The Effects of Instrument Lubricants on the Physical and Mechanical Properties of Resin-Based Composites

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Abstract

Uncured resin-based composites (RBCs) tend to stick to the placement instrument instead of cavity walls, potentially increasing void formation and margin discrepancy. Instrument lubricants (ILs) are used to overcome this problem, but they may affect the properties of RBC restorations. One hundred registered UK dentists were surveyed using a bespoke questionnaire to investigate their perspective on IL use and understand why and how they used them. Additionally, different laboratory investigations were conducted, based on the survey data, to test the effects of ILs on the physical and mechanical properties of RBCs. Two RBCs were treated with three classes of ILs —solvents, bonding agents and wetting resins —to investigate the effects different ILs have on the physical and mechanical properties of RBCs. Several areas were tested: degree of conversion, water uptake, Martens hardness, diametral tensile strength, microtensile bonding strength (μTBS), and the appearance of changes at the increment interface. The survey revealed that about 50% of the dentists used lubricants (32% response rate), of which bonding agents (67%) and wetting resins (33%) were most common. These were applied with microbrushes (47%) and by wiping the placement instruments (40%). The solvents, bonding agents and wetting resins created significant reductions in diametral tensile strength and Martens hardness, and increased water uptake compared to control groups fabricated without lubricants. The μTBS significantly reduced following treatment with solvents and bonding agents, but there was no reduction from the wetting resins. The respondents used lubricants to aid manipulation during placement. However, these materials have an impact upon physical and mechanical properties with solvents and bonding agents having the greater effect. Therefore, the use of ILs to manipulate the RBC should be limited or avoided.

Dedication

IN THE NAME OF ALLAH

And with His blessing

The All-Knowing, the Most-Wise

All praise to God whose grace sustains

I dedicate this work to the most important people in my life. The ones who have given me the motivation, drive, and energy to be where I am today.

First of whom are my **father** and **mother**. Although gone, their souls and memories are not forgotten.

And to my beautiful **family** whose continuous support was the catalyst for my achievements and success.

Finally, to my **friends** for being more than just friends. They became my second family during a journey full of ups and downs.

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List of Abbreviations

A	Acetone
ANCOVA	Analysis of covariance
ATR	Attenuated total reflection
B	Brush & Sculpt
BA	Bonding Agents
Bis-EMA	Ethoxylated bisphenol-A dimethacrylate
Bis-GMA	Bisphenol A glycidyl dimethacrylate
C	Control
CLD	Cross-linking density of polymerisation
CQ	Camphorquinone
CQC	Care Quality Commission
Cu	Copper
D3MA	Poly-decane-diol dimethacrylate
DC	Degree of conversion
DoC	Depth of cure
DTS	Diametral tensile strength
DW	Distilled water

E	Ethanol
EVA	Ethylene vinyl acetate
F	Filtek
FS	Flexural strength
FTIR	Fourier transform infrared spectroscopy
H	Harmonise
HEMA	2-hydroxyethyl methacrylate
HM	Martens hardness
I	Isopropyl
ILs	Instrument lubricants
LCU	Light curing unit
LED	Light-emitting diode
M	Modelling resin
MDP	10-Methacryloyloxydecyl dihydrogen phosphate
MIR	Mid infra-red
MPTMS	3-methacryloxypropyl trimethoxysilane
NHS	National Health Service
O	Optibond

OD	Optical density
PEGDMA	Polyethylene glycol dimethacrylate
PFM	Porcelain fused to metal
PMMA	Poly-methylmethacrylate
POSS	polyhedral oligomeric silsesquioxane
PPD	1-phenyl-1,2-propanedione
RBCRs	Resin-based composite restorations
RBCs	Resin-based composites
ROP	Ring-opening polymerisation
Rp	Fraction of the conversion of double to single bond in second
S	Scotchbond
SA	Initials of the principal researcher
SD	Standard deviation
SEBS	Styrene-ethylene/butylene-styrene
SEM	Scanning electron microscope
SL	Signum liquid
COIIA	Changes observation at increments interface area
TEGDMA	Triethylene glycol dimethacrylate

TPO	2,4,6-Trimethylbenzoyl-diphenylphosphine oxide
UDMA	Urethane dimethacrylate
UK	United Kingdom
WHO	World Health Organisation
WR	Wetting resins
WU	Water uptake
Zn	Zinc
γ-MPS	γ-(methacryloxy) propyltrimethoxy
μTBS	Microtensile bond strength

Chapter 1: Introduction

Many dentists encounter daily situations where they need to restore damaged dental tissues, and direct restorative materials are the main option for restoring function in a single visit (Opdam *et al.*, 2014). Different types of direct restorative materials have been introduced over the past 40 years and in dental practice resin-based composite (RBC) materials are widely used for restoring damaged teeth tissues (ADA Council on Scientific Affairs, 2003; Ástvaldsdóttir *et al.*, 2015). These materials have had satisfactory results when used to restore both anterior and posterior teeth (Alzraikat *et al.*, 2018). RBCs are currently seen as the best choice for avoiding mercury-containing restorative materials in direct placement restorations, and they can simultaneously enhance the aesthetics of direct restorations by their stability and longevity (Leprince *et al.*, 2013; Leprince *et al.*, 2014).

RBC materials have certain disadvantages related to their physical and mechanical properties, either before or after the polymerisation process they undergo when they are cured. With regard to handling, unset RBCs of different viscosity can stick to the instruments used to place them, creating difficulty for operators trying to condense and manipulate them. This behaviour raises the risk of increased inclusion of air or fluid and the subsequent creation of voids, and can also decrease the marginal adaptation of RBC materials to the prepared cavity. Different solutions have been introduced to reduce the stickiness of materials to the dental instruments used to place restorations. In the literature, the most widely reported solution is instrument lubricants (ILs) (Dunn, 2007; Barcellos *et al.*, 2008; Tuncer *et al.*, 2013; de Paula *et al.*, 2016; Sedrez-Porto *et al.*, 2016; Patel *et al.*, 2017). This method involves applying an IL either directly to the RBC or to the placement instruments. However, there is concern that ILs affect the

physical and mechanical properties of the RBC materials, impacting the stability and longevity of these restorations.

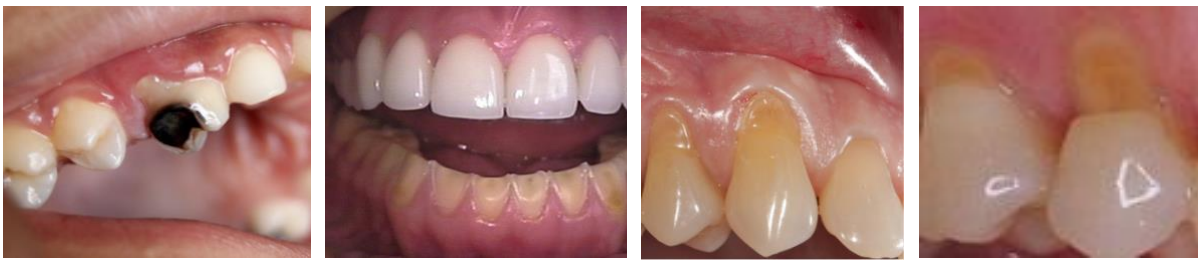
Certain points on this area of research require further elaboration in the literature, such as why and how UK dentists use ILs to manipulate RBC materials, and how ILs are perceived in this context. Furthermore, different types of RBCs can be combined with different ILs, and so this area also needs to be more fully investigated to determine and better understand the effects of ILs on the physical and mechanical properties of RBCs. Since there are few widely acceptable recommendations to help dentists decide on the use of ILs in their practice, it is hoped that this thesis will contribute in this regard.

After surveying why and how UK dentists use ILs, the study will conduct a laboratory investigation of the effects of the most frequently used ILs on the physical and mechanical properties of the most common RBCs. The project has three main phases: a narrative literature review summarising the previous work on ILs which will aid the manipulation of RBC materials; a survey of UK dentists answering questions on their knowledge and experience of using ILs in clinical practice; and, the aforementioned laboratory investigation.

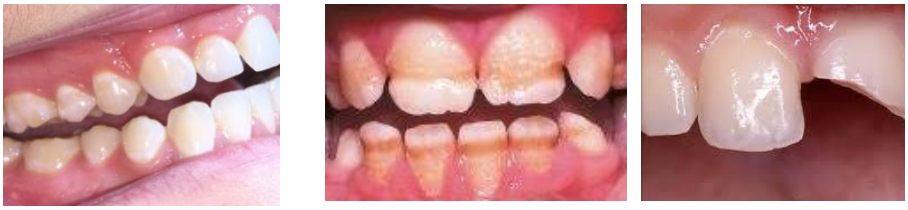
Chapter 2: Literature Review

2.1 Dental tissue defects

The hard dental tissues are most commonly damaged by caries but non-carious damage can also be caused by erosion, abrasion, abfraction, attrition, development defects and trauma. Figure 2-1 shows photographic examples of different kinds of tooth damage, for which a dentist may consider the use of a dental material such as a RBC (Americano *et al.*, 2017; Coupal, 2017; Janakiram;Deepan Kumar and Joseph, 2017; Wikipedia, 2020). The loss of hard tissues like enamel and dentine due to these causes leads to many critical problems for example patients experiencing pain. Essential functions such as mastication or the aesthetic of affected teeth can be affected, and issues may also arise with verbal communication, such that there is even a subsequent impact on the psychological health and social life of the affected person (Okoje *et al.*, 2012; Wong;Subar and Young, 2017). Therefore, replacing damaged tissues is important to restore optimum functionality and the best aesthetic outcome. RBCs are routinely used to repair such damage in dental practice, and indeed they can effectively address the consequences of damage to dental tissues (ADA Council on Scientific Affairs, 2003; Janakiram;Deepan Kumar and Joseph, 2017).



Caries Erosion Abrasion Abfraction



Attrition Development defects Dental trauma

Figure 2-1: Causes of dental hard tissues damages taken from Wikipedia, 2020

2.2 Restoration of damaged teeth tissue

The options available for treating damaged tissues are dependent on the area and progression of the tooth tissues damage (Horst, 2018; Philip;Suneja and Walsh, 2018). Once tissue damage has progressed beyond the ability of the tissue to be remineralised, then the only option available for tooth repair is a synthetic filling material. This will either be a direct restorative material placed by the dentist into the cavity, or an indirect restoration fabricated in a dental lab and then placed into the patient's mouth by the dentist. The most common materials used by dentists are direct restorative materials (DRM), through steps such as placement, finishing and polishing (ADA Council on Scientific Affairs, 2003; Demarco *et al.*, 2012).

2.3 Direct placement dental restorations

A number of materials have been used for this function throughout history but presently the two most commonly used classes are those based around alloys containing

mercury, termed dental amalgams, and glass-particle reinforced resin-based composites, called dental composites (Opdam *et al.*, 2010; Naghipur *et al.*, 2016). These two classes of DRM are used to restore damaged tissues with optimum functionality and aesthetics (ADA Council on Scientific Affairs, 2003; Pacheco *et al.*, 2018). Their development has involved mixed success in determining a suitable restorative material for damaged dental tissues, but they have been satisfactorily used in certain areas. For example, amalgam is a superior outcome in high-stress areas in the posterior teeth, while RBCs provide a better aesthetic outcome (Moraschini *et al.*, 2015; Naghipur *et al.*, 2016).

In addition to dental amalgam having superior mechanical and physical properties for restoring posterior teeth in comparison to RBCs, it also has greater stability and longevity in high-loaded areas in large class one and two cavities. Amalgam restorations within the oral cavity can be corroded in contact with oral fluids. The corrosion products can then accumulate in the gap between the restoration and the tooth material, potentially sealing the margins and reducing microleakage in this area and the occurrence of secondary caries. Indeed, the literature shows that dental amalgam restorations reduce the occurrence of secondary caries in high-risk patients a key cause of the failure of large restorations (Bernardo *et al.*, 2007; Chan *et al.*, 2010; Opdam *et al.*, 2010; Moraschini *et al.*, 2015; Wang;Habib and Zhu, 2017). Also, the placement of amalgam restorations is less technique sensitive, especially with the Zn-free amalgams, meaning they are easier to manipulate compared to RBCs (Naghipur *et al.*, 2016) because they can be placed and manipulated in the presence of saliva, (Opdam *et al.*, 2010).

However, dental amalgam has limitations related to the location of the restored tooth. It is generally used to restore specific teeth not in aesthetic areas, such as class one

and two cavities (ADA Council on Scientific Affairs, 2003; Khurshid *et al.*, 2015). Moreover, dental amalgam has lately been prohibited in some countries due to the environmental impact of Hg on health. In the literature, the evidence on the safety of amalgam is inadequate, despite some limited evidence noting that it may be linked to neurological disease, multiple sclerosis, and Parkinson's and Alzheimer's diseases (Mn, 2006), and may have some effect on the immunological and kidney functions. The main issue with its use in dental practice concerns amalgam handling because of mercury vapour (Uçar and Brantley, 2011; Alexander *et al.*, 2017), and indeed in the literature eliminating mercury-based amalgam is a matter of environmental protection rather than human toxicity. For this reason, the World Health Organization (WHO) recommends reducing the use of dental amalgam and supports the use of alternative materials. Also, the Minamata international convention in 2013 supported the banning of mercury and mercury products that would harm the environment and human health (Fisher *et al.*, 2018). Nevertheless, it remains an option for tooth restoration in some countries in certain clinical placement situations needing materials which utilize a less sensitive technique, and certain cases with a high risk of dental caries. Indeed, dentists still use dental amalgam for high-load areas such as posterior teeth (ADA Council on Scientific Affairs, 2003; Yip and Cutress, 2003; Uçar and Brantley, 2011; Van Landuyt *et al.*, 2011; Vervliet *et al.*, 2018).

2.4 Resin-based composites and their components

Due to the concerns mentioned in Section 2.3 on the use of dental amalgam in direct restorations, this section focuses on RBCs as a common type of DRM developed as an alternative to amalgam, especially for tooth restoration in aesthetic areas (Wang;Habib and Zhu, 2017; Wang *et al.*, 2018). RBCs were introduced as tooth-coloured restorations which come in various shades. This type of restorative material

underwent continuous development until satisfactory properties were achieved (Ferracane, 2011; Kubo, 2011; Leprince *et al.*, 2013; Sedrez-Porto *et al.*, 2016). The first use of acrylic-based material in dentistry was in the use of poly (methyl methacrylate) (PMMA) in denture fabrication, and this opened the way to the use of RBCs as restorative materials. Silicate cement was the first aesthetic DRM introduced in dentistry but it initially had inferior physical and mechanical properties in comparison to amalgam, especially regarding high solubility (Henschel, 1949). Much later, epoxy resin combined with aggregates of fused quartz or porcelain particles was introduced but it was slow to harden and so less suited to direct restoration (Bowen, 1956; Peutzfeldt, 1997; Pratap *et al.*, 2019). Resembling an epoxy, bisphenol A-glycidyl methacrylate (Bis-GMA) was also synthesised (Lavigueur;Christine and Zhu, 2011) but the epoxy group was replaced with a methacrylate group, as illustrated in Figure 2-2 (Markéta *et al.*, 2020). This material was introduced commercially in 1960 as new restorative material (Bowen, 1965). In 1970, a photo-polymerised resin composite system was introduced (Peutzfeldt, 1997) in parallel with the introduction of, and improvement to, adhesive bonding systems; the latter was an essential factor in the increased use of RBC restorations as DRM in dental practice (Ferracane, 2011; Kubo, 2011).

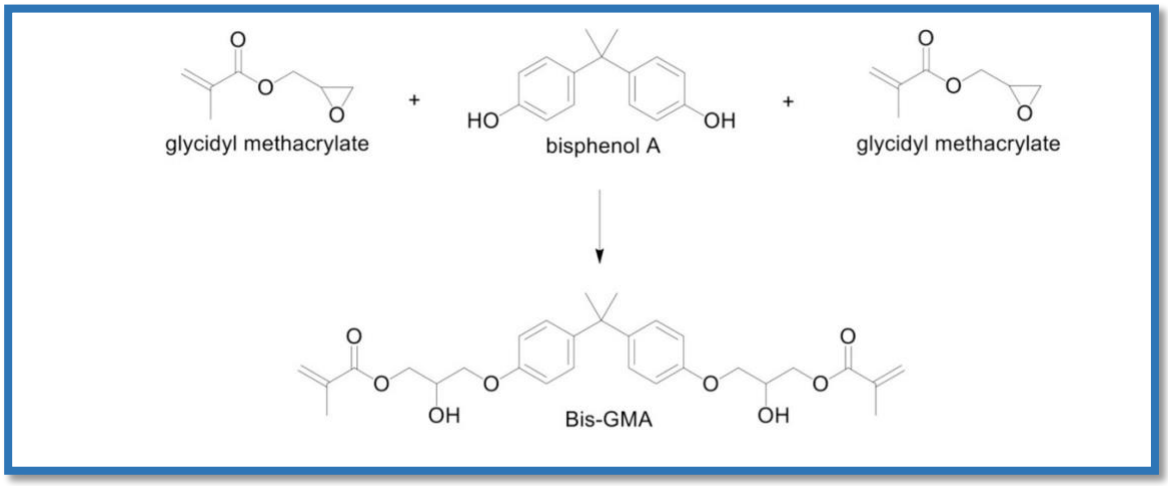


Figure 2-2: The chemical structure of the Bis-GMA from Markéta *et al.*, 2020

As reported in the literature from clinical practice, RBCs have many advantages and disadvantages as DRMs, the most important of which are summarised in Table 2-1 (Al-Sharaa and Watts, 2003; Eiriksson *et al.*, 2004; Ferracane, 2011; Van Landuyt *et al.*, 2011; Ferracane, 2013; Leprince *et al.*, 2013; Alvanforoush *et al.*, 2017; Barszczewska-Rybarek, 2019; Watts *et al.*, 2019).

