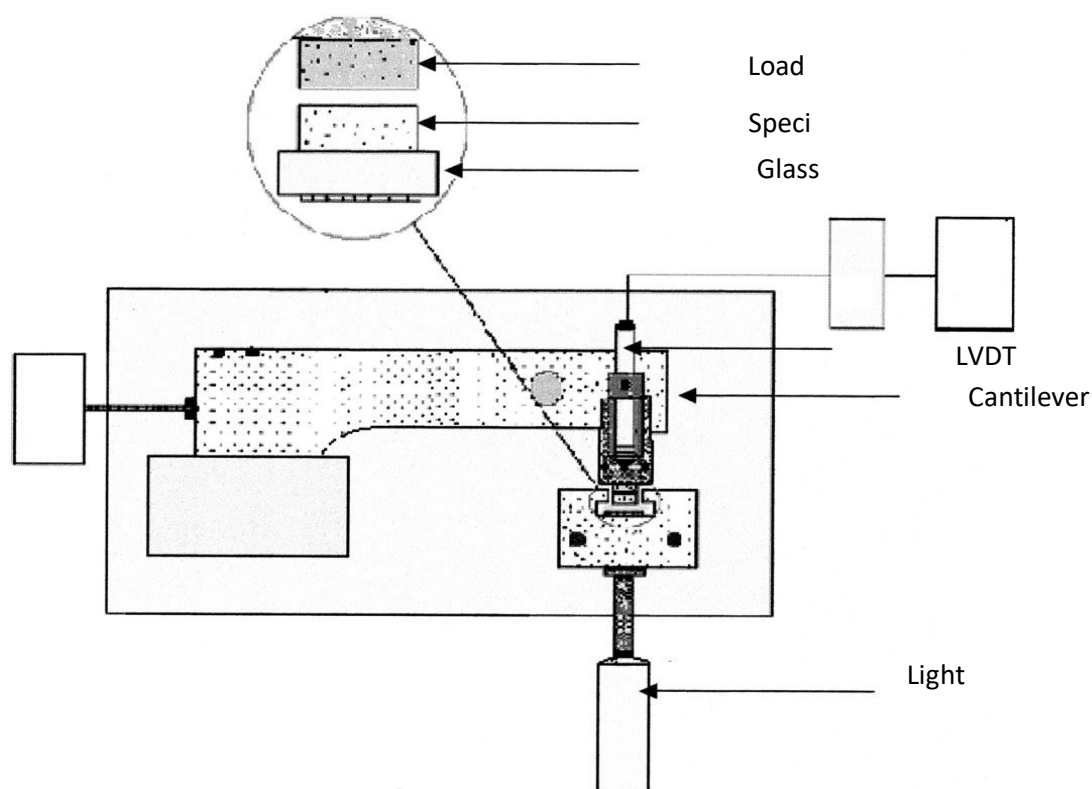


Figure-18 Single cantilever device (Bioman) (193)

This method was introduced by Watts et al. (193). In this test, a cantilever load cell is fitted with a rigid integral clamp. The lower part of the clamp is connected to a steel piston, which was sandblasted. On the other side, there is a glass plate, which is also sandblasted. The material is placed between these two sandblast surfaces to produce specimen disks. After polymerisation, the displacement of the free load of the load cell is measured with an LVDT. (Fig-18)

Fibre optic sensors:

In this method, a fibre optic (fibre bragg grating) sensor will be inserted in the middle of the mould and specimen. Stress after polymerisation will be measured for 10 min. (Fig-19) This is a suitable method since it is very easy to handle and the sensor is embedded in the sample and measures both polymerization residual stress and polymerization shrinkage stress. Fibre optics can be a great assist to material science(195).

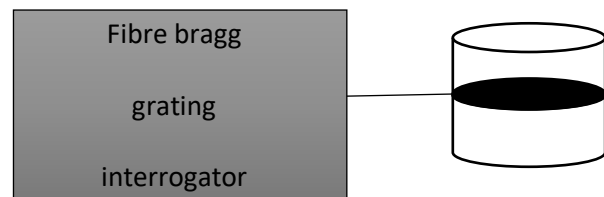


Figure-19 Schematic diagram of fibre sensor system (195)

2.8.7: Cuspal strain tests

Cuspal strain tests, examine cusp movement after restoration placement. Cuspal deflection is the result of polymerization shrinkage and its effect on adhesive interface and the compliance of cavity walls. (210, 211) Several factors can influence this stress such as size of cavity, C-factor, restorative material, bonding system, hydration of the tooth and sample conditions. (212-219) Also, it has been suggested that restoration technique can manage the amount of deflection stress. Many clinical approaches have been studied for example incremental placement of resin composites, soft start modes of curing lights and an intermediate liner.(211, 212, 214, 220) Also, new types of resin composites have been suggested by manufacturers to reduce this stress. Low-shrinkage resin composites are one of these products.(221)

Many researchers have studied methods to measure this deflection.(212-216) these methods included Fibre Bragg grating, strain gauges, profilometry and linear variable differential transformers. (212-216, 222-224) Researchers concluded that strain gauges are a precise method that can provide the best insights into cuspal deflection.(225-227)

Strain gauges:

Strain gauge is an instrument for measuring the strain in objects. Strain is calculated by dividing the length of stretch by original length of the sample. Strain gauges stretches with the material, thus recording a measurable change in electrical resistance. A positive value indicates a tensile strain and a negative value represents a compressive strain. Depending on the gauge strain is measured in one to three dimensions. (228)

This device has been used in several studies to measure the cuspal strain in resin composite restoration and determining the influence of low-shrinkage composite, thermal expansion, monomer ratio in composite and curing light intensity on cuspal flexure. (227, 229-235)

2.8.8: Thermal tests

Like most materials, dental composites expand with an increase in temperature, but like any other thermal expansion, this increase in dimension will be reduced as soon as the materials cools to reach the environment temperature. The difference between the thermal expansion coefficient of dental composites and tooth structure can cause microleakage formation.(195, 236) The most contribution to the change in temperature can be due to heat from exothermic polymerisation or curing light. The timing of this change is the same as polymerisation shrinkage so thermal expansion can compensate for the true polymerisation shrinkage and stress resulting from this shrinkage in the bonded surfaces at the time of light curing. However, when the restoration cools and reaches the oral environment temperature, it is expected that the shrinkage will be higher due to the additional shrinkage from the initial thermal expansion.

The literature lacks studies on this concept. This lack in literature and oversight can cause an underestimation in polymerisation shrinkage at the time of polymerising and the effect of this stress on the adhesive layer and remaining tooth structure of resin-based restoratives. For calculating this property, it is necessary to measure the core temperature of polymerised resin while exposed to curing light and record the temperature change. Using this data and linear shrinkage, the linear expansion coefficient(α) can be determined. With this coefficient exact expansion of resin composites can be determined. This result will make it possible to measure the true shrinkage of the materials.

Core or internal temperature of materials has been measured and investigated before in various studies.(237-240) One of the most reliable methods that has been used was thermocouple bare wires. When the fine wires are used, it does not affect the structure of the restoration. Also, they provide detail and reliable data.

Thermocouples are sensors, which measure temperature using the continuous current between two different base metals. Each Thermocouple consists of two different metal parts. The dissimilarity in electrical conduction of these metals, results in a measurable voltage. Due to the thermoelectric effect this voltage is temperature dependent. Thermocouples have different type and varieties. They are either in the shape of a wire or a probe. Wires have fast response and are more affordable. (241-244) They are categorised depending on their diameter, base metal and response . (241, 245)

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Chapter 3: Project I- A review on clinical success of resin composite restorations in posterior teeth - comparing periods: 1995-2005 and 2006-2016

3.1: Introduction:

Posterior resin composite restorations have an important place in clinical practice, their use has almost over taken silver amalgam, which was the main material used for these types of restoration in the past (1). Tooth-coloured materials are preferred by patients due to their aesthetic appearance. Furthermore, the biocompatibility of the mercury in amalgam has raised many questions and concerns from the consuming public (2), and the current FDI directive is to phase down the use of mercury containing components as part of the implementation of the Minamata Convention (3).

There has been an ongoing effort to improve the physical properties of these materials in an attempt to reduce clinical failure rates. Several modifications have been suggested such as: adding various-sized filler particles in order to reduce the brittleness and wear, use of siloxane monomers to reduce polymerisation shrinkage and introduction of the so-called 'bulk-fill' composites to simplify the restorative procedure (4-7). The modifications to composites have improved wear resistance and strength to create materials not dissimilar to dental amalgam, which has led to current resin composites being suitable as restorative materials for use in load bearing areas (7, 8).

The aims of this systematic review were to evaluate papers from 1995 - 2015 that assessed clinical performance of posterior composite restorations and compare results over the two last decades to determine whether the improvements have led to better clinical performance of posterior resin-based restorations.

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Comparison between published clinical success of direct resin composite restorations in vital posterior teeth in 1995–2005 and 2006–2016 periods

N Alvanforoush, J Palamara, RH Wong, MF Burrow

Melbourne Dental School, University of Melbourne, Melbourne, Victoria, Australia.

ABSTRACT

Composites are increasing in popularity as restorative materials. This growing role indicates the necessity of studies on their clinical outcome. In this study, clinical studies published on the performance of posterior composite restorations were included except those of less than a 24-month assessment period. Results of non-vital, anterior or primary teeth and cervical single-surface restorations were also excluded. Records about composite type, number of final recall restorations, failure/survival rate, assessment period and failure reasons were analysed for each decade. Overall survival/failure rates for studies in 1995–2005 were 89.41%/10.59% and for 2006–2016 were 86.87%/13.13%, respectively. In 1995–2005, the reasons for failure were secondary caries (29.47%) and composite fracture (28.84%) with low tooth fracture (3.45%) compared with reasons of failure in 2006–2016, which were secondary caries (25.68%), composite fracture (39.07%), and tooth fracture (23.76%). An increase in incidence of composite fracture, tooth fracture and need for endodontic treatment as failure reasons was noted in the latter decade in addition to a decrease in secondary caries, post-operative sensitivity, unsatisfactory marginal adaptation and wear. The overall rates of failure showed little difference, but the causes showed a notable change. This is believed to be a reflection of increased use of composites for larger restorations and possibly changes of material characteristics.

Keywords: Clinical success, composite restoration, failure reason, Posterior restoration, vital teeth.

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INTRODUCTION

Posterior resin composite restorations have established an important place in clinical practise and have continued to gain popularity. Resin composite use has almost superseded the previous posterior restorative material, namely, silver amalgam.¹ Compared with other posterior restorative materials, tooth-coloured resin composites are preferred by patients as a result of their increased desire for aesthetic restoration. In addition, patients have questioned the biocompatibility of the mercury in amalgam.² The advances of minimal intervention dentistry to conserve tooth structure have led to modified cavity preparations in association with improved bonding.

Any restorative material has a limited working life and replacing any restoration will cause further loss of tooth structure.³ Many factors affect longevity and success of a restoration, such as the material, patient and dentist factors, and cavity type.² Since resin composites were introduced, there has been an ongoing

challenge to improve their clinical performance, particularly for use in posterior teeth. This has seen the introduction of hybrid, packable, nano-filled, low-shrinkage and bulk-fill composites,^{4–8} but still the clinical effectiveness of posterior composite is questioned by many. Patient factors such as caries risk and occlusal stress can influence the long-term clinical success of a restoration.⁹ Also, a significantly higher failure rate for multi-surface and complex restorations compared with occlusal single-surface restorations has been demonstrated.²

According to Beck *et al.*,¹⁰ who reviewed studies on composite performance conducted over 19 years (1996–2015), the main reason for failure in the first 4 years of a composite restoration's life was fracture followed by marginal defects and secondary caries. In contrast, for older restorations (≥ 5 years) secondary caries and fracture were the predominant reasons for failure. However, in the last 20 years there have been substantial advancements and changes in composite materials and adhesive systems, which might have

influenced failure reasons and rates. The aim of this systematic literature review was to evaluate papers that assessed clinical performance of posterior composite restorations. Only papers published since 1995 that determined failure reasons and rates were evaluated in two periods, 1995–2005 and 2006–2016.

METHODS

In order to collect the related articles two separate search engines, PubMed and Medline Ovid, were used.

The search terms used for this review were: (('clinical evaluation' or 'clinical trial' or 'clinical success') and ('class I restoration' or 'class II restoration' or 'posterior composite') and ('composite' or 'resin composite')).

The same search terms were used for two time periods: 1995–2005 and 2006–2016 (Fig. 1).

After eliminating duplicate papers for the different search engines, several excluding factors were also applied: (i) laboratory-based; (ii) conducted entirely on non-vital teeth or results were not separable for vital teeth; (iii) conducted entirely on primary teeth or results were not separable for permanent teeth; (iv) conducted entirely on anterior teeth or results were not separable for posterior teeth; (v) of a duration less than 24 months; (vi) evaluating restored teeth with open laminate, tunnel preparation or cervical single surface (class V) restorations; and (vii) in a language other than English.

Exclusion of studies less than 24 months was based on the fact that failures increase significantly as study length increases and a short follow up can overestimate the clinical effectiveness/success of restorations.^{11,12}

After excluding unrelated papers and any previous articles to follow-up clinical trials, 64 papers remained. In addition, six papers were cited in these articles that fitted the search criteria. They were added by hand search. Out of these 70 articles, 30 belonged to the 1995–2005 period and 40 belonged to 2006–2016.

Extracted data from the contents of these papers were: composite brand and type; number of restorations placed at baseline and available at the final recall in each study; type of restoration (occlusal single-surface/multi-surface); observation period and numbers; and reasons for and rates of failure or overall success. All of this information was extracted from the texts and tables in the published papers and collated in two tables for each specified period (Tables 1 and 2).

Various types of composite were evaluated in these 70 papers.

Only studies that used the manufacturers' instructions for handling the materials and the accepted method of incremental insertion for non-bulk-fill composites regardless of use of lining such as glass ionomer cement with or without calcium hydroxide were included. No excluding factor was applied regarding cavity preparation, which varied from single occlusal surface to cusp build-ups and complex multi-surface cavities.

In articles that included a combination of either primary, non-vital or anterior teeth, only the results related to vital permanent posterior teeth were extracted and used. A similar procedure was followed with studies where direct and indirect composite restorations were placed by excluding the results related to indirect composite restorations.

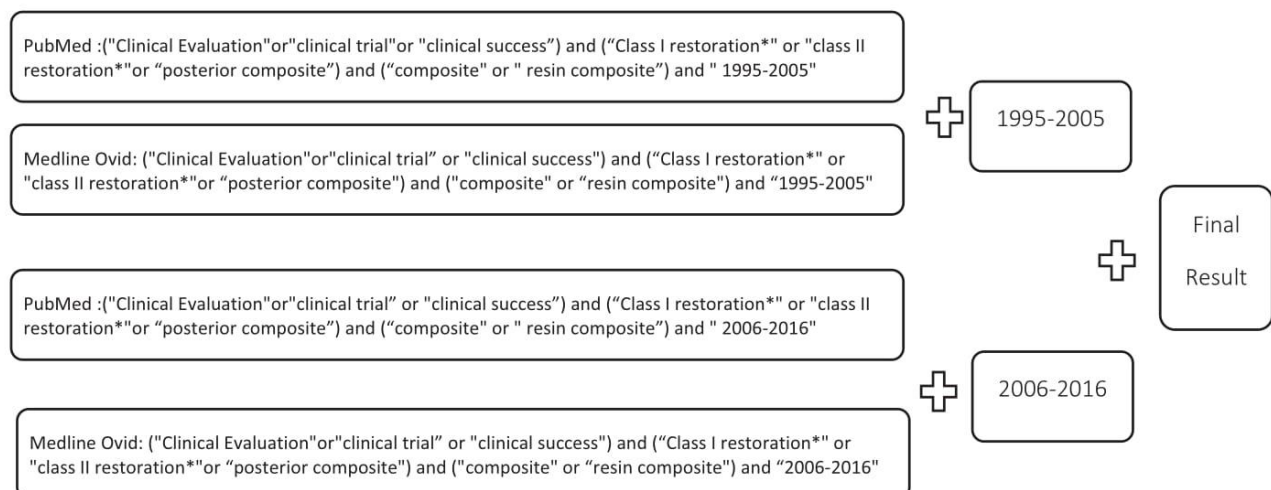


Fig. 1 Search.

Table 1. Summary of search results for 1995–2005

References	Composite type	No. of recall restorations	Failure rate or Survival	Length	No. of recall restoration per type		Failure reason								
					M*	S†	Composite fracture	Secondary caries	Tooth fracture	Marginal adaptation	Wear	Postoperative sensitivity and pain	Endodontic treatment	Unknown	Operator
18 23 24 20	Visio-Molar Hybrid	70	83% (s)‡	10 years	8	38		24							
	Prodigy Condensable	111		2 years	111	0	3	1							
	Occlusion Hybrid	37		10 years	22	15	1	6		1	11	4			
	Occlusion (PC 4575) Highly filled	117	21.4% (f)§, 77% (s)	10 years	74	43								25	
	+PC4502 Highly filled														
25	Estilux UV cure	85		17 years	–	–	2	3			3	2		9	1
	+Nuva-fl UV cure														
	+Nuva-fl PA UV cure														
	+Uvio-fl UV cure														
26	Tetric Ceram Hybrid	71	7.5% (f)	4 years	–	–	2		2						1
	Annual f.: 1.9%														
6	Surefil Hybrid Packable	33	81.3–92.0% (s)	3.5 years	23	31	6	2							
	+Spectrum Hybrid conventional														
27–28	Beautiful Universal	39		4 years	27	12					1				
29	Tetric Ceram	140	5% (f)	2 years	121	27	1	1				5			
	Highly dispersed														
30	Solitaire 2 Packable	70	95% (s)	2 years	70	0		1				2		1	
	+Solitaire 2 and flowline														
31	Z100	726		1–4 years	466	260									
28	Solitaire Packable	165	79.0% (s)	3 years	–	–	23					12			
32	Superlux Molar	82	8%	3 years	82	0	3	6	1			1			
	Midway-filled fine		+13% (f)												
	+P-50 APC														
	Compact filled														
5	ultra-fine														
	Herculite Hybrid fine	68		2 years	68	0									
	+Clearfil														
	Compact fine														
	+Visiomolar Hybrid														
33	Herculite XR hybrid	234	87% (s)	1–4 years	1100	109	21	13	7			4			
34,35	Prodigy packable	73	5% (f)	2 years	–	–					2				
	+Definite packable														
36,37	Z100	70	5.7% (f)	7 years	–	–		4							
	Compact-filled fine														
	+Clearfil RP Compact-fill														
	ultrafine														
	+Prisma TPH midway-filled ultrafine														
38	Z100 Compact-filled fine	90		6 years	23	67	5								
	+Tetric hybrid Compact-filled fine														
	+Christina midway-filled ultrafine														

(continued)

Table 1 continued

References	Composite type	No. of recall restorations	Failure rate or Survival	Length	No. of recall restoration per type		Failure reason								
					M*	St†	Composite fracture	Secondary caries	Tooth fracture	Marginal adaptation	Wear	Postoperative sensitivity and pain	Endodontic treatment	Unknown	Operator
19–39,40	Visio-Molar hybrid Surefil high density posterior	46 22	74.2–53.5% (s)	10 years 2 years	– 22	– 0	11	5							
41–43	Tetric hybrid +blend-a-lux hybrid +Pertac hybrid	30		3 years	23	7		4							
44	Superlux midway-filled fine +P-50 APC Compact-filled ultrafine	51	27.6% (f)	6–65 months	–	–	1	7	1	6		1			
21–45	Occlusion hybrid Heliomolar micro-filled +P-30 APC Hybrid +Herculite XR hybrid	37 161	40–50% 14.4%–9.3– 15.4% (f)	10 years 8 years	– –	– –	1 8	2 7		1	15	4 1		4	
46	Z100 +Clearfil RP +Herculite XR	104		2 years	0	104									
22	Lite-Fil P micro-filled +Heliomolar	72	S: 44.1% M: 55.5% (f)	10 years	9	34		8				1			
47	Solitaire 2 packable posterior	76	96% (s)	2 years	–	–	2								
48,49–50	Surefil packable posterior P-30 hybrid +Occlusin hybrid posterior +Clearfil compact-fill fine TPH hybrid	47 56	6% (f)	3 years 10 years	– 56	– 0	2	2						4	
51		29		2 years	29	0									

* Multi-surface.

† Single-surface.

‡ Survival rate.

§ Failure rate.

Table 2. Summary of search results for 2006–2016

References	Composite type	No. of recall restorations	Failure rate or Survival	Length	No. of recall restoration per type		Failure reason								
					M*	S†	Composite fracture	Secondary caries	Tooth fracture	Marginal adaptation	Wear	Postoperative sensitivity and pain	Endodontic treatment	Unknown	Operator
52	Estelite +G-aenial highly-filled flowable	40		3 years	30	10					1	1			
15	Visio-Molar hybrid	37	73.6% (s†) Annual f‡: 1.92%	15 years	–	–	13	7							
53	TPH	86	S: (s): 96.0% M: 72.1%	3 years	61	25	10		4	4					
14,54	Grandio nano-hybrid	65	96.9% (s)	10 years	65	0			1	1					
3,55	+Tetric Ceram hybrid Tetric evoceram nano-filled	114	80.7% (s) Annual f.: 1.9%	10 years	114	0	7	11	5			3			
56	+Tetric Ceram hybrid Filetek Silorane Low-shrinkage	100		2 years	0	100									
57	+Ceram X nano-hybrid	100		2 years	70	30									
58	Filetek Silorane low-shrinkage Filetek P-60 packable	39		3 years	21	18		1		1					
59	+Prodigy packable Tetric Ceram hybrid	54	3.7–7.4% (f)	4 years	–	–				1			2		
60	+Ceram X nano-hybrid Filetek Supreme nano-filler	91		2 years	–	–									
61	P-60	78		4 years	78	0	4		2				1		
62	Vit-L-essence micro-hybrid	69		2 years	69	0									
63	Filetek Supreme nano-filler	112	98% (s)	2 years	112	0	2								
64	+Tetric Ceram fine hybrid Tetric Ceram hybrid & Tetric flow	114	85.3% (s)	7 years	114	0	10	5	3						
13	+Tetric ceram Hybrid P-50 APC mini-filled hybrid +Herculite XR mid-filled hybrid	632	30% (f) Annual f.: 1.9–2.1% S: 22% M: 78% (f)	18 years	–	–	97	65	97					24	
65	Beautifil II S-PRG filler	31		3 years	65%	45%									
66–68	Quixfil posterior +Grandio Universal nano-hybrid	62		3 years	37	25	1	3							
69	Filetek Supreme nano-filled +Tetric Evo ceram nano-filled +Aelite Aesthetic nano-filled	67	Annual f.: 1.6%	5 years	26	41		1							
70	Filetek Supreme universal nano-filled	73	87.7% (s) 4.2% (f)	3 years	74	0	6	2					1		

(continued)

Table 2 continued

References	Composite type	No. of recall restorations	Failure rate or Survival	Length	No. of recall restoration per type		Failure reason						
					M*	S†	Composite fracture	Secondary caries	Tooth fracture	Marginal adaptation	Wear	Postoperative sensitivity and pain	Endodontic treatment
16,71	P-50 APC hybrid mid-filled +Herculite XR hybrid mini-filled Tetric Ceram HB & Tetric-flow	362	1.5–2.20% (f)	22 years	241	121	51	27	19	3			7
72	Quixfil posterior +Tetric Ceram hybrid	25	8% (f)	3.5 years	–	–				2	2		
73–75	Prodigy packable +Helionolar	83	2.7–0.6% (f)	4 years	70	13	3	1	4	1		1	
76	Alert packable +Surefil packable	432	Annual f.: 1.7%	3 years	153	279	4	15	2	2			
77,78	Esthet-X micro-hybrid +Point4 micro-hybrid	60	77% (s)	5 years	34	26	1	1					1
79	InTen-S low-shrinkage +Point4 micro hybrid	44	Annual f.: 1.5–1.60%	6 years	44	0							
80	Filetek Z350 nano-filled +Esthet-X nano-hybrid	51	Annual f.: 1.61–0.80–1.61%	3 years	0	51	1						
81	SDR flowable bulk-filled & Ceram X mono	25	Annual f.: 0.00–1.30%	2.5 years	25	0							
8	Filetek LS Low-shrinkage +Tetric Evoceram nano-filled	91		15 years	91	0	7	8					
82	Filetek Supreme nano-filled +p60 packable	107		5 years	107	0	2	1	5		3		
7	Filetek Silorane nano-hybrid	93	Annual f.: 1.61–0.80–1.61%	4.5 years	0	93		1		1	1	1	2
4	SDR flowable bulk-filled & Ceram X mono	104	Annual f.: 0.00–1.30%	3 years	76	28	1		1				
83	Filetek LS Low-shrinkage +Tetric Evoceram nano-filled	72		3 years	72	0	4						
84	Filetek Supreme nano-filled +p60 packable	60		3 years	0	60							
1	Filetek Silorane low-shrinkage	196	Annual f.: 1.2–1.0%	3 years	122	74	2	2	3				
85	SDR flowable bulkfil& Ceram X nano-hybrid	156	Annual f.: 1.7–1.2%	3 years	156	0	5						2
86	Filetek P90 +Quixfil posterior	67	3% (f)	56 months	36	31		1			1		
87	Filetek Z250 posterior +Filetek spectrum hybrid	80		3 years	0	80	6						

(continued)

Table 2 continued

References	Composite type	No. of recall restorations	Failure rate or Survival	Length	No. of recall restoration per type		Failure reason									
					M*	S†	Composite fracture	Secondary caries	Tooth fracture	Marginal adaptation	Wear	Postoperative sensitivity and pain	Endodontic treatment	Unknown	Operator	
17	Nulite F fiber reinforced packable + Alert fiber reinforced packable	72	25% (f)	6 years	72	0	5	5	4							
88	Beatfil universal giomer	108	5% (f)	3 years	72	36	2	1	1			1				
89	Solitaire packable porous +Alert fiber reinforced packable +Surefil hybrid packable +Filtek P60 packable +TPH spectrum hybrid packable	75	12.5% (f)	3 years	66	9	2				1					

* Multi-surface.

† Single-surface.

‡ Survival rate.

\$ Failure rate.

Some papers aimed to compare the performance of composites with another direct restorative material such as amalgam or calcium aluminate cement. In these cases, only the results related to composites were extracted and used.

In a few papers, failure rates for each reason were presented as a percentage rather than actual numbers; therefore, when a total number for failures was specified, reported percentages were converted to number of failed restorations.

Articles with periods of assessment greater than 5 years are displayed in Table 3. This table includes the publication date, assessment duration, overall success and failure rate, and, depending on availability, the annual failure rate. In several papers, these rates were calculated specifically for each composite brand or type, which are demonstrated as two separate results in one study.

RESULTS

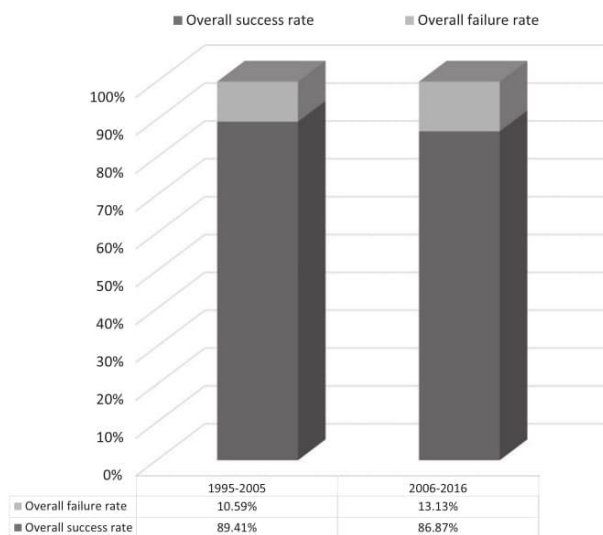
In the last two decades, numerous studies have been undertaken to evaluate performance of composites. However, the more recent studies showed that the clinical studies and methods of data collection have become more detailed and robust in the information provided. In the 1995–2005 period an annual failure rate was rarely calculated and the rate of unknown failure reason was high; on the other hand, in the 2006–2016 period more reports provided annual failure rates, case selection was more detailed and unknown causes of failure were reduced considerably (13.17% to 4.31%) (Fig. 2). The number of studies has increased by 33% (30 to 40) in the 2006–2016 period, which indicates the increase in relevance of composite for restoring posterior teeth as well as the need to improve the evidence base.

Twenty-eight different brands were used in the period 1995–2005 and 38 in 2006–2016. Several types of composites were included, such as mini-hybrid, micro-hybrid, nano-hybrid, nano-filled, micro-filled, mini-filled, midi-filled, fibre-reinforced, low-shrinkage (silorane-base), giomer, bulk-fill, flowable, packable, conventional, universal or posterior.

Most papers placed restorations in a combination of permanent molars and premolars. However, some only used molars, but no study solely used premolars. Recall durations varied from 2 to 22 years, with just over half of the papers identified being 3 years or less in duration. Overall, 3012 restorations were assessed at the final recalls in 1995–2005 and 4327 in 2006–2016. Respectively, 319 and 568 restorations were recorded as having failed (Fig. 2). Reasons for failure included restoration fracture, tooth fracture, secondary caries, postoperative sensitivity/pain, endodontic treatment, clinically unacceptable marginal

Table 3. Notable articles with long assessment period

	Publication date	Duration	Overall success rate	Overall failure rate	Annual failure rate
2006–2016					
Van de Sande, F. H. ¹³	2015	18 years	70%	30%	1.9%
Krämer <i>et al.</i> ¹⁴	2015	10 years	96.9%	3.1%	–
Van Dijken <i>et al.</i> ⁸	2015	15 years	77%	23%	1.6%
Dietz <i>et al.</i> ¹⁵	2014	15 years	73.6%	26.4%	1.5%
Van Dijken <i>et al.</i> ³	2014	10 years	80.7%	19.3%	1.92%
Da Rosa Rodolpho <i>et al.</i> ¹⁶	2011	22 years	64%	36%	1.9%
			74%	26%	2.20%
Van Dijken, J. W. ¹⁷	2006	6 years	75%	25%	1.50%
			87.2%	12.8%	–
1995–2005					
Gaengler <i>et al.</i> ¹⁸	2004	10 years	83%	17%	–
Gaengler <i>et al.</i> ¹⁹	2001	10 years	74.2–53.5%	25.8–46.5%	–
Lundin <i>et al.</i> ²⁰	1999	10 years	77%	21.4%	–
Raskin <i>et al.</i> ²¹	1999	10 years	50–60%	40–50%	–
Shimizu <i>et al.</i> ²²	1995	10 years	74%	26%	–

**Fig. 2** Overall results.

adaptation, wear, unknown factors and, finally, the operator; however, occasionally a combination of two factors was considered. The sum of failure reasons for the 1995–2016 period was 942, which included 110 teeth with two reasons for failure ($n = 100$, tooth and restoration fracture; $n = 4$, secondary caries combined with restoration fracture; $n = 3$, marginal and anatomical failure; $n = 2$, marginal adaptation failure and secondary caries; and $n = 1$, secondary caries with tooth fracture). Failure reasons and their percentages for each time period are shown in Fig. 3.

Overall failure rates showed a slight increase for the 2006–2016 period (Fig. 2), this was accompanied by a notable difference between principle failure reasons. In 1995–2005, there were two key causes for failure, secondary caries (29.47%) and composite

fracture (28.84%), in comparison with 2006–2016 where composite fracture (39.26%) had increased markedly compared with secondary caries (25.80%). In addition, there was an obvious difference between the rate of tooth fracture, postoperative sensitivity, wear and endodontic treatments: a notable increase in tooth fracture, a slight increase in endodontic treatment, a dramatic decrease in postoperative sensitivity as well as a noteworthy decrease in wear for the more recent studies (Fig. 3).