Advantages	Disadvantages
More aesthetic and tooth-coloured	Polymerisation shrinkage and poor dimensional stability
Bonding to tooth structure and less tooth structure loss (conservative)	Low wear resistance
Repairable	More water uptake and staining
Strengthening of the tooth structure	Technique-sensitive materials
Low cost in comparison to aesthetic indirect restorations	Tacking to placement instruments

Table 2-1: Summary of the advantages and disadvantages of the RBC materials

As shown in Table 2-1, RBC materials can be considered a suitable alternative to dental amalgam, as its improved aesthetic at low cost make it a popular choice in contemporary dental practice. It can be used in various situations to restore both

anterior and posterior damaged teeth and enhance their physical and mechanical properties and reparability (Opdam *et al.*, 2010; Ulla Pallesen, 2015; Pratap *et al.*, 2019). Direct RBC restorations provide more resistance against tooth fracture than amalgam by providing more intra-coronal damage tolerance limit and the fatigue resistance. Also, fillers are loaded in RBC materials in random directions that can help in distributing the load and stop crack propagation. The bonding area between the dentine and RBC also has enough strain capacity to relieve stress in the resin materials (Belli;Eraslan and Eskitascioglu, 2015). For these reasons, RBCs are a common choice in contemporary dentistry as direct restorations to restore damaged teeth, and as such are a suitable area of research for the current investigation.

In dental practice, RBC materials were introduced as two different types because of the mode of initiation of the polymerisation reaction, which was chemical- or light-activated. Two-paste chemical-cured RBC materials were the earliest form, but their long setting time, the tendency of void formation during the mixing, being hard to polish and the high tendency to discoloration made this form less preferred compared to the light cured RBCs. After the chemical-cured RBCs, the light-cured resin-based materials were introduced and they were easier to place incrementally and had better physical and mechanical properties compared to the chemically-cured materials (Ulla Pallesen, 2015). Such advances over the last five decades in RBCs and bonding agent systems have revolutionized modern dentistry (Barszczewska-Rybarek, 2019; Pratap *et al.*, 2019; Watts *et al.*, 2019).

Several dental light cure unit (LCU) generations have been used to cure RBC materials, and each new generation reduced the problems of the previous generation in order to meet the absorption spectrum of newly developed photoinitiators. Light-emitting-diodes or LED technology is the most recent in dental practice. LED LCUs are

available in dental practice in either mono- or polywave form (Leprince *et al.*, 2013). Monowave LED LCUs have a wavelength limited to 420–490 nm and match the camphorquinone (CQ) within the blue wavelength range at 465–470 nm narrow absorption peak. Polywave LED LCUs can also cover the wavelength range 380–420 nm in the near-ultraviolet region which extends to the violet visible light spectrum with a narrow absorption peak of 395–410 nm, giving a range of 395–510 nm to cover all types of photoinitiators. This matches both the CQ and alternative photoinitiator absorption spectrum, as shown in Figure 2-3 (Mills;Jandt and Ashworth, 1999; Santini *et al.*, 2012; Mohammed and Ario, 2015; Rueggeberg *et al.*, 2017).

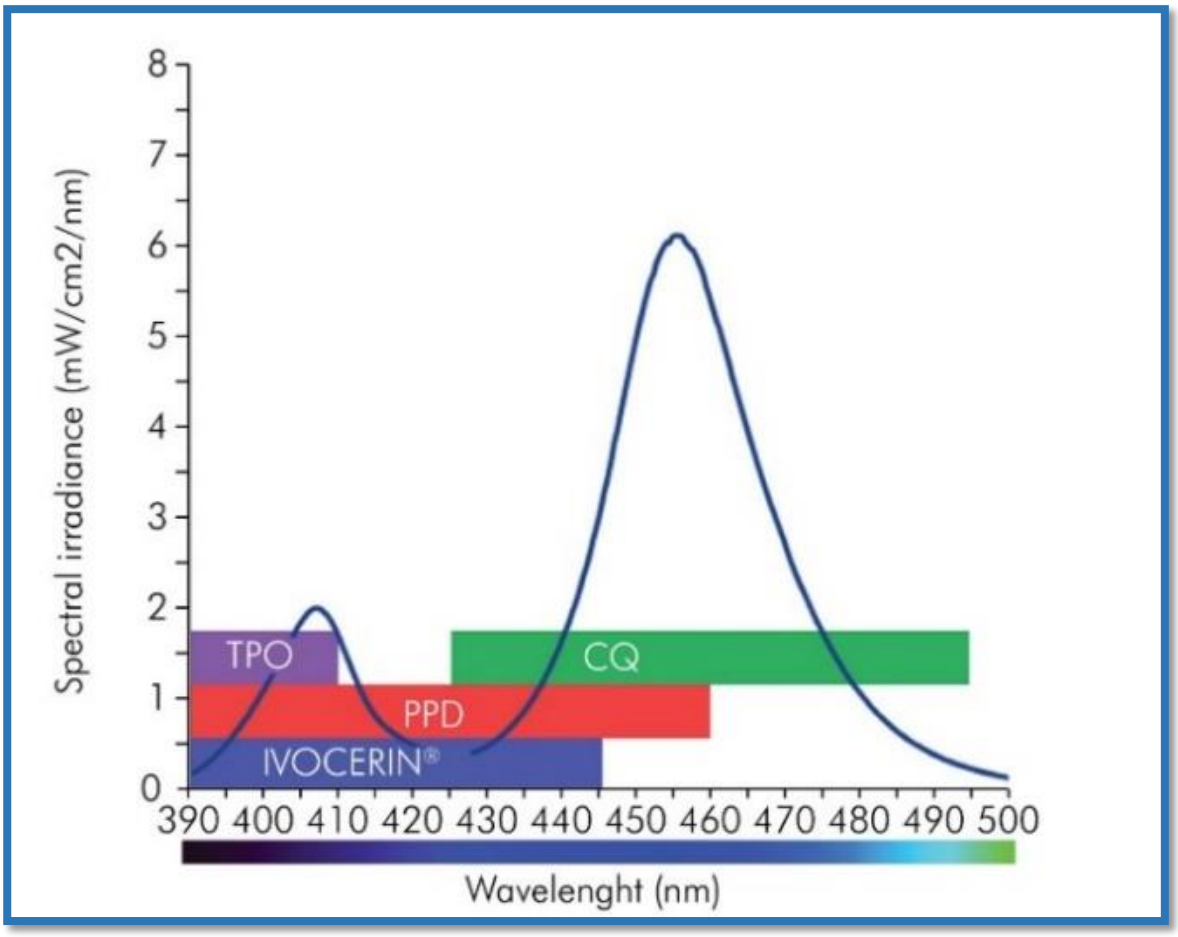


Figure 2-3: The different light absorption peaks of the CQ and alternative photoinitiators taken from Rueggeberg et al., 2017

Different factors such as the magnitude of the LCU irradiance and position in relation to the cured RBC restoration may affect the light intensity of an LCU, which in turn may affect the efficiency of the photo-polymerisation (Michaud *et al.*, 2014). Inappropriate tip positioning may reduce the irradiance and radiant exposure of the LCU received by the RBC restoration, reducing the depth of cure (DoC), degree of conversion (DC), and polymerisation cross-linking density (CLD) (Froes-Salgado *et al.*, 2009; Price;Felix and Whalen, 2010; Price *et al.*, 2011a; Leprince *et al.*, 2013). This can affect the adequacy of the polymerisation and the amount of energy delivered to the RBC materials, causing a noticeable diminution in the physical and mechanical properties (Leprince *et al.*, 2013).

The composite material is a material that contains a mixture of more than one material in three-dimension with a distinct interface separating the components (Phillps, 1997). As such, a dental RBC contains a mixture of three different materials: the organic matrix, inorganic fillers, and a coupling agent. The organic matrix, which includes the monomer system, an initiator system, and the stabiliser, represents the backbone of RBC materials. Inorganic fillers enhance and reinforce the physical and mechanical properties of RBC, while the coupling agent bonds the inorganic filler and organic resin (Peutzfeldt, 1997). Recent publications have outlined notable improvements in RBC materials (Leprince *et al.*, 2013; Meereis *et al.*, 2018), increasing the understanding of these main components and how they have been modified and improved to develop RBC materials with satisfactory properties.

The components of RBC react to set through the polymerisation of matrix monomers by generating a crosslinked polymer. This polymerisation process is triggered by exposing the photoinitiators, like CQ/amine system, to an appropriate light wavelength from the LCU to produce free radicals. Camphorquinone can only photo-initiate

polymerisation at a slow rate. Amines are utilised as co-initiators (electron donor) to speed up the polymerisation process. The polymerisation is started by the radicals that are generated. The amine-derived radicals are the must approach the reactive unsaturated bond in a monomer. These, in turn, react with each double carbon bond (C=C) to open it and generate a chain reaction (Figure 2-4).

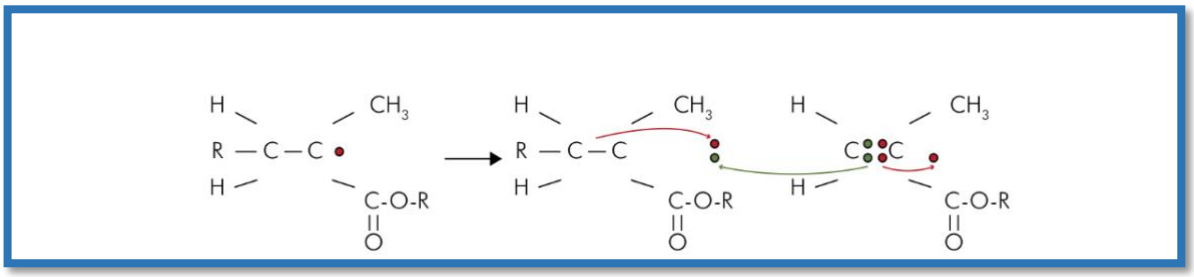


Figure 2-4: Polymer chain propagation from Rueggeberg et al., 2017

The polymerisation process includes three steps, initiation, propagation, and termination, during which the amount of converted monomer to polymer is termed the degree of conversion (DC) (Ferracane, 1985). Linear monomer addition occurs to form linear polymers which then crosslink to other polymers to form tri-dimensional polymers; these increase the physical and mechanical properties of the polymers (Rueggeberg *et al.*, 2017). However, the conversion of monomer to polymers has been shown to be less than 100% (Asmussen and Peutzfeldt, 2003) because the viscosity of the monomer is increased and changed from a viscous liquid to an elastic gel. This therefore reduces the movement of the free radicals and terminates new growth centres of polymer chains, as shown in Figure 2-5. A significant reduction in the rate of polymerisation (R_p, represents the fraction of the conversion of double bond to single per second to show the reaction speed) is called vitrification, whereby the polymerised materials are transferred from rubbery to glass state (Rueggeberg *et al.*, 2017). The amount of DC can be measured by the amount of (C=C) remaining in the polymer in

comparison to the initial stage (Hegde;Hegde and Malhan, 2008; Leprince *et al.*, 2013; Rueggeberg *et al.*, 2017; Barszczewska-Rybarek, 2019).

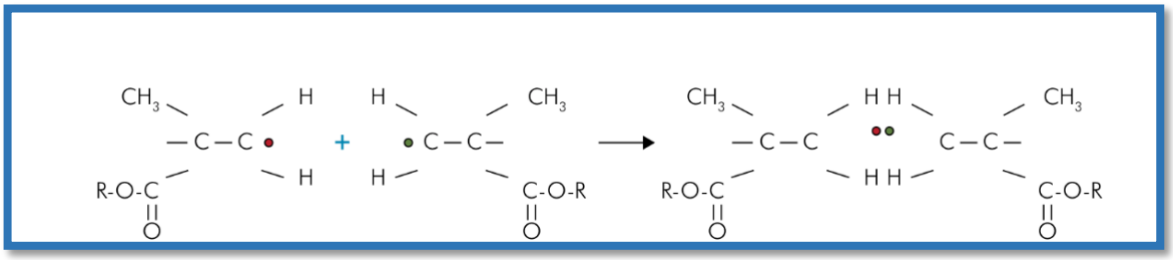


Figure 2-5: Chain termination due to free radical collision from Rueggeberg et al., 2017

2.4.1 Organic matrix

Different organic matrix forms have been used to produce dental RBC materials, including dimethacrylate and non-dimethacrylate monomers. The first of these is the most common and there are several forms such as Bis-GMA, urethane dimethacrylate (UDMA), and ethoxylated bisphenol-A dimethacrylate (Bis-EMA). Bis-GMA is a difunctional monomer of large molecular size and structure, causing it to have low volatility and diffusivity into tissues that can help reduce toxicity (Pratap *et al.*, 2019). It also has relatively lower polymerisation shrinkage, fast hardening and stiffer resin. (Barszczewska-Rybarek, 2009; Chan *et al.*, 2010; Leprince *et al.*, 2013). However, Bis-GMA has a high viscosity, which causes a lower DC due to restriction of the free radical movement, and less incorporation of the inorganic fillers; this is because hydrogen bonding results from the hydroxyl groups of the Bis-GMA structure. To reduce the viscosity of Bis-GMA, comonomers like triethylene glycol dimethacrylate (TEGDMA) are added. Increasing the distance between the methacrylate groups and the long and flexible chain of TEGDMA, as shown in Figure 2-6, creates lower viscosity in comparison to the aromatic rigid Bis-GMA monomer (Ertl *et al.*, 2010; Lavigueur;Christine and Zhu, 2011). The Bis-GMA monomer can be used alone or with another monomer like urethane dimethacrylate (UDMA). UDMA's viscosity is lower

than that of Bis-GMA and it also has a highly flexible chain of urethane linkages, as shown in Figure 2-7; this can increase the DC of RBC containing the UDMA in average from 76% to 87% in comparison to the Bis-GMA-based from 54% to 85%. Also, can increase the ability to load more inorganic fillers (Floyd and Dickens, 2006). In so doing, lower rates of polymerisation shrinkage occur, thus helping to avoid secondary caries and increasing the durability of restorations (Peutzfeldt, 1997; Lavigueur;Christine and Zhu, 2011; Vouvoudi;Baxevani and Sideridou, 2016)

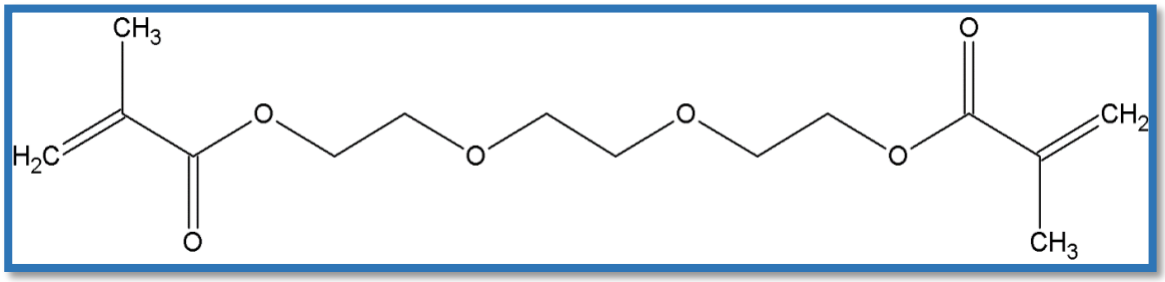


Figure 2-6: Chemical structure of TEGDMA from Floyd and Dickens, 2006

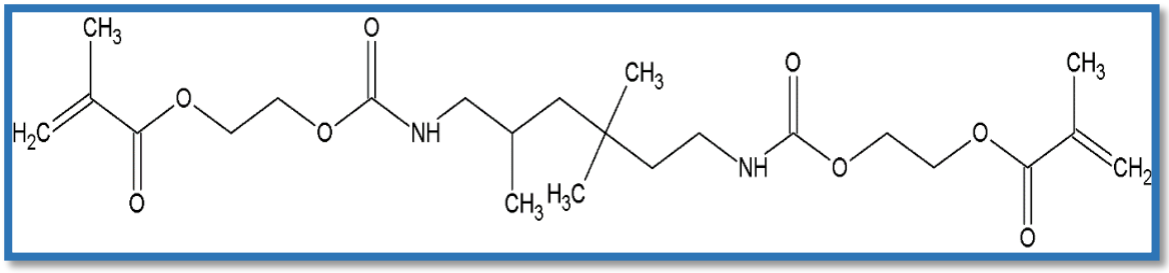


Figure 2-7: Chemical structure of UDMA from Floyd and Dickens, 2006

In Bis-GMA, the formation of the covalent and double bonds involved in the polymer cross-linking process causes unavoidable volumetric shrinkage (Agbaje;Shaba and Adegbulugbe, 2010; Xiong *et al.*, 2011). To reduce the drawbacks of Bis-GMA, non-dimethacrylates were introduced and one such is an oxirane and siloxane-based composite. This type of RBC has siloxane and oxirane monomers composed of a hydrophobic siloxane backbone with oxirane rings, and can undergo cationic ring-opening polymerisation (ROP) (Ilie *et al.*, 2006; Lavigueur;Christine and Zhu, 2011).

This double ROP can provide volume expansion due to the intermediate cationic polymerisation between the reacted rings.

2.4.2 Resin matrix: photoinitiators

The setting reaction of most RBC materials depends on their basic polymerisation mechanism. With methacrylates, free radical addition polymerisation is the most common. For vinyl groups, there is an electron-rich environment in the presence of a carbon-to-carbon double bond at the end of the monomer molecule, as seen in Figure 2-8. These types of material have an internal energy which needs to be released to react with other groups. This happens if free radical generators are used to create very reactive chemical species by producing a high density of electrons in the area of the reaction (Leprince *et al.*, 2013; Rueggeberg *et al.*, 2017). For this reason, a resin-based matrix contains other components, such as an initiator system, to activate polymerisation via the application of heat or light, or via the addition of chemicals acting as the catalyst for the initiators (Figure 2-9). A free electron is then formed in the outer orbit shield, ready to react with another electron and form a stable covalent bond (Figure 2-8) (Rueggeberg *et al.*, 2017). Light-activated initiator systems are the most recently used in dentistry, the primary function of which is to initiate polymerisation.

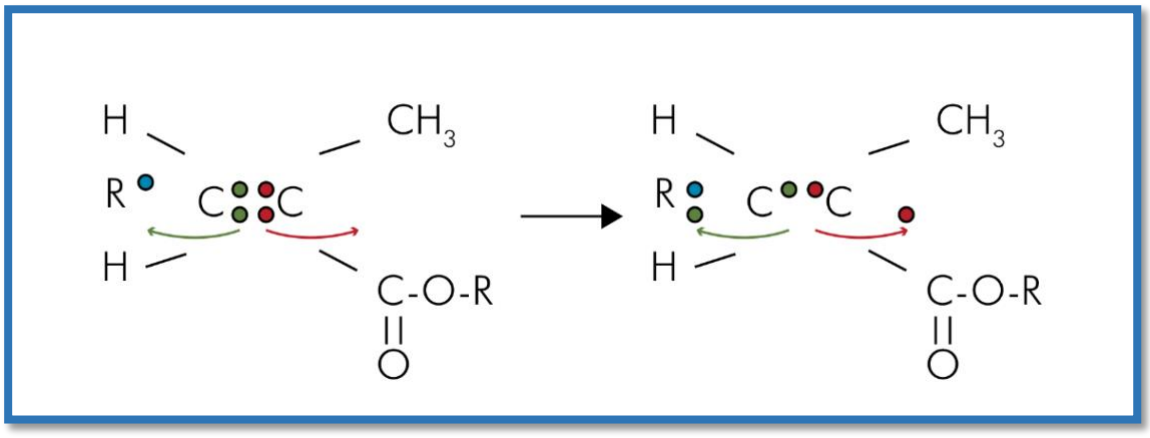


Figure 2-8: The photo-initiator system produces free radicals which initiate RBC polymerisation from Rueggeberg *et al.*, 2017

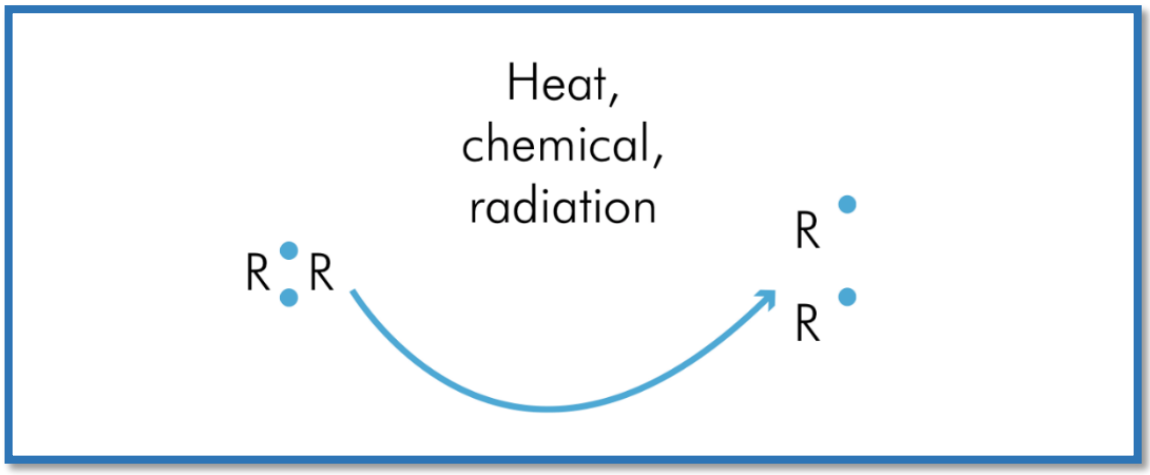


Figure 2-9: Different forms of initiators used in RBC from Rueggeberg *et al.*, 2017

The most popular photoinitiator is type 2 camphorquinone (CQ), and this activates the proton donor, which is a tertiary amine molecule, to produce free radicals (Figure 2-10) (Rueggeberg *et al.*, 2017; Barcelos *et al.*, 2020). However, due to its yellowish colour, CQ affects the long-term aesthetics of a restoration and so this type was combined or replaced by an alternative type 1 photoinitiator. For example, 2,4,6-trimethylbenzoyl-diphenylphosphine oxide (TPO) can be used alone, or with CQ (1-phenyl-1,2-propanedione—PPD) or germanium-based molecules such as Ivocerin (de Oliveira *et al.*, 2015). Derived from acylphosphine oxide and α-diketone, these alternative photoinitiators need no co-initiators to initiate polymerisation and are not yellowish like CQ (Ferracane, 2011; Leprince *et al.*, 2013). Even though all these photoinitiators have the same function, the absorption spectrum of CQ and the alternative initiators is different, with each having a different narrow absorption peak to initiate polymerisation, as illustrated in Figure 2-3. As such, a potential disadvantage of alternative photoinitiators is that they need to use the expensive polywave LCU to cover the wavelength of type 1 photoinitiators, in comparison to the monowave LCU which suits type 2 photoinitiators (de Oliveira *et al.*, 2015; Rueggeberg *et al.*, 2017; Pratap *et al.*, 2019)

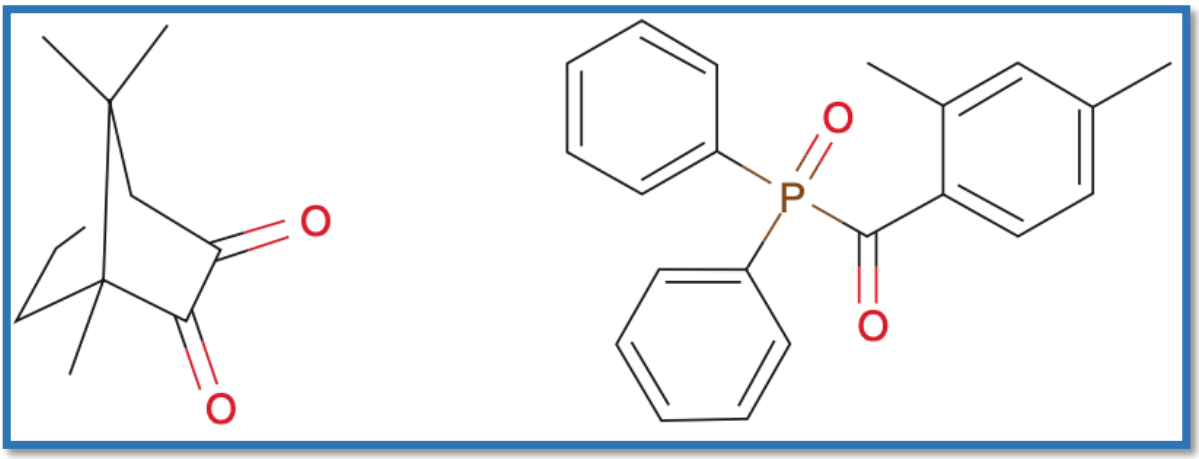


Figure 2-102-11: (A) The chemical structures of CQ and (B) TPO alternative photoinitiators of RBCs from Miletic, 2017

Alternative photoinitiators have a higher efficiency based on how many photons per initiator molecule can be absorbed within the provided wavelength. TPO has shown higher absorption efficiency at lower concentration in comparison to CQ due to the main structure of this type of photoinitiator, which has aromatic rings or chromophores present in the TPO (Figure 2-10) (de Oliveira *et al.*, 2015; Miletic, 2017; Rueggeberg *et al.*, 2017). Such systems have thus improved the depth of cure when mixed at a ratio of 1:1 and 1:4 with CQ. TPO has also shown a higher DC in Bis-GMA (78%) compered to CQ (65%), and exposure and storage time were unimportant for TPO but they were important for CQ (Rocha *et al.*, 2017; Vaidyanathan *et al.*, 2017). With PPD, as a photoinitiator this has a broader absorption into the blue spectral region, and mechanical properties like hardness and DC are enhanced by using alternative photoinitiators like TPO and PPD in comparison to CQ (Hadis;Shortall and Palin, 2012; Rocha *et al.*, 2017; Pratap, 2019). However, the use of type 1 photoinitiators combined with CQ produces higher DC in comparison to their separate use (Neumann *et al.*, 2006).

2.4.3 Inorganic fillers

As a significant portion of the weight or volume of most RBCs, inorganic fillers are another essential component, produced by grinding minerals like quartz, glass or sol-gel ceramics (Sakaguchi and Powers, 2012). All inorganic fillers and modifications of the components of the RBC matrix help to enhance the physical and mechanical properties of these materials, and indeed the effects of inorganic fillers on the properties of RBCs are well established in the literature. In this regard, the different characteristics of these fillers have been discussed in terms of how the size, shape, processing and loading volume of the fillers can be influential, such as reducing RBC polymerisation shrinkage. The literature also reports an improvement in RBC hardness and decreased water uptake when filler load is increased (Kim;Ong and Okuno, 2002; Rodrigues Junior *et al.*, 2007; Agbaje;Shaba and Adegbulugbe, 2010; Xiong *et al.*, 2011; Randolph *et al.*, 2016). However, the DC of the polymerised resin may be negatively affected by inorganic fillers due to light transmission through polymerised materials; this may be because of mismatching the refractive index and a change in the light velocity and direction when light passes through the interface area of the resin fillers (Leprince *et al.*, 2013). There is also the possibility that the viscosity may be increased, potentially affecting the mobility of the local monomer and leading to reduced DC (Lovell *et al.*, 1999; Turssi;Ferracane and Vogel, 2005; Randolph *et al.*, 2016). Because loaded fillers for RBCs have undergone many changes in shape, size and ratio through the history of the dentistry, a classification system was introduced based on the size of the RBC fillers (Figure 2-11) (Ferracane, 2011; Miletic, 2017).

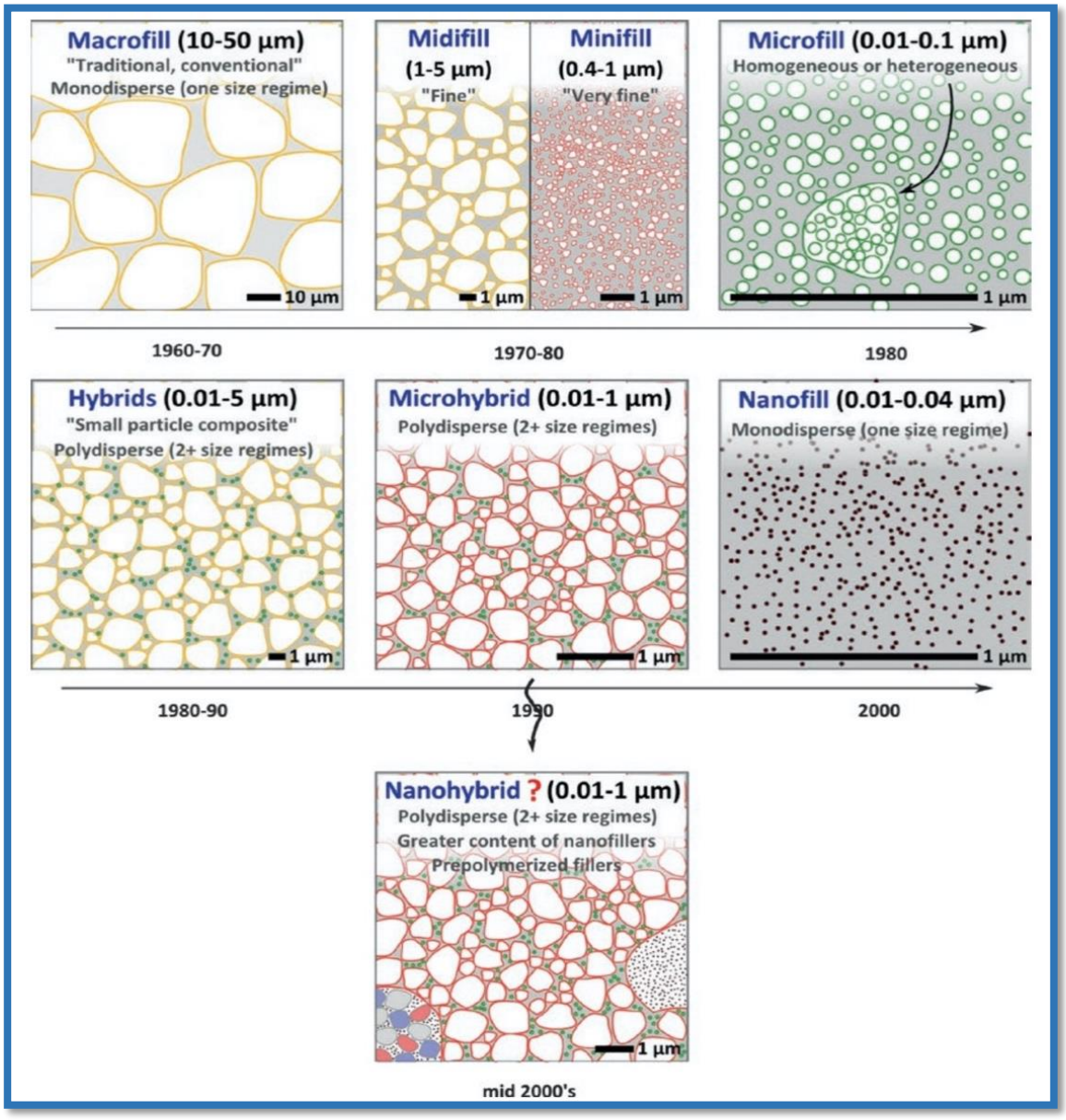


Figure 2-12: Classification of resin-based composite on the filler size taken from Miletic, 2017

The macro- and microfilled RBCs shown in Figure 2-11 were developed to provide better properties for RBCs. Macrofillers are a traditional composite filler ranging in size from 10–50 µm of ground radiopaque glass, quartz and ceramics and can even reach 100 µm. Such fillers are loaded into the resin matrix of the RBC at up to 70–80% wt, reinforcing the resin matrix and enhancing the mechanical properties of the RBCs. However, their key disadvantages are low wear resistance and the difficulty of

polishing set materials, which also discolour over time in comparison to later RBCs (Miletic, 2017; Alzraikat *et al.*, 2018). Microfilled RBCs were developed in the late 1970s to improve upon macro fillers. Instead of using milling and grinding techniques, fine silica microfillers were produced by hydrolysis and precipitation to create the fine size of this filler type. Microfilled RBC fillers range in size from microfill 40–50 nm, have a smooth surface, and are highly polishable and colour-stable compared to the macrofilled type. This makes microfilled RBCs suited to the restoration of damaged teeth tissues in aesthetic areas (Klapdohr *et al.*, 2005). However, their high surface area and lower filler loading create poor mechanical properties and increased thermal expansion; to overcome this problem, prepolymerised microfillers were added (Miletic, 2017).