DISCUSSION

This is a systematic review of research on clinical evaluations of posterior composite restorations of more than 24-month duration that were published since 1995. Laboratory-based work and studies that used open laminate or tunnel techniques were excluded. Articles were categorized into two time periods, 1995–2005 and 2006–2016, to reflect some of the technological changes that have occurred during the last 20 years. A change was obvious in number of articles in these two periods. An increase of 33.33% in 2006–2016 reflects the increasing placement of composite materials and the improvement in techniques.

A couple of different methods have been used for assessment of restorations: usually United States Public Health Service criteria and in some cases in the 2006–2016 period the FDI criteria.^{16,50,81} The overall trend in clinical evaluations has become more detailed, such as calculation of annual failure rates. Unknown causes of failure were overall listed at a rate of 13.17% in 1995–2005 but this had reduced to 4.31% in 2006–2016, which shows improved reporting and more detail of the recording of outcomes (Fig. 3).

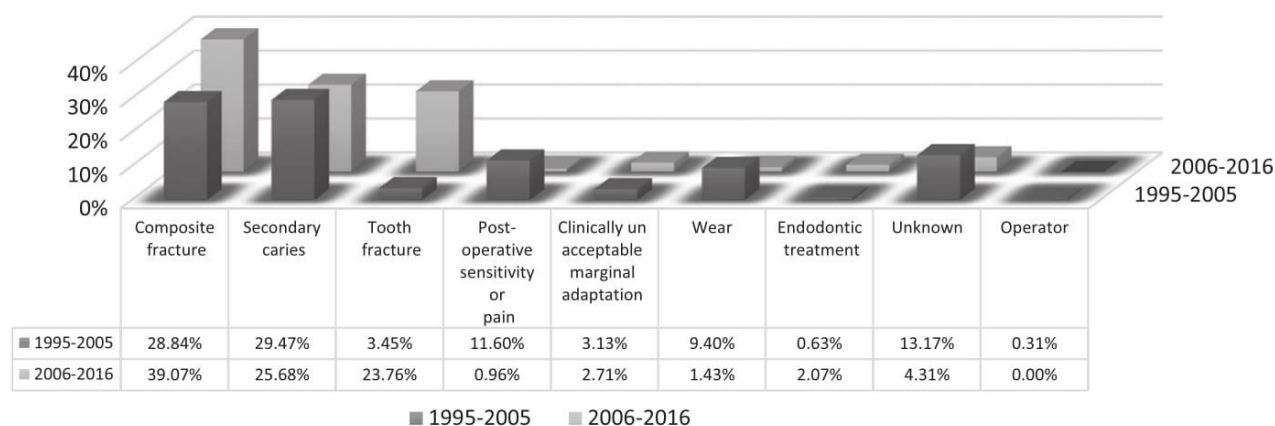


Fig. 3 Failure cause per period.

Most studies the search identified were short-term, of less than 2 years duration. Typically, the short study outcomes varied from the long-term results for composite restorations. More than half of the studies were 3 years or less in length, with 59.5% being in the 2006–2016 period. This is in line with the reported trend by Beck *et al.*,¹⁰ who indicated an increase in short-term clinical studies and lack of long-term reports. In a few studies of longer duration (from 10 to 22 years) the annual failure rates ranged 1.5–2.2%. This range is slightly lower than that reported by Opdam *et al.* in their systematic review of 2014, that stated a failure rate of 2.4% at 10 years.¹²

Regarding overall success rates, the report for the least successful material belongs to a packable composite, which is no longer commercially available. The best overall survival rates typically appeared in short-term studies. A total of 11 studies with different assessment durations had 100% success rate (seven 2 years, two 3 years, one 2.5 years and one 6 years). Lower failure rates in the early years following composite restoration placement are expected (annual failure rate: 1.8% at 5 years and 2.4% 10 years).¹² It is interesting to note that failure patterns tend to change during the lifespan of a restoration and do not have linear progression. Failure rate due to caries increases significantly as study length increases, whereas failure due to restoration fracture does not change markedly with time.¹²

According to overall failure rates for all studied restorations, which were 10.59% for 1995–2005 and 13.13% for 2006–2016, little difference was noted between these two periods, but the reasons for failure changed (Fig. 3). In 1995–2005, secondary caries and composite fracture were the predominant reason at rates of 29.47% and 28.84%, respectively, whereas in 2006–2016 although caries and fracture remained the main reasons for failure (25.68% and 39.07%), it was evident that fracture had increased. Both

secondary caries and restoration fracture showed similar ratios of change but in different directions, namely, a decrease in secondary caries and increase in restoration fracture (Fig. 4). The third most common reason for failure in 2006–2016 was tooth fracture, which changed from 3.45% in 1995–2005 to 23.76% in 2006–2016. Restoration fracture, secondary caries and tooth fracture are responsible for 88.51% of failures in the 2006–2016 period compared with 61.76% in the 1995–2005 period. This indicates that other factors such as wear, postoperative sensitivity and unacceptable marginal adaptation are becoming less important as failure reasons in more recent studies. Restoration fracture and tooth fracture together represent 62.83% of failures in the 2006–2016 period compared with 32.29% in 1995–2005. This change may be due to the increased and wider use of composite material in complex and multi-surface cavities, which are known to be more prone to tooth and restoration fracture.^{13,16} Including an extra surface in a restoration can be associated with a 30–40% increase in risk of failure.¹² As a result of this trend, an increase in endodontic treatments was also noted. Unfortunately, the specific distribution of the occlusal single-surface and multi-surface restorations in failed restorations was not always provided; therefore, it was not possible to determine clearer outcomes for failure of restoration types. In a few of papers, this information was listed as the number of restoration surfaces, but more specific descriptions of restorations were unclear. The difference in failure of restoration type was calculated in a few papers with higher failure rates for multi-surface restorations being evident. For example, Van de Sande *et al.* showed a 78% and 22% distribution of overall failure rates, respectively, for multi-surface and occlusal single-surface restorations after 18 years.¹³ Shimizu *et al.* showed 55.5% and 44.1% distribution of overall failure rates, respectively, for multi-surface and occlusal single-surface restorations after 10 years.²²

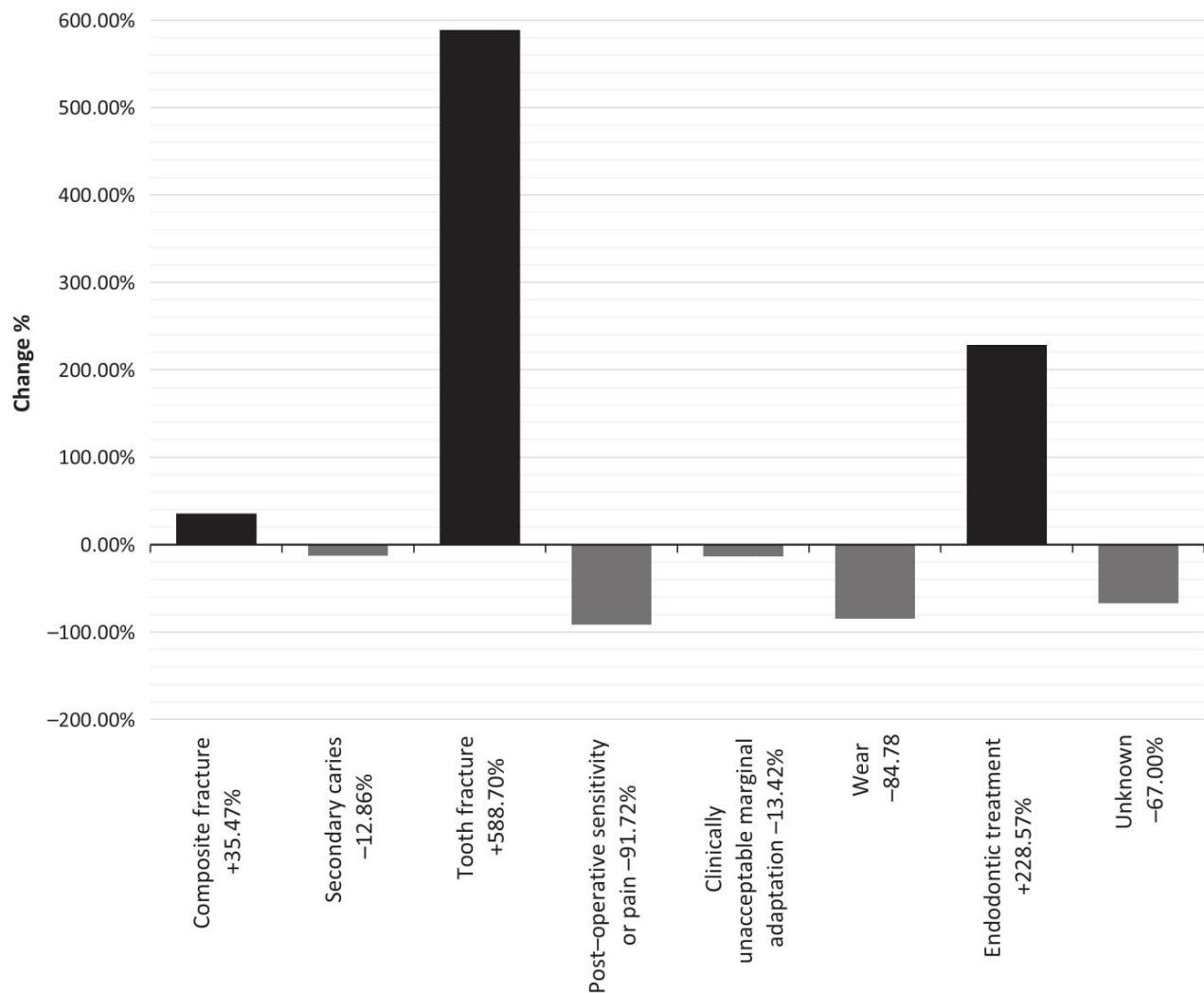


Fig. 4 Change of failure rates from 1995–2005 to 2006–2016.

One of the theories for cause of postoperative pain is formation of gaps between the cavity surface and restorative material allowing microleakage and fluid flow within dentinal tubules that is responsible for stimulating pulpal pain receptors. Consequently, an appropriate adhesive system that can seal dentinal tubules more effectively and can reduce postoperative pain, in addition to improving the marginal integrity of restorations, is important.⁹⁰ The significant change in postoperative sensitivity, which fell from 11.60% to 0.96% (Figs. 3 and 4), and slight decrease in clinically unacceptable marginal adaptation (3.13% to 2.71%) (Fig. 3) is most likely a result of improvements in adhesive systems during the last 20 years. Recent adhesive systems often show a reduction in technique sensitivity leading to less postoperative sensitivity,⁹¹ and an associated improvement in durability and sealing qualities.⁹²

Methods for measuring restoration wear either used a die cast at each recall session or a three-dimensional scanning method. The mean wear value varied from 2.72 μm per year¹⁵ to 16 μm per year²⁵ in the studies reviewed. Wear of composite materials is largely dependent on filler particle size, volume and resin matrix of the composite.^{90,93} Sarrett mentioned in his review that as a result of improvements in composite materials, wear and surface roughness are no longer significant clinical problems in posterior composite restoration longevity;⁹⁰ the current review confirms this statement, as failure due to wear and loss of anatomical form was reduced from 9.40% in 1995–2005 to 1.43% in 2006–2016 (Fig. 4).

In the second period, no failure or analysis of failure was reported due to operator influence. As described in previous reviews on clinical outcomes of posterior composites, this factor was not identified as

an influencing factor on restoration survival.^{2,10} The reason for this is that many of these studies are undertaken in controlled clinical settings and therefore do not reflect a typical private practice scenario.

Another factor that has been suggested to affect the life expectancy of a restoration is type of tooth, namely, molars compared with premolars. Some reviews showed that molars have a higher risk of failure¹² whereas others showed no difference.^{2,10} In the current review, several studies evaluated this aspect and found that the risk of failure was higher in molars^{13,16} where others reported no difference.^{8,19} It seems that a larger sample size and longer assessment period provides much better evidence for effect of tooth type on restoration survival.

A further factor that also has an effect on failure rates is the patient. In this category, the more influential factors are poor oral hygiene or high caries risk^{10,12} and high occlusal stress.^{9,10} Both of these factors can affect failure rates of restorations and reduce clinical longevity. In a meta-analysis, Opdam *et al.*¹² showed that restoration failure rates in patients with high, medium and low risk of caries after 10 years were 4.6%, 4.1% and 1.6%, respectively, which shows the influencing effect of caries risk on survival. In addition, Van de Sande *et al.*⁹ investigated the influence of patient risk factors on survival of posterior restorations. They reported a higher failure rate due to restoration fracture in patients with bruxism. Both factors must therefore be considered with respect to restoration success. Many of the papers included in this review considered these as excluding factors in patient selection, which means restoration success is not applicable to all patients. Another element that makes a difference is the evaluation of restorations on teeth with adjacent teeth and in a functioning occlusion. These conditions change the nature of the patient population, in view of the fact that some restorations are placed in absence of a neighbouring or opposing tooth.

As an established posterior restorative material, amalgam has been compared with posterior composite in several studies, where some showed better performance for amalgam^{50,94} and other similar to each other.^{95,96} Moraschini *et al.*, in a review on the clinical performance and longevity of amalgam and composite as posterior restorative materials, reported a mean success rate of 86.2% and 92.8%, respectively, for composite and amalgam in a mean 55-month assessment duration. As well as a shorter clinical life and greater degree of secondary caries for composites compared with amalgam, there was no significant difference between fracture rates.⁹⁷ This review showed similar survival rates with an average assessment duration of 5.17 years for 1995–2005 and 5.23 years for 2006–2016 (Fig. 2).

The overall success of posterior composite restorations showed satisfactory rates, with overall success rate of 89.41% for 1995–2005 and 86.87% for 2006–2016 for all studied restorations (Fig. 2). The range of the reported overall success rates for long-term studies (Table 3) improved in the 2006–2016 (minimum 64% to maximum 96.9%) compared with the 1995–2005 (minimum 50% to maximum 83%). This trend indicates that although composite materials are being used in multi-surface and more complex cavities, which are likely to show higher failure rates, the overall clinical performance seems to have improved in the last two decades. However, the necessity of longer assessment times for future studies is obvious because, as Opdam *et al.*,¹² Beck *et al.*,¹⁰ Brunthaler *et al.*² and many other authors have reported, with longer assessment periods there is a tendency of an increase in failure rates. Having this information will allow better case selection compared with what is currently known.

In conclusion, within the limitations of this review, comparing failure reasons in the last two decades has shown an important shift, reducing the significance of secondary caries, postoperative sensitivity and wear as failure factors in contrast to an increasing role of tooth fracture, restoration fracture and endodontic treatment as reasons for failure. The greater level of fracture may relate to the increase in size of restorations now being placed; however, more detailed data are needed. Further practise-based studies on posterior composite restorations are required with long assessment periods in combination with detailed failure reports on type and complexity of the studied restorations.

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Address for correspondence:

Professor Michael F. Burrow

Chair of Biomaterials

Melbourne Dental School

University of Melbourne

720 Swanston Street

Melbourne, Vic. 3010

Australia

Email: mfburrow@unimelb.edu.au

Chapter 4: Aims and hypotheses

Following the review of the literature it is clear there remains a number of questions to be answered. This thesis will aim to provide further evidence to assist with answering the following questions outlined below:

1. Based on the current literature, has there been a change in the reasons leading to failure of posterior resin-based restorations in the past decade?
2. Are GICs fracture toughness high enough to be used in load-bearing areas and Is there one fracture toughness test which may be best suited for the evaluation of GICs?
3. Does the use of artificial saliva as the storage medium influence fracture toughness values of GICs compared with distilled water as a comparative storage medium?
4. How does the heat generated during light polymerisation alter the core temperature of resin composite restorative materials, and does this temperature change pose a potential problem to the health of pulpal tissues?
5. How do differences in light polymerisation method influence the dimensional change and temperature of resin-based restorative materials?
6. Does a difference in the irradiance (intensity) by different types of light curing units influence polymerisation shrinkage of resin-based restorative materials?
7. Does thermal aging effect hardness, flexural strength and wear resistance of resin composite restorative materials?
8. How do differences in the method of light polymerisation of resin-based restorative materials affect the polymerisation contraction and subsequent movement of remaining tooth structure in prepared cavities?

To answer the above questions the following studies were developed:

1. A systematic review to evaluate the research literature published on the clinical performance of posterior resin composite restorations during the last 20 years.

2. A laboratory test to compare two methods of fracture toughness namely, Compact Tension and 4-point bending test on commercially available GICs and RM-GICs. In addition, the effect of storage media on fracture toughness of GICs and the benefits of resin coating use were investigated.
3. A laboratory test to study a method for measuring the core temperature of resin-based restorative materials during polymerisation, as well as determining polymerisation shrinkage stress and measuring dimensional changes when resin-based composite restorative materials were subjected to different types of polymerising units and methods of polymerisation.
4. A series of laboratory tests to measure the effect of thermal fatigue on the flexural strength, wear resistance and hardness of resin composite restorative materials.
5. A laboratory experiment to gain further understanding of strain development and movement of tooth structure when a bonded resin-based composite is placed in a prepared tooth cavity.

The hypotheses tested for each project were as follow:

1. ---
2. The null hypotheses were:
 - A- The two methods of fracture toughness (CT and 4-point bending test) provide similar outcomes when used to measure the fracture toughness of various GICs.
 - B- Artificial saliva as storage media has no significant effect on the fracture toughness of GICs.
 - C- Resin coating of GICs has no significant effect on fracture toughness values.
3. The null hypothesis was: different polymerisation unit irradiance and curing methods for resin composites have no significant effect on heat generated during polymerisation, no significant effect on shrinkage stress, or dimensional change.

4. The null hypothesis was: thermal fatigue has no significant effect on the flexural strength, wear resistance and hardness of resin composite restorative materials.
5. The null hypothesis of this study was that polymerisation unit irradiance or curing method has no significant effect on polymerisation contraction of resin composite materials and generation of strains within the surrounding tooth structure.

Chapter 5: Project II- Fracture toughness of glass ionomers

measured with two different methods

5.1: Introduction:

GICs are one of the popular restorative materials used in dentistry since their introduction in the 1970s (1, 2). Due to their fluoride-releasing characteristics and adhesion to tooth structure, they can have a significant positive influence on the success of dental treatment. Fluoride release from GIC has been suggested to reduce the occurrence of recurrent caries(3). Considering that the strength of GICs is lower than resin composites and due to the recent promotion of GICs as load-bearing restorations, fracture toughness of these materials becomes a more significant feature when considering restoration survival.

Kovarik et al. mentioned in their study on fracture toughness. This characteristic factor of materials is considered an intrinsic property and indicates a material's resistance to crack propagation (4).

Most of the studies conducted on fracture toughness have used specimens stored in 37°C distilled water which does not replicate the oral environment particularly well. There have been concerns raised due to the difference between the composition of saliva and distilled/deionised water, particularly with regard to differences in the ionic content which may be important for materials such as GIC, which easily react with the ions in their environment.

An important factor in the fracture toughness of composite materials is the inorganic content which can be determined via a standard method Ash test to measure the percentage fraction. In this test method, the organic part of the composite is burned-off in a furnace, and the remaining mineral content is measured as a weight per cent or volume percent. This content may influence or relate to the toughness of a material.

Another influencing factor on the crack propagation resistance or fracture toughness of materials is their inner structure which includes the pre-existing flaws in the material and molecular geometry

(4). GICs are known for their porous structure (5). Compared to other restorative materials such as resin composites this “swiss cheese” structure may reduce the fracture toughness dramatically.

Fujishima et al. (6) investigated the suitability of various fracture toughness methods and concluded that the double torsion method was the most reliable to provide information relating to crack propagation characteristics and give a ‘true value’ for the fracture toughness. However, this method was described as the most technique sensitive. Their study also concluded that the least technique sensitive methods were the Compact Tension (CT) test and 4-point bending test. Both these tests were able to provide useful information relating to crack propagation and reliable fracture toughness values. The outline of these tests is specified in ASTM E399-12e3 (7) and ISO 6872 (8) respectively. The method used for the CT test is similar to the method used by Kovarik et al. (4).

Shofu Inc. (Kyoto, Japan) introduced Giomers, which are resin-based composites containing pre-reacted glass said to exhibit features like a GIC glass. They have better aesthetic qualities and less tendency to dehydrate compared to GICs. Giomer contains urethane dimethacrylate (UDMA) as the main resin matrix component, hydroxyethyl methacrylate (HEMA) and pre-reacted glass (PRG) particles. The PRG is produced via a reaction between a polyacrylic acid and fluorosilicate glass. These pre-reacted glass particles are incorporated into the resin matrix to enhance its strength. (9-11)

Giomers exhibit higher strength compared to GICs. Also, it has been reported that this material shows the ability to release fluoride. However, Giomer lacks the “early burst” phase of fluoride (12, 13) which is a characteristic of all GICs. The water sorption is greater than resin composites and polyacid-modified resin composites (compomers) but lower than GICs (14). Also, the surface after finishing and polishing of Giomers is smoother than GICs, being comparable to resin composites and compomers (9). It has been suggested that Giomers have the benefits of an improved GIC with the disadvantage of needing an adhesive to bond to tooth structure. (11, 15)

The literature is still lacking information about the differences that may be caused by using artificial saliva as storage media instead of distilled water for GIC materials and how the storage medium may

influence physical property test outcomes. In addition, there is little information available regarding the variations in fracture toughness outcomes measured using different test methods.

The aims of this study were: 1- to compare two methods of fracture toughness (CT and 4-point bending test); 2- To determine the effect of the storage medium on the fracture toughness; 3- to evaluate the porosity of the GIC structure, and 4- To determine the percentage of inorganic content, of four different GIC materials and a Giomer.

The null hypotheses were: 1- There is no significant difference between fracture toughness results of two test methods, and 2- artificial saliva has no significant effect on fracture toughness of these materials compared to distilled water.

5.2: Additional tests:

5.2.1: Ash test:

Materials and methods:

The filler content of each sample was determined using a standard ash test following the method described in ISO 1172:1996. (16) Disk-shaped (12 mm Ø X 1.2±0.01 mm thickness) samples were prepared, mixed/light-cured per manufacturer's instruction and weighed on a precision balance (Precisa Gravimetrics AG, CH-8953 Dietikon, Switzerland) to a precision of ±0.0001 g. The samples were heated in an electric furnace (Combilabor CL-V, Heraeus, Hanau, Germany) to a temperature of 625°C maintained for 30 minutes to burn off the organic matrix of the composite. After cooling to the room temperature samples were placed in a vacuum flask containing silica gel for 12 hours. Samples were re-weighed, and the inorganic weight fraction (wt%) was calculated using equation-1. The volume of the sample was measured using a pycnometer for calculating the density (ρ_c) of each material and inorganic volume fraction (vol%).

$$Filler\ wt\% = \frac{W_1}{W_0} \times 100\% \quad \text{Equation-1}$$

$$Filler\ vol\% = \frac{\frac{V_1}{W_0}}{\rho_c} \times 100\% \quad \text{Equation-2}$$

(w_1 = Weight of the sample after ashing (g), W_0 = weight of the sample before ashing (g), V_1 = Volume of the sample after ashing (mm^3), ρ_c = density of the related resin composite (g/mm^3))

Results:

Table-7 Wt % for filler content of materials (17)

Material	Wt% filler (Mean $\pm$ SD)
ChemFil	68.95 $\pm$ 1.34
Riva HV	75.41 $\pm$ 1.01
Riva	72.96 $\pm$ 1.06
Equia	68.23 $\pm$ 0.73
Beautifil	66.12 $\pm$ 0.14

The wt% results of the ash test were reported in this project's paper (17)(Table-7). Vol% results were arranged in the below table (Table-8).

Table-8 Vol % for filler content of materials

Material	Vol% filler (Mean $\pm$ SD)
ChemFil	84.38 $\pm$ 1.63
Riva HV	79.13 $\pm$ 1.06
Riva	54.88 $\pm$ 0.80
Equia	96.81 $\pm$ 0.21
Beautifil	52.59 $\pm$ 0.56

Discussion:

The unusual high results for the vol% content indicated an error in the experimental process. So, vol% results were considered invalid. The results indicated a volume loss with the pycnometer method. This volume loss may be due to loss of some minerals in GICs such as P, F or Ca. These minerals are water soluble, so it was more appropriate to replace this medium with another material that had a similar density. Consequently, these results were excluded from the study.

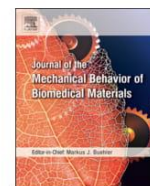
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Fracture toughness of glass ionomers measured with two different methods

Negin Alvanforoush, Rebecca Wong, Michael Burrow¹, Joseph Palamara*

The University of Melbourne – Melbourne Dental School, 720 Swanston St, Carlton, VIC 3053, Australia



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ABSTRACT

Glass Ionomer Cements (GICs) are brittle materials with low fracture toughness and strength. Therefore, understanding the fracture toughness is an important parameter to know, due to GICs being promoted for load-bearing restorations. Also, little is known about the effects of artificial saliva (AS) on fracture toughness of GICs. This project aimed to study the effects of storage and compare two different fracture toughness test methods, namely: Compact-Tension test and 4-point bending test. Samples were made from a Zinc-reinforced-GIC (ChemFil), High viscosity bulk-fill-GIC (HV) with and without LC-coating (EQUIA), Resin-Modified GIC (RM-GIC) (Riva-LC), HV-RMGIC (Riva-HV-LC) and flowable bulk-fill Giomer (Beautifil-Bulk Flowable) using a custom-made mould. Specimens were stored in either AS or distilled-water (DW) for 7 or 30 days. After storage, specimens were tested for fracture toughness. Results were analysed with Analysis of variance (ANOVA), Mann-Whitney and Weibull statistics. ANOVA showed a significant difference ($P < 0.05$) between methods, however the same trend among materials was observed in both methods. Some materials showed significantly higher values for samples stored in DW and others revealed the same trend but were not significant. Significantly higher ($P < 0.05$) mean fracture toughness values were observed for materials stored for 30 days. Since both fracture toughness test methods revealed the same fracture toughness strength order among materials, either test appears appropriate. GICs maturation led to a difference between 7 and 30-day results. The AS used had a high ionic strength compared to DW, which may have led to increased solubility of GICs and consequently to the reduced fracture toughness values. The results revealed that coating the GIC has a protective effect against storage media.

1. Introduction

Glass Ionomer Cements (GICs) are widely used in dentistry since their introduction in the 1970s (Wilson, 1991; Nicholson, 2016). They have been popular with dentists due to the fluoride-releasing characteristics and adhesion to tooth structure. Both of these qualities can have a significant influence on the success of dental treatment. Adhesion to tooth structure decreases microleakage and consequently bacterial infiltration along the tooth restoration interface that may lead to pulpal inflammation and recurrent caries. Also, fluoride release from GIC has been suggested to reduce the occurrence of recurrent caries (Raggio et al., 2016). In a previous study, it was revealed that recurrent caries was the main reason for failure of posterior resin-based restorations, but this has reduced somewhat, with tooth/restoration fracture now being the most common failure (Alvanforoush et al., 2017). Considering that the strength of GICs is lower than resin composites and due to the recent promotion of GICs as load-bearing restorations, fracture toughness of these materials becomes a more significant feature when considering restoration survival. Kovarik and Fairhurst

(1993) mentioned in their study on fracture toughness, this characteristic factor of materials is considered an intrinsic property and indicates a material's resistance to crack initiation and propagation.

Most of the studies conducted on fracture toughness have used specimens stored in 37 °C distilled water which does not replicate the oral environment particularly well. There have been concerns raised due to the difference between the composition of saliva and distilled/deionised water, particularly with regard to differences in the ionic content which may be important for materials such as GIC.

Fujishima and Ferracane (1996) investigated the suitability of various fracture toughness methods for composites and concluded that the double torsion method was the most reliable to provide information relating to crack propagation characteristics and give a 'true value' for the fracture toughness. However, this method was the most technique sensitive. Their study also concluded that the least technique sensitive methods were the Compact Tension (CT) test and 4-point bending test. Both these tests were able to provide useful information relating to crack propagation and reliable fracture toughness values. The outline of these tests is specified in ASTM E399-12e3 (ASTM, 2013) and ISO 6872

* Corresponding author.

E-mail address: palamara@unimelb.edu.au (J. Palamara).¹ Present address: Faculty of Dentistry the University of Hong Kong, Prince Philip Dental Hospital, 34 Hospital Road, Sai Ying Pun, Hong Kong.

(2015) respectively. The method used for the CT test is similar to the method used by Kovarik and Fairhurst (1993).

Shofu Inc. (Kyoto, Japan) introduced Giomers, which are resin-based composites containing pre-reacted glass said to exhibit features like a GIC glass such as, releasing fluoride. They have better aesthetic qualities and less tendency to dehydrate compared to GICs. Giomer contains urethane dimethacrylate (UDMA) as the main resin matrix component, hydroxyethyl methacrylate (HEMA) and pre-reacted glass (PRG) particles. The PRG is produced via a reaction between a polyacrylic acid and fluorosilicate glass. These pre-reacted glass particles are incorporated into the resin matrix to enhance its strength (Mitchell, 2010; Lien and Vandewalle, 2010; Walia et al., 2016).

Giomers exhibit higher strength compared to GICs. Also, it has been reported that this material shows the ability to release fluoride. However, Giomer lacks the “early burst” phase of fluoride (Gururaj et al., 2013; Yap et al., 2002) which is a characteristic of all GICs. The water sorption is greater than resin composites and polyacid-modified resin composites (compomers) but lower than GICs (McCabe and Rusby, 2004). Also, the surface after finishing and polishing of Giomers is smoother than GICs, which is comparable with resin composites and compomers (Mitchell, 2010). It has been suggested that Giomers have the benefits of an improved GIC with the disadvantage of needing an adhesive to bond to tooth structure (Walia et al., 2016; Gonulol et al., 2015).

The literature is still lacking information whether differences occur between artificial saliva and distilled water as storage media for GIC materials and how the storage medium may influence physical property test outcomes. In addition, there is little information available regarding the variations in fracture toughness outcomes in GICs measured using different test methods.

The aim of this study was to compare two methods of fracture toughness (CT and 4-point bending test) with four different GIC materials and a Giomer stored in either artificial saliva (without mucins) or distilled water to determine if the storage media or test method influences test results.

2. Materials and methods

The materials used in this research and their batch numbers and contents are presented in Table 1, and listed below:

1. Zinc-reinforced Conventional Glass Ionomer Cement (GIC) (ChemFil Rock, Dentsply Sirona, York, Pennsylvania, USA);
2. Auto-cure bulk fill Conventional high viscosity GIC and LC coating (EQUIA Forte Fil and EQUIA Forte Coat, GC Corp., Tokyo, Japan);
3. Resin Modified-Glass Ionomer Cement (RM-GIC) (Riva LC, SDI Ltd., Bayswater, Victoria, Australia);
4. Resin Modified-high viscosity Glass Ionomer Cement (RM-GIC) (Riva HV LC, SDI Ltd., Bayswater, Victoria, Australia);
5. Flowable Bulk fill Giomer (Beautifil-Bulk Flowable: Pre-reacted glass filled resin composite, Shofu Corp., Kyoto, Japan); and,
6. Auto-cure bulk fill Conventional GIC with high viscosity (HV) (EQUIA Forte Fil, GC Corp., Tokyo, Japan).

The storage times were determined as 7 and 30 days. Artificial saliva and distilled water were the storage media used. Half of the manufactured specimens were stored in 37 °C distilled water while the remaining half were stored in 37 °C artificial saliva without mucin using the formula:

50 mM NaCl + 0.5 mM CaCl₂ + 0.5 mM Na₂HPO₄· Adjusted to pH 7.0 (Shen et al., 2017).

A fresh batch of one litre was made weekly.

The storage media were changed every three days. Replacement media were heated to the same temperature as the specimens (37 °C), in order to prevent thermal shock.

A total of 720 samples in 48 groups were examined based on material, storage media, storage time and test method (n = 15, each group containing 15 samples).

2.1. CT test

Specimens required for the compact tension test were made using a custom-made polytetrafluoroethylene (PTFE) split mould (Figs. 1 and

Table 1
Specification of materials used.

Type	Product	Name	Manufacturer	Batch number	Composition
GIC	Auto-cure Zinc-reinforced Glass Ionomer Cement	ChemFil	DENTSPLY	15110007241 1511000724 1607000499 1607000503	Calcium-aluminium-zinc-fluoro-phosphor-silicate glass, Polycarboxylic Acid, Iron oxide pigments, Titanium dioxide pigments, Tartaric Acid, Water
GIC (HV) + Resin Coating	Auto-cure bulk fill GIC with high viscosity + Light-cure resin coating	EQUIA Forte Fil + EQUIA Forte Coat	GC	1602191 1606041 1608251 1608261 1610081	(polyalkenoic acid, fluoroaluminosilicate glass, Tartaric Acid, Water) [*]
RM-GIC	Light-cure Resin Modified-Glass Ionomer Cement	Riva	SDI	J1604055EG J1607201EG J1610033EG J1610173EG	2-Hydroxyethyl Methacrylate, Acrylic acid homopolymer, Dimethacrylate Cross-linker, Acidic Monomer, Tartaric acid, Glass powder
RM-GIC (HV)	Light-cure Resin Modified-Glass Ionomer Cement with high viscosity	Riva HV	SDI	K1604122EG K1607193EG K1611281EG K1610174EG	Acrylic acid homopolymer, 2-Hydroxyethyl methacrylate, Dimethacrylate Cross-linker, Acidic monomer, Tartaric acid, Glass powder
Giomer	Light-cure flowable Bulk-fill Pre-reacted glass filled resin composite	Beautifil-Bulk Flowable	Shofu	041507 101615 031717	Bis-GMA, UDMA, Bis-MPPEPP, TEGDMA, S-PRG filler based on fluoroboroaluminosilicate glass, Polymerization initiator, pigments
GIC (HV)	Auto-cure bulk fill GIC with high viscosity	EQUIA Forte Fil	GC	1703151 1608251 1702131	(polyalkenoic acid, fluoroaluminosilicate glass, Tartaric Acid, Water) [*]

* Exact composition N/A.

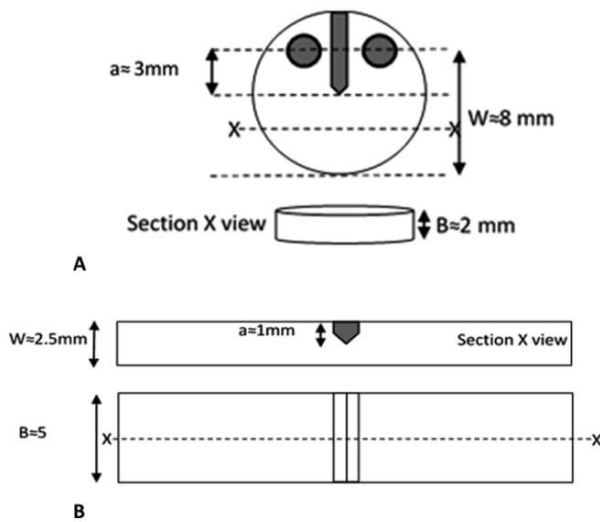


Fig. 1. Diagram of the specimen design and geometry: A – CT test specimen, B – 4-point bending test specimen.

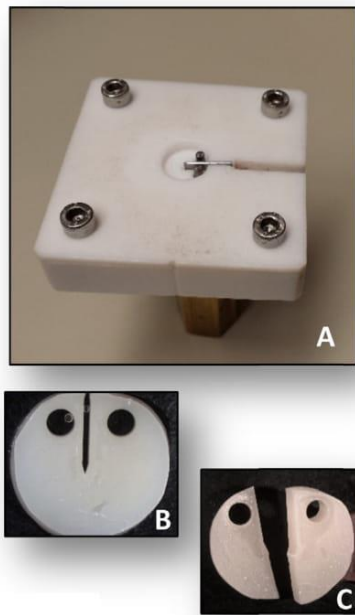


Fig. 2. A – PTFE Mould with metal plate. B – Specimen before test. C – specimen after test.

2). The mould was filled with GIC, and excess was expressed with a mylar strip under finger pressure. A metal plate with a V-shaped edge was placed in the prefabricated notch space in order to create a V-shaped notch in the cement (Fig. 2A). The light-cured specimens were polymerised as per manufacturers' instructions with a LED curing light unit with a light intensity of 1200 mW/cm² (Bluephase, Ivoclar Vivadent) for 20 s in three overlapping sections on each side (total 120 s). The self-cured samples were left in the mould for the recommended setting time according to the manufacturers' instructions. The specimens were carefully removed from the split mould, and then any irregularities were removed by gentle grinding with 1200-grit abrasive papers under water. Specimens were checked for any voids or cracks under $\times 10$ magnification and discarded when identified. A precrack was established by using a carbon steel surgical blade No.10 blade

(Swan-Morton LTD. Sheffield, England) under hand pressure in the V-shaped notch as the last stage of specimen preparation and before placing in the storage medium. For samples that needed coating, the blade was covered with coating material before preparation of the precrack and light-cured afterwards as per manufacturers' instructions. Following this procedure, all surfaces of these specimens were coated and light-cured. A new blade was used for producing the precrack after preparation of every six samples.

Specimens made of each material were randomly allocated to each group. After storage (7 or 30 days), the dimensions for calculating K_{Ic} were measured with a digital micrometer to ± 0.001 mm (for "B", adjacent to the notch) (Fig. 1) and under a Stereomicroscope (Leica Microsystems, Wetzlar GmbH, Germany) to ± 0.1 mm ("W" at least in 3 locations) (Fig. 1). During the test, specimens were maintained wet by covering with damp cotton pellets.

After test "a" (crack length) (Fig. 1) was calculated by measuring to a tolerance of ± 0.1 mm under a Stereomicroscope (Leica Microsystems, Wetzlar GmbH, Germany) (Fig. 1). Fracture toughness (K_{Ic} (MPa $\sqrt{m}$)) of specimens was calculated by using the maximum load at failure on a universal testing machine (INSTRON, Model:5544, Canton, MA, USA) with a crosshead speed of 0.5 mm/min and following formula:

$$K_{Ic} = \frac{F_c \cdot f\left(\frac{a}{w}\right)}{BW^{0.5}} \quad (1)$$

(F_c = maximum load at fracture (MN), B = specimen thickness (m), W = dimension from the un-notched edge of the specimen to the plane centerline of the loading holes (m), a = distance between notch and centre of the hole (crack length) (m),

$$f\left(\frac{a}{w}\right) = \frac{\left(2 + \frac{a}{w}\right) \left[0.76 + 4.8 \frac{a}{w} - 11.58 \left(\frac{a}{w}\right)^2 + 11.43 \left(\frac{a}{w}\right)^3 - 4.08 \left(\frac{a}{w}\right)^4\right]}{\left(1 - \frac{a}{w}\right)^{1.5}} \quad (\text{ASTM, 2013}).$$

2.2. 4-point bending test

A custom-made aluminium and stainless-steel mould was made for the four-point bend test (Fig. 3A).

A total of 24 groups were examined based on material, storage media and storage time ($n = 15$).

Samples were made using the custom-made split mould (Fig. 3). The mould was filled with restorative material, and excess was expressed with a mylar strip and a glass slab under finger pressure. The light-cured specimens were cured using the same method as CT samples, and the self-cured samples were left in the mould for the recommended setting time by the manufacturers' instructions. The specimens were removed from the split mould, and any irregularities were removed with 1200-grit abrasive paper with water lubricant and checked for any voids or cracks under $\times 10$ magnification. The production method of the precrack was similar to the CT test method: using a carbon steel No.10 surgical blade under hand pressure in the V-shaped notch as the last stage of specimen preparation and before storage. For Equia with coating groups, the specimens were coated using the same technique as for the CT test. A new blade was used for producing the precrack after preparation of every six samples.

The fracture toughness (K_{Ic} (MPa $\sqrt{m}$)) was calculated using maximum load at failure in this formula:

$$K_{Ic} = \frac{F_c}{B\sqrt{w}} \cdot \frac{S_1 - S_2}{w} \cdot \frac{3\sqrt{\alpha}}{2(1-\alpha)^{1.5}} \cdot Y^\circ \quad (2)$$

where F_c = fracture load (MN), S_x = span (m) ($x = 1$: outer span; $x = 2$: inner span), B = specimen width (m), $\alpha = \frac{a}{w}$, a = notch depth (crack length) (m), w = specimen height (m), Y° = stress intensity shape factor = $1.9887 - 1.326\alpha - (3.49 - 0.68\alpha + 1.35\alpha^2)\alpha(1-\alpha)(1+\alpha)^{-2}$ (ISO 23146-2012-2, 2012).

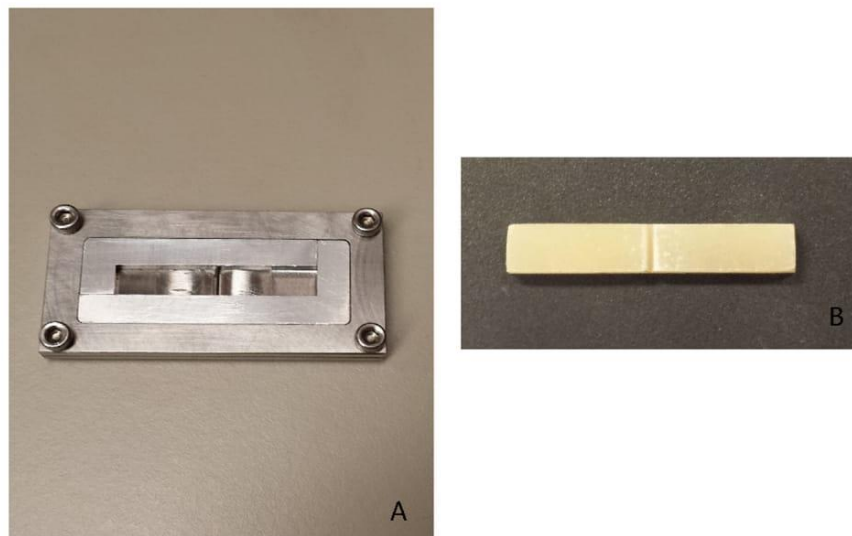


Fig. 3. A. 4-point bending test mould. B. Specimen for 4-point bending test.

2.3. Filler content (standard ash test)

The filler content was determined using a standard ash test in accordance with ISO 1172:1996 (1996). Disk-shaped samples were prepared and weighed using a precision balance (Precisa Gravimetrics AG, CH-8953 Dietikon, Switzerland), then placed in an electric furnace (Combilabor CL-V, Heraeus, Hanau, Germany) and heated to 625 °C. This temperature was maintained for 30 min to burn out the organic matrix. After cooling to room temperature samples were placed in a vacuum with silica gel for 12 h. Samples were re-weighed, and the inorganic weight fraction (wt%) was calculated using Eq. (3)

$$\text{Filler wt\%} = \frac{W_1}{W_0} \times 100\% \quad (3)$$

(W_1 = Weight of sample after burning (g), W_0 = weight of sample before burning (g)).

2.4. Statistical analysis

Data were checked for normal distribution and analysed with Levene, ANOVA, Tukey and Mann-Whitney U tests (SPSS, V23, IBM). In addition, the Weibull cumulative distribution function of Excel software was used (Excel 2016, Microsoft) (Eq. (3)).

$$F(x, \alpha, \beta) = 1 - e^{-\left(\frac{x}{\beta}\right)^\alpha} \quad (4)$$

In this test, Eq. (4) was used for the Weibull cumulative distribution, considering α = Shape parameter/Weibull modulus, β = characteristic

life and e = K_{Ic} value. Afterwards, the table of Weibull modulus was created.

3. Results

3.1. Test method

Test methods revealed a statistically significant difference in K_{Ic} value of materials. However, the trend between materials in each test method is the same. The K_{Ic} in descending rank was Beautifil > Riva > Equia with coating > Riva HV > Chemfil > Equia without coating for each test method.

3.1.1. CT test

In this test method, the results of each material regardless of medium and storage time showed a significant difference in K_{Ic} ($P < 0.05$). The same outcome was observed for storage times (regardless of material and medium) ($P < 0.05$), but no difference of K_{Ic} was observed between two storage media ($P > 0.05$).

Mean values of K_{Ic} of CT test groups showed a higher value in groups stored in DW, with all materials giving a higher value in DW and 30-day compared to AS and 7-day groups. (Except: Equia with coating) (Table 2).

3.1.2. 4-point bending test

The results for 4-point bending test are displayed in Table 3. The same trend as for the CT test was observed. Except for Equia with

Table 2
Mean value of K_{Ic} (MPa $\sqrt{m}$) $\pm$ SD for CT test.

Material	Storage condition			
	Artificial saliva		Distilled water	
	7-days	30-days	7-days	30-days
ChemFil	0.43 $\pm$ 0.06 ^{a, A}	0.49 $\pm$ 0.06 ^{a, B, C}	0.45 $\pm$ 0.05 ^{a, A, B}	0.50 $\pm$ 0.03 ^{b, C}
Riva HV	0.67 $\pm$ 0.11 ^{b, C, A}	0.73 $\pm$ 0.09 ^{b, A, B}	0.72 $\pm$ 0.12 ^{b, C, A, b}	0.76 $\pm$ 0.07 ^{c, B}
Riva	0.75 $\pm$ 0.09 ^{c, A}	0.78 $\pm$ 0.04 ^{b, A, B}	0.77 $\pm$ 0.11 ^{c, A, B}	0.82 $\pm$ 0.06 ^{c, B}
Equia with resin coating	0.60 $\pm$ 0.08 ^{b, A}	0.82 $\pm$ 0.25 ^{b, B}	0.67 $\pm$ 0.16 ^{b, A, B}	0.80 $\pm$ 0.23 ^{c, B}
Equia without coating	0.35 $\pm$ 0.06 ^{a, A}	0.37 $\pm$ 0.07 ^{a, A}	0.36 $\pm$ 0.05 ^{a, A}	0.38 $\pm$ 0.07 ^{a, A}
Beautifil	1.04 $\pm$ 0.10 ^{d, A}	1.18 $\pm$ 0.10 ^{c, B}	1.05 $\pm$ 0.13 ^{d, A}	1.21 $\pm$ 0.10 ^{d, B}

* Materials with the same lower case superscript letter are not significantly different for each media and storage time, that is within each column ($P > 0.05$).

* Values with the same upper case superscript letter are not significantly different in each material group, that is within each row ($P > 0.05$).

Table 3Mean value of K_{Ic} (MPa $\sqrt{m}$) $\pm$ SD for 4-point bending test.

Material	Storage condition			
	Artificial saliva		Distilled water	
	7-days	30-days	7-days	30-days
ChemFil	0.31 $\pm$ 0.06 ^{a, A}	0.44 $\pm$ 0.06 ^{a, B, C}	0.38 $\pm$ 0.08 ^{b, B}	0.50 $\pm$ 0.06 ^{b, C}
Riva HV	0.58 $\pm$ 0.07 ^{b, A}	0.63 $\pm$ 0.12 ^{b, A, B}	0.60 $\pm$ 0.06 ^{c, A}	0.72 $\pm$ 0.12 ^{c, B}
Riva	0.72 $\pm$ 0.05 ^{c, A}	0.73 $\pm$ 0.07 ^{b, A}	0.76 $\pm$ 0.06 ^{d, A, B}	0.80 $\pm$ 0.09 ^{c, B}
Equia with resin coating	0.78 $\pm$ 0.22 ^{c, A}	0.73 $\pm$ 0.10 ^{b, A}	0.71 $\pm$ 0.11 ^{d, A}	0.74 $\pm$ 0.08 ^{c, A}
Equia without coating	0.26 $\pm$ 0.09 ^{a, A}	0.35 $\pm$ 0.11 ^{a, B, C}	0.27 $\pm$ 0.09 ^{a, A, B}	0.38 $\pm$ 0.05 ^{a, C}
Beautifil	1.02 $\pm$ 0.06 ^{d, A}	1.07 $\pm$ 0.07 ^{c, A, B}	1.04 $\pm$ 0.07 ^{e, A, B}	1.09 $\pm$ 0.07 ^{d, B}

* Materials with the same lower case superscript letter are not significantly different for each media and storage time, that is within each column ($P > 0.05$).* Values with the same upper case superscript letter are not significantly different in each material group, that is within each row ($P > 0.05$).

coating the results showed higher K_{Ic} value in DW and 30-day compared to AS and the 7-day groups in all materials.

3.2. Storage media – AS and DW

In each storage medium, the outcomes for the different test methods were not significantly different, on the other hand, the effect of time was significant ($P < 0.05$).

Storage medium was observed to have a significant effect on ChemFil, Riva and Riva HV with higher values in DW, while no significant difference for Beautifil and Equia was observed.

3.3. Storage time – 7 and 30-days

Analysis of each storage period showed a significant effect. With higher values in 30-day storage time ($P < 0.05$).

3.4. Filler content (standard ash test)

The calculated wt% for filler content of materials is arranged in Table 4. wt% in descending order are: Riva HV > Riva > ChemFil > Equia > Beautifil.

3.5. Weibull modulus “m”

The Weibull moduli are displayed in Table 5. The highest m values were seen for Beautifil, and Riva (Respectively: Mean values of m: 14.42 and 13.01) and lowest were for Equia (Mean values of m: with coating 6.20 and without coating 5.14). Chemfil and Riva HV revealed intermediate values between these materials (Respectively: Mean values of m: 8.03 and 8.20) (Table 5).

One sample of each material was randomly selected, and the thickness was reduced to 1.11 ± 0.08 mm then using a light microscope, images with $10\times$ and $40\times$ magnification were collected to show the particle size and density of fillers within materials. Using the transmitted light technique more images were collected to illustrate the void distribution and size with transmitted light (Fig. 4).

Table 4
wt% for filler content of materials.

Material	wt% filler (Mean $\pm$ SD)
ChemFil	68.95 $\pm$ 1.34
Riva HV	75.41 $\pm$ 1.01
Riva	72.96 $\pm$ 1.06
Equia	68.23 $\pm$ 0.73
Beautifil	66.12 $\pm$ 0.14

Table 5

Weibull modulus “m” for each group.

	Chemfil	Riva	Riva HV	Equia with coating	Equia without coating	Beautifil
4-point						
30 AS	7.35	11.29	5.48	8.13	2.70	15.98
30 DW	9.08	9.69	6.32	9.90	7.86	17.84
7 AS	4.78	15.69	9.19	4.21	2.30	18.16
7 DW	5.05	14.44	11.59	7.39	3.46	15.94
CT						
30 AS	9.75	19.60	8.70	3.56	5.70	10.95
30 DW	11.45	15.31	11.05	3.87	5.41	8.60
7 AS	8.21	9.58	6.67	7.88	5.39	12.95
7 DW	8.56	8.44	6.58	4.69	8.33	14.96
Mean	$\pm$ S- D	8.03 $\pm$ 2.27	13.01 $\pm$ $\pm$ 3.8- 7	8.20 $\pm$ 2.29	6.20 $\pm$ 2.40	5.14 $\pm$ 2.23
	14.42 $\pm$ 3.36					

4. Discussion

4.1. Test methods

As outlined by Kovarik and Fairhurst (1993) the method of creating the precrack has a significant effect on the measured values. Several studies have measured the fracture toughness of GICs using different test methods and different ways to prepare the precrack. The results of these studies revealed different fracture toughness values for the same materials, but the same order among materials was consistently observed (Fujishima and Ferracane, 1996; Kovarik and Muncy, 1995; Yamazaki et al., 2006; Knobloch et al., 2000; Lloyd and Mitchell, 1984; Baig et al., 2015; Mese et al., 2016; Ilie et al., 2017). This difference may be explained by the use of the various methods of pre-crack creation since it has been reported that the sharpness of a precrack will affect the fracture toughness values in association with the specimen geometry (Kovarik and Fairhurst, 1993; Fujishima and Ferracane, 1996; Ferracane et al., 1987; Bagheri et al., 2010). On the other hand, as Quinn and Quinn (2010a) discussed in their paper, the sample size and the volume of the material that precrack has to propagate to complete fracture has a critical rule in the value of K_{Ic} . This fact indicates that higher K_{Ic} value is expected in samples with larger final crack compared to the initial precrack since these samples had to overcome a larger volume of material to complete the fracture.

In the current study, test method results also revealed a statistically significant difference, which confirms the results of previous studies. CT test method resulted in a higher mean value of K_{Ic} for each material compared to 4-point bending test method. This is with the exception of Equia with coating groups (Fig. 1) (Table 6). The exclusion of Equia with coating can be due to structural difference of sample. Although the

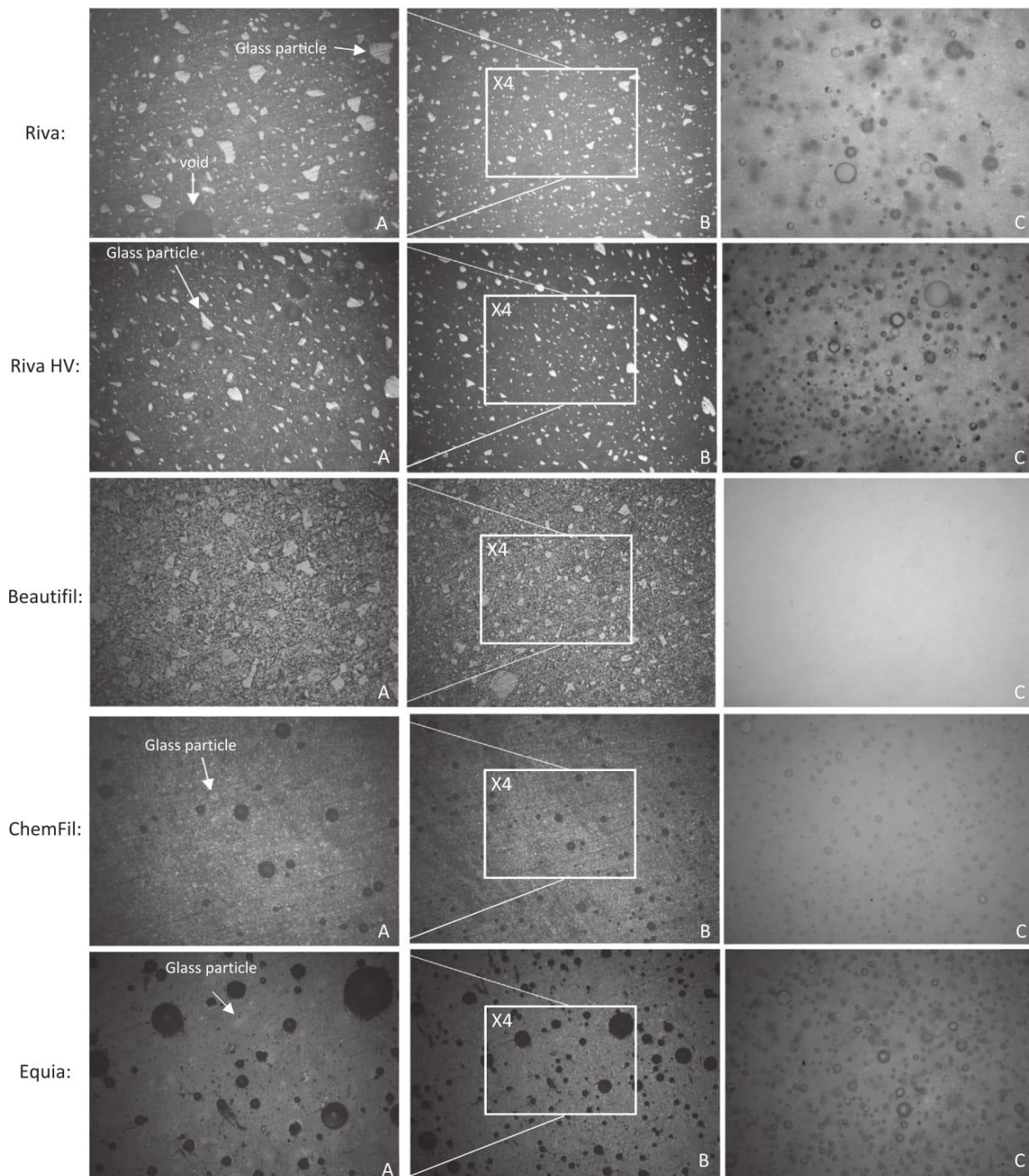


Fig. 4. Microscopic image ($\times 40$) of samples with direct light (A), Microscopic image ($\times 10$) of samples with direct light (B) and inverted reflected method (C) – (Thickness of samples = 1.11 ± 0.08 mm).

mean values of test methods for each material are different, the order of materials based on K_{IC} is the same. So, comparing the results of various studies on fracture toughness for ranking of the materials is possibly a more valid method of comparison than a direct comparison of the absolute K_{IC} values. Consequently, both techniques are believed to be suitable for measuring K_{IC} .

4.2. Storage media

The importance of the storage medium for GICs has been discussed extensively in the literature (Mitchell, 2010; Miyazaki et al., 1996; Serra et al., 1994; Earl et al., 1989, 1985; Nicholson and Czarnecka, 2007; Sadaghiani et al., 2009). However, the effect of artificial saliva on fracture toughness of these materials is a subject that has not been previously studied in depth. The artificial saliva used in this study, excluded mucin to prevent any effects caused by bacterial growth. The

Table 6
K_{IC} (MPa $\sqrt{m}$) Mean value $\pm$ SD for each testing method.

Material	Method	Mean value $\pm$ SD
ChemFil	CT test	0.47 $\pm$ 0.06
	4-point bending	0.41 $\pm$ 0.10
Riva HV	CT test	0.72 $\pm$ 0.10
	4-point bending	0.63 $\pm$ 0.11
Riva	CT test	0.79 $\pm$ 0.08
	4-point bending	0.75 $\pm$ 0.07
Equia with resin coating	CT test	0.72 $\pm$ 0.21
	4-point bending	0.74 $\pm$ 0.14
Equia without resin coating	CT test	0.37 $\pm$ 0.06
	4-point bending	0.32 $\pm$ 0.10
Beautifil	CT test	1.12 $\pm$ 0.13
	4-point bending	1.06 $\pm$ 0.07

ionic content for the chosen solution was similar to saliva and revealed high activity concentrations of ions such as Cl⁻, Na⁺, Ca²⁺ and HPO₄²⁻, on the solubility of the dental materials and in turn on their mechanical properties.

The storage medium showed different effects on individual materials as outlined below (regardless of storage time):

- Chemfil, Riva and Riva HV showed a significant difference between K_{IC} values of samples stored in AS and DW with, DW storage gave higher results.
- Equia with coating showed no significant difference between the two storage media.
- Beautifil and Equia were not significantly different, however, a similar trend as Chemfil, Riva and Riva HV occurred exhibiting higher K_{IC} for the groups stored in DW.

The exception of Equia with coating group was most likely due to the coating material. In previous studies, it was shown that the use of coating materials could affect the physical properties of GIC (Miyazaki et al., 1996; Serra et al., 1994; Earl et al., 1989, 1985; Nicholson and Czarnecka, 2007; Gorseta et al., 2016). A study by Gorseta et al. (2016) showed no significant difference between the flexural strength of GIC samples stored in various storage media with the same coating, which was similar to the findings in the current study. It may, therefore, be concluded that the coating material protected the sample from the effects of artificial saliva that seems to have affected the physical properties of GIC in the non-coated groups. Also, This study results indicated a significant improvement ($P < 0.05$) in K_{IC} due to the presence of coating. This finding suggests that coating material protects the sample from any environment and enhances the fracture toughness of the GIC significantly.

Lower fracture toughness in samples stored in artificial saliva (other than coated groups) might be explained by the differences between ionic content and concentration (i.e. the ion activity) of the two storage media. The presence of ions such as Cl⁻, Na⁺, Ca²⁺ and HPO₄²⁻ in the saturated artificial saliva solution used in this study may have resulted in changes in the surface properties of the GICs by increasing solubility of these materials and decreasing their strength leading to a reduction in their fracture toughness. It has been reported that the presence of calcium and phosphorus ions in saliva results in the inclusion of more calcium in the cement matrix after 10 years of clinical service (Shiozawa et al., 2013; Zanata et al., 2011). Other studies indicated more cracks in the surface, increased surface hardness and a whiter appearance for GICs stored in a 42.7 wt% CaCl₂ solution and saliva compared to water as the storage medium (Shiozawa et al., 2013; Okada et al., 2001). It has been proven that ions such as Ca²⁺ and PO₄³⁻ are absorbed into the GIC matrix and react with the non-reacted carboxylate groups remaining in the cement matrix, which provides an explanation as to the alteration in appearance and surface hardness (Shiozawa et al., 2013; Okada et al., 2001). Also, it has been described

that these changes occur in the subsurface region and are similar to the process of forming an intermediate layer that is attached to both the tooth surface and GIC (Crisp et al., 1976; Ngo et al., 1997). All these reactions and processes may result in reduction of the fracture toughness of these materials.

Given the similar ion content of the AS used in this study and saliva and the difference in the effect of two storage media on fracture toughness mean values of each material, it can be said that the effect of storage media is dependent on the type of material and the presence of a coating layer.

Considering the small difference in fracture toughness mean values of the two storage media, it can be concluded that any effect is likely to be very small in the oral environment. From the results obtained the concern using DW instead of AS in studies such as this is most likely not justified, and therefore the use of DW as a storage medium in future investigations seems appropriate.

It appears that the effect of the coating affords protection from the storage media or surrounding environment, this may be important clinically as it may also protect the GIC surface from other ionic attack and potential surface deterioration. The coating protects the surface of the sample from the storage environment in addition to filling surface defects and reducing solubility. Considering the high ion content of the artificial saliva was used in this study, it can be concluded that coating is a useful and beneficial barrier to protect GICs from the storage medium.

4.3. Storage duration

The 30-days storage period resulted in higher mean fracture toughness values compared to 7-days storage. This is mainly due to the maturation of the GIC. Many studies indicate that the maturation process results in improvement of physical properties of GIC notably in the first 24 h and can continue for as long as one year (Sadaghiani et al., 2009). During a GICs' life, storage medium has a significant role in keeping the material hydrated in order to prevent cracking and crazing of the surface (Sadaghiani et al., 2009; Burrow, 2013).

So the higher mean value for fracture toughness in the 30-days groups confirms the maturation of GICs which may influence restoration success immediately after placement. This result indicates that increased storage time leads to a higher fracture toughness for GICs. Even Beautifil gave a significantly higher fracture toughness value after 30-days storage. There has been a discussion in the literature on the long-term physical properties of resin-based restorative materials (Curtis et al., 2008; Drummond et al., 2004), where it has been reported that the likelihood of hydrolytic degradation may result in softening of the resin matrix due to water sorption (Curtis et al., 2008) and degradation of the silane interface between fillers and matrix (Drummond et al., 2004). Loss of fillers or interface degradation could lead to microcrack formation that may degrade the mechanical properties of materials such as Giomers. However, in this study and other recent studies, this theory was not confirmed (Ilie and Stawarczyk, 2016). It was reported by Ilie and Stawarczyk (2016) that with time the microhardness of Giomers can improve. In addition, in the current study, an improvement in fracture toughness of Beautifil was observed with the longer storage time. This change might be due to the S-PRG structure, where the pre-treated particles may have a protective surface layer that resists degradation.