In the early 1980s, new types of RBC were introduced with a broad range of filler sizes that included a combination of macro- and microfillers, made of different types of macrofiller silicate particles, quartz, and glass within a range of 1–50 µm and amorphous silica microfillers sized 40 µm. These filler sizes were reduced through the 1980s (Miletic, 2017); the macrofillers of 1–5 µm were termed midifills and those of 0.6–1 µm minifills. This type of RBC displays good physical and mechanical properties, showing more wear resistance and better optical properties. Due to the low radiopacity and abrasiveness against enamel of quartz particles, these were replaced by other types of filler such as barium-glass and ytterbium/yttrium trifluoride fillers, to enhance the radiopacity and possibility of fluoride release. However, this change made no significant difference from the perspective of fluoride release (Miletic, 2017). In the 1990s, universal composite, called microhybrid RBC, was introduced with small filler particles added to the hybrid composite, which has more favourable physical and

mechanical properties for restoring anterior and posterior teeth (Klapdohr *et al.*, 2005; Beun *et al.*, 2007; Chan *et al.*, 2010; Ferracane, 2011; Miletic, 2017).

In the mid-2000s, nanotechnology was used to create nanofilled and nanohybrid RBCs from different kinds of silica and/or zirconia fillers sized 5–20 nm and containing discrete and non-agglomerated particles. This type also contains fused and agglomerated nanoclusters of an average size of 0.6–10 µm. Nanohybrid RBC contains larger silica and zirconia particles ranged 0.6–1 µm, as well as nanoclusters and prepolymerised fillers. Later, technology such as sol-gel was used to increase the loading of nanofillers into the RBC to over 80 wt% with ultrafine particles. In so doing, this helped to counteract issues, such as polymerisation shrinkage, but also increased the properties of strength, microhardness and wear resistance which are so essential in posterior occlusal applications (Schmidt *et al.*, 2000; Ferracane, 2011; Khurshid *et al.*, 2015; Miletic, 2017).

2.4.4 Coupling agents

Two RBC components, the organic matrix and inorganic fillers, are chemically different and so bonding between them is difficult. The silane coupling agent has bifunctional groups which provide covalent bonding with an organic matrix from one end, and it can bond to reactive materials, for example methacrylate, epoxy and acrylate. Another end with OH groups can react with inorganic fillers, as illustrated in Figure 2-12 (Miletic, 2017), and this stabilises and enhances the mechanical properties of RBCs. The transfer and distribution of stresses between the different phases of RBC, such as from the organic matrix to the stiffer filler particles, has improved the performance of these materials. Previously, the presence of silane materials between the two main parts would have increased the chance of void formation and thus potentially weaken the

cohesion of the organic matrix to the inorganic fillers. To overcome this, the fillers are salinized to enhance the wettability of filler surfaces and improve bonding to the organic part of the RBC. This technique has enhanced the strength of the RBC materials by about 50% (Arksornnukit;Takahashi and Nishiyama, 2004).

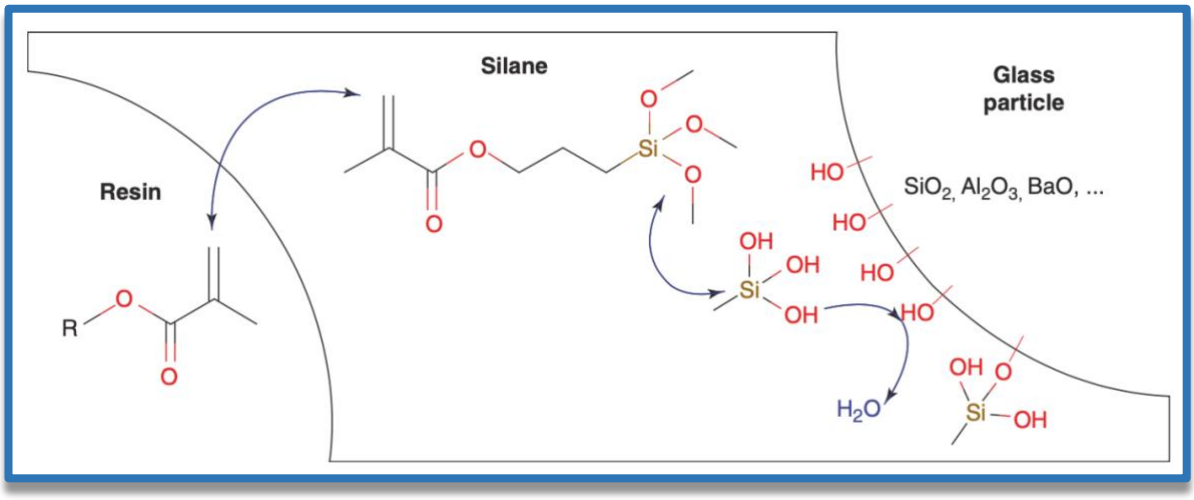


Figure 2-13: Coupling agent function of the organic matrix and inorganic fillers from Miletic, 2017

Different types were introduced like γ-(methacryloxy) propyltrimethoxy silane (γ-MPS) and 3-methacryloxypropyl trimethoxysilane (MPTMS) (Aydinoglu and Yoruc, 2017; Miletic, 2017). These materials are hydrophobic, resist solvents and improve bonding between the matrix and inorganic fillers; their flexibility also increases the performance of the coupling agents (Matinlinna;Lassila and Vallittu, 2007; Aydinoglu and Yoruc, 2017). Using MPTMS improves the mechanical properties of RBCs, including compressive strength, angular flexural strength, flexural strength, elasticity modulus and DC compared to non-silanized fillers (Antonucci *et al.*, 2005; Matinlinna;Lassila and Vallittu, 2006; Aydinoglu and Yoruc, 2017).

2.4.5 Resin-based composite viscosity

To overcome some of the drawbacks of RBCs, especially polymerisation shrinkage and poor handling, many modifications have been made to the resin matrix and inorganic fillers. Since the viscosity of RBCs plays an important role in their use and handling in dental practice, RBCs of different viscosity have been introduced. Currently, the RBC viscosities used in dental practice can be classified into three main groups according to the organic matrix and inorganic fillers loading ratio, as shown in Figure 2-13. These RBC types of different viscosity can be recognised by the clinician with no investigation equipment (Miletic, 2017).

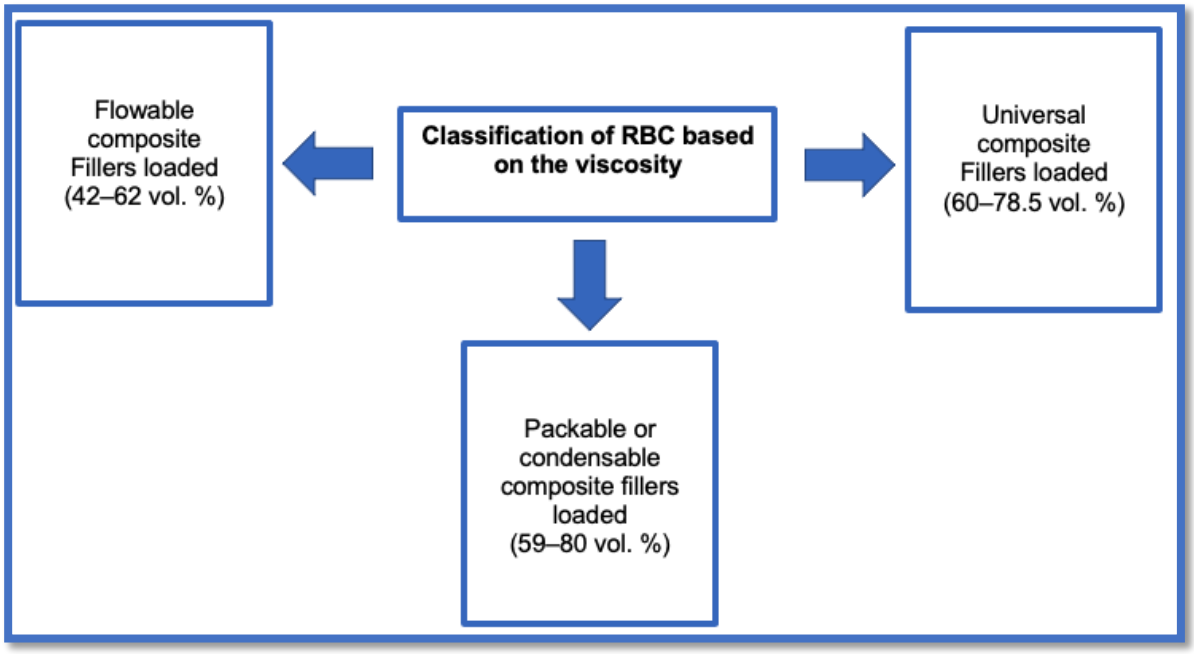


Figure 2-14: Classification of resin-based composite based on viscosity

Universal RBCs are the most widely used direct restorative materials in dental practice. Introduced early in the 21st century, these have undergone numerous developmental stages for the purpose of obtaining materials with better properties (Figure 2-13). Universal RBCs are highly loaded (60–78.5 vol. %) to increase the physical and mechanical properties (Hervás-García *et al.*, 2006). In the literature, it is suggested

that the placed increments are not increased by more than 2 mm, to help the curing light penetrate the unset materials and to enhance the DC of the cured monomers. As such, satisfactory results have been obtained with coloured restorations in both anterior and posterior teeth (Jassé *et al.*, 2019). However, this type still presents polymerisation shrinkage, especially in larger restorations, depending on the number of cavity walls involved. This shrinkage is the main reason for secondary caries and discolouration at the margins of restorations, leading to the need for their replacement (Leprince *et al.*, 2013; Bucuta and Ilie, 2014).

The second type of RBC shown in Figure 2-13 is flowable, introduced to provide materials with less viscosity and so increase the wettability on tooth surfaces. These materials are injectable and can be used in areas that are difficult to restore with more viscous RBC types. Flowable composite has the same small size particle as the universal hybrid RBC but a lower concentration of filler (42–62 vol. %). The impact of this is enhanced handling of the materials and the ability to reach all the surface irregularities. It can also help to apply the material in thin layers to decrease the occurrence of air voids. However, in the literature, flowable composite is considered to have a higher risk of polymerisation shrinkage due to the low filler loading. For this reason, it is recommended for use in cervical margin restorations and minimum occlusal contact areas, or as a cavity liner in class one and two restorations with a universal RBC on the top. It has also been used to bond orthodontic brackets, stabilize fractured teeth, repair defects in old RBC restorations, and opaque the colour of a repaired porcelain fused to metal (PFM) crown (Hervás-García *et al.*, 2006; Kusai Baroudi, 2015).

The third RBC shown in Figure 2-13 is packable. This form was developed for use in high-stress areas like posterior tooth restorations, and can be packed into the cavity in the same way as an amalgam restoration. These RBCs have the potential to provide a better contact point and achieve good occlusal anatomy of the restored teeth (Hervás-García *et al.*, 2006). The difference with packable RBC is the morphology and loading of the inorganic fillers at a ratio of 59–80 vol. %, and it has been suggested that some forms of this type of RBC have a greater depth of cure (4-5 mm) than universal resin composites (2 mm) (Miletic, 2017; Haugen *et al.*, 2020). Therefore, packable RBCs are more suited to posterior teeth restorations, especially class two, due to their physical and mechanical properties and the packability in a deep cavity (Lopes *et al.*, 2002). However, concerns include the increased potential for poor adaptation and voids associated with microleakage because of higher viscosity (Peutzfeldt, 1997; Lee;Son and Um, 2003; Lee;Um and Lee, 2006; Chan *et al.*, 2010; Chesterman *et al.*, 2017). By looking through the different categories of RBCs currently in use in dental practice, the differences in their viscosity can be considered an important point in the handling of the materials themselves and the placement process of RBC restorations.

2.5 Different placement techniques of RBC materials

Nowadays, the high demand for RBC restorations of damaged anterior and posterior teeth tissues makes them the most widely used direct restorative materials (Leprince *et al.*, 2013). The revolution in RBCs has been led by advances in different types of RBC and the variety in viscosity levels (Al-Ahdal;Silikas and Watts, 2014), caused by changes to the main components and subsequently improved physical and mechanical properties. Also, material application and manipulation during the placement process can be achieved in different ways so that each type is placed easily and safely (Chan

et al., 2010; Hickey *et al.*, 2016; Meereis *et al.*, 2018). This helps dentists use the right application and a better placement technique with the RBC which has the best properties and longevity for the patient.

The success of an RBC restoration can be affected by many different factors related to the properties of the material or the bonding system used. Also involved is the position of the LCU and the matching of the emitted wavelength to the photoinitiator of the cured materials, so that polymerisation of the RBC materials is initiated. Moreover, the technique selected for placing the materials will depend on the type and viscosity of the RBC and the location of the restoration. This selection has noticeable effects on the physical and mechanical properties and longevity of an RBC restoration.

The different RBCs used in dental practice and mentioned in the literature have drawbacks which can reduce the physical and mechanical properties of RBC restorations. Some of these can produce a lower DC or polymerisation shrinkage, which, as stated, are essential factors in the longevity and quality of the RBC restoration. This shrinkage is the main reason for marginal leakage, which increases the risk for secondary caries and marginal discolouration. These two problems are identified as the main reasons for replacing RBC restorations. Different methods have been tried to minimize these drawbacks, either by modifying the main components of the RBC or using different techniques to apply the materials, reduce polymerisation shrinkage and provide better DC.

2.5.1 The incremental technique

Of the techniques for applying RBCs to restore teeth, one of the most commonly used is the incremental placement of multiple layers of a 2mm thickness of RBC. This placement is thought to enhance the DC by permitting more curing light to penetrate

and increase the conversion of monomers to polymers. The technique is also believed to be an effective way of reducing the occurrence of post-curing stress in RBC materials between the materials and cavity walls, thus diminishing the possibility of microleakage (Roopa;Usha and Vedhavathi, 2011; Narene, 2014).

The incremental technique can be divided into different techniques. In the horizontal layering technique, the materials are placed in 2mm layers and then cured. One drawback of this method is an increase the ratio of the bonded surfaces to non-bonded, which is known as high C-factor. (Figure 2-14b). To avoid this issue, the oblique layering technique can be used (Figure 2-14e). The RBC materials are placed in a wedge shape and cured on the occlusal surface and cavity walls, and this limits the drawback that occurs with the horizontal technique. The vertical technique shown in Figure 2-14a was introduced to reduce the gingival space that can occur from polymerisation shrinkage. In this technique, a small amount of material is placed vertically onto one of the cavity walls, and the RBC is cured from the opposing side of the wall. Other placement techniques have been suggested in the literature, such as centripetal build-up, split-increment horizontal layering, and successive cusp build-up. All of these techniques work on reducing the effects of C-factors, polymerisation stress and void formation, and are illustrated in Figures 2-14(D–C) to explain the placement of the increments of the RBC materials in the cavity.

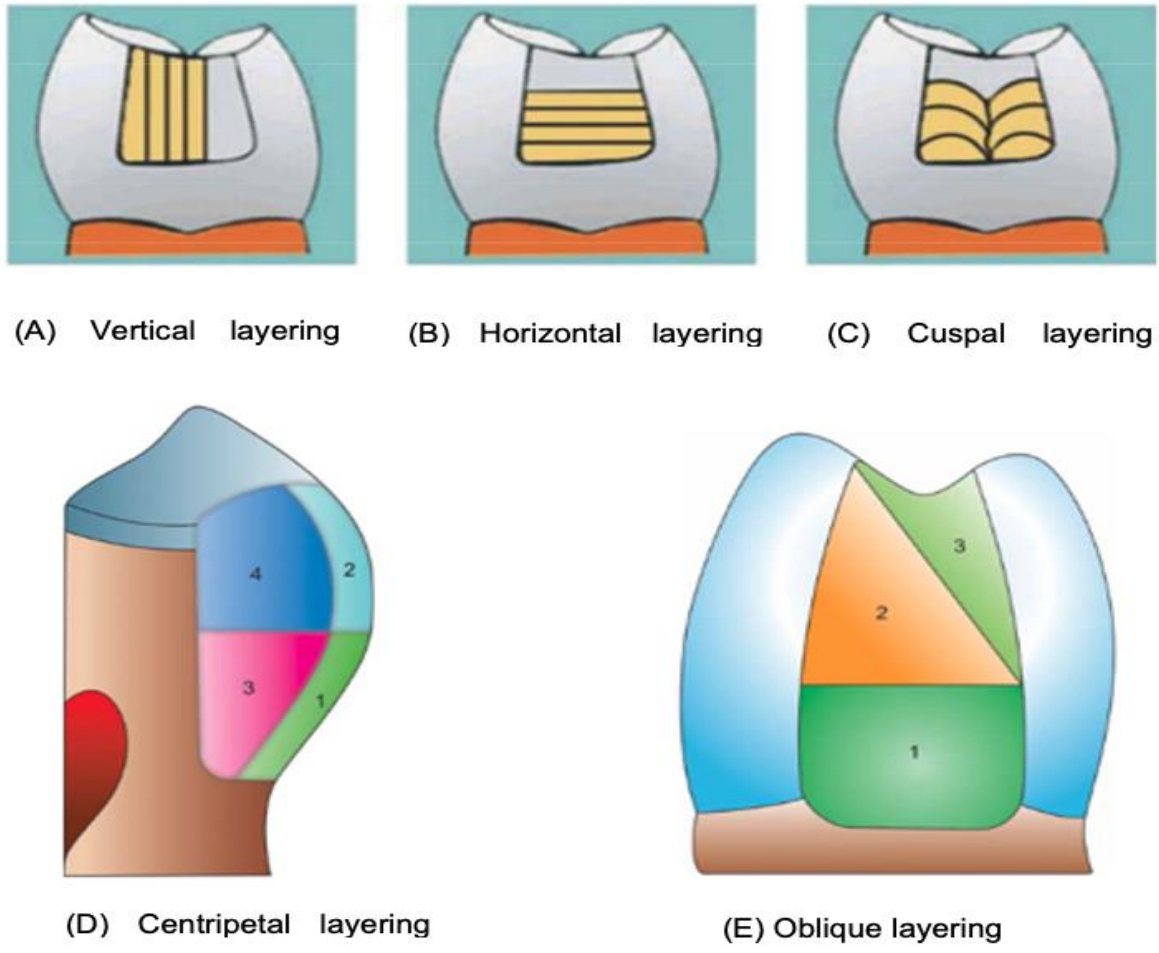


Figure 2-15: Different incremental techniques A, B and C taken from Narene, 2014 and D and E from Roopa;Usha and Vedhavathi, 2011

Another incremental technique is known as three-site, and again this was mainly developed to overcome polymerisation shrinkage and void formation. It works by curing the RBC materials through a clear matrix with reflective wedges to ensure better results. The use of wedge-shaped increments can reduce the stress on the walls by reducing the C-factor of the restored cavity. The literature has shown how this technique is followed by an obliquely placed layer to decrease stress and reduce the occurrence of polymerisation shrinkage (Giachetti *et al.*, 2006; Yadav *et al.*, 2019).