4.4. Weibull modulus “m”

Weibull test is a common experiential expression of the cumulative probability of survival at a specific K_{IC} (Ilie and Hickel, 2007; Weibull, 1951). As Quinn et al. mentioned, a high modulus, or steep slope of the graph, is associated with a narrow strength distribution. So, materials with high Weibull moduli (m) are more predictable and less likely to fracture and fail at a stress much lower than the mean value (Quinn and

Quinn, 2010a). Higher m values (or steeper slopes) were observed for Beautifil while the lowest was recorded for Equia (Table 5). Consequently, based on the outcomes of this study it seems that Beautifil is likely to provide less variability as a restorative material which may translate to better reliability from the aspect of fracture. Therefore lower m values could indicate that a material or restoration failure due to fracture could occur at lower forces than reported loads at failure. This low reliability in GICs compared to a Giomer may be explained by the fact that strength is a function of the porosity of the material. Also, homogeneity of porosity distribution in the mix with a secondary effect possibly being caused by the particle size distribution and filler content can influence the reliability of the restorative materials (Fig. 4). The location and proximity of voids in the samples may also act as initiation sites for fast crack propagation and a fracture to occur at lower loads than expected. This is one reason why the GICs show lower m value. On the other hand, in a more homogeneous and less porous material such as Giomer, the reliability is higher. Another influential factor is the filler glass particle size and content. The larger size of these particles may increase the fracture toughness value of the material up to a maximum filler volume per cent similar to resin based composites (Ilie et al., 2012) as the crack front would curve or dissipate between particles while a smaller particle size would appear to decrease in the K_{Ic} values (Fig. 4). However, the fracture origins of filled resin were shown to be manifold, including particle agglomerates associated with mixing, wetting, distribution of filler phase, air bubbles, resin rich areas, properties of filler particle (Quinn and Quinn, 2010b). Ash test and Weibull analysis revealed the following trend in filler content and m value:

Filler content wt%: Riva HV > Riva > ChemFil > Equia > Beautifil
 M value: Beautifil > Riva > Riva HV > ChemFil > Equia

As expected, these trends indicate that a higher filler content in GICs results in a higher m value. The exception to this fact is Riva HV. Although this GIC had a higher filler content compared to Riva showed a lower m value. It has been reported before that the powder to liquid ratio can influence the porosity of GICs (Fleming et al., 2003; Torabzadeh et al., 2011). So, this change in ranking can be explained by the consistency of this material. The high viscosity or higher powder to liquid ratio increases the chance of producing bubbles and voids after mixing. Fig. 4 also illustrates the more porous nature of Riva HV compared to Riva.

Considering the results of this study the use of GICs in load-bearing areas needs careful consideration. The Weibull moduli are low for GICs compared with resin composites (Rodrigues et al., 2008), which results in less reliability of their fracture toughness values and consequently, it is possible that restoration failure due to fracture is likely to occur at lower loads than the reported mean values. Comparing the reported Weibull moduli from previous studies on resin composites indicates higher average results in contrast to the lower m values of GICs. (Rodrigues et al., 2008) Hence, from laboratory evidence, it would seem more suitable to choose alternative materials than GICs in load bearing areas, such as Giomer or resin composites which show greater reliability of failure under higher loads.

5. Conclusion

In conclusion, the results related to materials stored at 30-days demonstrated a significantly higher mean fracture toughness compared to 7-days storage. On the other hand, results of this study showed no significant difference between fracture toughness values in different storage media. Trends suggest a higher value in DW groups compared to AS storage, however, this may relate to the AS formula used. Groups with coated specimens did not follow this trend, indicating that coating GIC will provide some protection. Either K_{Ic} test method seems suitable to evaluate K_{Ic} values for materials such as GICs.

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Chapter 6: Project III- Change in temperature and polymerisation stress of resin composites related to irradiance protocol from light curing units

6.1: Introduction:

Dental resin composites are becoming the most common direct restorative materials used clinically since the Minimata Treaty (1) was implemented. However, polymerisation shrinkage and the effect it may have on bonded surfaces in a cavity, including the potential for gap formation remains a concern. (2) Where there is little tooth compliance, polymerisation shrinkage stress at these surfaces can remain after curing as a residual shrinkage stress, (3, 4) such that a restored tooth remains under stress even when there is no functional load on it. This stress is capable of causing marginal gap formation leading to microleakage that may create an environment suitable for recurrent caries initiation which is one of the main failure reasons of posterior restorations. (5-7) Also, polymerisation stress can contribute to the formation of enamel crazing, postoperative pain and cuspal flexure. (3, 8)

The magnitude of polymerisation shrinkage stress of resin composites is influenced by many properties such as filler content, matrix chemistry, degree of conversion and polymerisation kinetics. (3, 9-13) Many approaches have been introduced to reduce shrinkage stress based on the following concepts; modifying material formulation, different placement techniques and slow start/ramped curing. (14-20) Changes in the formulation that reduced polymerisation shrinkage were to increase filler content with nanometre-sized fillers (16, 17) or increasing the monomer molecular weight by replacing triethylene glycol dimethacrylate (TEGDMA) with urethane dimethacrylate (UDMA) and bisphenol-A polyethylene glycol diether dimethacrylate (Bis-EMA). (3, 18) TEGDMA has a relatively low molecular weight which results in a higher number of double bonds per unit of weight and creates a high degree of cross-linking and a greater shrinkage. (21, 22) The improvement in the resin matrix

has led to an increased light penetration depth which made the bulk placement of materials possible. Hence, the introduction of the so-called 'bulk-fill' resin composites. The thickness of the resin-bulk when restoring a cavity can vary between 4 to 5 mm depending on the material, with some requiring a surface veneer layer with a conventional resin composite. (21, 23, 24) A matrix resin which was suggested to have lower polymerisation contraction is the organic modified ceramic (ORMOCER). Ormocer matrix-based composites are hybrid polymers containing an organic-inorganic bond. It has been reported that the reduced amount of organic resin reduces polymerisation shrinkage. (14, 25)

Research has focussed on differences between curing light (spectrum) and irradiance, with most studies finding that Quartz-Tungsten-Halogen (QTH) curing lights produced an inferior result for resin-based material polymerisation compared with recent Light Emitting Diode (LED) curing lights. This is partly due to the higher output intensity (irradiance) of LED curing units at the required wavelength. (26) Several curing methods for polymerisation have been incorporated into LED curing units such as the ramp or soft-start approach which is based on a short, low intensity (irradiance) light application that is ramped up to the maximum light intensity at the final exposure. (27) It has been suggested that these methods can reduce the rate of shrinkage and temperature increase of the restorative material during polymerisation. (28-30) Since the light intensity (irradiance) only increases gradually to the maximum output, the result is a slower activation of the radicals associated with the conversion of the monomer to polymer thus prolonging the 'gel stage' of polymerisation. (31-34) However, this ramp mode extends the chairside time, which in some circumstances may not be feasible. The new light curing devices with high irradiance are presented as a means to reduce the chairside time for polymerisation by reducing the curing time to 3 seconds. However, the high irradiance light radiation raises concerns regarding the effect on polymerisation shrinkage of resin composites and temperature increases during polymerisation.

Dental resin composites expand with increased temperature, but like any other thermal expansion, this dimensional change will revert as soon as cooling occurs. The difference between the thermal coefficient of expansion of dental resin composites and tooth structure is thought to be a contributory

factor of microleakage. (35, 36) The greatest change in temperature for a bulk of resin composite is typically during polymerisation and exposure to the curing light radiant energy output. Resin composite polymerisation is an exothermic reaction causing an increase in temperature within the material. Also, light curing units produce thermal energy in addition to the light energy. As a result, the initial polymerisation shrinkage occurs simultaneously with an expansion caused by the temperature increase in the material. Depending on the rates of expansion and contraction this overlap will cause a compensation in dimensional changes. This dilemma becomes more influential when the resin composite has a greater bulk. Bulk volumes of resins produce more heat due to the increased mass of the polymerisation reaction, and the heat may stay *in situ* for longer due to the volume of the material. In the small incremental placement technique recommended for conventional composites, heat dissipation occurs more quickly, and the restoration equilibrates to the surrounding temperature faster. As a result, the heat generated becomes a critical consideration for the success of the restoration. The phenomenon of compensation due to heat differentials has rarely been considered in previous studies. This gap in the literature and oversight may cause an underestimation of the stresses present along tooth-restoration bonded interfaces at the time of polymerisation and after a material cools.

The aim of this study was to simultaneously investigate core temperature and shrinkage stress of bulk-fill and conventional resin composites exposed to four different irradiance protocols.

6.2: Methods and materials:

6.2.1: Polymerisation stress test and thermal analysis:

Five resin composite restorative materials were used in this study, their type and composition are outlined in Table-9. In this experiment in the 1.8 mm curing thickness category, one further material

was tested (SDR). Due to the low viscosity of this material, the preparation of samples with 5mm curing thickness was not possible, for the selected C-factor.

A total of one hundred and thirty-two specimens were prepared in 44 groups based on material, curing thickness and light curing irradiance. (n=3)

Table-9 Specification of materials.

Type	Product	Name	Manufacturer	Lot #	Shade	Composition
Universal composite	light cured radiopaque universal composite	Kalore	GC	1604261	A2	DX-511 monomer, di-UDMA, DM(37) Urethane dimethacrylate (UDMA) – ethoxylated bisphenol-A dimethacrylate (Bis-EMA) – dimethacrylate – butylated hydroxytoluene (BHT) – Uv light absorber – stabilizer (38)
Bulk-fill composite	highly filled bulk-fill composite	SonicFill 2	Kerr	6120927	A2	BisGMA, TEGDMA, BisEMA(39) Silicon dioxide – glass, oxide, chemicals – Zirconium compound (as Zr) – Ytterbium trifluoride (40)
Universal composite	universal Nano-ceramic restorative	Ceram X	DENTSPLY	1611000471	A2	Methacrylate modified polysiloxane (organically modified ceramic)(ORMOCER) - Dimethacrylate resins (UMA, Bis-EMA, TEGDMA) - Fluorescent pigment - UV stabilizer - Stabilizer - Camphorquinone - Ethyl-4(dimethylamino) benzoate - Bis-(4-methyl-phenyl) -iodonium hexafluorophosphate - Barium-aluminum-borosilicate glass - Ytterbium fluoride - Iron oxide pigments and titanium oxide pigments according to shade (41, 42)
Anterior composite	light cured restorative composite	Clearfil Majesty ES-2	Kuraray Noritake Dental Inc	C20029	A2	bisphenol A di glycidyl methacrylate - Silanated barium glass filler - Pre-polymerized organic filler -Hydrophobic aromatic dimethacrylate - Hydrophobic aliphatic dimethacrylate - dl-Camphorquinone - Accelerators - Initiators -Pigments (43)
Bulk-fill composite	bulk-fill ultra-universal restorative composite	Aura	SDI	170170	Universal	acrylic monomers as: diurethane dimethacrylate - triethylene glycol dimethacrylate - 2,2-bis[4-(2-methacryloxy) ethoxy] phenyl] propane(44)
Flowable composite	posterior bulk-fill flowable base	SDR	DENTSPLY	1610131	Universal	Modified UDMA, BisEMA, TEGDMA (37) Polymerisable dimethacrylate resins - Polymerisable tri methacrylate resins - Polymerisable urethane dimethacrylate - Colourants are synthetic inorganic iron oxides. (45)
Bonding agent	universal self-etch adhesive	Clearfil SE Bond 2	Kuraray Noritake Dental Inc	7J0011	--	bisphenol A di glycidyl methacrylate - 2-hydroxyethyl methacrylate - 10-Methacryloyloxydecyl dihydrogen phosphate - Hydrophobic aliphatic dimethacrylate - Colloidal silica - dl-Camphorquinone - Initiators -Accelerators(46)

The light curing devices and the polymerisation method used are listed below:

1. LED curing light continuous (LED C) 1500 mW/cm², Radii Plus+, SDI Limited, Bayswater, Australia, (wavelength 440-480nm wavelength peak wavelength 460nm),
2. LED curing light with ramp (LED R) 1500 mW/cm², Radii Plus+, SDI Limited, Bayswater, Australia, (wavelength 440-480nm wavelength peak wavelength 460nm)
3. QTH curing light continues (QTH) 560 mW/cm², XL 3000, 3M ESPE, Seefeld, Germany, (wavelength 400-500nm wavelength peak wavelength 470nm) and
4. LED curing light seven by 3-second periods with a 2-second interval between exposures (HI) 3200 mW/cm², Valo cordless, Ultradent Products Inc. UT, USA, (wavelength 395-480nm wavelength peak wavelength 465nm, 445nm and 405nm).

Two pairs of polymethylmethacrylate rods, 30 mm in length, were prepared. One pair had a diameter of 1.8 mm whilst the other was 5.0 mm. One surface of each rod was roughened with 1200-

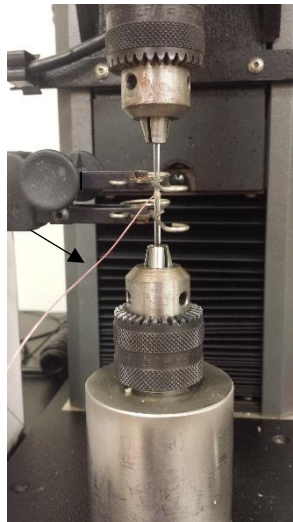


Figure-20 1.8 mm diameter rods with maintained distance (1.44 mm) using extensometer (E) and inserted thermocouple to monitor temperature (arrow)

grit abrasive paper to ensure good adhesion between the rods and restorative material. A pair of rods with the same diameter was fixed to a universal testing machine (Model 5942; Instron, Norwood, MA).

The abraded surfaces of the rods were coated with a resin adhesive (Clearfil SE Bond 2, Adhesive, Kuraray Noritake Dental Inc. Tokyo, Japan) and light cured as per manufacturer's instructions. The restorative material was placed on the lower rod and distance of 1.44 mm for 1.8 mm diameter rods and distance of 4.00 mm for 5.0 mm diameter rods (Configuration-factor = 0.62) was achieved by manual adjustment (Fig-20).

Excess resin composite was removed using a plastic instrument, so the surface remained flush with the rod circumference. The distance between the rod ends was maintained with the aid of a linear strain gauge extensometer linked to the testing machine software (Bluehill3 Materials Testing Software; Instron, Norwood, MA). A fine thermocouple wire (Precision Fine Bare Wire Thermocouple Type J, IRCO-003, OMEGA) was inserted into the uncured resin composite (Fig-20) and output was recorded with NI SignalExpress software (NI-DAQmx, NI SignalExpress, 2014, V 14.0.0 National Instruments) via conditioning module (Hi-speed USB Carrier (NI USB-9162) and module (NI cRIO-9211), National instruments). After 80 s recording time to stabilise the temperature, the resin composite sample was light cured from one side using one of the four irradiances described. Hence a curing thickness of 1.8 mm, which meets the maximum requirement of 2 mm layer for conventional resin composites, and a, 5 mm curing thickness, which is recommended for use with most bulk-fill composites, was used. The sample was exposed for 40 s, with the distance between the light tip and

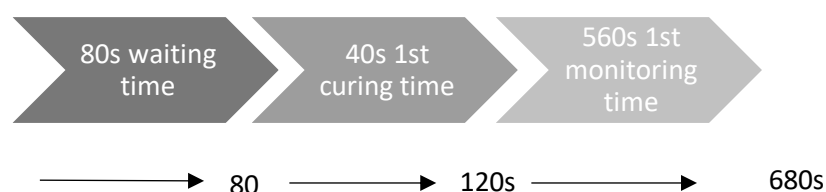


Figure-21 Test Timeline and process for each sample

composite surface kept at approximately 0.5 mm. The radiant energy for LED R, LED C, QTH and HI light units for this duration AS indicated by the factory was as followed respectively: 63.75J/cm², 60 J/cm², 22.4 J/cm² and 67.2 J/cm². The signal from the thermocouple and universal testing machine were monitored simultaneously and recorded for 600s, during and after curing. (Fig-21)

- A. The **final polymerisation shrinkage stress (Final δ_{pol} (MPa))** of materials was calculated using equation-1 (Table-10) (Fig-22).

$$\text{Final } \delta_{pol} = \frac{F_{Final}}{\pi r^2} \quad \text{Equation-1}$$

- B. The **core temperature** changes of the samples were reported in two categories:

1. The **maximum temperature (Max T (°C))**
2. The maximum changes in temperature (ΔT (°C)) (Table-10) (Fig-23), equation 2.

$$\Delta T = \text{Max } T - \text{room } T \quad \text{Equation-2}$$

All the above values were calculated for each sample for the 600s monitoring period.

6.2.2: Filler content (Standard Ash test):

The filler content of each sample was determined using a standard ash test following the method described in ISO 1172:1996. (47) Disk-shaped (12 mm $\varnothing$ X 1.20 $\pm$ 0.01 mm thick) samples were prepared and weighed (Precisa Gravimetrics AG, CH-8953 Dietikon, Switzerland) to a precision of $\pm$ 0.0001 g. The samples were heated in an electric furnace (Combilabor CL-V, Heraeus, Hanau, Germany) to a temperature of 625°C maintained for 30 minutes to burn out the organic matrix of the composite. After cooling to room temperature, samples were placed in a vacuum chamber containing silica gel for 12 hours then re-weighed, and the inorganic weight fraction (wt%) was calculated using equation-3. The volume of the sample was measured using a pycnometer for calculating the density (ρ_c) of each material and inorganic volume fraction (vol%).

$$Filler\ wt\% = \frac{W_1}{W_0} \times 100\% \quad \text{Equation-3}$$

$$Filler\ vol\% = \frac{\frac{V_1}{\rho_C}}{\frac{W_0}{\rho_C}} \times 100\% \quad \text{Equation-4}$$

(W_1 = Weight of the sample after ashing(g), W_0 = weight of the sample before ashing(g), V_1 = Volume of the sample after ashing (mm³), ρ_C = density of the related resin composite(g/mm³))

6.2.3: Statistical analysis:

Final δ pol, Max T and ΔT of each sample were calculated. Mean values of each group in each category were calculated and arranged in table-10. Data were analysed using ANOVA, Bonferroni, Tukey and Kruskal Wallis tests ($\alpha = 0.05$). (SPSS, V23, IBM).

6.3: Results:

Table-10 Calculated Mean value $\pm$ SD

	$\Delta T(^{\circ}C)$	Max T($^{\circ}C$)	Final δ pol (MPa)	$\Delta T(^{\circ}C)$	Max T($^{\circ}C$)	Final δ pol (MPa)
	Mean value $\pm$ SD	Mean value $\pm$ SD	Mean value $\pm$ SD	Mean value $\pm$ SD	Mean value $\pm$ SD	Mean value $\pm$ SD
	1.8mm depth of cure			5mm depth of cure		
Kalore						
QTH	14.12 $\pm$ 1.15 ^a	37.50 $\pm$ 1.12 ^a	1.62 $\pm$ 0.19 ^a	16.05 $\pm$ 1.85 ^a	39.89 $\pm$ 1.80 ^a	2.40 $\pm$ 0.22 ^a
LED C	18.29 $\pm$ 2.40 ^a	41.41 $\pm$ 2.67 ^a	2.39 $\pm$ 0.41 ^a	14.37 $\pm$ 2.97 ^a	38.07 $\pm$ 3.06 ^a	2.06 $\pm$ 0.88 ^a
LED R	15.84 $\pm$ 3.74 ^a	39.03 $\pm$ 3.79 ^a	2.12 $\pm$ 0.40 ^a	14.94 $\pm$ 0.70 ^a	39.43 $\pm$ 1.31 ^a	2.25 $\pm$ 0.32 ^a
HI	54.95 $\pm$ 9.05 ^b	79.04 $\pm$ 9.10 ^b	4.84 $\pm$ 0.58 ^b	35.73 $\pm$ 3.92 ^b	58.99 $\pm$ 3.89 ^b	5.35 $\pm$ 0.35 ^b
Sonicfil I						
QTH	14.46 $\pm$ 1.54 ^a	38.58 $\pm$ 1.39 ^a	2.91 $\pm$ 0.26 ^a	17.10 $\pm$ 1.68 ^a	40.63 $\pm$ 1.52 ^a	4.66 $\pm$ 0.21 ^{a,b}
LED C	14.70 $\pm$ 3.19 ^a	37.75 $\pm$ 2.90 ^a	2.52 $\pm$ 0.34 ^a	19.36 $\pm$ 1.23 ^a	43.34 $\pm$ 1.38 ^a	4.18 $\pm$ 1.66 ^{a,b}

<i>LED R</i>	13.93±1.89 ^a	37.11±2.16 ^a	2.94±0.34 ^a	15.78±1.42 ^a	39.31±1.51 ^a	3.70±0.43 ^a
<i>HI</i>	52.40±4.68 ^b	75.86±4.85 ^b	4.28±0.75 ^b	35.64±5.24 ^b	59.27±5.32 ^b	6.42±0.41 ^b
Ceram X						
	ΔT(°C)	Max T(°C)	Final δpol (MPa)	ΔT(°C)	Max T(°C)	Final δpol (MPa)
	Mean value ± SD	Mean value ± SD	Mean value ± SD	Mean value ± SD	Mean value ± SD	Mean value ± SD
	1.8mm depth of cure			5mm depth of cure		
<i>QTH</i>	14.83±3.00 ^a	37.07±2.78 ^a	3.97±0.47 ^a	21.47±1.40 ^a	44.86±1.60 ^a	4.73±0.75 ^a
<i>LED C</i>	17.87±3.21 ^a	40.64±3.10 ^a	3.12±0.32 ^a	19.17±1.38 ^a	43.54±1.21 ^a	4.01±0.97 ^a
<i>LED R</i>	14.77±0.41 ^a	38.29±0.69 ^a	3.04±0.30 ^a	17.44±0.85 ^a	41.03±0.50 ^a	3.49±0.66 ^a
<i>HI</i>	57.93±2.15 ^b	81.60±2.31 ^b	5.23±0.54 ^b	37.89±4.67 ^b	61.76±5.03 ^b	7.37±0.57 ^b
Clearfil						
<i>QTH</i>	13.54±2.78 ^a	36.63±3.30 ^a	2.94±0.22 ^a	15.55±1.01 ^a	38.69±1.04 ^a	3.22±0.12 ^a
<i>LED C</i>	17.86±2.71 ^a	41.12±2.10 ^a	2.89±0.43 ^a	16.77±0.43 ^a	41.17±0.44 ^a	2.89±0.60 ^a
<i>LED R</i>	17.83±1.56 ^a	41.53±1.75 ^a	2.37±0.20 ^a	14.95±0.81 ^a	38.59±0.95 ^a	2.54±0.68 ^a
<i>HI</i>	51.93±3.87 ^b	75.60±4.11 ^b	4.52±0.45 ^b	34.33±3.66 ^b	58.31±3.77 ^b	6.01±0.29 ^b
Aura						
<i>QTH</i>	13.22±2.13 ^a	36.04±1.84 ^a	2.63±0.17 ^a	18.32±0.75 ^a	42.56±0.79 ^a	4.28±0.24 ^{a,b}
<i>LED C</i>	15.06±2.21 ^a	38.20±1.87 ^a	2.92±0.46 ^a	19.40±1.82 ^a	43.75±1.86 ^a	4.69±0.67 ^{a,b}
<i>LED R</i>	14.99±2.00 ^a	38.49±1.50 ^a	2.47±0.55 ^a	16.24±3.74 ^a	40.15±3.58 ^a	3.62±1.02 ^a
<i>HI</i>	51.96±1.32 ^b	75.42±1.19 ^b	4.92±0.30 ^b	36.75±0.85 ^b	60.65±0.59 ^b	5.95±0.97 ^b
SDR						
<i>QTH</i>	19.99±4.60 ^a	42.01±4.89 ^a	1.91±0.72 ^a	---	---	---
<i>LED-C</i>	21.97±1.45 ^a	44.52±0.77 ^a	2.55±0.95 ^{a,b}	---	---	---
<i>LED-R</i>	19.83±7.74 ^a	42.40±7.73 ^a	2.54±0.31 ^{a,b}	---	---	---
<i>HI</i>	40.54±0.58 ^b	62.96±0.48 ^b	4.18±0.27 ^b	---	---	---

* a,b,c=Within each material those groups with the same superscript are not significantly different

6.3.1: Final polymerisation shrinkage stress (Final δ pol)

The final polymerisation stress (δ pol) after the 600 sec measurement period for each material revealed significantly different values among the irradiance protocols. The highest value for δ pol occurred for the HI irradiance for all materials while the lowest δ pol varied among the other three curing irradiances.

QTH was the only curing irradiance that showed significant differences ($p < 0.05$) between materials for the 1.8 mm curing thickness. However, for the 5 mm curing thickness QTH and HI showed a significant difference ($p < 0.05$). The highest final δ pol value was observed in Ceram X for all curing irradiances at the 1.8 mm specimen thickness, but for only HI and QTH at 5 mm thickness. The lowest value for δ pol was obtained for Kalore in all curing irradiances with the exception of the HI at 1.8 mm where SDR displayed the lowest final δ pol. (Table-10) (Fig-22).

For the 5 mm sample thickness, higher mean values of Final δ pol were observed for each material compared to 1.8 mm sample thickness.

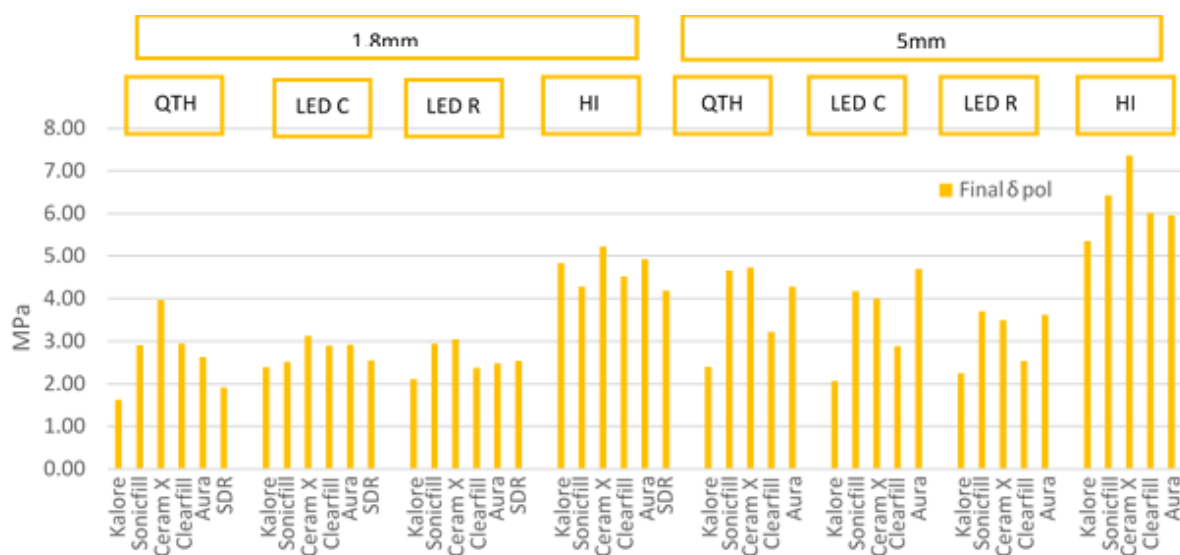


Figure-22 Final δ pol mean values (MPa)

6.3.2: Core temperature:

Max T and ΔT :

Both temperature subgroups (Max T and ΔT) showed significantly different values ($p < 0.05$) between curing irradiances for each material, excluding SDR. The highest temperature for each material was observed in the HI irradiance whilst the lowest varied for each material, with the lower irradiance condition.

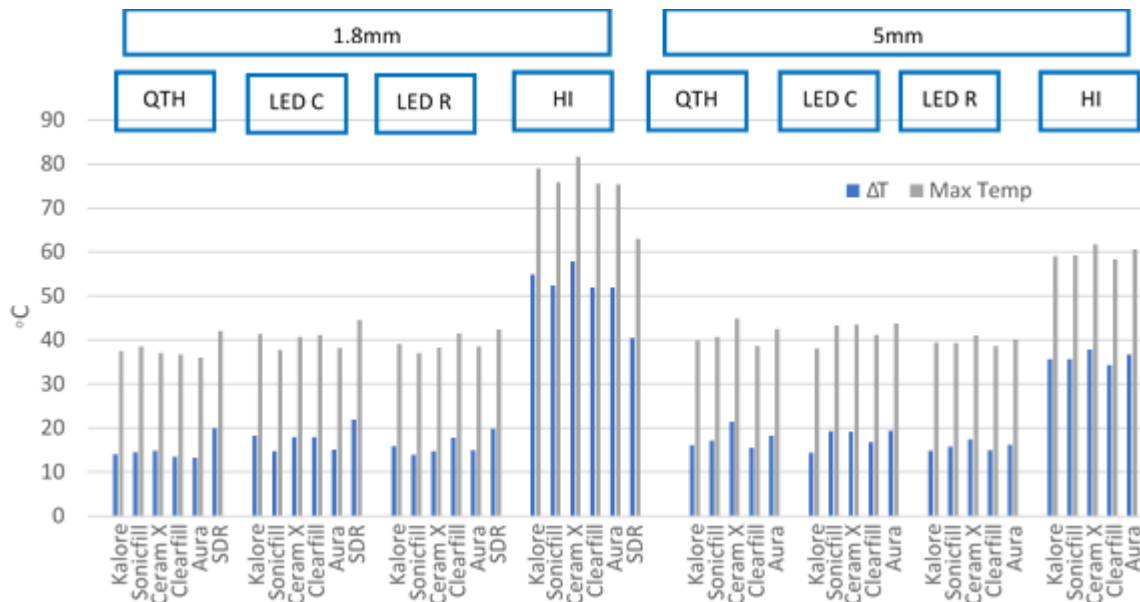


Figure-23 Core temperature mean values (°C)

Among all materials, results of the highest temperature values, Ceram X exhibited the largest differences. However, the least changes in temperature varied between 1.8 mm and 5 mm specimens' thicknesses (respectively: Aura, Kalore) (Table-10) (Fig-23).

The only polymerisation method that showed a significant difference ($p < 0.05$) in temperature categories between materials in 1.8 mm thickness was LED-C. SDR produced the maximum value in temperature categories for each protocol. The exception was the HI, between all materials, the

highest temperature values for this protocol was reported for Ceram X and the lowest for SDR (Table-10) (Fig-22).

The recommended exposure time for the HI curing unit is no more than 2 exposures of 3 seconds each. However, in order to obtain a similar radiant energy exposure, the same curing times as the other light curing irradiances required seven exposures. Nevertheless, the data were also recorded for the temperature change from the first 2 exposures of 3 seconds which are presented in table-11. The trends for HI in relation to maximum temperature and temperature change among tested composites were the same as the 40s exposure time, with largest values recorded for Ceram X and smallest varied depending on the sample thickness (1.8mm: SDR, Aura and 5mm: Kalore).

TABLE-11 MAX T, ΔT AND HE OF HI METHOD AFTER ONLY 2 CYCLES.

HI (MEAN VALUE)	1.8mm depth of cure		5mm depth of cure	
	Max T(°c)- 2 cycles	ΔT (°c)- 2 cycles	Max T(°c)- 2 cycles	ΔT (°c)- 2 cycles
	61.83±1.51	38.16±1.58	47.83±3.14	24.10±3.01
KALORE	61.26±3.70	37.16±3.65	43.60±2.39	20.34±2.37
SONICFILL	60.75±1.70	37.30±1.55	49.94±2.21	26.31±2.20
CERAM X	63.83±2.62	40.15±2.59	50.61±3.39	26.74±3.06
CLEARFIL	60.31±3.75	36.65±3.52	45.35±0.90	21.37±0.81
AURA	63.01±1.46	39.56±1.55	49.66±1.18	25.75±1.45
SDR	56.79±2.43	34.37±2.19	---	---

In the 5 mm sample thickness, lower mean values of maximum temperature and temperature change were seen for each material compared with the 1.8 mm thickness.

6.3.3: Filler content (Standard Ash test):

The calculated Wt % and Vol % for filler content of the tested resin composites are shown in

Table-12. Vol% in ascending order is:

TABLE-12 WT % AND VOL % FOR FILLER CONTENT OF RESIN COMPOSITES.

Ceram X < SDR < Kalore < Aura < Clearfil
< Sonicfill. Whereas Wt % in ascending
order is: Clearfil < SDR < Kalore < Ceram
X < Sonicfill < Aura.

MATERIAL	Wt% filler (Mean ± SD)	Vol% filler (Mean ± SD)
KALORE	69.66±0.06	53.67±1.29
SONICFILL	71.50±0.076	62.08±2.63
CERAM X	70.19±0.08	51.30±0.48
CLEARFIL	58.61±0.19	57.85±2.86
AURA	72.42±0.01	54.56±1.32
SDR	64.66±0.26	51.76±2.22

6.4: Discussion:

6.4.1: δ pol:

The mean shrinkage stress (δ pol) ranking for the resin composites tested for all the curing irradiances with the 1.8mm thickness in descending order is Ceram X > Aura > Clearfil > Sonicfill > SDR>Kalore. For the 5 mm thickness the order is Ceram X > Sonicfill > Aura > Clearfil > Kalore, with Sonicfill the only material that changed (SDR not tested). Most materials showed a significant effect on stress when the high-intensity curing irradiance was used. The highest mean stress values for δ pol was observed for the HI irradiance, and whilst the lowest differed for each material. The significantly higher stress values recorded for the HI irradiance are related to the high intensity output of the curing light which was reported in previous studies(18, 20, 26). The high intensity light causes a rapid polymerisation and stiffness increase that cannot be compensated by the flow of the stressed resin composite structure. A greater volume or bulk of the resin composite results in a higher stress. This effect was generally observed in the current study as a larger difference between polymerisation shrinkage stress resulting from the HI irradiance and the other low irradiances (intensity) in the 5 mm thickness samples, which have a greater volume. The lowest values of δ pol for Aura, Clearfil, Ceram X were observed when samples were cured using the LED R irradiance protocol. When comparing the

results of 5 mm and 1.8 mm sample thicknesses, the ranking of Sonicfill for polymerisation shrinkage rose to a higher level. Also, a larger difference was seen between the final δ_{pol} of bulk-fill materials with the change in sample thickness compared with the conventional resin composites. In other words, a greater volume of material results in a higher final δ_{pol} which is more noteworthy when the material is a bulk-fill resin composite. This difference may be due to the reduced monomer to polymer conversion for the conventional resin composites for the 5 mm thickness. Among the conventional resin composites, a greater difference in final δ_{pol} between two curing thicknesses was seen for Ceram X, which revealed similar final δ_{pol} values to the bulk-fill resin composite in the 5 mm thickness and significantly higher values in the 1.8 mm thickness. As a universal resin composite, this material is possibly under polymerised in the 5 mm thickness, and this is the reason for the lower ranking of this material in final δ_{pol} in 5mm thickness.

In the 1.8 mm sample thickness a drop in the values were seen with HI irradiance during the curing period. These results are believed to be due to the higher thermal expansion from the heat produced from the light exposure and rapid polymerisation reaction. During HI irradiance in this sample thickness, sufficient initial curing (stiffness) has taken place to allow stress to build up and allow the thermal expansion to exceed the polymerisation shrinkage and show a negative value for stress (i.e. expansion) (Fig-24&25).

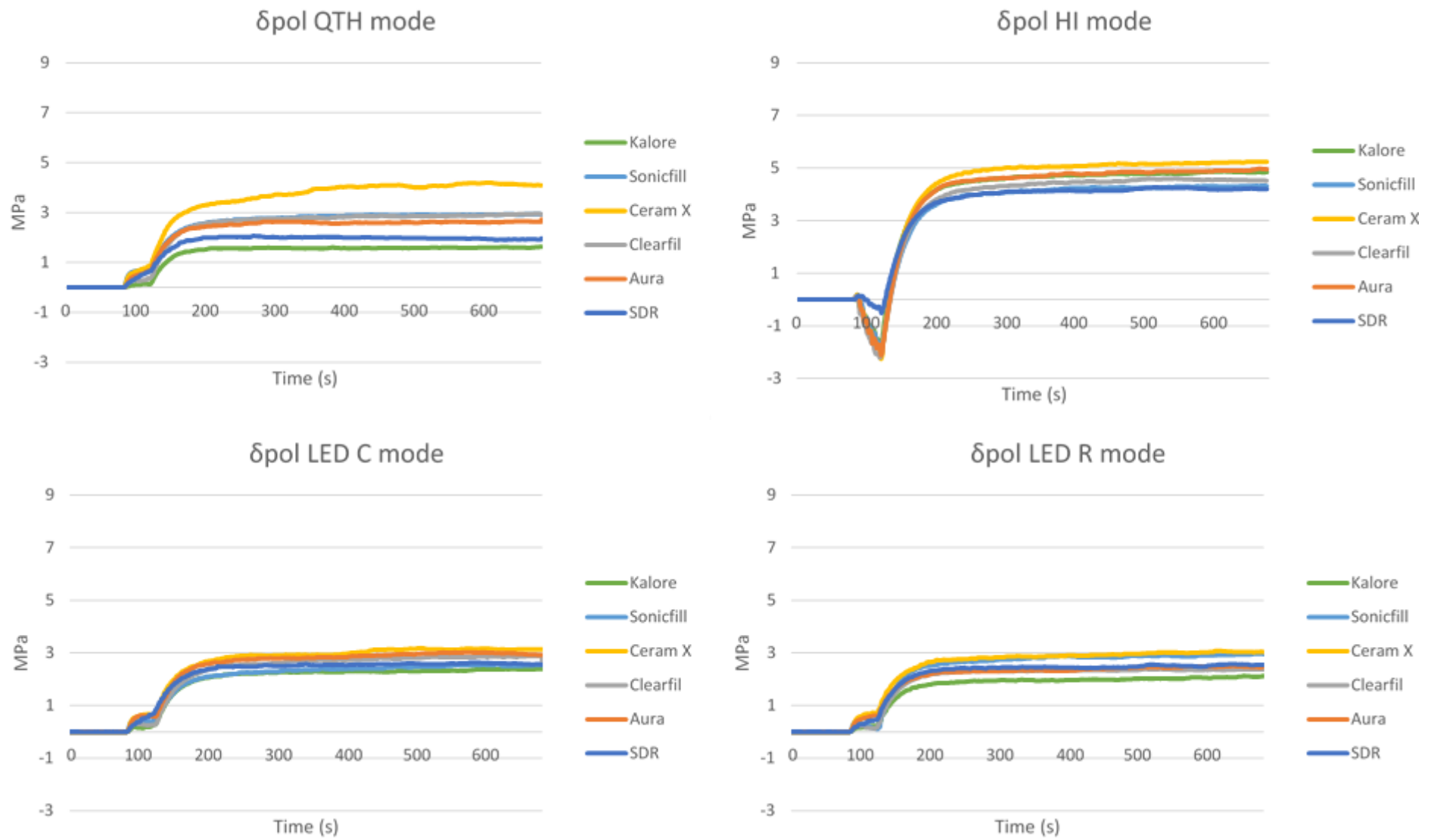


Figure-24 δ pol during the testing time for each sample (1.8mm depth of cure)

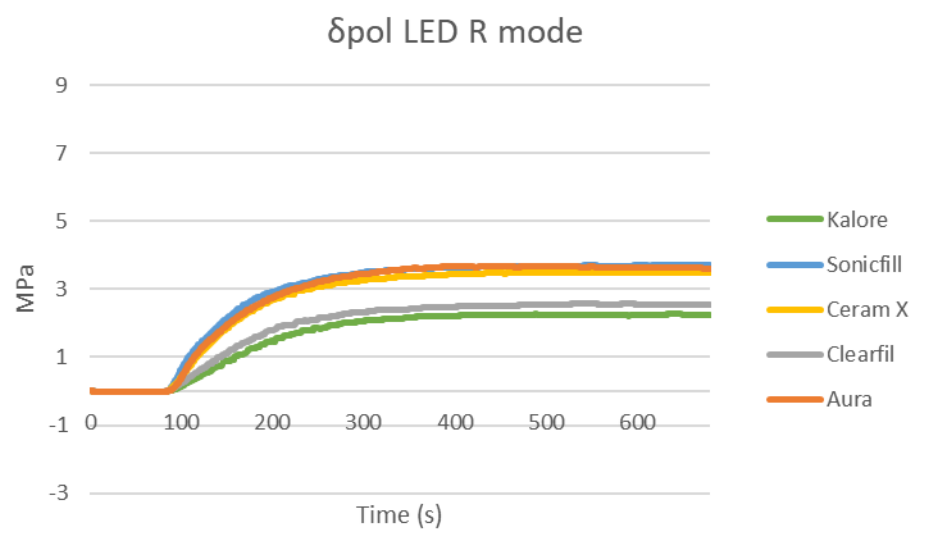
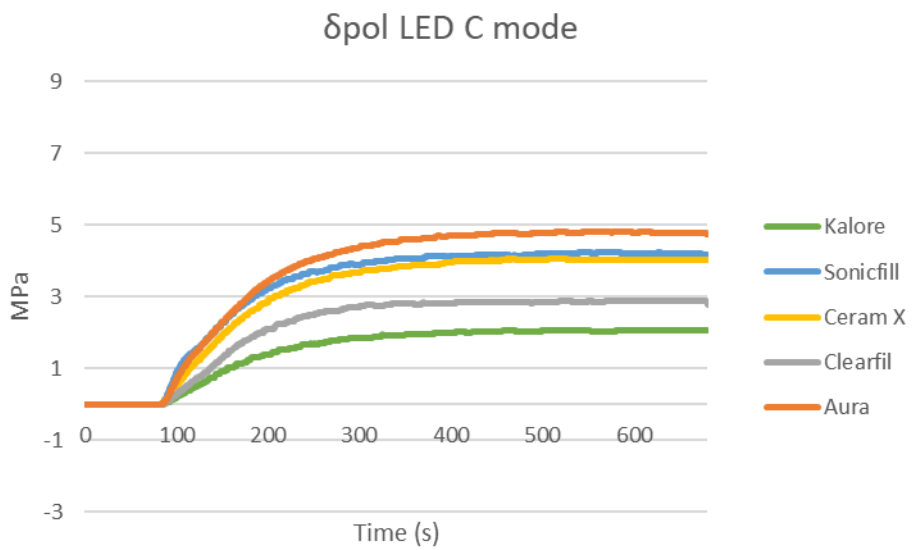
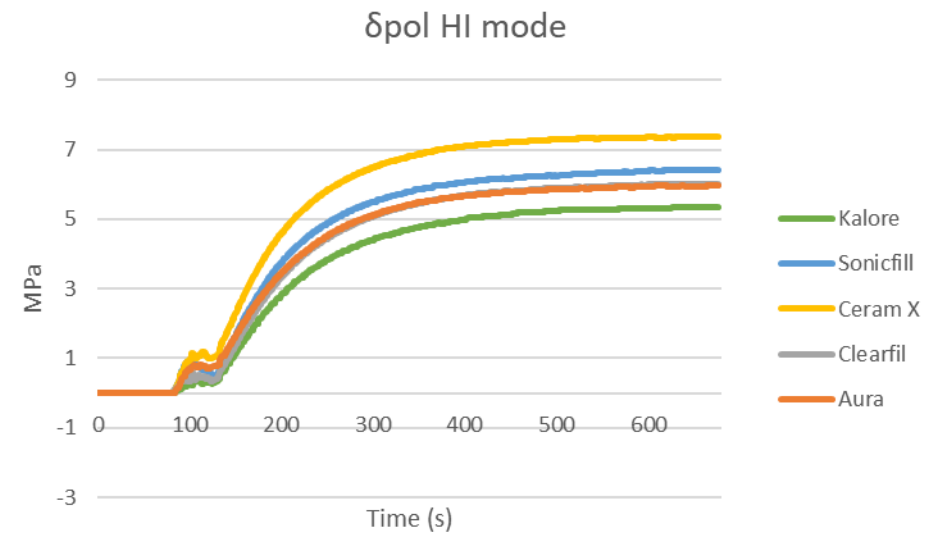
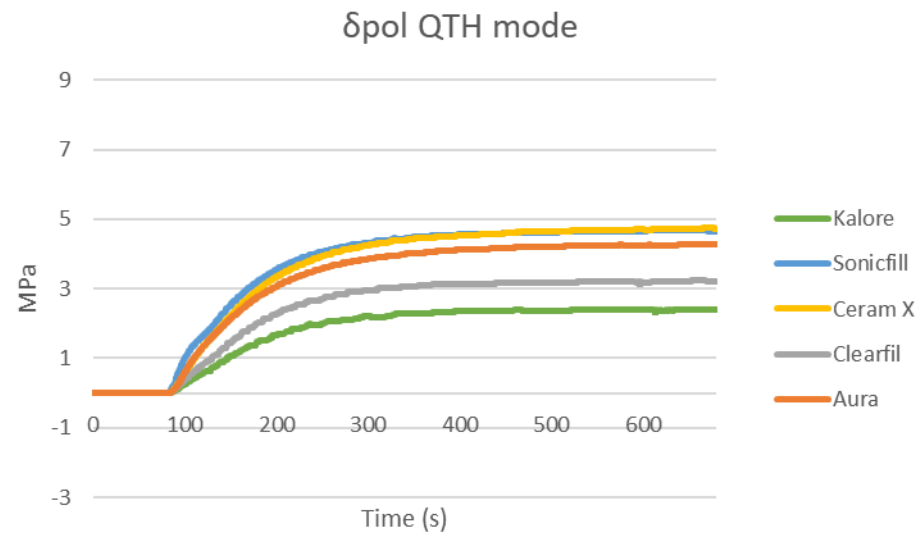


Figure-25 δpol during the testing time for each sample (5mm depth of cure)

QTH was the only irradiance that showed a significant difference in final δpol for the materials tested in both thicknesses. QTH has the lowest irradiance and radiant energy. Also, a correlation between the mean values of final δpol and Max T in the HI group is another indication that output irradiance and radiant energy have an influence on final δpol , which was possible to calculate due to the new method that was used in this study of measuring the temperature change and polymerisation shrinkage stress simultaneously. The HI irradiance generates a greater photothermal component due to its high irradiance and a greater rate of formation of free radicals increasing the rate of polymerisation which increases the temperature and rate of diffusion of a greater number of free radicals produced in the bulk of the resin composite. The increase in resin composite thickness results in an increase in δpol given that maximum curing has been achieved in the resin composite. This maximum degree of conversion is expected to occur in the 1.8mm thickness but not for 5 mm thickness where there may be a material dependence between bulk fill and conventional resin composites. This material dependence may also be reflected at low irradiance and radiant energy values for QTH unit where the degree of conversion may not be optimised.

In previous studies, a reduction in shrinkage using the soft start curing protocol for polymerisation of resin composites has been confirmed (27-29). The slow initial cure of LED R was aimed to reduce shrinkage based on the gradual increase in the output intensity of the light curing unit, thus allowing time for the resin composite to compensate against ongoing shrinkage of polymerisation by allowing the unpolymerised (gel stage) monomer to flow and relieve internal stresses. Ranking of Mean δpol values in this study for curing units, for all the materials with a 1.8 mm thickness in descending order is HI, LED C, QTH and LED R. However, with the 5 mm thickness LED C was noted to have a stronger influence in stress reduction, with mean δpol values in descending order changed to HI, QTH, LED R and LED C for the larger thickness.

The ranking of mean δpol values varied for each material. The common trend was that all materials exhibited the highest δpol value when polymerised

with the HI irradiance regardless of sample thickness. Consequently, it was concluded that this irradiance and irradiant energy results in the highest polymerisation shrinkage stress regardless of resin composite and material bulk volume, as the energy exposure for LED-R, LED-C and HI were similar (63.75J/cm², 60 J/cm², and 67.2 J/cm² respectively).

The difference in ranking of materials between Wt% and Vol% of filler content is due to the various densities(ρ) of the fillers. A lower Vol% filler content indicates a greater Vol% of resin which may lead to greater shrinkage and as a result a higher δ pol. A higher mean value for Ceram X was observed in the final δ pol. Since Ceram X contains pre-polymerised fillers and these fillers are organic parts, they are not included in the ash test results. As reported by the manufacturing company these percentages are: Vol%=59-61 and Wt%= 77-79 (42). The high value of Ceram X in final δ pol may not be explained by the lower Vol% filler content compared to the other resin composites in this study. The reason for this difference may be the monomer used in the resin matrix being an ORMOCER which was only present in Ceram X. (Fig-24&25)

6.4.2: Core temperature:

Polymerisation is an exothermic reaction (48, 49) when using the same light curing irradiance a higher Max T or ΔT for a material may indicate a greater degree of conversion or a greater percentage of monomer in different materials. Furthermore, the quantity of visible light photoinitiator (for example camphorquinone and amino co-initiator) or inhibitor (e.g., butylated hydroxytoluene) can also influence the degree of conversion and curing rate (18). In this study, the irradiant output for the HI irradiance and LED C were similar. However, the radiant energy from the QTH light was approximately one-third that of the other two curing irradiances. This difference may affect the rate of polymerisation and the degree of conversion (50).

In general, the highest temperature values were recorded for the HI irradiance, whilst the lowest were generally seen for the QTH and LED R irradiances. Ranking of mean ΔT value is in the following order: HI> LED C> LED R $\approx$ QTH. This pattern is not surprising since the light curing device produces heat in addition to light (51), which is related to the irradiance and radiant exposure from the light

curing units. Halogen globes produce more heat compared to LED lights which are more efficient in the conversion of electrical energy to (blue) light. However, the heat of a halogen globe with an irradiance of 560 mW/cm² is much lower than the HI or LED units with the irradiance of 3200 mW/cm² and 1500 mW/cm² respectively. Resulting in lower photo thermal effect and lower radiant energy.

Consequently, higher values for temperature categories in the HI irradiance are expected. Lower values (ΔT) were observed for the bulk-fill resin composites (Sonicfill and Aura) compared to other materials in 1.8mm thickness, but for 5mm thickness the ranking changed to the highest after Ceram X. Ceram X had the highest mean value in both temperature categories, which is believed to be due to the different resin content as explained above.

The mean value of ΔT and Max T for each material decreased for the 5 mm thickness. The biggest change was seen for the HI irradiance, while the smallest change was for QTH. This variation may be due to the penetration depth of the light and heat output produced by the curing units. For a constant irradiance a thicker bulk of composite results in lower core temperature rise, since the bulk of the restorative material acts as thermal insulation. So, for a sample thickness of 5mm, there is a greater mass of material between the light and the thermocouple in the core of the sample. The light intensity reduces rapidly as a function of distance through resin composite which results in a lower rise in temperature due to the heat generated by the photothermal effect. The exothermic reaction will also be slowed which results in a similar core temperature to 1.8 mm thickness or lower (Fig-23).

The ΔT and Max T values showed a significant variation among materials when QTH light was used whereas the other curing irradiances did not show significant differences among materials.

A typical ΔT for LED C, LED R and QTH ranged between 14 to 21 °C, but for HI the ΔT ranged from 34°C for Clearfil with 5 mm thickness to 58°C for Ceram X 1.8 mm thickness. This large increase in temperature difference gives some cause for concern, as such a high temperature change may cause changes to tooth structure or more importantly pulpal tissue, especially when blood supply may be reduced due to the vasoconstrictor from local anaesthetic solutions. The extremely high temperature was seen in the 1.8 mm thickness. ΔT of HI irradiance samples in this category ranged between 52 to

58°C, in contrast to a range of 14 to 38°C for the same curing irradiance in 5 mm sample thickness. The lower temperature for the 5 mm thickness may also be attributed to an incomplete or slower rate of curing during the polymerisation process along with the shielding (insulating) of the thermocouple.

It has been reported that human collagen can be partially denatured when exposed to a temperature of 80°C for a minute and completely denatured when a temperature greater than 80°C occurs for one minute. (52) The mean ΔT value for Ceram X with HI curing irradiance for 1.8 mm thickness attained 58 °C. Considering that the baseline temperature was room temperature (24°C), it can be concluded that this increase in temperature may reach 80°C clinically when body temperature is 37°C.

The factory has recommended the HI curing light to be used for only one or two exposures of 3s for each layer. However, in the current study in order to standardise the curing times (40s), this device was used for seven 3s exposures over a 40s period, which would not be undertaken clinically.

ΔT and Max T values of the HI irradiance after 2 cycles of 3 seconds each are shown in table-11. The reduction in the Max T mean value for all materials was 15°C (from 77°C to 62°C) in the 1.8 mm thickness and 12°C (from 60°C to 48°C) in the 5 mm sample. A greater temperature reduction will occur with longer time between the exposure as the majority of heat is generated during light irradiance and not due to the exothermic reaction. As reported by Seltzer et al. a 5.5°C increase of the intra-pulpal temperature can cause a significant rise in pulpal necrosis (53). The reported ΔT and Max T results raise concern regarding the heat generated during curing of resin composites. When the core temperature of a resin composite sample increases by 27°C in a 5 mm bulk thickness after 2 curing cycles, the probability of pulpal tissue damage increases during restoration placement when a thin pulpal floor exists. Hence, caution is advised particularly with new high irradiance devices. Further studies on the temperature changes in resin composites at the surface of a restoration and tooth structure are essential to gauge the amount and rate of heat transfer and temperature rise during polymerisation of resin composite at high irradiation levels.

6.5: Conclusion:

In conclusion, Ceram X displayed a higher δ_{pol} compared with other materials in this study. This reflects the importance of the composition of this resin composite. However further tests need to be carried out to determine if the HI irradiance correlates to a higher degree of conversion of the monomer to polymer.

The irradiance, radiant exposure and mass/ thickness of material to be polymerised all appear to be important parameters in influencing the final δ_{pol} and temperature changes of the test materials. The simultaneous investigation of the temperature change and contraction stresses leads to a better understanding of final δ_{pol} in resin composite materials. High irradiance curing units will enable shorter clinical curing times to be used but it is important to adhere to cooling intervals between irradiation exposures to avoid unwanted thermal interactions to the pulp.

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Chapter 7: Project IV- Effect of thermal ageing on flexural strength, wear, and hardness of bulk-fill and conventional resin composites

7.1: Introduction:

Composite fracture has become the main reason for the failure of posterior resin-based restorations in recent years based on outcomes from a systematic review clinical studies (1). Flexural strength is one of the influencing properties of restorative materials that may indicate the tendency of restoration fracture during the function. So, understanding the effects of ageing on this property may provide useful information to aid in predicting the life of a restoration. Different methods have been used to accelerate artificial ageing of resin composites. Each technique uses one, or a combination of different stress factors, such as thermal stresses, storage media, fatigue (mechanical) stress via loading, abrasion or exposure to light (2, 3). Thermal ageing in a solution such as water will lead to both hydrolytic and thermal degradation. This degradation may influence the formation of microcracks at the interface between the filler and polymer matrix (4). This ageing method has been used in many studies to illustrate the durability of restorative materials (2- 4).

Wear is an important factor for large restorations, especially those under high masticatory loads such as restorations with functional cusp involvement, or patients with bruxism (5, 6). An ideal restorative resin composite will have similar tribological behaviour to enamel. This similarity results in less vertical dimension loss in occlusion and greater strength for the restored tooth (7). These are all important and influential factors in the success of a restoration, hence a better understanding of how resin composites wear, especially the newer bulk-fill materials will aid in the better understanding clinical performance of these materials.

This project will evaluate the biaxial flexural strength, abrasive wear and hardness of conventional and bulk-fill composites after thermal ageing to determine whether differences between these two types of resin composite differ.

TABLE-13 SPECIFICATION OF MATERIALS.

Type	Product	Name	Manufacturer	Lot #	Exp. Date	Shade	Wt% filler (Chapter 6)	Vol% filler (Chapter 6)	filler size	Composition
Anterior composite	light-cured restorative composite	Clearfil Majesty ES-2	Kuraray Noritake Dental Inc	C20029	07-2018	A2	58.61±0.19	57.85±2.86	Silanated barium glass filler (average: 0.7 µm)(8)	bisphenol A di glycidyl methacrylate - Silanated barium glass filler -Pre-polymerized organic filler -Hydrophobic aromatic dimethacrylate - Hydrophobic aliphatic dimethacrylate - di-Camphorquinone - Accelerators - Initiators -Pigments (9)
Bulk-fill composite	bulk-fill ultra-universal restorative composite	Aura	SDI	171123	01-2020	Universal	72.42±0.01	54.56±1.32		acrylic monomers as: diurethane dimethacrylate - triethylene glycol dimethacrylate - 2,2-bis[4-(2-methacryloxy) ethoxy] phenyl] propane(10)
Universal composite	light-cured radiopaque universal composite	Kalore	GC	1604261	04-2019	A2	69.66±0.06	53.67±1.29	400nm 100nm 700nm 17µm(11)	DX-511 monomer, di-UDMA, DM(12) Urethane dimethacrylate (UDMA) – ethoxylated bisphenol-A dimethacrylate (Bis-EMA) – dimethacrylate – butylated hydroxytoluene (BHT) – Uv light absorber – stabiliser (13)
Universal composite	universal Nano-ceramic restorative	Ceram X	DENTSPLY	1705001011	05-2019	A2	70.19±0.08	51.30±0.48	spherical, pre-polymerized SphereTEC™ fillers 15µm(14)	Methacrylate modified polysiloxane (organically modified ceramic)(ORMOCER) - Dimethacrylate resins (UMA, Bis-EMA, TEGDMA) - Fluorescent pigment - UV stabilizer - Stabilizer - Camphorquinone - Ethyl-4(dimethylamino) benzoate - Bis-(4-methyl-phenyl) -iodonium hexafluorophosphate - Barium-aluminum-borosilicate glass - Ytterbium fluoride - Iron oxide pigments and titanium oxide pigments according to shade (14, 15)
Bulk-fill composite	highly filled bulk-fill composite	SonicFill 2	Kerr	6120927	09-2018	A2	71.50±0.076	62.08±2.63	4µm(16)	BisGMA, TEGDMA, BisEMA(17) Silicon dioxide – glass, oxide, chemicals – Zirconium compound (as Zr) – Ytterbium trifluoride (18)

7.2: Methods and materials:

Samples were divided into 10 groups depending on temperature and material. The materials tested and their composition are displayed in Table-13.

Biaxial flexural test:

Disk-shaped specimens (12 mm $\varnothing$ $\times$ 1.5 mm thick) were made using a stainless-steel mould. And

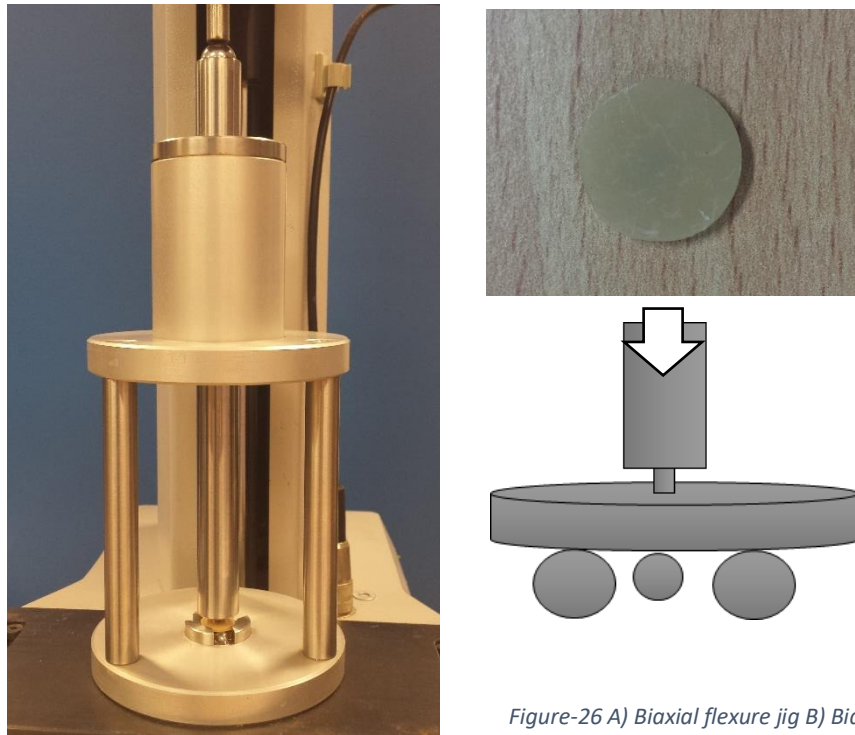


Figure-26 A) Biaxial flexure jig B) Biaxial flexure test sample C) Diagram of sample under load

light cured as per manufacturer's instruction. Half of the specimens were stored in distilled water at 60°C, and the other half were kept in distilled water at 37°C as control groups (n=6). The water uptake of materials determined the storage time. The samples were weighed until all groups attained a stable weight for water sorption (28 days).

After the storage period (28 days), the bi-axial flexural strength of specimens was measured with piston on 3 balls biaxial test jig using equation-1 as described in ISO 6872 (19). (Fig-26)

$$\delta = (-0.2387P(X-Y))/b^2$$

Equation-1

(δ =maximum centre tensile stress(MPa), P=total load at fracture (N), b=specimen thickness at fracture origin (mm), $X=(1+\nu) \ln(r_2/r_3)^2+[(1-\nu)/2] (r_2/r_3)^2$, $Y=(1+\nu) [1+\ln(r_1/r_3)^2]+(1-\nu)(r_1/r_3)^2$, $\nu=0.25$ (Poisson's ratio), r_1 =radius of support circle(mm), r_2 =radius of loaded area(mm), r_3 =radius of specimen(mm)

Hardness test:

One fractured piece of each flexural test sample was subjected to a 300g load for 10s for 3 indentations with Micro-Knoop Hardness (KH) Tester (n=6). (Average was recorded as KH Number.)

Wear test:

Disk-shaped specimens (8 mm diameter $\times$ 1.2 mm thick) were prepared (n=3) and embedded in polymethylmethacrylate resin (Cold curing acrylic, Vertex- Dental B.V., The Netherlands). After polymerisation, the sample surface was polished by gentle grinding with 1000 and 2000-grit SiC abrasive papers under water. Specimens were imaged with a light microscope (Leica Microsystems, Wetzlar GmbH, Germany) and the profile was scanned using a contact profilometer (Surface roughness tester SJ-400, Mitutoyo Corporation, Kawasaki, Kanagawa, Japan) in three parallel lines 2.5mm long separated by 1.5mm for each scan. (determined using a micrometre, precision ± 0.001 mm).