Previous studies have suggested that an incremental technique is one of the more common techniques for applying RBCs to cavities (Tiba *et al.*, 2013; Bucuta and Ilie,

2014). Several laboratory studies and clinical strategies have supported using this technique, which is thought to help to give more curing depth and thus better DC and physical and mechanical properties. Also, the 2mm thickness layers will reduce the stress on the wall of the cavity due to polymerisation shrinkage (Kwon;Ferracane and Lee, 2012; Van Dijken and Pallesen, 2016; Yadav *et al.*, 2019). Use of this technique will reduce the relative ratio of bonded to non-bonded walls of the prepared cavity, and so reducing the C-factor, which will reduce cusp deflection and marginal discrepancies (Roopa;Usha and Vedhavathi, 2011; Yadav *et al.*, 2019).

Despite these advantages, the incremental technique has shown some drawbacks in RBC placement. For example, the placement of multiple increments may be associated with the use of ILs to reduce the handling problems encountered with placing multiple layers of RBC, and there is also the strong possibility of contamination and void formation between the layers of materials. Further, this technique can be time consuming in comparison to the bulk technique and so is not preferred by clinicians (Van Dijken and Pallesen, 2016).

2.5.2 The bulk-fill technique

As an alternative to the above techniques and materials, bulk-fill RBC and the placement technique were introduced to enable the placement of materials with a greater thickness of 4–6 mm in a single increment. The manufacturers used different strategies to achieve more light transmission and flowability, and their materials have low or high viscosity and can be cured to a depth of 4mm. The material can be placed in 4 mm increments and flowable RBC can be used as an optional liner (Figure 2-15a); the proximal and occlusal surface can then be covered with universal RBC, as shown in Figure 2-15b. Other advances with bulk-fill material include sonic-activated RBC,

which can be placed in 5 mm increments (Figure 2-15c), and the fact that some bulk-fill materials are dual cure and can be placed in one large increment (Figure 2-15d). However, the drawback with this RBC is that the aesthetic is poor, and so an outer layer of universal composite is needed to enhance the aesthetic of restorations (Chesterman *et al.*, 2017; Reis *et al.*, 2017). Despite the disadvantage, it has been claimed that bulk-fill RBC can save time and effort, which are essential in dental practice (Chesterman *et al.*, 2017).

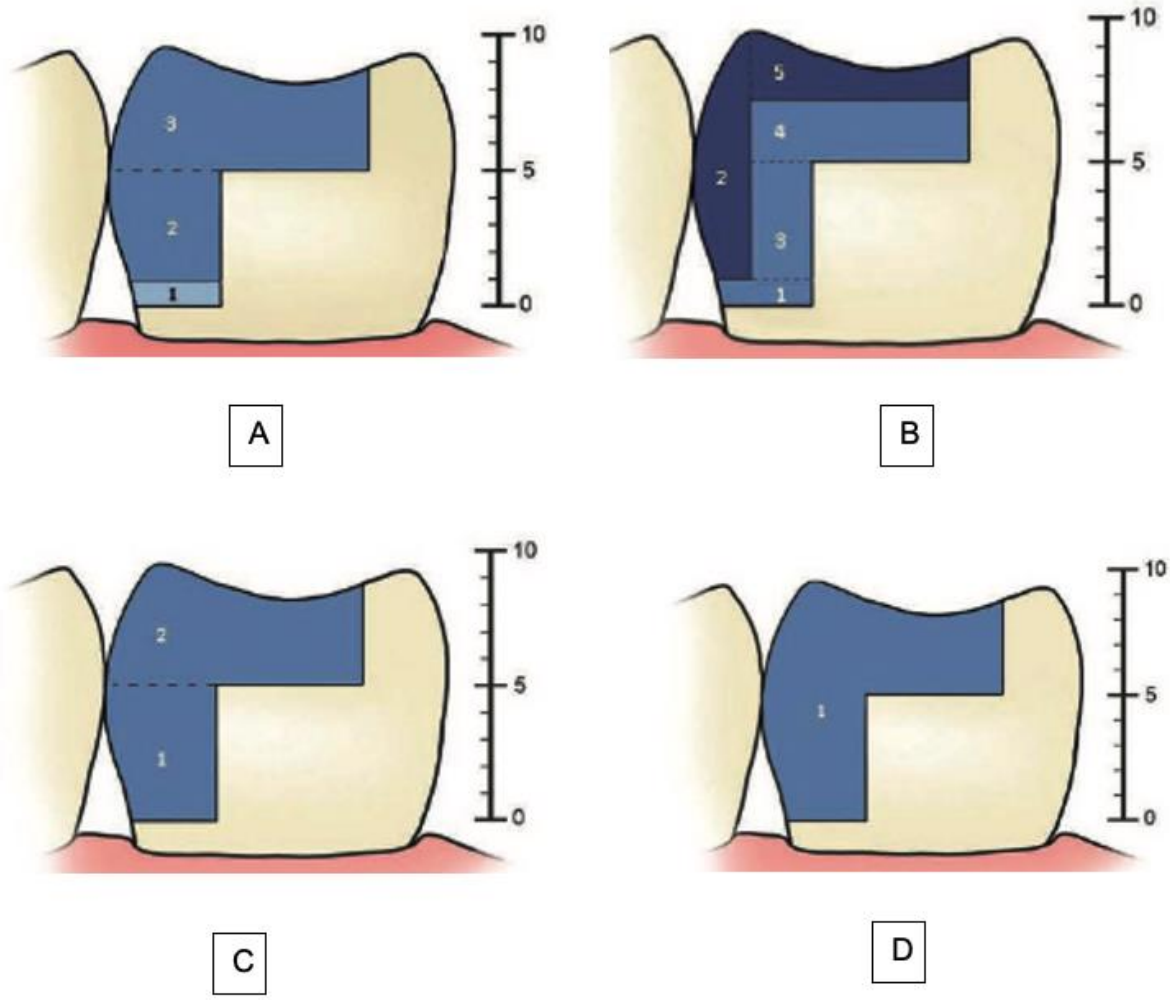


Figure 2-16: Different technique of bulk-fill restorative materials taken from Chesterman *et al.*, 2017

Although less than universal RBCs, the DC of bulk-fill material is still acceptable at about 55% (Furness *et al.*, 2014; Leprince *et al.*, 2014). Moreover, its clinically acceptable values increase after 24 hours with a 4mm depth of cure (Par *et al.*, 2015). While the incremental technique is considered standard for the placement of the RBCs, several clinical trials and *in vitro* studies have reported no significant difference in the performance of both techniques (Loguercio *et al.*, 2019; Veloso *et al.*, 2019; Santis *et al.*, 2020; Durão *et al.*, 2021). These studies found that some bulk-fill materials have an acceptable depth of cure when the hardness of the placed materials was tested. Bulk-fill also has values comparable to international standard parameters for flexural strength, solubility, and water uptake (Tiba *et al.*, 2013; Furness *et al.*, 2014; Kelic *et al.*, 2016; Van Dijken and Pallesen, 2016; Karaman;Keskin and Inan, 2017; Rothmund *et al.*, 2017; Jassé *et al.*, 2019; Loguercio *et al.*, 2019; Veloso *et al.*, 2019).

More negatively, some bulk-fill RBCs lack the colour stability of universal RBC (Tiba *et al.*, 2013), and at 4mm thickness they increase monomer elution time (Rothmund *et al.*, 2017). Bulk-fill RBCs hardness values, when measured at deepest points, are also lower than those measured for RBCs placed using the incremental technique (Lazarchik *et al.*, 2007). Additionally, while there is no significant difference in low C-factor cavities, RBCs placed using the incremental technique shown significantly better bond strengths between tooth structure and RBCs in high C-factor cavities in comparison to bulk-fill placed RBCs. The reason for this improved bond strength with the incremental technique in the high C-factor cavities is that each increment placed provides more free surface to help relieve polymerization stress. So, that would help to reduce the stress at the bonding area between the tooth structures and RBC restoration. That will reduce the restoration fracture, cusps deflection and marginal discrepancies (Ferracane, 2008; Narene, 2014; Han and Park, 2018).

Throughout the literature regarding the placement techniques, most studies were *in vitro* or systematic review papers. They have discussed the different placement techniques or the effects of using them in the longevity of placed direct RBC restorations. All reviewed studies had different aims, like the effect of the varied placement techniques or the effects of using them in the longevity of placed direct RBC restorations. They tested many aspects like the effect of placement techniques on the amount of the cuspal deflection and how are some of the placement techniques can reduce this issue. Also, some of them investigated how the different placement technique can change the occurrence of microleakage at the margins of RBC restorations. Furthermore, some of reviewed studies discussed the effects of the placement techniques on the depth of cure during the RBC restorations placement. Also, how that can increase the RBC restorations longevity in clinical situations. From what has been found in the literature the use of an incremental placement technique appears to be supported more greatly to place RBC restorations. Also, using some specific techniques that were discussed earlier can increase the benefits from using them compared to the other techniques as discussed previously.

2.6 Manipulation challenges with RBC

One problem encountered by clinicians when using RBCs is that the unset material is sticky, meaning that when they are applied to a tooth, they tend to stick to the placement instrument rather than the cavity walls (Rosentritt *et al.*, 2014). This can lead to voids and discrepancies at the margins of the restoration. The stickiness of the material to a surface in general results from force or the work that occurs between them, depending on the strength of the bonding and attraction between the molecules of both parts. In RBCs, stickiness is related to the matrix viscosity and filler volume of the monomers. RBCs of low viscosity and more wettability when in contact with an

instrument surface have a higher chance of sticking to the instrument compared to high viscosity RBC. Also, the resistance that occurs against the debonding force of the materials plays a role in this issue (Opdam *et al.*, 1996; Al-Sharaa and Watts, 2003; Lee;Um and Lee, 2006; Ertl *et al.*, 2010; Kaleem;Satterthwaite and Watts, 2011). Another influential factor is the type of organic matrix used and the interlocking and interfacial interaction between it and the inorganic fillers (Lee;Um and Lee, 2006). For example, Bis-GMA, one of the most popular monomers used in RBC materials matrix and the backbone of the resin, has been identified as one of the most viscous monomers. This is because of the hydroxyl group in the monomer, which has the hydrogen bonding interaction and the polarity that will lead to an increase in the viscosity of RBCs materials (Park *et al.*, 2011). When this monomer is diluted with another monomer of lower viscosity, such as TEGDMA, the overall monomer viscosity reduces; however, the cost of this is an increase in the tendency of the RBC to stick to placement instruments, and a subsequent increase in the manipulation difficulty of unset RBCs during placement. That is because the presence of TEGDMA in the resin boosted pre-gel viscous flow (low viscosity) and reduced RBC viscosity. Also, there is no aromatic group in the central part of the molecule of TEGDMA and presence of (C-O-C) linkages in the molecule make it has lower viscosity. (Feilzer and Dauvillier, 2003; Ertl et al., 2010; Rosentritt et al., 2014). The volume, weight, shape and size of the inorganic fillers also play an essential role in the viscosity of the RBC.

Unset RBC has a greater tendency to stick to placement instruments than bonded dentin for different reasons, such as because the adhesion of some unset RBC materials to the instruments is higher than the cohesive strength of low viscosity RBC (Kaleem;Satterthwaite and Watts, 2011; Rosentritt *et al.*, 2019). Moreover, if the surface area increased and the diameter of the placement instrument's end is more

than 2 mm and the force applied on the placed RBC materials more than 2N, this will increase the stickiness of RBC as that was shown in literature (Rosentritt, 2019). The type of manufacturing material of the placement instrument also plays a role (Steele;McCabe and Barnes, 1991; Ertl *et al.*, 2010; Rosentritt *et al.*, 2019).

Resin composites behave as non-Newtonian viscoelastic fluids, showing shear thinning behaviour. As viscosity decreases under shear loading, resin composites become more fluid during adaptation to the tooth structure. (Loumprinis *et al.*, 2021). Different ways to reduce RBC sticking to placement instruments were investigated. Some of those methods were changing and modifying the main components of the RBC. Manufacturers have reduced matrix viscosity while removing stickiness by modifying filler content and employing various matrix monomer compositions. So, that can help to increase the viscosity and reduce the chance of attaching the RBC to the placement instruments. (Al-Sharaa and Watts, 2003; Rosentritt *et al.*, 2014; Loumprinis *et al.*, 2021). Also, another method focused on coating the placement instruments surfaces by using non-stick layers, as shown in Figure 2-16. This method works by the lowering surface energy of the placement instruments, which can diminish the possibility of sticking the placed materials to the dental instruments. This feature can reduce the adhesion and friction force between the placed RBC materials and placement instruments. So, that can help to make the manipulation of RBC materials easier (Steele;McCabe and Barnes, 1991; Navabpour *et al.*, 2006; Sun *et al.*, 2007; Leppäniemi *et al.*, 2017; Rosentritt *et al.*, 2019).

The instruments made from materials such as gold, aluminium oxide or titanium nitride were introduced as coated instrument to reduce sticking placed RBC materials to the placement instruments. Also, for the same purpose, the disposable instruments made from polypropylene and styrene-ethylene/ butylene-styrene triblock-copolymers

(SEBS); and synthetic foam pads synthesised from polyethylene and poly (ethylene vinyl acetate) (EVA) were introduced (Figure 2-16) (Steele;McCabe and Barnes, 1991; Liu and Kim, 2014). The recent studies investigated the effects of non-stick coating materials like polytetrafluoroethylene (PTFE), doped diamond-like carbon (DLC) that were used as coating materials. Also, superhydrophobic surface subjected to micro structuring by etching, then coated by non-stick materials prepared by using atomic layer deposited (ALD) technique. The coating materials prepared with this technique showed promising results. The ALD reduced the pull-out force by about 45%, and the amount of adherent restorative material by about 90%. So, that would lead to reduced adhesion of RBC to the surface of placement instruments (Matinlinna *et al.*, 2006; Saha *et al.*, 2015; Leppäniemi *et al.*, 2017). Thus, although some of the coated instruments have not shown significant effects and have not been established as a definitive solution, some of them introduce promising results and need further investigation (Steele;McCabe and Barnes, 1991; Leppäniemi *et al.*, 2017).

Titanium nitride coated instrument | Disposable modelling tips | Disposable foam pad

Figure 2-17: Potential solutions for managing how RBC sticks to dental instruments from Health, 2016; Club, 2018

Other potential solutions to tacking are wetting resins, bonding agents, and instrument lubricants, the latter in the form of organic solvents like ethanol, acetone and isopropyl alcohol (Sneed and Draughn, 1980; Tjan and Glancy, 1998; Gorge Perdigao, 2006; Dunn, 2007; de Paula *et al.*, 2016; Patel *et al.*, 2017). Bonding agents in one, two and three steps have also been used (Tjan and Glancy, 1998; Gorge Perdigao, 2006; Barcellos *et al.*, 2011; de Paula *et al.*, 2016; Münchow *et al.*, 2016; Patel *et al.*, 2017), as have wetting resins (Gorge Perdigao, 2006; Barcellos *et al.*, 2008; Tuncer *et al.*, 2013). All these may overcome the drawbacks of RBC stickiness, either by applying them to the surface of placement instruments or directly onto the RBC itself (Dunn, 2007; Ferracane *et al.*, 2017; Patel *et al.*, 2017).