Then the weight of samples was measured via a precision balance (Precisa Gravimetrics AG, CH-8953 Dietikon, Switzerland, precision ± 0.001 g). Afterwards, the samples were subjected to a linear wear test using a Taber linear Abraser (Taber industries, NY, USA). The abrasive element was enamel from a buccal cusp of an intact lower first premolar obtained from a 12-year-old after informed consent was received. (The enamel of the buccal cusp was imaged before each test, under a microscope too eliminate the chance of facet formation.) Distilled water was the lubricating medium used, the sliding distance of the cusp on the composite samples was approximately 5.08 mm, with a load of 21N for 20,000 cycles at an average speed of 2.45 mm/s. Afterwards, the sample was weighed,

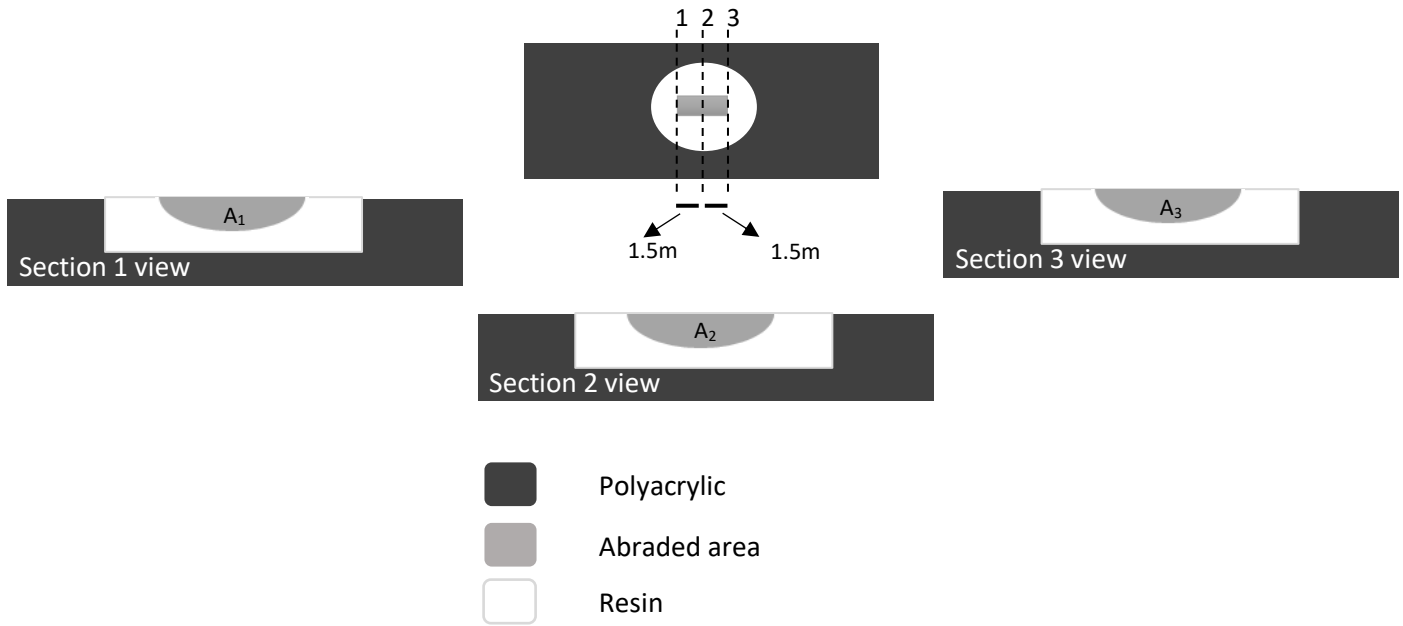


Figure-27 Diagram of Wear test sample indicating the middle area of abraded volume (A_2) and lateral areas of abraded volume (A_1 and A_3)

and the abraded surface was imaged with a light microscope and analysed with the profilometer. As previously described. Vertical areas (A_1 , A_2 and A_3) of abraded volume were calculated using the depth of 3 scan lines with 3mm width then the volume loss (ΔV) was calculated using Equation-2. The wear volume coefficient (K_V) and wear mass coefficient (K_M) were calculated using equations 3 and 4, which is based on the Archard equation (20-22)

$$\Delta V_1 = (((A_1 + A_2) / 2 \times 1500) + ((A_2 + A_3) / 2 \times 1500)) \quad \text{Equation-2}$$

$$K_V = \Delta V_2 / (P \cdot L) \quad \text{Equation-3}$$

$$K_M = \Delta M / (P \cdot L) \quad \text{Equation-4}$$

(where ΔV_1 = lost volume (μm^3), A_1 , A_3 = lateral area of abraded volume (μm^2), A_2 = Middle area of abraded volume (μm^2), ΔV_2 = lost volume (mm^3), K_V = wear volume coefficient (mm^3/Nm), P = applied load (N), L = sliding distance (m) K_M = wear mass coefficient (g/Nm), ΔM =lost mass (g))

Statistical analysis:

Flexural strength(δ), K_v , K_M and KHN of each sample were calculated. Mean values of each group in each category were calculated and analysed using ANOVA, Bonferroni, Tukey and Kruskal Wallis tests ($\alpha = 0.05$). (SPSS, V23, IBM).

7.3: Results:

All calculated data are displayed in Table-14.

TABLE-14 BIAXIAL FLEXURAL STRENGTH, KHN AND WEAR COEFFICIENT MEAN VALUES

TEMPERATURE (°C)	Material	Biaxial flexural strength δ (MPa) (n=6)	KHN (n=6)	Wear volume coefficient K_M (mm ³ /Nm10 ⁻⁶) (n=3)	Wear mass coefficient K_v (g/Nm) (n=3)
		Mean value $\pm$ SD	Mean value $\pm$ SD	Mean value $\pm$ SD	Mean value $\pm$ SD
37	Clearfil	71.07 $\pm$ 3.94 ^{a*}	38.98 $\pm$ 10.97 ^a	234.40 $\pm$ 18.62 ^a	0.10 $\pm$ 0.06 ^a
	Aura	78.16 $\pm$ 7.36 ^{a, b}	57.91 $\pm$ 3.16 ^{a, b}	270.00 $\pm$ 32.70 ^{a, b}	0.16 $\pm$ 0.03 ^a
	Kalore	88.58 $\pm$ 8.19 ^b	58.76 $\pm$ 9.45 ^{a, b}	382.86 $\pm$ 289.78 ^b	0.16 $\pm$ 0.06 ^a
	Ceram X	116.91 $\pm$ 5.13 ^c	68.48 $\pm$ 19.58 ^b	243.20 $\pm$ 52.24 ^{a, b}	0.07 $\pm$ 0.06 ^a
	Sonicfill	75.15 $\pm$ 8.52 ^a	68.69 $\pm$ 11.01 ^b	345.87 $\pm$ 113.61 ^{a, b}	0.07 $\pm$ 0.03 ^a
60	Clearfil	65.35 $\pm$ 13 ^a	48.53 $\pm$ 6.75 ^{a, b}	493.78 $\pm$ 35.71 ^{a, b}	0.15 $\pm$ 0.15 ^a
	Aura	78.23 $\pm$ 18.18 ^a	44.40 $\pm$ 6.57 ^a	318.51 $\pm$ 24.04 ^a	0.07 $\pm$ 0.03 ^a
	Kalore	85.43 $\pm$ 17.58 ^a	64.86 $\pm$ 11.61 ^{b, c}	1025.34 $\pm$ 510.59 ^{b, c}	0.05 $\pm$ 0.01 ^a
	Ceram X	88.86 $\pm$ 16.74 ^a	88.67 $\pm$ 12.12 ^d	292.19 $\pm$ 194.11 ^a	0.10 $\pm$ 0.14 ^a
	Sonicfill	79.78 $\pm$ 12.23 ^a	77.37 $\pm$ 17.07 ^{c, d}	387.77 $\pm$ 201.28 ^{a, b}	0.28 $\pm$ 0.05 ^a

* a,b,c =within each temperature category those groups with the same superscript are not significantly different

Biaxial flexural strength δ :

The biaxial flexural strength was significantly different (p -value < 0.05) among tested materials in the 37°C group. Lower flexural strengths were observed for samples stored in 60°C for Clearfil, Kalore and Ceram X, whereas the bulk-fill materials showed little variation (Aura and Sonicfill) and were not different ($p > 0.05$). The trend in materials ranking was similar in both temperature groups: Ceram X > Kalore > Sonicfill = Aura > Clearfil.

Hardness:

Hardness values revealed significant differences (p -value < 0.05) among tested materials. Higher values were recorded for the 60°C samples, except for Aura. A similar trend in the material ranking was observed between the two ageing groups: Sonicfill, Ceram X>Kalore >Clearfil, Aura.

Wear:

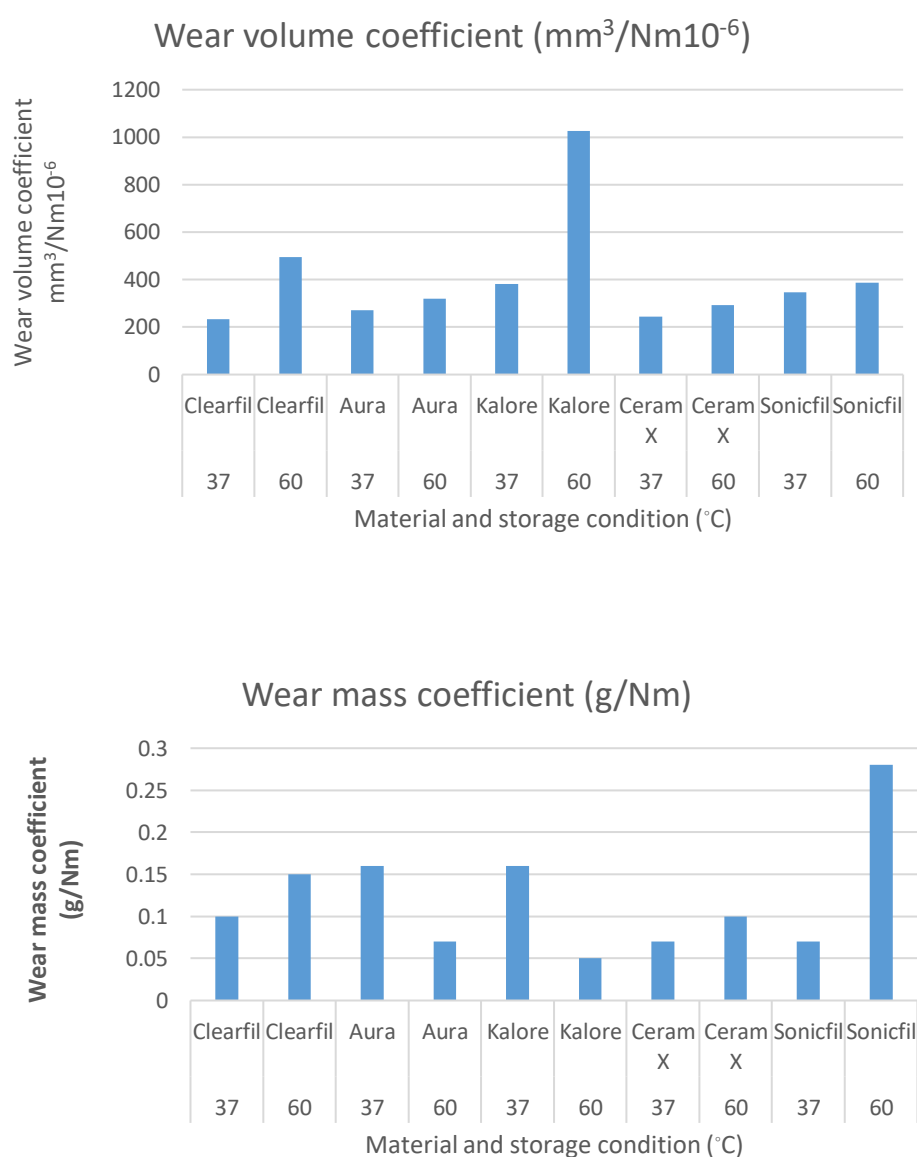


Figure-28 Wear coefficients

Wear volume coefficient- In this category, values were significantly different among materials ($p < 0.05$). Higher volume wear coefficient was determined for samples stored at 60°C, however, the magnitude of this difference was varied for each material. The ranking of materials was the same between two ageing categories with the exception of Clearfil (Kalore > Sonicfill > Aura > Ceram X). The wear volume coefficient varied between 234.40 and 382.86 mm³/Nm × 10⁻⁶ for 37°C group and 292.19 and 1025.34 mm³/Nm × 10⁻⁶ for 60°C group, which indicated a larger deviation between materials in the latter group.

Wear mass coefficient- This coefficient revealed no significant ($p > 0.05$) difference between material. Also, the ranking of material for each temperature group was not the same. The results for 37°C group was between 0.07 and 0.16 g/Nm. And 60°C group results were ranged from 0.05 to 0.28 g/Nm. Variation in group 60°C is much larger than the group with the storage 37°C temperature.

7.4 Discussion:

Flexural strength is one of the important measures of strength for dental materials since a considerable amount of this flexural stress occurs during mastication (4, 23). This type of test is reported to be more accurate due to concerns involving the wedging stress and counter moments produced by friction, at the loading points in 3-point bending test and also replicates a more similar situation to occlusal forces (24).

The differences between the flexural strength of materials are due to dissimilarities in resin composition and filler. Fillers can affect the physical properties by size, volume and type. However, the storage medium also has an important effect on flexural strength. (9, 10) Materials in this study had an inverse correlation between flexural strength and Vol% filler content. The higher Vol% filler content resulted in lower flexural strengths. On the other hand, the filler mass revealed not a strong correlation with this physical property (R^2 : Biaxial flexural strength δ vs Vol% in 37°C = 0.57, Biaxial

flexural strength δ vs Vol% in 60°C = 0.28, Biaxial flexural strength δ vs Wt% in 37°C = 0.11, Biaxial flexural strength δ vs Wt% in 60°C = 0.61). Based on previous studies, the filler morphology and molecular composition of filler are also important factors when assessing flexural strength (4, 23). Due to the higher Vol% particles may be very close to each other creating a weakness in strength as there is little or no matrix coupling to keep the particles together so the load cannot be transferred throughout the material.

Conventional resin composites (Clearfil, Kalore and Ceram X) in this study revealed a lower flexural strength after the 60°C ageing. On the other hand, the bulk-fill composites (Sonicfill and Aura) revealed a significantly ($P < 0.05$) higher flexural strength. However, the ranking between materials did not change between the two temperature groups. The improvement of flexural strength of the bulk-fill composite may be due to their matrix composition, which is different from conventional composites.

Thermal ageing has been used in many studies to illustrate the durability of restorative materials. These methods are not an indication of the exact time of failure and deterioration, but they give us a perspective on what changes in physical properties may occur in a material as it ages. The findings of this study and previous studies indicate a decline in flexural strength of conventional resin composites when materials were exposed to various means of ageing (4, 25, 26). In contrast, the bulk-fill resin composites showed a slightly higher flexural strength after the ageing.

In posterior restorations and restorations in high load-bearing areas, restorative materials with lower flexural strength could possibly fail more easily and earlier due to fracture, so it is important to choose a material with a durable flexural strength that changes little over time.

KHN increased in those groups that were exposed to the 60°C ageing, however, Aura was the exception. Previous studies concluded that the effect of ageing due to exposure to a higher temperature storage solution seems to vary among resin composites (27). In the current study, the only material that showed a deterioration in hardness after exposure to 60°C ageing was Aura. The hardness of a material is not only dependent on the matrix and filler content but is also dependent on the arrangement or alignment and size of the fillers at the surface. Consequently, lower hardness values

may indicate a lower polymerisation in resin. Also, several factors influence the ageing resistance of resin composites, such as surface area and amount of the fillers and the strength of the coupling agent of the filler to the resin matrix (28). In this study, Ceram X, Kalore and Sonicfill have the largest filler size and greater hardness values. High hardness values of Ceram X may also be due to the special ceramic fillers in this material. Also, Ceram X contains pre-polymerised particles which result in a higher degree of polymerisation of resin monomer (resin conversion) and higher surface hardness. A recent study showed that unfilled UDMA/TEGDMA polymers have higher flexural strength than BisGMA/TEGDMA polymers, likely due to increased crosslink density in the polymer network. (29). This is in line with the results of this study which indicates low values for Clearfil, which mostly contains BisGMA, compared to other resin composites which contain UDMA. All these differences are based on the components of the material.

As previous studies suggest, composites differ significantly in ageing behaviour as well as mechanical and physical properties, which both are based on the components of the material (30). The smallest increase of K_v after thermal ageing (60°C) was observed for Sonicfill, Aura and Ceram X in ascending order. So again, the bulk-fill resin composites in this study revealed a smaller susceptibility to thermal ageing. Wear of resin composite is dependent on the properties/composition of the matrix material, the degree of conversion, the content/properties of the filler, the filler-matrix interface bonding, and the relative abrasiveness of filler against the matrix (31) (Table-13). In two-body wear tests of resin composite, the wear is directly related to the filler fraction and fracture toughness (32). Excluding Ceram X, which has a different monomer system (Ormocer), all tested materials showed an inverse correlation between K_v and Wt% filler in the 60°C group. Ceram X revealed the lowest K_v among tested materials which may be an indication of better coupling of the filler and matrix in this material. As mentioned in a study by Kruzic et al. (31) some resin matrixes showed significantly less wear, which may be the reason for reduced wear results noted for Ceram X and bulk-fill composites in this study. Also, Ceram X contains pre-polymerised particles which result in a higher resin conversion and higher surface hardness which may result in lower wear.

The results of the K_M did not correlate with the K_V . Also, a large variation was seen in the results for some materials. This discrepancy was believed to be due to the embedding material. The PMMA embedding resin has a high-water uptake when it is immersed in the distilled water (storage medium). After storage samples were taken out, tapped dried and weighed after 5 minutes of drying at room temperature. During the test, samples were maintained in an environment of 99% relative humidity (i.e., they were not immersed in distilled water). After approximately 25 hours duration of the test, the water content of the PMMA resin was affected and thus altered the weight of the samples. As a result, the K_M results were not reliable as time was needed for the PMMA to stabilise in relation to water imbibition.

Within the limitation of this study it may be concluded that, the influence of thermal ageing on microhardness was material dependent, and a higher wear coefficient and lower flexural strength were seen in the conventional resin composites of this experiment. Bulk-fill resin composites of this study revealed no effect to thermal ageing on biaxial flexural strength and smaller negative impacts on wear coefficient. It can also be concluded that bulk-fill resin composites in this study revealed better physical properties and better resistance to deterioration by artificial ageing compared to conventional composites.

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Chapter 8: Project V- Cuspal strains of teeth restored with different bulk-fill resin composites and modes of light curing

8.1: Introduction:

Resin composite use has grown in popularity as a posterior restorative material. This increase in use had led to composite being placed in larger and more complex restorations, thus magnifying the shortcomings of these materials. To reducing the failure of these restorations, we need to have a better understanding of their negative properties such as the polymerisation shrinkage. Polymerisation shrinkage of resin composite has been associated with microleakage, debonding, secondary (recurring) caries and postoperative sensitivity (1). Many methods have been used to reduce the stresses which are caused by polymerisation contraction such as, incremental placement technique for resin composite, slow-start polymerisation using ramp mode for the curing light output and more recently the modification of materials formulation by including monomers that can 'stretch', thus reducing the stress rate during shrinkage (2-8). With the introduction of bulk-fill composites, a concern regarding the polymerisation shrinkage and depth of cure of the bulk composite has been raised (9-11). These concerns were heightened when the high irradiance (intensity) curing lights also became available. The combination of an increased bulk or volume of resin composite and rapid polymerisation in just a few seconds may lead to a substantial rise in the retained stresses due to the rapid shrinkage creating detrimental forces at the bonded tooth-resin interface. Also, these new high intensity LED curing lights are more likely to produce higher temperatures in the pulpal chamber, similar to the high irradiance plasma arc curing systems. As mentioned in the results of previous projects (chapter 6) of this thesis, the high intensity light cure rapidly increases the core temperature of the resin composites.

A method that has been used to measure the polymerisation contraction stress is cuspal flexure. In this test, the clinical cavity morphologies are replicated, which is one of the necessities to measure

an accurate polymerisation shrinkage stress(8). The importance of replicating the clinical situation is based on the influential factors in the production of stress. The amount of stress in the resin composite is dependent on various material physical and mechanical properties including modulus of elasticity, bonding and degree of conversion. But also, the shape of remaining tooth structure has a critical role in stress relief depending on the amount of compliance in the tooth. As a result, replicating the clinical situation in the laboratory results in a more realistic measurement of stress that can be extrapolated to clinical outcomes (12, 13).

Currently, the evidence remains limited concerning the influence of the use of bulk volumes of resin-based materials cured with a high irradiance LED light and how this may influence the remaining tooth structure and pulpal temperature.

The null hypotheses: 1- Different irradiances of LED curing devices do not affect the cuspal strain, and movement; 2- Different magnitude of irradiances from light curing devices do not affect the temperature change in the pulp chamber; 3-Hardness of bulk-fill resin composites after light curing do not vary in less than 4mm depth (lower hardness indicates a lower monomer conversion and an indication of not enough polymerisation).

8.2: Pilot study:

8.2.1: Aims:

A pilot study was conducted to determine the predominant directions of the strain in the tooth cusps after a cavity was prepared in order to select the most suitable single strain gauge placement.

8.2.1.1: Methods and materials:

Rectangular rosettes strain gauge with 3 measuring grids, and Linear Variable Differential Transformer (LVDT) were used to measure cuspal strain and movement, respectively. Specimens were prepared on 3 intact premolars. These teeth were stored in 0.5% Chloramine T solution for 2 weeks and then in distilled water at 4 °C before use. A MOD slot cavity, 4mm wide and 4 mm deep with a flat pulpal floor was prepared with a high-speed handpiece using a cylindrical diamond bur (HORICO, 806

314 110 534, Berlin, Germany). The prepared teeth were mounted in nylon mounting rings 2mm below the cementoenamel junction (CEJ) to replicate the relationship of teeth to the boney support that would occur in the oral cavity. Strain gauge rosettes (WA-06-030WR-120, Micro-measurements, USA) were attached to the buccal and lingual cusps with the strain gauge adhesive (M-bond 200, Micro-measurements, USA). In addition, two small pits were prepared in Buccal and Lingual cusps to locate and place the LVDTs. The prepared sample was mounted on an orienting jig with LVDTs attached. The tip of each LVDT rod was placed in contact at the point of the pits on each buccal or lingual cusp.

Cavities were restored with an adhesive and resin composite following the manufacturers' instructions. The cavity surface was bonded using Clearfil SE bond 2 (Kuraray Noritake Dental Inc., Tokyo, Japan) and restored with a resin composite: Aura (bulk-fill ultra-universal restorative composite, SDI, Bayswater, Australia). After placing a 2mm layer of the resin composite, a thermocouple wire (Precision Fine Bare Wire Thermocouple Type J, IRCO-003, OMEGA) was placed in the centre of the pulpal floor, and the rest of cavity was filled with the restorative material. The restored cavity was then light cured with high intensity LED curing light (3200 mW/cm² (HI), Valo cordless, Ultradent Products Inc. UT, USA) for 3-second periods with a 2-second interval between each exposure. (Fig-29)

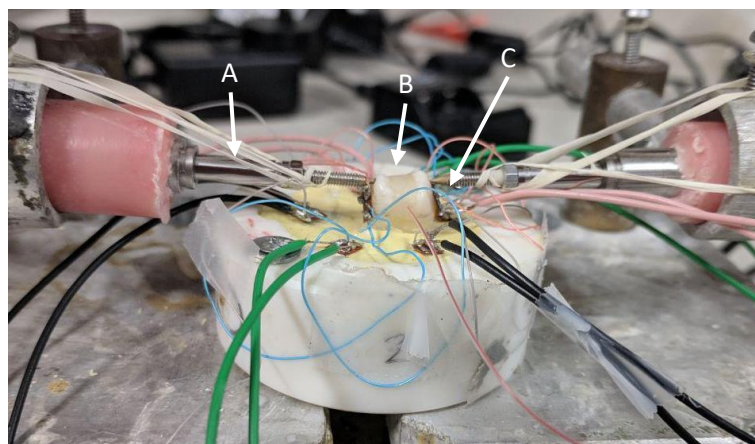


*Figure-29 Pilot study: restored sample A- LVDT rod B- Restored tooth
C- Strain gauge*

Change in temperature during bond application and composite polymerisation were recorded with the thermocouple wire and NI SignalExpress software (NI-DAQmx, NI SignalExpress, 2014, V 14.0.0

National Instruments) via conditioning module (Hi-speed USB Carrier (NI USB-9162) and module (NI cRIO-9211), National Instruments, USA). The strain and movement of the cusps were recorded synchronously using bonded strain gauges, LVDT rods and Labview software (LabVIEW7 Express, National Instruments, 2003) via conditioning device (SC-2345, signal conditioning with configurable connectors, National Instruments).

8.2.1.2: Results:

The results of the pilot study indicated that most of the strain was in the buccolingual direction. A smaller amount of strain was seen in the mesiodistal direction and almost nothing in the axial direction. (Fig-30)

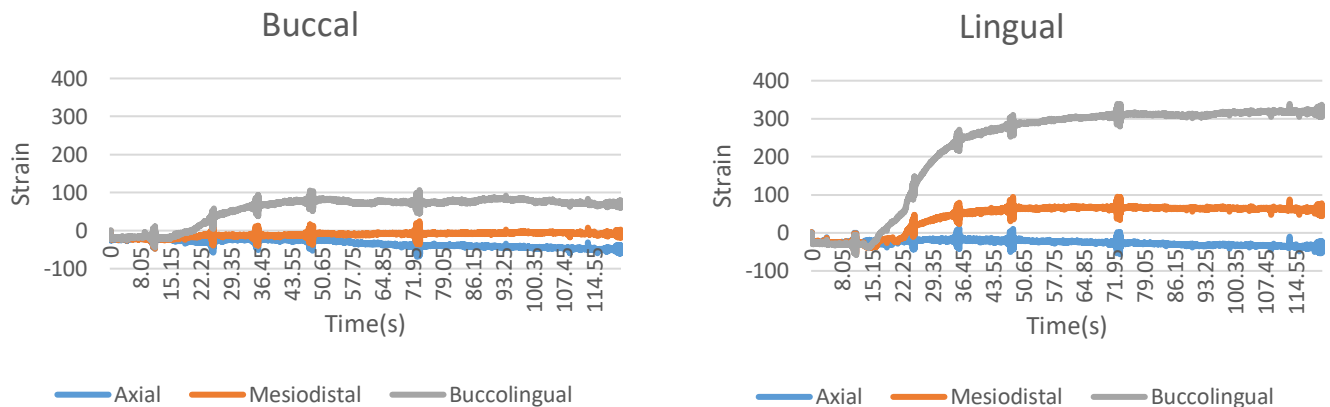


Figure-30 Strain diagram of Buccal and Lingual cusp of sample #1

8.2.1.3: Discussion:

Since a noteworthy amount of the change in strain was seen in the buccolingual direction. A single strain gauge was selected for the experiment to record the strain in the buccolingual direction.

During the restoration process, due to the packing of the final layer, the position of the thermocouple was changed. Also, the core temperature change of restoration revealed limited valuable clinical knowledge. So, the placement of thermocouple was altered to the pulp chamber, which would provide more clinically relevant information.

8.3: Methods and materials:

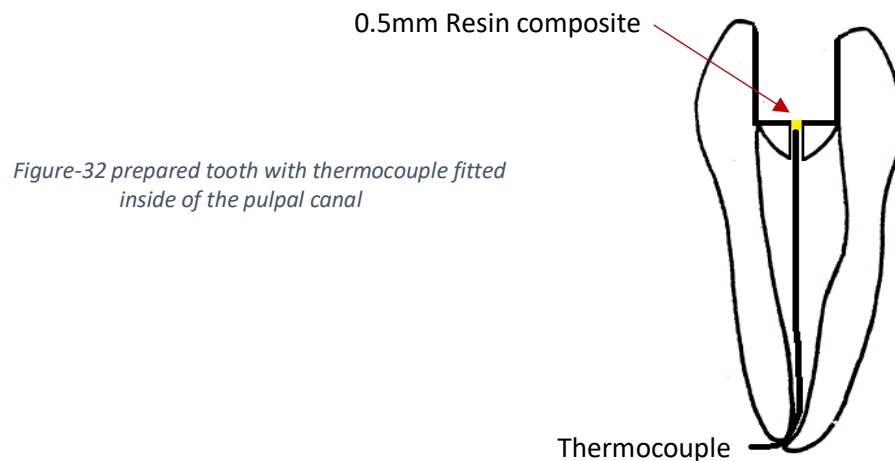
Based on the pilot study, single strain gauges and Linear Variable Differential Transformer (LVDT) were chosen as suitable methods to measure cuspal strain and movement, respectively (Fig-31).



Figure-31 A-Mounted tooth with fitted LVDTs and strain gauges. B- Schematic view of fitted strain gauges and LVDTs

Cavities were prepared on 24 intact premolars with closed apices in 4 groups based on restorative material and curing light unit (n=6). The teeth were collected in accordance to the ethics regulation (Ethics ID: 1748725) teeth were initially stored in 0.5% Chloramine T solution for 2 weeks and changed to distilled water at 4 °C before use. A MOD slot cavity, 4mm wide and 4 mm deep with a flat pulpal floor was prepared with a high-speed handpiece under water spray with a cylindrical fine-grit diamond bur (HORICO, 806 314 110 534, Berlin, Germany)). A perforation to the pulp chamber was produced using a round diamond bur (HORICO, 806 314 001 534, Berlin, Germany), and the root canal was enlarged with endodontic files (Stainless steel, K-files, Ultradent, USA) for the fitting and placement of a thermocouple into the pulp chamber and root canal, through the apex. A thermocouple wire (Precision Fine Bare Wire Thermocouple Type J, IRCO-003, OMEGA) was fixed 0.5 mm below the pulpal floor with a resin bonding agent (SE Bond (Table-15)), and it was covered with resin composite (Aura) (Fig-32). Then the prepared teeth were mounted in nylon mounting rings 2 mm below the cementoenamel junction (CEJ) to replicate the relationship of teeth to the supporting alveolar bone in the oral cavity. Single strain gauges (CEA-06-032uw-120, Micro-measurements, USA) were attached

to the buccal and lingual cusps with strain gauge adhesive (M-bond 200, Micro-measurements, USA), then two small pits were prepared using a round diamond bur (HORICO, 806 314 001 534, Berlin, Germany) on the buccal and lingual enamel surfaces of the respective cusps for LVDTs location and placement. The prepared sample was mounted on an orienting jig with the LVDTs attached. The tip of



each LVDT rod was placed in the pits on the buccal and lingual cusps. (Fig-31)

Cavities were restored as per manufacturers' instructions, using the materials listed in table-15 and following the curing methods outlined below:

Table-15 Specification of materials.

Type	Product	Name	Manufacturer	Lot #	Shade	EXP	Composition
Bulk-fill composite	highly filled bulk-fill composite	SonicFill 2	Kerr	6759070	A2	2020-01	BisGMA, TEGDMA, BisEMA(14) Silicon dioxide – glass, oxide, chemicals – Zirconium compound (as Zr) – Ytterbium trifluoride (15)
Bulk-fill composite	bulk-fill ultra-universal restorative composite	Aura	SDI	171123	Universal	2020-11	acrylic monomers as: diurethane dimethacrylate - triethylene glycol dimethacrylate - 2,2-bis[4-(2-methacryloxy) ethoxy] phenyl] propane(16)
Bonding agent	Universal self-etch adhesive	Clearfil SE Bond 2	Kuraray Noritake Dental Inc	000086	--	2021-12	bisphenol A di glycidyl methacrylate - 2-hydroxyethyl methacrylate - 10-Methacryloyloxydecyl dihydrogen phosphate - Hydrophobic aliphatic dimethacrylate - Colloidal silica - dl-Camphorquinone - Initiators - Accelerators(17)

1- light-emitting diode (LED) curing light continuous 1500 mW/cm² for 20 seconds (MI), radii plus+, SDI Limited, Bayswater, Australia.

2-LED curing light two 3-second periods with 2-second interval 3200 mW/cm² (HI), Valo cordless, Ultradent Products Inc. UT, USA.

Change in temperature during bond application, and composite placement and curing were recorded with the thermocouple wire and NI SignalExpress software (NI-DAQmx, NI SignalExpress, 2014, V 14.0.0 National Instruments, USA) via conditioning module (Hi-speed USB Carrier (NI USB-9162) and module (NI cRIO-9211), National Instruments). The strain and movement of the cusps were recorded using bonded strain gauges, LVDT rods and Labview software (LabVIEW7Express, National Instruments, 2003) via conditioning device (SC-2345, signal conditioning with configurable connectors, National Instruments) for 120s.

Temperature change (ΔT), total strain change (ΔS) and total dimensional change of the cusps (ΔD) were calculated using the following formulas:

1- Temperature change (ΔT) = maximum recorded temperature - initial recorded temperature;

2- Total strain change (ΔS) = (final recorded buccal cusp strain - initial recorded buccal cusp strain) + (final recorded lingual cusp strain - initial recorded lingual cusp strain);

3- Total dimensional change (ΔD) = (final recorded buccal cusp dimensional position - initial recorded buccal cusp dimensional position) + (final recorded lingual cusp dimensional position - initial recorded lingual cusp dimensional position)

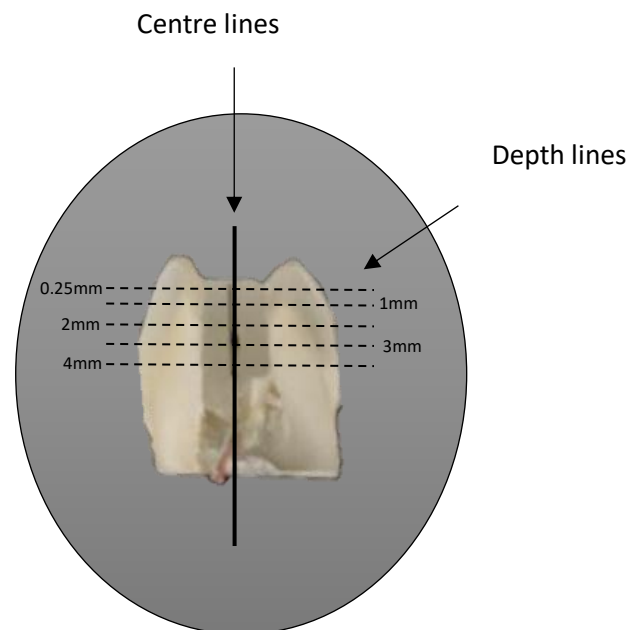
Hardness test:

Figure-33 Sections for hardness test

After the strain and temperature test were completed, samples were sectioned buccolingually in 2mm thick sections. The central 2mm slice of the composite restoration was subjected to a Vickers hardness test analysis by using a 50g load with 10 second dwell time, in 5 different depth at the centre line of restoration on both sides (Fig-33).

Both curing light devices were tested with a radiometer (CE Curing light meter 200, Rolence enterprise Inc., Taiwan) to determine the irradiance intensity output.

Statistical analysis:

Data were analysed with Levene's normality test, *t*-test, Mann-Whitney U test and Pearson test for correlation using SPSS software (SPSS, V23, IBM).

8.4: Results:

Radiometer test:

Both curing light devices, which were used in this study, were tested for the irradiance output intensity. The maximum value for the MI and HI method were 1250 and 2000 mW/cm², respectively.

Table-16 Calculated Mean value± SD

Materials	Curing method	ΔT (°C)	Total ΔS	Total ΔD (μm)
Aura	HI	5.78±0.92	257.85±142.35	74.87±10.09
	MI	4.38±0.76	221.08±99.21	71.61±10.09
Sonicfill	HI	5.79±0.92	277.60±133.53	73.24±5.35
	MI	4.23±0.98	259.36±141.90	66.73±9.60

Temperature change (ΔT), Total strain change (ΔS), Total dimensional change (ΔD) (n=6)

The calculated mean values of the results are presented in table-16&17.

Table-17 Calculated Mean value± SD

Materials	Curing method	Hardness 0.25mm depth	Hardness 1mm depth	Hardness 2mm depth	Hardness 3mm depth	Hardness 4mm depth	Hardness ratio (%) 4mm to 0.25mm
Aura	HI	68.60±7.04	67.39±5.56	61.61±4.00	59.23±5.60	48.40±2.04	71.06±6.39
	MI	73.84±10.50	61.75±4.03	59.42±8.29	53.21±10.07	49.44±4.46	68.16±11.93
Sonicfill	HI	94.77±15.90	89.42±8.92	87.06±7.59	81.44±4.00	69.84±5.78	75.08±11.10
	MI	95.99±14.81	83.74±9.16	82.09±11.66	82.21±15.33	77.89±8.74	82.13±10.12

Higher ΔT, ΔS and ΔD mean values were seen in groups using HI curing method, only ΔT variation was significant. Higher ΔS values were observed for Sonicfill compared with Aura. But none of these differences were statistically significant (CI=0.95).

Light curing process in the bonding stage and the composite stage were started at 90s and 10s respectively (Fig-34). Same trends were seen in ΔT during bond and resin composite light curing process (Fig-34). Higher peak values for each material were observed when they were light cured with HI curing protocol. The reduction in temperature before light curing in the bonding process was due to air drying of primer prior to application of bonding resin.

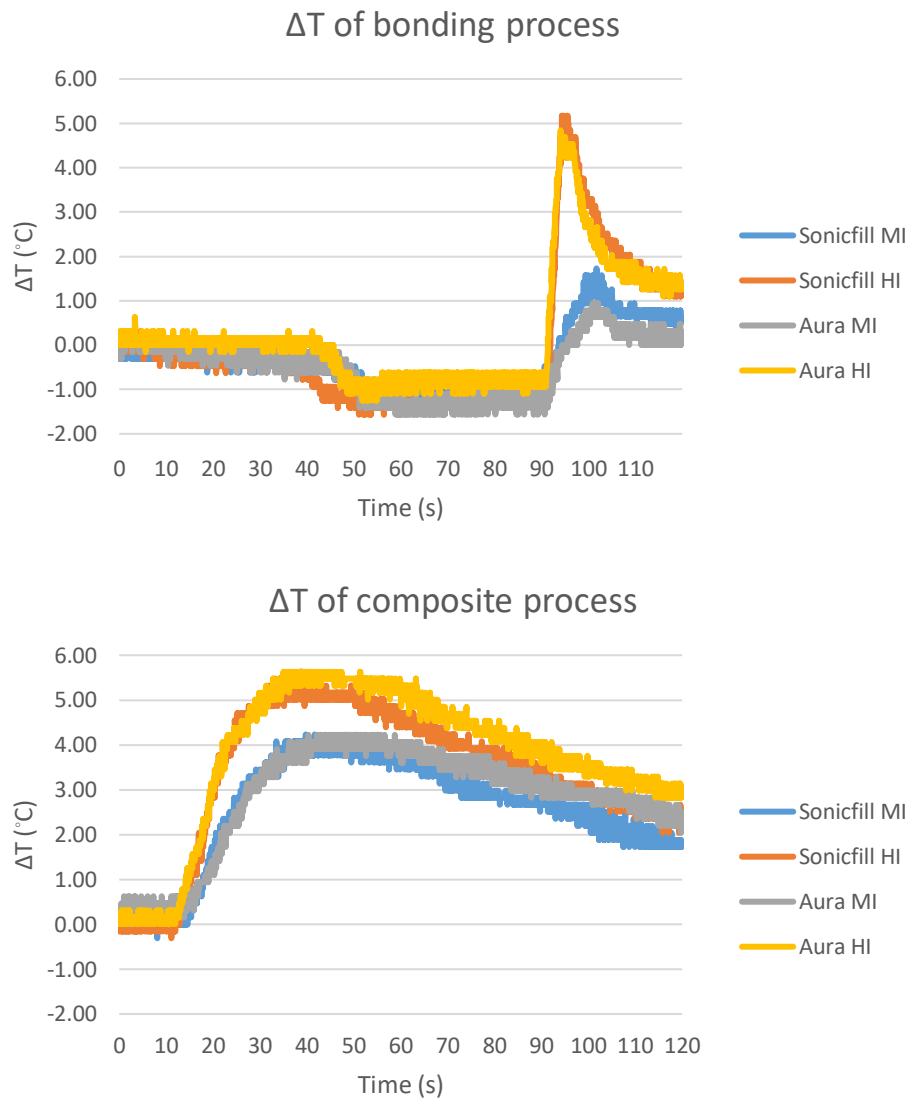


Figure-34 Mean ΔT of Bonding and composite light curing process

Hardness:

The differences between the hardness of 0.25 mm depth and 4mm depth are similar in all groups with higher values in groups with HI as curing method. The statistical analysis revealed a significant positive correlation between these two groups of hardness.

The significance of the curing method's effect on the hardness of each layer was different for each material tested. For Aura, the curing method resulted in a significant difference between hardness at the 3, 1, and 0.25mm depths. Moreover, for Sonicfill, it significantly varied among the 4, 2 and 1mm depths.

8.5: Discussion:

The peak irradiance intensity of the curing light devices was 1250 and 2000 mW/cm² for MI and HI devices, respectively. The reported intensity of MI light curing device by the manufacturer is 1500 mW/cm² (18). The lower value recorded for the MI device may be due to the ageing of the device, possible damage to the fibre-optic light tip. However, the large difference between reported 3200 mW/cm² and recorded 2000 mW/cm² irradiance intensity for the HI curing device is due to the test method used by the company for measuring output. (19). The Valo curing light has three different curing modes, namely standard 'output' at 1000 mW/cm², high power at 1400 mW/cm² and Xtra power at 3200 mW/cm² as reported by the company. The manufacturer stated the peak irradiance intensity for the standard and high-power modes were measured using a Demetron radiometer, similar to the method used in this research to test light output. However, for the Xtra power mode peak intensity, the method of output measurement was performed using an integrating sphere, which is an optical device consisting of a hollow spherical cavity. The interior of the sphere is coated with a diffuse white reflective material to measure the power of total light radiation from all directions, which will include the scattered light. This different measuring method will, therefore, result in a large difference in the measurement output and may be regarded as misleading. It is customary for companies to report the irradiance intensity of dental curing lights via radiometer measurements, such as for the two lower intensity modes of the Valo curing light or radii plus (MI curing method device). Hence the output provided for the Xtra power output is not comparable with the other conventional curing irradiances. The results from the current work indicate the output when a radiometer is used is only 2000 mW/cm².

Consequently, the total radiant exposure for the curing methods based on measured peak irradiance (intensity) in this study were approximately, HI: 12 J/cm² and MI: 25 J/cm². The lower values of hardness at 4mm depth in the HI groups compared with the MI groups of the same material may be a result of the lower energy output of this curing method. These lower values may indicate that the two 3-second exposures recommended by the manufacturer are insufficient for 'adequate'

polymerisation of bulk-fill resin composites. On the other hand, the temperature changes observed with the high intensity curing method means that a longer exposure time may further increase the chances of causing pulpal damage.

The ratio of the hardness value of the 4 mm depth to the 0.25 mm for all groups was as follow: Aura HI (71.06%), Aura MI (68.16%), Sonicfill HI (75.08%) and Sonicfill MI (82.13%). The only group with a higher than 80% ratio, indicated by ISO 4049 (20) was Sonicfill MI. A gradual decrease of hardness with depth was evident in almost all groups, which is the result of the reduction in light penetration and consequently light attenuation in deeper parts of the restoration (Fig-35).

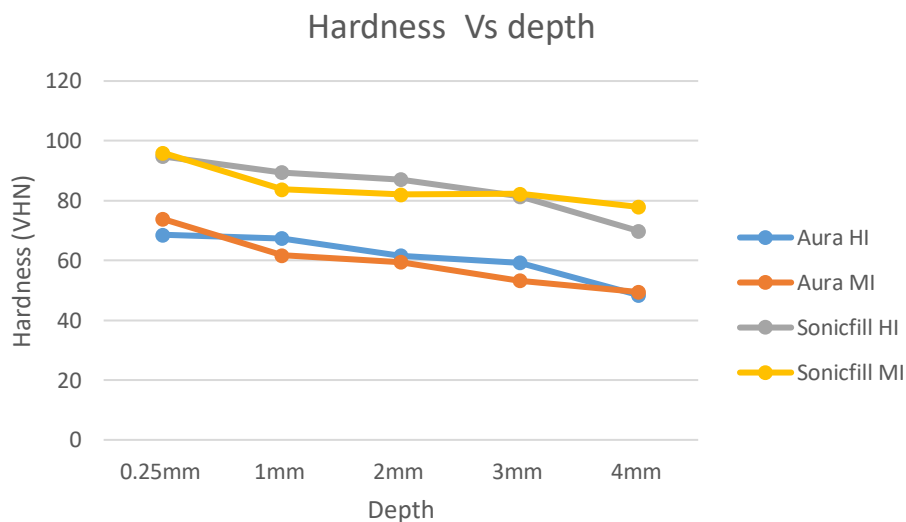


Figure-35 Hardness VS depth trend diagram

Although the hardness of 4mm depth and 0.25 depth layer are significantly different, there was a strong positive correlation (Pearson correlation:0.74) between them. The lower value in the deepest layer was seen in groups with HI curing method for each material. In addition, Besegato et al. found (21) that regardless of the composition of a bulk-fill resin composite, the light-curing protocol with higher irradiance and shorter exposure time (higher power density) resulted in greater polymerisation shrinkage and lower hardness. As mentioned above, this difference in hardness may be due to the lower radiant exposure (energy) in this curing protocol compared to the MI protocol.

For the change in temperature during curing (ΔT), significantly higher values were observed in all the HI groups. The significant difference between change in temperature for the two curing methods for the same material was approximately 1.5 °C.

In a study by Zach and Cohen (22), it was revealed that an increase of 5.5 °C in intra-pulpal temperature resulted in irreversible pulp damage in 15% of the tested animals. So this change in temperature has been considered as the critical point of temperature rise in experiments since the Zach and Cohen study (23-25).

The change in temperature for the HI curing method for both composites showed a more than 5.5 °C (critical point) increase in temperature during polymerisation of the restorative material (table-16 and Fig-34). This change in temperature may be enough to cause irreversible changes in pulpal tissues, particularly if the remaining dentine thickness is minimal, e.g., in the order of 0.5 mm thick. One of the shortcomings of this study that should be considered is the variation in the thermal conductivity of dentine as a function of depth. The results of temperature change in the pulp chamber may be affected by this difference in the clinical situation. However, from a clinical standpoint, the use of this light for curing, particularly in deep cavities, should be approached with great caution.

In this study, a large MOD slot was prepared on maxillary premolar teeth in order to thin the tooth structure as a means to magnify cuspal deflection (Volume of the cavity $\approx 64 \text{ mm}^3$). As mentioned in previous studies, the degree of cuspal deflection is directly related to the amount of tooth loss. In addition, as the cavity size increases, more restorative material is required, which results in greater shrinkage forces and consequently more cuspal deflection (25)

Similar to previous studies, the total strain change (ΔS) and Total dimensional change (ΔD) values indicated an inward movement of the remaining tooth cusps (10). All samples displayed a higher ΔS and ΔD for the HI curing method compared with the MI method for the same material. As a result, the null hypothesis: "Different intensities of light curing devices do not affect the cuspal strain, and movement" was rejected. The higher values for the HI curing method indicate rapid polymerisation of the restorative material, which was a result of shorter exposure time greater amount of energy

within the short time frame. Higher ΔS values were observed for Sonicfill compared to Aura in this experiment. It is important to mention again that the stress is a multifactorial property which is dependent on physical and mechanical properties of the material such as modulus of elasticity, bonding and degree of conversion and the shape of the tooth and cavity. So, this variation may be an indication of a difference in modulus of elasticity. Also, it may be said that natural teeth have differences in dimension and quality of structure, which, may also affect the stress results obtained.

There are few studies on cuspal flexure in relation to polymerisation shrinkage of bulk-fill resin composites (9, 10, 26, 27). Some have reported that no differences were observed between the cuspal flexure of conventional and bulk-fill resin composites (10, 27) and some studies on cuspal movement reported a higher movement for teeth restored with conventional resin composite compared to bulk fill (9, 26). Since the methods of measuring cuspal flexure and movement in these studies were different from this experiment, a direct comparison of results is very difficult to make. These methods included: Fiber Bragg grating sensors, 3D scanning and twin channel deflection measuring gauge.

One of the main shortcomings of tests on natural teeth is that the natural teeth can show large variations in size, dentine or enamel quality, as well as internal structural differences. These differences may influence the results and change the test environment and response for each sample. Also, due to the limitations of the number of extracted teeth obtained, it was not possible to include conventional resin composites and other light curing methods. So further investigations are recommended to compare the effects of conventional and bulk fill composites on cuspal flexure.

In conclusion, the null hypotheses were rejected, different irradiances of light curing devices affected cuspal strain, cusp movement and temperature change in the pulp chamber. Hence, it is important to carefully choose a suitable curing light and method for clinical application, especially in deeper cavities. Also, the hardness of bulk-fill resin composites after light curing seems to gradually decrease as a function of depth, with significantly lower values at a depth of 4mm were recorded in this study. Based on these results, the materials tested would appear to have failed the requirements set out for the depth of cure for the ISO Standard 4049. Based on the finding of this study, the tested

bulk-fill materials are not recommended for 4mm deep restorations and increments with less than 4mm thickness should be placed to achieve 80% hardness at depth.

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Chapter 9: General discussion and conclusion

The most common material that is currently used for direct posterior restorations is resin composite (1). As mentioned in previous chapters, the recently introduced materials in this category are the bulk-filled resin composites. To better understand the polymerisation shrinkage, flexural strength, hardness and wear of these new materials, three experiments were designed, which were reported in chapters 6 to 8. Comparing the bulk-fill resin composites to conventional resin composites that were tested, the bulk filled materials, in general, showed higher flexural strength after ageing, a lower wear volume coefficient, reduced hardness when measured at a depth of 4 mm from the material surface (chapter 7 & 8). It was observed that in comparison with the conventional materials, the bulk-filled materials showed no significant difference in polymerisation shrinkage stress values (chapter 6). By using a hardness test, the bulk-fill resin composites revealed that they had not reached 'adequate' polymerisation at 4mm, which was measured as a reduction in hardness (chapter 8) (2).

In several previous studies, higher flexural strength (3) and the same or lower polymerisation shrinkage stress (4-10) was reported for bulk-fill compared with conventional resin composites. Although the test methods are different among studies, the same trends were observed. The experiments in this thesis also showed the same patterns in the results.

Flexural strength is considered the best measure of strength for dental materials and is defined as the maximum stress during flexion that a material can resist before failure (11). As previously reported, the composition of resin composites, such as filler content and resin matrix have an important influence on flexural strength (12). Higher flexural strength of a resin composite may be due to lower filler content, type or amount of resin in the restorative material. The same inverse correlation between Vol% filler and flexural strength was observed in the results of chapter 7. This may be due to a max amount of filler volume that can be added to the resin matrix after which a detection in mechanical properties occurs. Also, the finding of this study and previous studies indicates a decline in flexural strength of conventional resin composites when materials were exposed to various forms of accelerated ageing (13-15). In contrast, the bulk-fill resin composites showed a

slightly higher flexural strength after the ageing tests. The lower physical properties of materials after water storage is a result of the separation of polymer chains by water molecules (16). Water causes deterioration in resin composites by weakening the silane interface and leaching filler particles or softening the resin matrix due to hydrolysis (14). If filler particles and their interface with resin matrix are stable, the weak physical properties may be the result of plasticisation of the resin; this water imbibition continues until saturation is attained (17, 18). All of these factors result in a reduction in physical properties. So, the fact that flexural strengths of bulk-fill resin composites showed higher flexural strength after ageing may be due to a greater resistance of the resin matrix to hydrolysis, plasticisation or stronger connection at the silane interface. As reported by previous studies, flexural strength is a good indicator for the durability of material under stress, and it has been shown to correlate well with clinical longevity (19). So, it can be said that the improvement of flexural strength after ageing in the bulk-fill composites may result in better clinical longevity. This needs to be tested further with extensive long-term clinical evaluations.

Previous studies indicated that abrasive wear tends to lead to an increase in surface roughness and alter the surface topography of resin composites. A result of increased roughness will be greater formation of biofilm on the tooth and consequently an increase in the risk for recurrent caries (20-22). Wear volume coefficient of resin composites is also highly dependent on the composition of these materials, and due to factors such as: resin matrix, quality of silane interfacial adhesion, filler size, shape and quantity (20, 23, 24). A lower wear volume coefficient in bulk-fill resin composite also can be an indication of their stronger connection at the silane interface.

It has been suggested that the higher depth of cure in bulk-fill resin composites may be a result of an increased refractive index mismatch between resin and filler or a reduction in the amount of pigments and an enlarged the filler size (25-27). Most prior studies have reported a sufficient hardness value up to a 4mm depth for the bulk-fill composites (7, 8, 28, 29), which is in contrast to what was determined in the current study. There are some studies that have investigated the effect of high irradiance (intensity) LED light irradiation on bulk-fill composites (30-32). In the study by Daugherty et

al. (32), the effect of short exposure time with a high intensity light was studied, and that study showed a lower degree of polymerisation at a 4mm depth for the bulk-fill composites. In addition, Besegato et al. (30) found that regardless of the composition of a bulk-fill resin composite, the light-curing protocol with higher irradiance and shorter exposure time (higher power density) resulted in greater polymerisation shrinkage and lower hardness. These results are similar to those reported in the current work. This variation between reported results in studies is based on the dependence of this physical property on not only the resin composite composition but on other factors such as the total radiant energy output of the curing light. In another study, the benefits of a longer exposure time were investigated and concluded that longer light irradiation times such as 30s produces radiant energies which are in line with the recommendations from the current scientific literature, leading to adequate curing efficiency (33). It may, therefore, be concluded that short exposure time with high intensity curing lights leads to insufficient total radiant energy output to achieve satisfactory polymerisation in the deepest layers of bulk-fill composites.

The work in chapter 8 only investigated bulk-fill resin composites. In the literature, there is experimental work that compared the cuspal flexure of teeth restored with conventional or bulk-fill composites (28, 34-36). These studies showed there no differences in cuspal flexure of these two types of resin composite (28, 34), while there have been others which demonstrated less cuspal movement for bulk-filled composites (6, 35, 36). During polymerisation, the monomer molecules combine together to form a polymer network, that results in a smaller distance between monomer molecules within the formed polymeric structure. This reduction results in a densely cross-linked polymer and polymerisation shrinkage. When polymerisation shrinkage occurs within a restoration that is bonded to cavity surfaces, mechanical stresses will develop and be transmitted to the tooth-restoration interface (36-39). The polymerisation shrinkage stresses and subsequent cuspal flexure caused by resin composites can be related to their resin matrix, filler content, polymerisation kinetics, degree of conversion, and modulus of elasticity (7, 40). It was proposed that a higher filler content should be used to reduce resin volume, but it is not an efficient approach to reduce post-gel polymerisation

shrinkage (41) therefore, another approach was used to reduce the polymerisation shrinkage, which was a modified chemistry that slows the rate of polymerisation (7). Immediate lower polymerisation shrinkage and consequently lower cuspal flexure of bulk-fill resin composites are highly desirable due to these chemical modifications. Limited studies have reported an extended monitoring period for polymerisation shrinkage. In a study by Ide et al. (42) light curing results in less volumetric polymerisation shrinkage of bulk-fill composites immediate after curing, however, this value increased rapidly after 24 h to the same level as conventional resin composites. It has been reported that due to post-cure polymerisation in the first 24h after light curing, the degree of conversion of monomer to polymer in the resin composite increases (43). This later polymerisation is a result of the late reaction of remaining trapped radicals after light exposure (44). With time, these radicals reach pendant groups and their reaction results in additional conversion (42, 45). So, a reduced immediate polymerisation shrinkage is a result of the initial lower degree of conversion, which will increase after 24h due to post-curing polymerisation. It can be said the difference in the reported results on polymerisation shrinkage, and consequent cuspal flexure may be resolved after a longer monitoring time, and similar values of cuspal flexure would be seen for conventional and bulk-fill resin composite. Therefore, further studies are required to monitor the polymerisation shrinkage and cuspal flexure of teeth restored with bulk-fill resin composites for longer periods of time.

Another influential factor on the physical properties of resin composites that was investigated in this thesis was a recently introduced high irradiance (intensity) LED curing light device. The rapid exposure of high intensity devices (high power density output during exposure) caused higher final polymerisation shrinkage stress, higher strain and greater movement in remaining tooth cusps in all tested materials, although the total radiant energy output of this light curing method was half of the 'medium' irradiance (intensity) device, namely the conventional light curing method (chapter 6&8).

As reported in earlier studies, a high power density results in highly crosslinked polymers in which the developing network severely limits the mobility of the unreacted radicals. The rate of free radical polymerisation follows a characteristic pattern throughout the course of the reaction due to mobility

limitations. This pattern shows an early decrease in the rate of radical usage and an increase in the number of radicals. As the network develops further, radicals become trapped, and the polymerisation rate decreases and this results in a highly crosslinked polymer with lower conversion and unreacted monomer and radicals (45, 46), which results in higher polymerisation shrinkage stress consequently higher strain and greater movement of remaining tooth cusps. So, the power density of the curing protocol will strongly influence the physical properties of resin composites.

Another aspect that was tested in relation to the high irradiance (intensity) LED light curing device was temperature. This device increased the core temperature of resin composites to a concerning level and was much higher than the other light curing devices used. The rise in pulp chamber temperature was tested, revealing a higher temperature rise compared to the conventional LED light curing method (HI:5.8°C MI:4.4°C and 4.2°C). In the study by Zach and Cohen (47), they showed that an increase of 5.5 °C in intra-pulpal temperature could result in irreversible pulp damage in 15% of the tested animals. This change in temperature has been considered as the critical point value for maximum temperature rise in experiments (48-50). The greatest changes in temperature within the pulp chamber were associated with the high irradiance (intensity) curing light that recorded a temperature change of up to 5.8°C, which is above the critical point based on Zach and Cohen's work.

Other studies investigating high intensity LED curing light devices revealed adverse effects on all materials. There are very limited experiments in the literature investigating high intensity LED curing light units with an irradiance of more than 1600 mW/cm²(32). Considering the findings of the current study on negative effects of such high intensity curing lights on temperature change in the pulp chamber, polymerisation shrinkage stress, cuspal flexure and movement of remaining tooth structure, further studies are required to investigate the effects of high irradiance (intensity) LED curing light devices and to determine their clinical limitations for irradiance and exposure time.

In contrast to laboratory studies, clinical findings of bulk-fill resin composites are limited, and with a short monitoring time (51-57). One clinical trial has a monitoring period of 10 years (58). In this study, a success rate of 80.0% was reported for 45 multi-surface posterior bulk-fill resin composite

restorations which had a lower success than the 86.9% success rate of conventional resin composite restorations reported in chapter 3 (59). The main reported failure reasons were the same for conventional and bulk-fill resin composite restorations, namely: fracture and recurrent caries. This lower success rate may be due to some shortcomings that were found in the series of studies performed here, such as lower hardness at 4 mm deep. More evidence and longer observation times of clinical outcomes are necessary to determine the clinical performance of bulk-fill resin composites. So, further clinical experiments with longer duration are necessary.

In conclusion, bulk-fill resin composites possess similar physical properties compared with conventional resin composite. However, questions have been raised with regard to adequate polymerisation at a depth of 4 mm particularly with high irradiance and low radiant exposure. It can be said that they defeat their main purpose, bulk placement.

Also, the reduction in polymerisation time by the higher intensity curing light devices causes higher polymerisation stress and greater temperatures in tooth structure and the pulp chamber. So, care must be taken when using high irradiance (intensity) LED curing devices such that sufficient radiant energy is given to the resin composite to achieve the required degree of conversion. However, temperature increase in the resin composite, which can be conducted to the pulp, must be considered as the limiting factor in delivering sufficient energy at high irradiance for shorter period. So, it may be wiser to add a few seconds to a restorative procedure time and use a conventional intensity light curing device with a lower irradiance to achieve better physical properties and reduce the danger of overheating the pulpal tissues.

9.1: Future work:

Based on the outcomes of this series of studies, the following future experiments are required to expand the knowledge on this topic:

1. A study on polymerisation shrinkage and cuspal flexure of teeth restored with bulk-fill resin composites for 24 h.
2. A clinical investigation on the cuspal movement of teeth restored with conventional and bulk-fill resin composites.
3. A study to measure and compare the cuspal flexure of teeth restored with conventional and bulk-fill resin composites.
4. An investigation on the effect of high intensity lights on physical properties of other resin composite materials.
5. An animal study on the effects of high-intensity light curing devices on pulpal tissue.
6. More clinical studies on the long-term success of multi-surface bulk-fill resin composite restorations.