The addition of lubricants to RBC during placement may, however, have adverse effects on its physical and mechanical properties. Patel *et al.* (2017) reported that the use of instrument lubricants increases water uptake and reduces the strength of the restoration, while de Paula *et al.* (2016) found that it may affect the DC and cross-linking density (CLD). The use of alcohols as instrument lubricants has been shown to be incompatible with Bis-GMA- and UEDMA-based RBCs as the adhesive and cohesive strength of the RBC was reduced. Alcohols as organic solvents can solve the RBC matrix and diminish its connection to inorganic fillers (Tjan and Glancy, 1998). Also, using unfilled resin as an instrument lubricant may jeopardise the marginal integrity of an RBC restoration. Moreover, using such materials has the potential to trap more air between the increments, and adding any foreign materials can cause contamination of the monomer matrix and affect polymerisation (Liebenberg, 1999; Tuncer *et al.*, 2013; de Paula *et al.*, 2016; Patel *et al.*, 2017).

2.7 Types of instrument lubricant

Various instrument lubricants have been investigated *in vitro* with regard to stickiness and simplifying the placement and sculpting of RBC restorations (Gorge Perdigao, 2006), shown in Table 2-2.

Lubricant agents	Studies suggesting these agents
One step bonding	Tjan and Glancy (1998); Gorge Perdigao (2006); Barcellos *et al.* (2008); Barcellos *et al.* (2011); Patel *et al.* (2017)
Two step bonding	Tjan and Glancy (1998); Barcellos *et al.* (2008); de Paula *et al.* (2016); Patel *et al.* (2017)
Three step bonding	Gorge Perdigao (2006); de Paula *et al.* (2016); Patel *et al.* (2017)
Absolute ethanol	de Paula *et al.* (2016); Patel *et al.* (2017)
70% ethanol	Tjan and Glancy (1998); Gorge Perdigao (2006); de Paula *et al.* (2016)
Bisco modelling resin	Tuncer *et al.* (2013)
Acetone	Gorge Perdigao (2006)
Tescera sculpting resin	Gorge Perdigao (2006)
70% isopropyl alcohol	Sneed and Draughn (1980); Tjan and Glancy (1998); Gorge Perdigao (2006); Dunn (2007)
Composite wetting resin	Barcellos *et al.* (2008)

Table 2-2: Instrument lubricants as investigated in previous *in vitro* studies

The use of instrument lubricants (IL) when placing RBCs can have adverse effects on the physical and mechanical properties of these materials, but new and different types and brands continue to arrive on the market for dentists to use. Most of the materials shown in Table 2-2 were investigated *in vitro* studies and have had demonstrable effects on the physical and mechanical properties of RBCs materials. ILs used in previous studies have significantly reduced the tensile strength of the tested

specimens due to the effect of these materials on the internal components of the treated RBC (Sneed and Draughn, 1980; Tjan and Glancy, 1998; Gorge Perdigao, 2006). Also, the use of a lubricant when manipulating RBC can affect the DC and density of cross-linking. In some situations, IL can increase the DC but at the same time reduce the density of cross-linking, leading to poorer physical and mechanical properties in the polymerised material. The effect of lubricants on the DC and cross-linking of different RBCs has been linked to the type of RBC matrix and the components of the lubricant (de Paula *et al.*, 2016). Various types of RBC containing different monomers, when treated with ILs, have reacted differently and produced varying DC values. Also, ILs affected the RBC's surface hardness, roughness, and colour stability when test materials were manipulated. All these effects differ depending on the tested RBC matrix and the loaded fillers. The presence of ILs on uncured RBC can affect cross-linking, as mentioned, and reduce the hardness values of treated RBC (Tuncer *et al.*, 2013). Water uptake and diametral tensile strength (DTS) are also affected when ILs are used between the increments as their presence can increase water uptake and reduce the DTS (Patel *et al.*, 2017). The presence of ILs also diminishes the flexural strength of treated RBC materials (Dunn, 2007). Thus, from the literature, many physical and mechanical properties have been investigated, and can affect the stability and longevity of RBCs treated with ILs.

Studies in the literature investigated the changes in the main components of the RBC itself to reduce the sticking of the RBC to the placement instruments (Al-Sharaa and Watts, 2003; Ertl *et al.*, 2010; Kaleem;Satterthwaite and Watts, 2011; Rosentritt *et al.*, 2014; Loumprinis *et al.*, 2021). Moreover, a small number of sources discussed coated dental instruments with non-stick materials to be considered a possible solution for this problem (Steele;McCabe and Barnes, 1991; Leppäniemi *et al.*, 2017). Providing

different types of RBC with varied viscosity or introducing instruments coated with non-stick materials would not have critical effects on the physical and mechanical properties as that can occur when using the ILs. That is because those two methods can have no effects on the main components of the placed RBC materials, because there are no foreign substances used during the RBC materials manipulation and curing. So, that can reduce the materials contamination and the possibility of polymerisation process interruption or voids formation. Therefore, the use of ILs to manipulate the RBC still has more essential aspects that need to have further investigation. There was limited evidence identified regarding the using of the ILs and it does not cover all aspects of the influence of ILs on the physical and mechanical properties of RBC or their impact on RBC restoration longevity.

Studies have investigated the effects of ILs on some of the RBC properties. They showed ILs had varied outcomes regarding the consequences of those materials on RBC restorations' essential physical and mechanical properties. So, that makes the investigation of using these materials necessary, mainly since the previous studies did not use many ILs to test them on different types of RBCs. Such a limitation of evidence makes decision-making for dentists challenging concerning whether to use ILs or not. In addition to laboratory investigations, a survey-based study would have the potential to gather data from UK dentists to identify why and how these materials are used, which types are most frequently used, and with which application techniques.

2.8 Questionnaire-based surveys

A questionnaire-based survey is a popular method of investigating a target population's attitude, behaviour and background (McColl *et al.*, 2001). With this method, collected data can be interpreted via quantitative or qualitative methods to provide insights into the area of investigation. Questionnaires are frequently used to survey groups of

individuals to investigate their understanding about a certain topic, and the selection of an appropriate type and study design has an impact on the collected data. The review of the literature provided in more depth in the next section has shown that a questionnaire with standardised, systematic, and formal questions is useful in this area of research. To help draw valid inferences about the whole population, the questionnaire should collect data from target population with appropriate methods (Horowitz and Sedlacek, 1974; Etikan and Bala, 2017). In turn, this enhances the collected data's validity and reliability and can reduce bias (Tan and Burke, 1997).

2.8.1 Data collection by questionnaire

Questionnaires can be used to collect different types of information on the sample, such as gender, ethnicity, and marital status, as well as information on the sample's behaviour, and events in the past, present, or future. Other common areas of data collection include the beliefs and knowledge of the participants regarding certain issues or health matters, or their life or work, or the society in which they live. Other information regarding attitudes, opinions and reasons can be collected via a questionnaire-based survey. So, it can be considered a well-tested data collection instrument in many research areas (Fishbein, 1967; Goodrich, 1979; McColl *et al.*, 2001; Lietz, 2010; Rowley, 2014).

In the dental and healthcare fields, questionnaire surveys have provided valuable information on general, dental, and oral public health. For example, in dental practice, questionnaire-based surveys have been conducted on applied restorative techniques and materials to restore endodontically treated teeth (Naumann *et al.*, 2016). Another research theme is the effects of instrument dimensions on the extent to which RBC materials stick to placement instruments, and how this influences the manipulation of

RBC materials (Rosentritt *et al.*, 2019). However, this research did not investigate the dentists' opinion regarding the use of ILs in their daily practice and how and why they used them. Also, the opinions of dental students on the use of RBC restorative materials in posterior teeth, instead of metal-based materials, were investigated in different European countries (Lynch *et al.*, 2010b). A questionnaire has also been used to evaluate factors influencing dentists' treatment decisions (Chisini *et al.*, 2019). From these examples, we can see that such surveys are helpful in the health and dental care fields in terms of assessing and understanding issues on dental and oral health services, to facilitate better patient care.

2.8.2 Questionnaire-based surveying and the collection of high-quality data

The literature shows that questionnaire-based surveys must be developed carefully. In this section, the different stages of their creation are highlighted to emphasise their importance to the quality of the final data collection instrument, and to helping the researcher answer their study questions (Goodrich, 1979; McColl *et al.*, 2001; Rattray and Jones, 2007; Lietz, 2010; Etikan and Bala, 2017). The stages are as follows:

1. Defining the objectives/aims of the study
2. Reviewing the literature on the topic of the study
3. Defining the concept to be tested precisely
4. Designing the study and its steps appropriately
5. Determining the hypotheses to be investigated and their requirements
6. Selecting the most useful method of collecting the data
7. Designing the instrument questionnaire
8. Piloting the questionnaire to refine it
9. Selecting the sample from the target population

10. Performing data collection
11. Processing and analysing the data
12. Interpreting and reporting the survey findings.

The steps for preparing and performing the survey are flexible in sequence, such as when piloting reveals that changes are needed (Fanning, 2005). So, the questionnaire-based survey can be helpful instrument for collecting data, but that needs to be processed in careful stages to obtain high quality data. In this way, the research questions can be answered with data that represents the target population (McColl *et al.*, 2001; Fanning, 2005).

Besides these stages, other elements of questionnaire-based survey design help with the collection of high-quality data, for example, by precisely determining the sample population that will represent the target population with adequate sampling frame. A suitable study design will also positively affect the collected data and result from the survey (McColl *et al.*, 2001; Bradburn, 2004; Aday, 2006; Etikan and Bala, 2017). By evaluating the validity and variability of the questions, the precise questions can be posed to determine the participants' views on the topic being researched (Litwin, 1995). Survey management is an essential factor to help manage the survey's timeline and its cost-effectiveness, again to enhance the quality of the collected data (Etikan and Bala, 2017). All these elements are ideally considered to strengthen the data obtained from a questionnaire-based survey.

2.8.3 Different methods for data collection

Various means of collecting survey data can be employed, including a face-to-face interview, telephone interview or self-completion questionnaire, the latter via post, a captive audience, or by e-mail or the internet. Each way has its own advantages and

disadvantages, and none has been shown to be superior. For instance, face-to-face interviews are intensive and can be expensive, requiring more resources and trained staff, and may not cover a wide range of the targeted population (McColl *et al.*, 2001; Mathers;Fox and Hunn, 2009). Self-completion questionnaires can be paper- and postal-based and are thus more efficient at covering more participants from broader areas, and are also cheaper than face-to-face surveys (Kazi and Wardah, 2012). The postal survey method can also be quicker than interview surveys and allows for late questionnaires to be returned via follow up requests (Gonzenbach and Mitrook, 1996; Asch;Jedrziewski and Christakis, 1997). This type has other added advantages: interviewer bias can be avoided, in contrast to face-to-face interviews; no special skills or equipment are needed in their administration; participants have more time to complete the questions and thus better answers may be produced; and, participants have high anonymity, thus reducing bias or any embarrassment that may affect their engagement (Mathers;Fox and Hunn, 2009). At the same time, there are disadvantages: postal questionnaires may be lost or misdirected; participants may have difficulty interpreting the questions; and, there is the risk of a low return rate (McColl *et al.*, 2001; Kazi and Wardah, 2012). Despite the disadvantages of the paper-based and self-completion method, it remains suited to collecting data from a target population. However, the selection of the survey method in any study must be made on a survey-by-survey basis to ensure the quality and cost goals of each survey are met (Thwaites Bee and Murdoch-Eaton, 2016).

2.8.4 Question forms and questionnaire presentation

The questions in a questionnaire must be clear, straightforward, and easy to understand, and creating them is not simple as they must be in a form that helps the research meet its aims and objectives. At the same time, they need to be presented in

a logical sequence to collect the requested data. The type of questions used in the survey can influence the judgement tasks and processing of the survey (Bickart, 2001; Bradburn, 2004; Mathers;Fox and Hunn, 2009). Different question forms can be used to ask about a concept, opinion, behaviour, or attitude, and the most widely used are open and closed types. Open questions are designed to allow the respondents to express their opinions more flexibly within the scope of the topic of the question, and it is important that this form is written so that the participant is not led to think that one answer is more acceptable than another. With closed questions, a list of answers is provided from which the respondent can choose. Each question form has its strengths and weaknesses. When preparing the questions, researchers need to consider the linguistic and cognitive abilities of the target audience, and avoid complexity, ambiguity, leading and double-barrelled questions.

Even if the survey questions are well written and presented in a logical order, an inappropriate questionnaire layout and formatting can lead to a low response rate. As such, the questionnaire needs to be presented in a way that considers the respondent's concerns and is serious about collecting the information required on a specific topic. The researcher must ensure the questionnaire has well formatted document to enhance engagement. For example, by using a stapled booklet format and well-presented cover with sufficient information regarding who is conducting the survey and what its title is. The survey should contain full guidance on completion by giving clear instructions, to help respondents complete the questionnaire easily (Goodrich, 1979; Brace, 2004). Finally, it should be as succinct as possible.

2.8.5 The return rate and how to increase it

The processing of the survey includes three components: the researcher (surveyors), the participants (surveyees), and the survey tools (modes). All these elements are involved in the development, delivery, completion, and return of the survey (Fan and Yan, 2010). Achieving a high return rate is an essential factor in being able to generalise the results collected from the sample to the wider population, and to reduce the possibility of response bias. It is also important for efficiency and reducing the cost of implementing the survey (Rattray and Jones, 2007; Johnson and Wislar, 2012).

Several methods have been proposed to increase the rate of response in questionnaire-based surveys. Asch;Jedrziewski and Christakis (1997) and Dykema *et al.* (2011) suggest: writing a cover letter and personalising it; sending a letter in advance of the questionnaire; using certified mail; providing a stamped, addressed return envelope; and, using coloured paper. Other studies have proposed motivating participants to be more engaged in the survey by providing incentives, such as a pen, a lottery or prize draw, sweets or money (Clark;Khan and Gupta, 2001; Robertson;Walkom and McGettigan, 2005; Vannette and Krosnick, 2018). Some studies show that using incentives increases the response rate in different modes of survey-based studies (Beydoun *et al.*, 2006; Dirmaier *et al.*, 2007; Mann;Lynn and Peterson, 2008). The prepaid monetary incentives were more effective than promised incentives, but there was no evidence showing how much it should be to give a better response rate (Singer and Ye, 2013). The effect of the incentives on the quality of response was not clear and more studies were recommended to investigate their effects on the validity and reliability (Singer and Ye, 2013; Yu *et al.*, 2017). However, while using incentives may be innocuous in some situations, in others it is not, such as when the research is degrading for the participant, or when consent for participation is

dependent on the amount of the incentive received. In the latter case, a large incentive may be the only reason for participation (Grant and Sugarman, 2004). Indeed, some ethics committees believe that most incentives are unacceptable because they can exert pressure on the respondents to participate and could be viewed as being overly coercive (Edwards, 2010; Yu *et al.*, 2017; Vannette and Krosnick, 2018). Also, in a health survey, the incentives have the potential to bias the response, affecting the quality of the collected data that measures the incidence and prevalence of the diseases (Yu *et al.*, 2017).

From all the previous points raised regarding the questionnaire-based survey, the literature has shown it can be a helpful instrument to investigate different subjects. There were different types of information collected by conducting questionnaire-based studies. Broad topics in dentistry have been investigated to answer research questions. This data collection method has key steps to get the most beneficial results and high-quality data. However, there is flexibility to move between those steps to improve the survey's outcome and solve any problems. A questionnaire can be used in different modes, and the postal/self-completion and paper-based questionnaire is one of the most applied methods to cover a large target population. There is no clear superiority for one mode to another; the selection of used mode depends on different factors like the aims and the budget of the survey. The target sample can be surveyed using different question types like open or closed-ended questions depending on the survey aims. Questionnaires should be designed and organized in proper sequence with clear and straightforward unambiguous language. This type of survey is also ideally to be short and precise to encourage the participants to complete the survey. The response rate from this type of study is an essential element to increase the data quality and extract effective results from this data. A low response rate is one of the

most significant disadvantages of the paper-based survey. However, some methods can help increase the response rate without violating the main aims of the study and the ethical aspect.

2.8.6 Dentistry survey studies

In dentistry, a diversity of topics has been investigated using questionnaires, such as:

- The condition of oral and dental public health services, and risk factors affecting oral health and their impact on participants' health (Martins *et al.*, 2016);
- An evaluation of the factors and resources affecting dentists and their productivity in dental practice;
- Occupational burnout and work engagement in dental practice, specifically in the UK (Holmes *et al.*, 2008; Stone *et al.*, 2014);
- Teaching, learning and assessment on dentistry courses (Lynch *et al.*, 2007; Lynch *et al.*, 2010a);
- The teaching of the placement of RBCs and criteria for selecting RBCs instead of other restorative materials (Lynch *et al.*, 2010b);
- The substantial increase in the placement of RBCs in dental practice compared to amalgam, especially in the UK (Gilmour;Evans and Addy, 2007);
- The selection of restorative materials for posterior teeth in dental practice and use of layering materials under RBC restorations (Blum;Younis and Wilson, 2017);

  The influence of placement instrument dimensions on the handling of dental composite materials (Rosentritt *et al.*, 2019).