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Appendixes

Raw data of Chapter 5: Project II- Fracture toughness of glass ionomers measured with two different methods

(K _{IC} (MPa√m)) of 7 days storage and 4-point bending test						
	Beautifil	Chemfill	Equia with coating	Equia without coating	Riva	Riva HV
AS1	1.02	0.32	0.61	0.26	0.71	0.56
AS2	0.94	0.22	0.86	0.20	0.70	0.48
AS3	1.05	0.36	0.70	0.06	0.71	0.52
AS4	0.98	0.34	0.74	0.28	0.68	0.54
AS5	1.02	0.31	1.06	0.30	0.75	0.62
AS6	0.91	0.17	0.67	0.13	0.78	0.51
AS7	1.07	0.40	0.59	0.20	0.74	0.52
AS8	1.05	0.35	0.78	0.28	0.70	0.56
AS9	1.06	0.31	0.86	0.37	0.72	0.68
AS10	1.12	0.36	0.55	0.24	0.83	0.55
AS11	1.08	0.26	0.90	0.37	0.66	0.69
AS12	1.11	0.29	0.63	0.26	0.72	0.62
AS13	0.95	0.35	0.84	0.46	0.67	0.53
AS14	0.99	0.34	1.36	0.27	0.79	0.69
AS15	1.02	0.22	0.57	0.25	0.67	0.62
DW1	1.03	0.23	0.59	0.44	0.78	0.50
DW2	1.01	0.27	0.66	0.26	0.89	0.50
DW3	1.05	0.46	0.62	0.25	0.76	0.63
DW4	0.94	0.32	0.60	0.24	0.74	0.56
DW 5	1.02	0.28	0.66	0.21	0.77	0.56
DW 6	1.09	0.41	0.84	0.31	0.76	0.52
DW 7	0.99	0.34	0.78	0.20	0.70	0.60
DW 8	1.11	0.37	0.67	0.23	0.73	0.71
DW 9	0.95	0.39	0.72	0.16	0.82	0.58
DW 10	1.02	0.44	0.72	0.24	0.72	0.65
DW 11	1.23	0.45	0.87	0.26	0.72	0.46
DW 12	1.03	0.46	0.94	0.16	0.82	0.59

	Beautifil	Chemfill	Equia with coating	Equia without coating	Riva	Riva HV
DW 13	1.09	0.35	0.79	0.42	0.79	0.65
DW 14	1.10	0.43	0.72	0.41	0.64	0.65
DW 15	1.03	0.47	0.57	0.33	0.76	0.67

(K _{IC} (MPa√m)) of 30 days storage and 4 point bending test						
	Beautifil	Chemfill	Equia with coating	Equia without coating	Riva	Riva HV
AS1	1.00	0.40	0.60	0.39	0.63	0.55
AS2	1.05	0.43	0.73	0.32	0.68	0.73
AS3	1.19	0.41	0.67	0.27	0.70	0.74
AS4	0.98	0.51	0.68	0.18	0.68	0.84
AS5	1.06	0.58	0.77	0.34	0.82	0.44
AS6	1.04	0.45	0.77	0.33	0.68	0.66
AS7	1.11	0.48	0.76	0.29	0.88	0.75
AS8	1.17	0.49	0.74	0.40	0.73	0.70
AS9	1.12	0.30	0.61	0.11	0.78	0.38
AS10	1.14	0.47	0.86	0.38	0.75	0.58
AS11	1.11	0.41	0.81	0.43	0.79	0.57
AS12	0.96	0.42	0.62	0.38	0.60	0.65
AS13	1.05	0.45	0.63	0.49	0.73	0.65
AS14	1.11	0.36	0.96	0.50	0.76	0.67
AS15	0.94	0.45	0.77	0.50	0.78	0.64
DW1	1.13	0.44	0.81	0.40	0.94	0.74
DW2	1.03	0.40	0.75	0.38	0.82	0.91
DW3	1.15	0.57	0.98	0.35	0.58	0.69
DW4	1.13	0.48	0.73	0.37	0.83	0.83
DW 5	1.12	0.55	0.74	0.45	0.77	0.73
DW 6	0.97	0.60	0.79	0.39	0.80	0.76
DW 7	1.02	0.49	0.66	0.37	0.89	0.80
DW 8	1.08	0.60	0.67	0.42	0.75	0.84
DW 9	1.01	0.47	0.76	0.31	0.88	0.82
DW 10	1.15	0.43	0.71	0.35	0.90	0.75
DW 11	1.09	0.53	0.74	0.46	0.76	0.60
DW 12	1.24	0.50	0.61	0.40	0.74	0.46
DW 13	1.10	0.55	0.68	0.40	0.78	0.62
DW 14	1.05	0.45	0.77	0.46	0.74	0.60
DW 15	1.13	0.52	0.72	0.27	0.82	0.61

(K _{IC} (MPa√m)) of 7 days storage and CT test						
	Beautifil	Chemfill	Equia with coating	Equia without coating	Riva	Riva HV
AS1	1.01	0.41	0.63	0.40	0.82	0.89
AS2	1.09	0.38	0.60	0.39	0.71	0.73
AS3	1.03	0.39	0.56	0.33	0.73	0.79
AS4	0.95	0.35	0.54	0.35	0.70	0.74
AS5	1.13	0.40	0.50	0.34	0.72	0.74
AS6	0.92	0.43	0.53	0.46	0.80	0.67
AS7	1.08	0.33	0.57	0.41	0.76	0.69
AS8	1.14	0.44	0.84	0.38	0.67	0.65
AS9	1.15	0.48	0.60	0.40	0.90	0.73
AS10	1.11	0.44	0.53	0.42	0.64	0.52
AS11	1.11	0.47	0.62	0.29	0.73	0.62
AS12	1.19	0.47	0.58	0.34	0.58	0.62
AS13	0.93	0.56	0.63	0.24	0.86	0.57
AS14	0.89	0.45	0.68	0.31	0.72	0.69
AS15	0.89	0.49	0.55	0.25	0.84	0.44
DW1	1.22	0.43	0.69	0.34	0.55	0.73
DW2	1.08	0.52	0.53	0.44	0.75	0.67
DW3	0.80	0.39	0.70	0.44	0.79	0.69
DW4	1.25	0.47	0.56	0.31	0.95	0.87
DW 5	1.10	0.49	0.56	0.37	0.75	0.77
DW 6	1.11	0.43	0.67	0.41	0.80	0.76
DW 7	1.15	0.51	0.50	0.32	0.83	0.79
DW 8	1.08	0.37	1.11	0.38	0.68	0.73
DW 9	1.17	0.42	0.79	0.37	0.81	0.44
DW 10	1.06	0.33	0.86	0.35	0.90	0.76
DW 11	0.98	0.41	0.64	0.27	0.90	0.75
DW 12	0.85	0.52	0.76	0.40	0.83	0.71
DW 13	0.86	0.50	0.47	0.35	0.79	0.71
DW 14	1.05	0.47	0.58	0.36	0.72	0.84
DW 15	1.03	0.43	0.58	0.35	0.82	0.70

(K _{IC} (MPa√m)) of 30 days storage and CT bending test						
	Beautifil	Chemfill	Equia with coating	Equia without coating	Riva	Riva HV
AS1	1.20	0.49	0.56	0.30	0.82	0.89
AS2	1.17	0.49	1.21	0.43	0.77	0.79
AS3	1.19	0.47	0.83	0.37	0.83	0.73
AS4	1.28	0.47	0.74	0.32	0.76	0.85
AS5	1.21	0.53	0.55	0.40	0.81	0.76
AS6	1.30	0.51	0.71	0.50	0.85	0.73
AS7	1.26	0.41	0.84	0.24	0.80	0.62
AS8	1.11	0.46	1.01	0.39	0.81	0.79
AS9	1.26	0.52	0.94	0.37	0.74	0.67
AS10	1.05	0.58	0.57	0.37	0.74	0.63
AS11	1.14	0.45	1.31	0.43	0.77	0.67
AS12	1.19	0.43	0.58	0.37	0.74	0.68
AS13	1.26	0.42	1.11	0.37	0.85	0.60
AS14	0.93	0.50	0.56	0.26	0.73	0.68
AS15	1.14	0.61	0.71	0.44	0.73	0.88
DW1	1.24	0.45	0.72	0.36	0.70	0.68
DW2	1.27	0.50	1.18	0.37	0.87	0.71
DW3	1.05	0.50	0.99	0.39	0.88	0.78
DW4	1.13	0.50	0.56	0.49	0.88	0.78
DW 5	1.14	0.51	0.50	0.42	0.80	0.81
DW 6	1.31	0.52	0.60	0.28	0.80	0.77
DW 7	1.16	0.48	1.03	0.37	0.86	0.64
DW 8	1.20	0.48	0.56	0.44	0.83	0.83
DW 9	1.33	0.55	1.01	0.40	0.80	0.82
DW 10	1.10	0.49	1.17	0.22	0.81	0.70
DW 11	1.26	0.58	0.76	0.43	0.88	0.86
DW 12	1.29	0.65	0.60	0.37	0.96	0.69
DW 13	1.21	0.52	0.68	0.36	0.84	0.86
DW 14	1.36	0.49	0.74	0.32	0.79	0.68
DW 15	1.15	0.52	0.82	0.47	0.81	0.87

Raw data of Chapter 6: Project III- Thermal and depth of cure analysis of linear shrinkage and polymerisation shrinkage stress of resin composites related to method and type of curing light

Ash test		
Material	wt %	vol %
SDR	64.47	49.65
SDR	64.96	54.08
SDR	64.55	51.57
Clearfil	58.65	57.76
Clearfil	58.78	60.76
Clearfil	58.40	55.03
Kalore	69.61	55.02
Kalore	69.66	53.55
Kalore	69.74	52.44
Ceram X	70.15	50.96
Ceram X	70.13	51.86
Ceram X	70.28	51.10
Sonicfill	71.41	63.72
Sonicfill	71.52	59.05
Sonicfill	71.56	63.48
Aura	72.41	53.82
Aura	72.41	53.78
Aura	72.43	56.09

1.8mm depth				
Material	Method	$\Delta T(^{\circ}\text{C})$	Max Temp ($^{\circ}\text{C}$)	Final δ_{pol} (MPa)
Kalore	QTH	13.04	36.58	1.58
Kalore	QTH	13.98	37.17	1.83
Kalore	QTH	15.34	38.75	1.45
Sonicfill	QTH	16.19	40.18	3.17
Sonicfill	QTH	13.23	37.66	2.64
Sonicfill	QTH	13.96	37.91	2.91
Ceram X	QTH	11.37	33.86	4.12
Ceram X	QTH	16.57	38.88	3.44
Ceram X	QTH	16.56	38.46	4.35
Clearfil	QTH	16.25	39.68	3.11
Clearfil	QTH	10.70	33.13	3.01
Clearfil	QTH	13.68	37.07	2.69
Aura	QTH	13.82	36.86	2.43
Aura	QTH	10.85	33.93	2.75
Aura	QTH	14.99	37.34	2.70
Kalore	LED C	17.67	41.09	1.94
Kalore	LED C	20.94	44.23	2.47
Kalore	LED C	16.26	38.92	2.75
Sonicfill	LED C	12.37	35.32	2.48
Sonicfill	LED C	18.34	40.95	2.88
Sonicfill	LED C	13.40	36.99	2.20
Ceram X	LED C	16.32	39.12	3.18
Ceram X	LED C	21.56	44.20	3.42
Ceram X	LED C	15.72	38.60	2.78
Clearfil	LED C	15.59	40.11	2.85
Clearfil	LED C	20.86	43.53	3.34
Clearfil	LED C	17.14	39.72	2.49
Aura	LED C	15.34	38.52	2.67
Aura	LED C	17.12	39.89	3.45
Aura	LED C	12.73	36.19	2.64
Kalore	LED R	13.93	36.63	2.56
Kalore	LED R	20.15	43.40	1.99
Kalore	LED R	13.43	37.04	1.79
Sonicfill	LED R	14.64	37.70	3.33
Sonicfill	LED R	11.78	34.71	2.79
Sonicfill	LED R	15.36	38.91	2.70
Ceram X	LED R	14.51	37.50	2.76
Ceram X	LED R	14.55	38.77	3.36
Ceram X	LED R	15.24	38.60	3.01
Clearfil	LED R	19.55	43.41	2.26
Clearfil	LED R	17.44	41.24	2.61
Clearfil	LED R	16.51	39.94	2.24

Material	Method	$\Delta T(^{\circ}\text{C})$	Max Temp ($^{\circ}\text{C}$)	Final δ_{pol} (MPa)
Aura	LED R	13.36	37.19	1.86
Aura	LED R	14.39	38.15	2.92
Aura	LED R	17.22	40.14	2.64
Kalore	HI	51.88	75.88	5.10
Kalore	HI	47.83	71.95	4.17
Kalore	HI	65.14	89.30	5.23
Sonicfill	HI	47.84	71.06	4.11
Sonicfill	HI	52.20	75.76	3.63
Sonicfill	HI	57.19	80.76	5.10
Ceram X	HI	59.40	83.04	5.07
Ceram X	HI	58.92	82.83	5.83
Ceram X	HI	55.46	78.94	4.78
Clearfil	HI	55.26	79.13	4.01
Clearfil	HI	52.85	76.58	4.74
Clearfil	HI	47.68	71.08	4.82
Aura	HI	53.43	76.67	5.22
Aura	HI	51.61	75.27	4.62
Aura	HI	50.86	74.31	4.92

5mm depth				
Material	Method	ΔT (°C)	Max Temp (°C)	Final δ_{pol} (MPa)
Kalore	QTH	17.55	41.25	2.62
Kalore	QTH	13.99	37.85	2.18
Kalore	QTH	16.60	40.58	2.39
Sonicfill	QTH	17.81	41.25	4.53
Sonicfill	QTH	18.30	41.74	4.54
Sonicfill	QTH	15.18	38.90	4.91
Ceram X	QTH	23.05	46.68	5.59
Ceram X	QTH	20.96	44.24	4.26
Ceram X	QTH	20.40	43.67	4.33
Clearfil	QTH	14.38	37.48	3.25
Clearfil	QTH	16.14	39.25	3.08
Clearfil	QTH	16.11	39.33	3.32
Aura	QTH	17.42	41.67	4.29
Aura	QTH	18.72	42.83	4.51
Aura	QTH	18.79	43.17	4.03
Kalore	LED C	15.49	38.89	2.02
Kalore	LED C	16.62	40.63	2.96
Kalore	LED C	11.01	34.68	1.20
Sonicfill	LED C	20.77	44.89	5.32
Sonicfill	LED C	18.59	42.26	2.28
Sonicfill	LED C	18.71	42.86	4.94
Ceram X	LED C	20.56	44.69	4.99
Ceram X	LED C	19.14	43.64	4.00
Ceram X	LED C	17.80	42.28	3.05
Clearfil	LED C	16.41	40.92	3.37
Clearfil	LED C	16.66	40.91	3.09
Clearfil	LED C	17.22	41.67	2.21
Aura	LED C	17.30	41.61	3.92
Aura	LED C	20.39	44.98	5.18
Aura	LED C	20.39	44.98	5.18
Kalore	LED R	14.23	40.78	2.62
Kalore	LED R	15.64	39.37	2.06
Kalore	LED R	14.94	38.16	2.06
Sonicfill	LED R	17.32	40.96	3.27
Sonicfill	LED R	15.51	39.00	4.13
Sonicfill	LED R	14.51	37.98	3.69
Ceram X	LED R	18.07	41.23	3.95
Ceram X	LED R	17.78	41.40	3.78
Ceram X	LED R	16.47	40.45	2.74

Material	Method	ΔT (°C)	Max Temp (°C)	Final δ_{pol} (MPa)
Clearfil	LED R	15.02	38.09	1.76
Clearfil	LED R	15.73	39.69	3.03
Clearfil	LED R	14.11	38.00	2.84
Aura	LED R	11.93	36.03	2.55
Aura	LED R	18.67	42.52	4.59
Aura	LED R	18.13	41.90	3.71
Kalore	HI	31.62	54.76	4.94
Kalore	HI	39.43	62.42	5.55
Kalore	HI	36.15	59.79	5.55
Sonicfill	HI	30.00	53.46	6.03
Sonicfill	HI	36.56	60.45	6.39
Sonicfill	HI	40.35	63.90	6.86
Ceram X	HI	38.44	62.32	7.30
Ceram X	HI	42.26	66.49	7.97
Ceram X	HI	32.96	56.48	6.83
Clearfil	HI	32.83	56.68	5.70
Clearfil	HI	31.66	55.63	6.06
Clearfil	HI	38.49	62.63	6.27
Aura	HI	37.42	61.06	4.85
Aura	HI	35.79	59.98	6.67
Aura	HI	37.03	60.92	6.33

Raw data of Chapter 7: Project IV- Effect of thermal ageing on flexural strength, hardness and linear wear of bulk fill resin composites

Storage Temperature(°C)	Material	Flexural strength (MPa)	KHN
37	Clearfil	71.46	41.10
37	Clearfil	69.68	39.43
37	Clearfil	67.69	37.27
37	Clearfil	70.34	22.30
37	Clearfil	68.59	56.67
37	Clearfil	78.65	37.10
37	Aura	70.41	54.67
37	Aura	80.72	61.80
37	Aura	67.81	57.47
37	Aura	82.00	57.70
37	Aura	86.64	61.37
37	Aura	81.35	54.43
37	Kalore	74.49	63.93
37	Kalore	90.85	65.70
37	Kalore	84.18	46.47
37	Kalore	90.02	64.43
37	Kalore	96.58	65.30
37	Kalore	95.36	46.71
37	Ceram X	118.10	96.47
37	Ceram X	110.57	79.63
37	Ceram X	122.74	53.37
37	Ceram X	122.78	44.33
37	Ceram X	113.93	78.47
37	Ceram X	113.34	58.60
37	Sonicfill	74.00	62.13
37	Sonicfill	74.71	69.37
37	Sonicfill	80.85	50.47
37	Sonicfill	72.68	80.43
37	Sonicfill	61.64	72.07
37	Sonicfill	87.01	77.70
60	Clearfil	48.49	40.90
60	Clearfil	52.27	48.33
60	Clearfil	64.70	44.23
60	Clearfil	85.81	52.63

Storage Temperature(°C)	Material	Flexural strength (MPa)	KHN
60	Clearfil	75.03	59.70
60	Clearfil	65.84	45.40
60	Aura	43.53	49.17
60	Aura	79.30	48.73
60	Aura	76.67	40.80
60	Aura	94.09	49.90
60	Aura	86.06	44.83
60	Aura	89.73	32.97
60	Kalore	76.36	46.40
60	Kalore	59.02	71.70
60	Kalore	110.15	77.67
60	Kalore	96.20	68.67
60	Kalore	81.06	55.47
60	Kalore	89.77	69.27
60	Ceram X	91.64	107.83
60	Ceram X	88.68	97.80
60	Ceram X	94.48	82.33
60	Ceram X	99.69	75.00
60	Ceram X	102.38	81.20
60	Ceram X	56.27	87.83
60	Sonicfill	74.18	95.53
60	Sonicfill	71.92	59.57
60	Sonicfill	76.13	78.43
60	Sonicfill	67.37	88.80
60	Sonicfill	99.62	88.20
60	Sonicfill	89.48	53.67

Storage Temperature(°C)	Material	Volume wear coefficient mil(mm ³ /Nm)	Mass wear coefficient (g/Nm)
37	Clearfil	256.23	0.15
37	Clearfil	219.11	0.13
37	Clearfil	260.06	0.15
37	Aura	448.89	0.25
37	Aura	380.77	0.28
37	Aura	398.08	0.18
37	Kalore	789.47	0.28
37	Kalore	398.31	0.14
37	Kalore	614.80	0.22
37	Ceram X	358.74	0.16
37	Ceram X	347.49	0.02
37	Ceram X	244.00	0.11
37	Sonicfill	554.93	0.09
37	Sonicfill	619.30	0.16
37	Sonicfill	293.85	0.06
60	Clearfil	452.80	0.02
60	Clearfil	510.37	0.11
60	Clearfil	518.18	0.32
60	Aura	294.96	0.05
60	Aura	317.57	0.10
60	Aura	343.01	0.06
60	Kalore	435.91	0.04
60	Kalore	1331.67	0.05
60	Kalore	1308.43	0.05
60	Ceram X	516.33	0.03
60	Ceram X	180.78	0.26
60	Ceram X	179.45	0.02
60	Sonicfill	192.45	0.23
60	Sonicfill	594.52	0.33
60	Sonicfill	376.33	0.27

Raw data of Chapter 8: Project V- Cuspal strains of teeth restored with different bulk-fill resin composites and modes of light curing

Material	Curing method	$\Delta T (^{\circ}\text{C})$	Total ΔS	Total $\Delta R (\mu)$
Aura	HI	6.55	269.11	78.12
Aura	HI	6.56	368.83	78.13
Aura	HI	4.70	458.88	68.36
Aura	HI	6.56	94.94	87.89
Aura	HI	5.63	245.04	78.12
Aura	HI	4.68	110.32	58.59
Aura	MI	5.63	146.48	68.36
Aura	MI	4.70	123.88	68.36
Aura	MI	3.76	245.04	78.12
Aura	MI	4.70	326.43	68.36
Aura	MI	3.75	138.35	58.59
Aura	MI	3.76	346.32	87.89
Sonicfill	HI	6.57	235.10	78.12
Sonicfill	HI	6.57	234.19	68.36
Sonicfill	HI	6.57	209.78	68.36
Sonicfill	HI	4.70	205.26	68.36
Sonicfill	HI	4.70	232.39	78.13
Sonicfill	HI	5.63	548.86	78.13
Sonicfill	MI	4.70	177.23	58.60
Sonicfill	MI	3.76	203.45	68.36
Sonicfill	MI	4.70	183.56	58.59
Sonicfill	MI	3.76	525.35	58.59
Sonicfill	MI	5.63	152.81	78.12
Sonicfill	MI	2.82	313.77	78.12

Material	Curing method	Hardness (VHN) 4mm depth	Hardness (VHN) 3mm depth	Hardness (VHN) 2mm depth	Hardness (VHN) 1mm depth	Hardness (VHN) 0.25mm depth
Aura	HI	45.98	50.32	58.10	60.52	59.28
Aura	HI	50.73	67.82	58.73	66.65	69.68
Aura	HI	48.97	60.12	63.17	77.42	63.33
Aura	HI	46.13	60.32	57.98	66.28	73.37
Aura	HI	50.40	58.67	67.85	68.22	78.93
Aura	HI	48.20	58.17	63.82	65.28	67.00
Aura	MI	55.92	57.47	64.37	67.38	65.90
Aura	MI	46.30	53.90	56.60	55.70	70.07
Aura	MI	46.58	70.40	73.80	64.65	92.57
Aura	MI	46.28	49.73	54.37	61.07	75.93
Aura	MI	54.37	45.67	56.22	61.93	75.52
Aura	MI	47.17	42.07	51.15	59.75	63.07
Sonicfill	HI	79.63	81.42	92.65	102.35	94.28
Sonicfill	HI	67.52	77.83	80.87	95.10	83.45
Sonicfill	HI	62.80	76.75	85.90	77.07	77.08
Sonicfill	HI	70.90	87.42	81.63	84.63	92.52
Sonicfill	HI	71.73	84.42	99.65	92.00	98.13
Sonicfill	HI	66.43	80.83	81.67	85.35	123.17
Sonicfill	MI	90.35	107.83	100.52	93.33	113.22
Sonicfill	MI	83.08	80.08	92.48	86.23	104.07
Sonicfill	MI	74.12	90.75	76.02	91.47	99.10
Sonicfill	MI	68.57	79.38	72.18	85.27	69.22
Sonicfill	MI	68.98	65.83	73.02	69.12	97.10
Sonicfill	MI	82.27	69.40	78.30	77.03	93.22

**THE UNIVERSITY OF MELBOURNE
HUMAN RESEARCH ETHICS COMMITTEE**

**APPLICATION SUMMARY FOR
APPROVAL OF HUMAN RESEARCH**



1. ADMINISTRATION DETAILS

ETHICS ID: 1748725.1

TITLE: Cuspal strains of teeth restored with different bulk-fill resin composites and modes of light curing

APPLICATION TYPE: Project Application **RESPONSIBLE RESEARCHER:** Burrow, PROF Michael Francis

RESPONSIBLE HEAG: Melbourne Dental School **HESC:** Medicine and Dentistry

ADMINISTERING DEPARTMENT: 5110 - Melbourne Dental School **ADMINISTERING CENTRE:** (if applicable)

2. PROJECT DETAILS

PROJECT TYPE: Supervised Student Research Project - PhD

RESEARCH INVOLVES: Collections or use of biospecimens

BRIEF DESCRIPTION: The aim is to determine the effect of different curing lights and resin-based materials on the cuspal movement of posterior teeth after restoration placement. Resin composites are showing improved long-term clinical success. Their failure is mainly fracture or secondary caries. In view of this, polymerization shrinkage effects on cuspal movement are considered influential on clinical outcomes and remain one of the factors where more evidence is needed. This study aims to gain a better understanding of how resin-based materials contract and tooth structure moves. Strain gauges will be placed on teeth to measure material effects during polymerisation.

PROPOSED DURATION OF WHOLE RESEARCH PROJECT: **From:** SEP-2017 **To:** SEP-2018

PROPOSED DATE TO COMMENCE DATA COLLECTION: 01-Mar-2017

3. PERSON DETAILS

Responsible Researcher

Name	Burrow, Prof Michael	Department	5110 - Melbourne Dental School
Person Type	Staff	Centre	
Phone Number	9341 1474	Email Address	mfburrow@unimelb.edu.au
Qualifications	Bachelors Degree, Adelaide University Masters (Coursework), Adelaide University Masters (Research), University of Melbourne PhD, Tokyo Medical and Dental University Post Doctorate, Adelaide University		
Experience & Skills Relevant to the Project	dental material experience, resin-based restorative material experience		
Additional Training Required			
Ethics Training Already Undertaken			

Student Researcher

Name	Alvanforoush, Negin	Department	
Person Type	Student	Centre	
Phone Number		Email Address	n.alvanforoush@student.unimelb.edu.au
Qualifications			
Experience & Skills Relevant to the Project	dental material experience		
Additional Training Required			
Ethics Training Already Undertaken			
Student Supervisor(if applicable)	Prof. Burrow		

Co researcher

Name	Wong, Dr Rebecca	Department	5110 - Melbourne Dental School
Person Type	Staff	Centre	
Phone Number	93411517	Email Address	rhkwong@unimelb.edu.au
Qualifications	Bachelors Degree, University of Melbourne Graduate Diploma, University of Melbourne Post Graduate Certificate, University of Melbourne Doctorate (Research), University of Melbourne Masters (Coursework & Research), University of Melbourne Masters (Coursework & Research), University of Melbourne		
Experience & Skills Relevant to the Project	dental material experience, resin-based restorative material experience		
Additional Training Required			
Ethics Training Already Undertaken			

Co researcher

Name	Palamara, A/Prof Joseph	Department	5110 - Melbourne Dental School
Person Type	Staff	Centre	
Phone Number	93411532	Email Address	palamara@unimelb.edu.au
Qualifications	Bachelors Degree (Honours), Monash University PhD, Monash University		
Experience & Skills Relevant to the Project	dental material experience, resin-based restorative material experience		
Additional Training Required			
Ethics Training Already Undertaken			

4. ADDITIONAL QUESTIONS

5. ATTACHMENTS

PLEASE ENSURE YOU ATTACH A PAPER COPY OF EACH OF THE FOLLOWING ATTACHMENTS:

Category	Description	Attached Via Themis	Hard Copy Only
Additional Module		Yes	No
Application		Yes	No
Consent Form		Yes	No
Miscellaneous	Updated PLS included	Yes	No
Miscellaneous		Yes	No
Plain Language Statement		Yes	No



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HUMAN RESEARCH ETHICS COMMITTEE**

ANNUAL REPORT for 2017

ADMINISTRATION DETAILS

APPLICATION TYPE:	Project Application	ETHICS ID:	1748725
APPROVAL CATEGORY:	HESC	ANNUAL REPORT NO:	1
RESPONSIBLE HEAG:	Melbourne Dental School	ANNUAL REPORT FOR YEAR:	2017
HESC:	Medicine and Dentistry	SUBMITTED BY:	Negin Alvanforoush
MAXIMUM EXPIRY DATE:	17 October 2022	STATUS OF ANNUAL REPORT:	Approved (set at submission)

1. PROJECT DETAILS

TITLE: Cuspal strains of teeth restored with different bulk-fill resin composites and modes of light curing

Role	Name	Person Type	Department/Organisation
Responsible Researcher	Burrow, Prof Michael	Staff	5110 - Melbourne Dental School
Co researcher	Palamara, A/Prof Joseph	Staff	5110 - Melbourne Dental School
Co researcher	Wong, Dr Rebecca	Staff	5110 - Melbourne Dental School
Student Researcher	Alvanforoush, Negin	Student	

2. PROJECT STATUS

2.1 Status of Project: Continuing - data collection not yet complete

Estimated Completion Date: 14 February 2019

If the data collection phase is COMPLETE and no further interaction with participants is envisaged submission of this report will then constitute the FINAL ethics report for the project.

If the project is NOT YET COMMENCED note that it is necessary to complete and submit the report, including the questions relating to research over the past twelve months.

If the project is NEVER COMMENCED note that it is only necessary to complete the field requesting an explanation as to why the project was abandoned. No further questions will need to be completed.

2.2 Statement of progress.

The Collection of teeth is in progress.

3. IN THE LAST TWELVE MONTHS

3.1 Have all the conditions of approval required by the HEAG and Human Ethics Sub-Committee been met? If

NO please provide details below.

Yes

- 3.2 Have there been any changes to procedures or direction of the approved protocol (including the source, number and manner or recruiting participants)? If YES please provide details below.**

No

3.2.1 Has approval been sought for these changes?

If approval has NOT been sought, please note that before continuing approval for this project can be granted a Request for Amendment to the project must be submitted to your HEAG. This can be done by completing and submitting your request via Themis Research.

- 3.3 Have there been any changes to the consent procedures or documentation that were approved in the original protocol since your last report? If YES please provide details below.**

No

- 3.4 Has the project been monitored as detailed in the approved protocol? If NO please provide details below.**

Yes

- 3.5 Has data been stored securely, as detailed in the original protocol? If NO please provide details below.**

Yes

- 3.6 Have there been any incidents/unforeseen problems during the project, including**

- data security problems
- adverse effects on participants
- unforeseen problems of an ethical nature
- withdrawal of participants from the project which has had a significant impact on the research (either because of the proportion of participants that have withdrawn, or because of the reasons for their withdrawal)

If YES please provide details below

No

- 3.7 Have any complaints been made about the conduct of the project? If YES please provide details below and explain what action was taken by researchers.**

No

- 3.8 How will your research findings be disseminated (e.g., conference papers, publications)?**

Conference paper and publication

4. CONTINUING PROJECTS

- 4.1 Do you propose to make any amendments to the project including SIGNIFICANT changes to the plain language statement(s) and consent form(s) that will need approval for use in the coming year?**

Not required

If 'YES' you will need to submit a Request for Amendment to your HEAG before new work can proceed. This can be done by completing and submitting your request via Themis Research.

- 4.2 Have you reviewed (and updated where necessary) all current advertisements, plain language statements and consent forms to be used in the coming year (e.g., dates, names and contact details of researchers, etc.)?**

Yes

- 4.3 Can you please confirm that the relevant contact details for the HREC are included in documentation to be provided to participants?**

Yes

Note that it is a requirement of approval that participants be provided with contact details for the approving HREC. These details should be included in any plain language statements or other documentation provided to participants. (Note that the relevant contact number for the Executive Officer, HREC, at The University of Melbourne is 8344 2073.)

- 4.4 Please confirm that all people or groups (for example, co-researchers, research assistants, schools, hospitals, organizations) involved in recruitment have been instructed in the informed consent procedures approved by the approving HEAG/HESC?**

Yes, people/groups involved in recruitment have been instructed in informed consent procedures

5. OTHER COMMENTS

- 5.1 Please include any additional comments you may wish to make regarding this research.**



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HUMAN RESEARCH ETHICS COMMITTEE**

ANNUAL REPORT for 2018

ADMINISTRATION DETAILS

APPLICATION TYPE:	Project Application	ETHICS ID:	1748725
APPROVAL CATEGORY:	HESC	ANNUAL REPORT NO:	2
RESPONSIBLE HEAG:	Melbourne Dental School	ANNUAL REPORT FOR YEAR:	2018
HESC:	Medicine and Dentistry	SUBMITTED BY:	Negin Alvanforoush
MAXIMUM EXPIRY DATE:	17 October 2022	STATUS OF ANNUAL REPORT:	Approved (set at submission)

1. PROJECT DETAILS

TITLE: Cuspal strains of teeth restored with different bulk-fill resin composites and modes of light curing

Role	Name	Person Type	Department/Organisation
Responsible Researcher	Burrow, Prof Michael	Staff	5110 - Melbourne Dental School
Co researcher	Palamara, A/Prof Joseph	Staff	5110 - Melbourne Dental School
Co researcher	Wong, Dr Rebecca	Staff	5110 - Melbourne Dental School
Student Researcher	Alvanforoush, Negin	Student	

2. PROJECT STATUS

2.1 Status of Project: Continuing - data collection not yet complete

Estimated Completion Date: 15 August 2019

If the data collection phase is COMPLETE and no further interaction with participants is envisaged submission of this report will then constitute the FINAL ethics report for the project.

If the project is NOT YET COMMENCED note that it is necessary to complete and submit the report, including the questions relating to research over the past twelve months.

If the project is NEVER COMMENCED note that it is only necessary to complete the field requesting an explanation as to why the project was abandoned. No further questions will need to be completed.

2.2 Statement of progress.

We are still collecting extracted teeth.

3. IN THE LAST TWELVE MONTHS

3.1 Have all the conditions of approval required by the HEAG and Human Ethics Sub-Committee been met? If

NO please provide details below.

Yes

- 3.2 Have there been any changes to procedures or direction of the approved protocol (including the source, number and manner or recruiting participants)? If YES please provide details below.**

No

3.2.1 Has approval been sought for these changes?

If approval has NOT been sought, please note that before continuing approval for this project can be granted a Request for Amendment to the project must be submitted to your HEAG. This can be done by completing and submitting your request via Themis Research.

- 3.3 Have there been any changes to the consent procedures or documentation that were approved in the original protocol since your last report? If YES please provide details below.**

No

- 3.4 Has the project been monitored as detailed in the approved protocol? If NO please provide details below.**

Yes

- 3.5 Has data been stored securely, as detailed in the original protocol? If NO please provide details below.**

Yes

- 3.6 Have there been any incidents/unforeseen problems during the project, including**

- data security problems
- adverse effects on participants
- unforeseen problems of an ethical nature
- withdrawal of participants from the project which has had a significant impact on the research (either because of the proportion of participants that have withdrawn, or because of the reasons for their withdrawal)

If YES please provide details below

No

- 3.7 Have any complaints been made about the conduct of the project? If YES please provide details below and explain what action was taken by researchers.**

No

- 3.8 How will your research findings be disseminated (e.g., conference papers, publications)?**

publication

4. CONTINUING PROJECTS

- 4.1 Do you propose to make any amendments to the project including SIGNIFICANT changes to the plain language statement(s) and consent form(s) that will need approval for use in the coming year?**

Not required

If 'YES' you will need to submit a Request for Amendment to your HEAG before new work can proceed. This can be done by completing and submitting your request via Themis Research.

- 4.2 Have you reviewed (and updated where necessary) all current advertisements, plain language statements and consent forms to be used in the coming year (e.g., dates, names and contact details of researchers, etc.)?**

Yes

- 4.3 Can you please confirm that the relevant contact details for the HREC are included in documentation to be provided to participants?**

Yes

Note that it is a requirement of approval that participants be provided with contact details for the approving HREC. These details should be included in any plain language statements or other documentation provided to participants. (Note that the relevant contact number for the Executive Officer, HREC, at The University of Melbourne is 8344 2073.)

- 4.4 Please confirm that all people or groups (for example, co-researchers, research assistants, schools, hospitals, organizations) involved in recruitment have been instructed in the informed consent procedures approved by the approving HEAG/HESC?**

Yes, people/groups involved in recruitment have been instructed in informed consent procedures

5. OTHER COMMENTS

- 5.1 Please include any additional comments you may wish to make regarding this research.**