These examples of dentistry research show the importance and effectiveness of questionnaire-based surveys for investigating different topics on dental materials as

well as dentists' opinions of them. Indeed, the literature has covered certain aspects of dentists' perceptions regarding the use of the RBC as a direct restorative material. However, the review of the literature on questionnaire surveys in dentistry has not revealed any published data on the use of ILs in dental practice. Nor has it identified any questionnaire-based surveys of dentists' opinions of the stickiness of the RBCs they manipulate, and thus there has been no focus on the effects of this issue on dentists' placement techniques in daily practice. Moreover, there is no indication of the methods and materials used in practice to overcome the problem of RBC tacking to placement instruments.

2.9 Evaluation of the physical and mechanical properties of RBC materials

In previous decades, *in vitro* tests have played a very effective role in providing essential data on important aspects of the physical and mechanical properties of RBCs, leading to advances in their development (Leprince *et al.*, 2013; Armstrong *et al.*, 2017; Ferracane *et al.*, 2017; Ilie *et al.*, 2017). Some of these tests have evaluated the effects of instrument lubricants (IL) on the properties of treated RBC materials (Rosatto *et al.*, 2015). For example, diametral tensile strength (DTS) and flexural strength (FS) have been evaluated concerning the effect of ILs on the strength and durability of RBC materials. Moreover, the effect of ILs on hardness and roughness has been tested, for example to evaluate the hardness of the surface of RBCs treated with instrument lubricants, which have been shown to influence resin composite hardness (Tuncer *et al.*, 2013; Heintze *et al.*, 2017).

The consequences of instrument lubricant use on the DC and the cross-linking density (CLD) of the polymer have been evaluated concerning the effect on the quality and longevity of restorations (de Paula *et al.*, 2016). This factor shows the importance of the monomers' DC to a polymer. At the same time, there must be a high level of CLD

to increase the RBCs' physical and mechanical properties (Ferracane, 2006) because, without CLD, even if the DC is achieved, the structure of the polymerised RBC restoration is affected. This then leads to a reduction in the physical and mechanical properties by increasing the possibility of RBC plasticisation through the extrinsic chemical substances (Leprince *et al.*, 2013; de Paula *et al.*, 2016). In this regard, CLD was evaluated in terms of the amount of reduction in the hardness values of RBC before and after immersion in solvents for 24 hours. Spectroscopic techniques were used and these detected the effect of the instrument lubricants on the DC of the RBCs (de Paula *et al.*, 2016). Furthermore, the adhesive and cohesive strength of the RBC materials was tested via a tensile strength test, showing reduced cohesion between incrementally placed layers of RBC materials (Tjan and Glancy, 1998; Barcellos *et al.*, 2011). In the same vein, several other properties have been tested, such as water uptake and colour stability (Brace, 2013; Patel *et al.*, 2017). However, in general more research is required to fully evaluate the effects of different ILs on the physical and mechanical properties of RBC materials, and laboratory investigation is a common method of doing so. Different laboratory tests have evaluated properties such as strength, toughness, and fracture and wear resistance.

2.9.1 Attenuated total reflection Fourier transform infrared spectroscopy

A high level of polymerisation or degree of conversion is critical in the determination of the properties and potential performance of RBC materials. The DC is represented in the percentage of carbon converted from double- to single-bonded. Monomer to polymer conversion will not be 100%, as some of the monomers remain unreacted. The extent of polymerisation is quantified by comparing the amount of remaining carbon double bonds in the polymer structure to the initial monomer amount (Leprince *et al.*, 2013). Standard methods have been used in previous studies to measure the

DC with spectroscopic techniques, either with a mid-infrared Fourier transform (FTIR) or Raman spectroscopy, to evaluate the quantity of remaining carbon double bonds.

FTIR is based on an interferometer which uses a Fourier transform to convert an interferogram into a spectrum, and it is considered the most contemporary infrared spectrometer. Its main drawback is its level of destructiveness and elaborate sample preparation (Ferracane and Greener, 1984; Berthomieu and Hienerwadel, 2009; Coulibaly *et al.*, 2016; Miletic, 2017). Attenuated total reflection (ATR) was added to avoid long specimen preparation and additional steps in testing. Also, all materials to be tested can be placed directly, even uncured RBC, and the DC can be measured during the curing process. An infrared beam is directed at a certain angle onto the crystal and the tested specimen, whose surface must be entirely tied to the ATR crystal, so all reflected infrared penetrates the tested specimen. The absorption occurs on the crystal surface in contact with the specimen, as illustrated in Figure 2-17 (Bruker, 2011; Miletic, 2017).

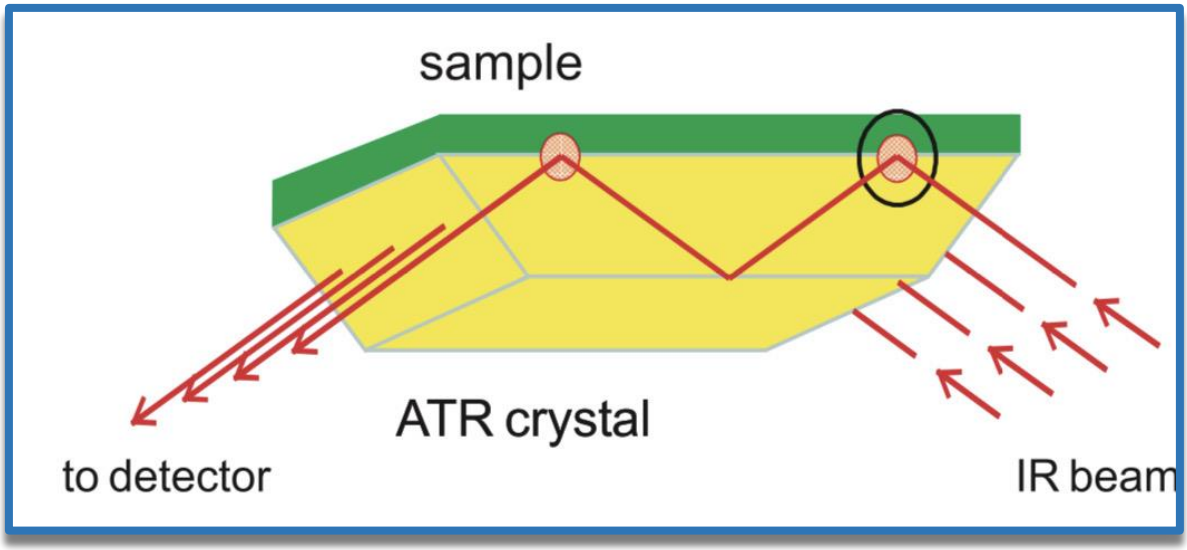


Figure 2-18: Attenuated total reflection in contact with a sample from Bruker, 2011

The surface of the tested specimen should by water-free to avoid interfering with DC measurements because water has the same absorption spectrum (Smith, 2011; Miletic, 2017). Elimination of the effects of the water on the values of scanned specimens has been investigated (Moraes *et al.*, 2008), either by using nitrogen gas to dry the surface of the ATR crystal or by using the near-infrared region. This region has lower absorptivity and a higher path length, which allows measurements to be taken through the bulk of the specimen. However, this region needs more internal standards and only evaluates the DC in the vibration band 6164 cm^{-1} of $=CH_2$ (Leprince *et al.*, 2013). In the literature, even near-infrared has shown benefits, although the mid-infrared region has mostly been used to measure the DC of RBC materials (Stansbury and Dickens, 2001; Trujillo;Newman and Stansbury, 2004; Miletic, 2017). The mid-infrared spectral region mostly uses 400–4,000 cm^{-1}, and is the most common method of measuring DC in RBC materials by quantifying the vibrational band of 1,637 cm-1 intensity changes. ATR-FTIR detects the C=C stretching vibrations directly before and after the curing of RBCs (Smith, 2011; Alshali;Silikas and Satterthwaite, 2013; Miletic, 2017). The areas under the curves or the height of the peak at 1,607 and 1,637 cm^{-1} of cured and uncured RBC is used to calculate the DC percentage, according to Eq.1 (Ruyter and Svendsen, 1978; Leprince *et al.*, 2013; Ferracane *et al.*, 2017):

$$\text{The degree of conversion} = \left(1 - \frac{\text{cured (area under } 1637/\text{area under } 1607\text{)}}{\text{uncured (area under } 1637/\text{area under } 1607)}\right) \times 100$$

(Eq. 1)

A previous study has suggested that using instrument lubricants affects the DC of RBCs. In this study, ATR-FTIR was used to determine the DC and so to evaluate the influence of the ILs on the properties of the RBC. The use of different bonding agent

systems and ethanol as an IL was combined with different RBCs, and it was found that some of these affected the DC in comparison to the control group (de Paula *et al.*, 2016). Moreover, ATR-FTIR has been used in several studies to evaluate the conversion of monomers to polymers under different circumstances for different resin-based materials (Ferracane, 1985; Turssi;Ferracane and Vogel, 2005; Xiong *et al.*, 2011; Santini *et al.*, 2012; Oliveira *et al.*, 2014; Al-Zain *et al.*, 2017; Ferracane *et al.*, 2017).

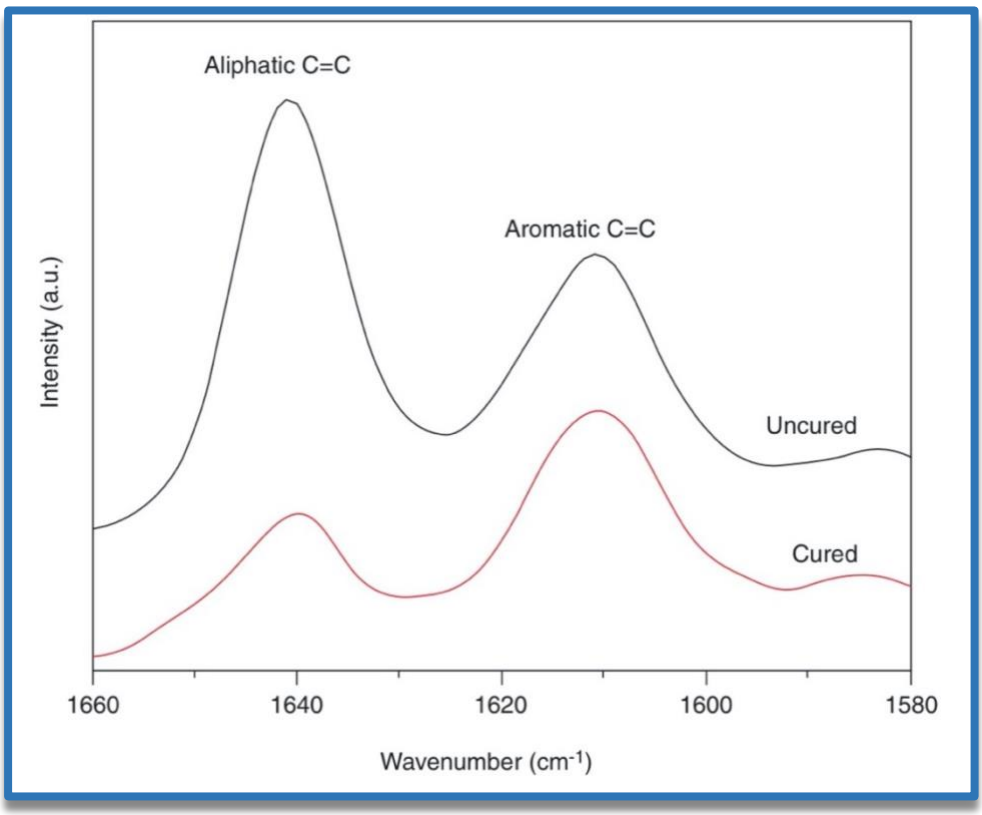


Figure 2-19: The reference bands aromatic 1,607 cm^{-1} and aliphatic 1,637 cm^{-1} of the infrared used to determine the DC of cured/uncured RBCs taken from Miletic, 2017

2.9.2 Water uptake

During the polymerisation process, not all monomers are converted to polymers (Asmussen and Peutzfeldt, 1998; Leprince *et al.*, 2013; Cornelio *et al.*, 2014), and unreacted monomers in RBC restorations may leach into the surrounding fluid. This then becomes an appropriate place for microporosities and increased water uptake.

RBCs are considered hydrophilic in current monomers such as TEGDMA, and this increases the possibility of water uptake because of the polarity of the monomers in the RBC matrix. This polarity can cause hydrogen to bond with water molecules from the surrounding fluids and increase the amount of water entering RBC restorations (Sideridou;Achilias and Karabela, 2007; Decky *et al.*, 2009). Common RBC polymers have been ranked according to volume increase from water uptake, as follows: poly-TEGDMA > poly-Bis-GMA > poly-UDMA > poly-Bis-EMA > poly-decane-diol dimethacrylate (D3MA) (Sideridou;Karabela and Vouvoudi, 2008). Water uptake can cause the fillers to debond from the resin matrix due to increased pressure stress from the fluid on the bonding, thus negatively affecting the physical and mechanical properties of the RBC (Ito *et al.*, 2005; Drummond, 2008; Van Landuyt *et al.*, 2011; Rahim *et al.*, 2012; Mansouri and Zidan, 2018).

The ISO standard limit for RBC water uptake has been determined in the literature to be about 40 µg/mm^3 (Rahim *et al.*, 2012). Increased water uptake induces hydrolytic degradation, and many *in vitro* studies have shown a decrease in flexural, modulus, tensile strength and the DTS of RBCs stored in water for different durations (Yiu *et al.*, 2004; Ito *et al.*, 2005; Sideridou;Karabela and Bikiaris, 2007; Patel *et al.*, 2017). Also, water uptake can reduce the hardness of RBC materials, depending on their organic matrices, after being stored in water (Porto *et al.*, 2013). Further effects can also occur with the colour stability of the RBC materials (Mansouri and Zidan, 2018).

The application of external materials during RBC placement can contaminate the RBC due to increased water uptake, and thus affect its physical and mechanical properties. Previous studies have investigated the effect of IL on the properties of RBC materials (Tuncer et al., 2013; de Paula et al., 2016; Patel et al., 2017), and water uptake was tested in this regard to investigate the effect of IL on the longevity of the RBC materials

(Patel et al., 2017). The water uptake in literature was measured by using the standard method which is recommended by the ISO 4049 for polymer-based restorations. This method calculates the values for water uptake in micrograms per cubic millimetre for all tested specimens. The other method was measured by calculating the percentage weight change of tested specimens. Both methods can evaluate the amount of the water that has been taken into the RBC. They could calculate the water uptake of the tested RBC materials and detect the different in specimen before and after the water immersion. However, the methodology for measuring hygroscopic expansion and solubility of resin-based restorative materials is only specified in ISO standard 4049 for resin-based dental filling materials (Tyas;Jones and Rizkalla, 1998; Patel *et al.*, 2017; Shen and Ralph Rawls, 2022).

2.9.3 Hardness measurements

Using instrument lubricants for shaping and scalping the surfaces of RBC restorations before curing can affect the physical and mechanical properties of restoration surfaces. Since the outer layer is most exposed to saliva and other fluid in the oral cavity, this will affect the hardness and longevity of the restoration (Tuncer *et al.*, 2013; de Paula *et al.*, 2016). Hardness tests can be on the macro-, micro- or nano-scale and have been used to evaluate the surface hardness of the materials. The hardness tests introduced in previous studies are either Vickers or Knoop tests. Both measure the hardness through the indentations created by the indenter of the machine on the surface of the specimen. Some studies showed a good correlation between the hardness of RBCs materials and degree of monomer conversion (Ferracane, 1985; Neves *et al.*, 2002; Leprince *et al.*, 2012; Leprince *et al.*, 2013). Also, the impact of the light source is not well known, and so investigations of hardness are used to examine the relationship by using bottom/top ratios (B/T). The bottom to top surface ratio should

be 80% or more for the material to be considered sufficiently cured throughout. The evaluation of the hardness of 1mm thickness showed bottom/top values at 100%. Increasing the thickness of RBC materials by up to 4mm gradually reduced the values of hardness, depending on the thickness of tested specimens (Monte Alto *et al.*, 2006; Leprince *et al.*, 2012; Leprince *et al.*, 2013). Such tests show the relationship between the depth of cure, DC and hardness of tested RBC materials (Neves *et al.*, 2002; Leprince *et al.*, 2013; Kelic *et al.*, 2016; Al-Zain *et al.*, 2017; Ilie *et al.*, 2017).

The Martens hardness test, formally known as the universal hardness test, is mentioned in the literature regarding the evaluation of resin composite (International Organization for Standardization, 1995). Despite not being used as much as the Vickers and Knoop tests, some studies have shown that the results of the HM test were more reliable (Shahdad *et al.*, 2007). Vickers and Knoop tests relied on readings taken visually by an operator to determine the borders of the indentations under the microscope. This view was likely affected by the resolution of the vision of the device or the operator's perception, as well as by other factors such as the type of RBC, elastic speed recovery, time, and the size and quantity of the fillers. The concept of the Martens test was applying a force of the indenter on the surface of the tested specimen for 30 seconds. The test force and the indenter displacement were calculated during the test procedure, thus eliminating the effect of the elastic recovery of the RBC (Shahdad *et al.*, 2007; Bürgin;Rohr and Fischer, 2017).

2.9.4 Diametral tensile testing

In aesthetic areas and on posterior teeth, RBC restorations of damaged tissue receive a high load due to mastication (Chan *et al.*, 2010; Ferracane, 2011; Kubo, 2011). All introduced RBCs should be tested for various properties such as diametral tensile,

flexural, wear resistance, and tensile strengths, and fracture toughness (Leprince *et al.*, 2013). The results of these tests can be influenced by the different components of the RBC materials, including the resin's organic matrix and inorganic fillers. The type, size and amount of loading of the fillers can also influence the strength values of the tested RBC materials (Peutzfeldt, 1997; Chan *et al.*, 2010; Leprince *et al.*, 2013; Pratap *et al.*, 2019).

The diametral tensile strength test (DTS, also called the Brazilian test) has been used to measure the strength of RBCs. This test has been discussed in the literature regarding specimen types, for example disc and beam, as well as other limitations that can affect strength values, especially the centre of the region of failure (Darvell, 1990). The DTS test is considered a valid test that indirectly measures tensile force (Darvell, 1990; Wang and Cao, 2018), and was developed to test brittle materials that cannot be tested with a high quality uni-axial tensile test. However, the DTS depends on the accuracy of the specimen preparation because the specimen needs to be uniformly loaded over its entire surface. This is because all the specimen's surfaces must receive the same compressive load through the plates, as shown in Figure 2-19 (Ilie *et al.*, 2017). Also, only specimens which split into equal two parts are considered to have been accurate. Therefore, the surface area was reduced by doing disk- rather than cylindrical-shaped specimens to reduce the risk of an inaccurate fracture. The mechanism of the DTS depends on the transfer of compression strength to tensile strength, which helps to test the tensile strength of cylindrical specimens (Ilie *et al.*, 2017).

DTS has been used to test the strength of RBC and is considered a valid test of brittle materials by the Academy of Dental Material (Ilie *et al.*, 2017); DTS values reported in the literature are 30–55 MPa (Casselli *et al.*, 2006). The DTS test has been used in

several studies to evaluate the strength of RBC built-up materials and compare the strength of different types of light-cured RBC materials. It has also been used to evaluate the effects of RBC thickness and the distance of the light cure from the materials (Combe *et al.*, 1999; Della Bona *et al.*, 2008; Medikasari, 2018). Repaired RBC strength has also been measured using the DTS test (Imbery *et al.*, 2014), as have the effects of ILs on the strength of RBC when using materials to facilitate the manipulation (Sneed and Draughn, 1980; Patel *et al.*, 2017). Other studies have used DTS tests to evaluate the effects of different activation techniques on the polymerisation of RBC materials (Casselli *et al.*, 2006), and the strength of RBC bioactive materials (Alrahlah, 2018). Despite its limitations, these studies show that DTS remains an option for evaluating the strength of RBC materials in *in vitro* testing.

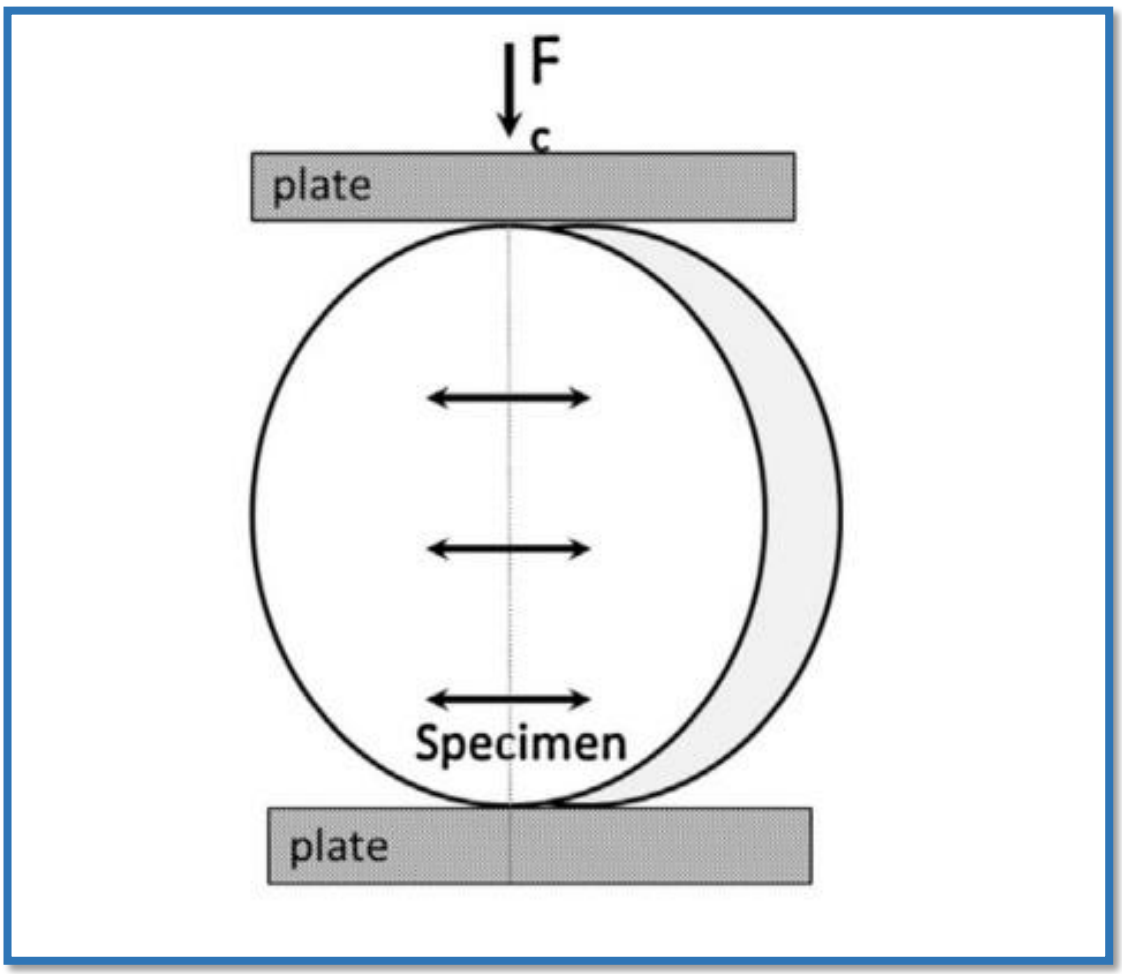


Figure 2-20: The diametral tensile test from Ilie *et al.*, 2017

2.9.5 Microtensile testing

Shear and tensile force tests are generally used to evaluate bonding strength. While the shear load test may produce a non-uniform load at the edge of the tested bonding interface during the test, the tensile test has been ranked as one of the best bonding strength tests for evaluating materials under tensile load by the Academy of Dental Materials. The tensile test has also been used to evaluate several themes, including: the bonding strength of different surfaces and materials (Ilie *et al.*, 2017; Sano *et al.*, 2020); the bonding of RBC materials and dentin (the most common area of research) (Cho and Dickens, 2004; Nayif *et al.*, 2008; Ramos *et al.*, 2016; Armstrong *et al.*, 2017; Mousavinasab *et al.*, 2018); the bonding strength of RBC materials to enamel and other substrates like ceramics and RBC (Özcan *et al.*, 2007; Isolan *et al.*, 2014; Yu *et al.*, 2019); the effects of different placement techniques on the strength of bonding RBC materials to tooth structures (Niu *et al.*, 2009); and, the effects of ageing on the strength of bonded surfaces (Casagrande *et al.*, 2006; Sideridou;Karabela and Bikiaris, 2007).

With micro-tensile testing, specimens have been prepared to reflect various shapes and geometries, with the hourglass shape being the first of these (Sano *et al.*, 2020). When the specimens are made narrower at the interface area of the bonded surfaces, the bonding strength depends on the surface area for bonding. The small size of the specimens used in this test can give the advantage of purely tensile strength, which is better than when directed at the union interface due to lack of distortion (Sano *et al.*, 1994; Araújo *et al.*, 2018). It was also found that tensile strength is reduced when the thickness of the specimen increases due to the force being distributed on a large area which may have more defects (Sano *et al.*, 2020).

Other specimens have been prepared by sectioning macro specimens produced in a block or cylindrical shape into multiple micro specimens (Armstrong *et al.*, 2017). Such cross-sections should be about 1 mm for non-trimmed stick-shape specimens, shown in Figure 2-20a, but can be larger for trimmed, dumbbell- (Figure 2-20b) and hourglass-shaped specimens (Figure 2-20c) (Armstrong *et al.*, 2017). In the literature, a modified micro-tensile test specimen with a diameter of 300 μm × 300 μm and a cross-section of 0.09 mm^2 has been reported (Özcan *et al.*, 2007; Fernandes *et al.*, 2014; Ilie *et al.*, 2017; Araújo *et al.*, 2018). All the produced specimens must be checked under the microscope to exclude any specimens exhibiting defects that could encourage early failure. The geometry of the specimens can affect the bonding strength values of the tested materials, and so the preparation of specimen stage has a critical effect on the data collected (Armstrong *et al.*, 2017; Araújo *et al.*, 2018)

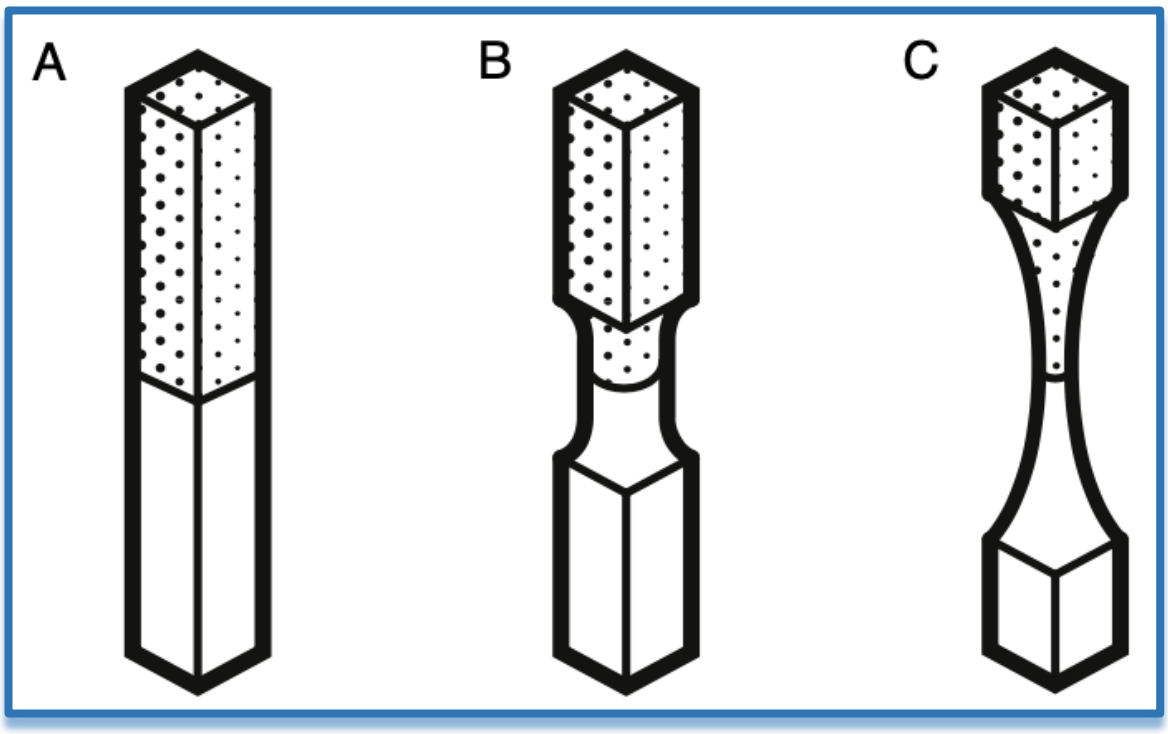
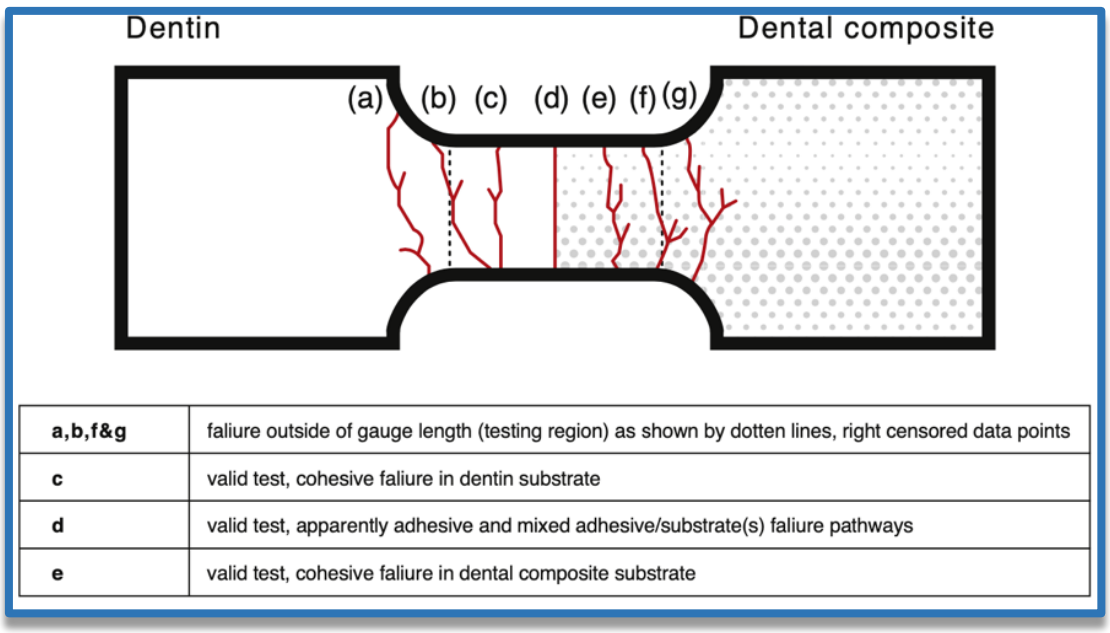


Figure 2-21: Different shapes of the micro-tensile test specimens: (A) Untrimmed; (B) Trimmed stick-shaped; (C) dumbbell and hourglass taken from Armstrong *et al.*, 2017

Most of specimens fracture when submitted to a loading rate of 0.5–1.0 mm/min. The fracture of a tested specimen can be effected within 5–10 seconds after the machine reaches preload force. It is important to ensure all the data start at the same point for more accurate and easier processing. Different patterns of failure in tested specimens are illustrated in Figure 2-21, according to the location of fracture (Von Fraunhofer, 2012). Fractures in the tested RBC materials away from the area of the interface bonding are called cohesive failure, while adhesive failures occur when specimens fail or fracture at the area of the adhesive bonding interface. A failure can also be due to the adhesive and RBC materials together. However, if the fracture is at the area glued to the attachment, the specimen is considered an invalid test (Von Fraunhofer, 2012; Dao Luong *et al.*, 2016; Armstrong *et al.*, 2017; Araújo *et al.*, 2018).



a,b,f&g	faliure outside of gauge length (testing region) as shown by dotten lines, right censored data points
c	valid test, cohesive faliure in dentin substrate
d	valid test, apparently adhesive and mixed adhesive/substrate(s) faliure pathways
e	valid test, cohesive faliure in dental composite substrate

Figure 2-22: Fracture failures in specimens by location taken from (Armstrong *et al.*, 2017)

In the literature review, most of the studies that have been conducted were previously discussed and were *in vitro* tests. The vast majority of tests used to investigate the physical and mechanical properties were laboratory-based studies. There were *in vivo*

studies used to test the properties of the dental materials, but they were not as many as the *in vitro* studies used in this area. The correlation between the clinical and laboratory tests can help to develop a new material rapidly. However, in literature there was no strong correlation between the laboratory tests and short/long term clinical performance evaluation of RBC materials (Bayne, 2011).

The clinical studies have some properties that can be evaluated by observing a surface changes, margins, occlusion, and interproximal contact. However, the tests that have been discussed above as laboratory tests were contributed previously through the dental materials development by evaluating the bulk properties of RBC materials like the physical and mechanical properties. They have provided the ability to predict the RBCs behaviour and failure. Also, they have investigated the effects of external factors that would compromise the RBC materials physical and mechanical properties. The laboratory tests still have some limitations, like the different circumstances in the oral cavity that can be different from the laboratory situation. However, the ways of simulating the oral conditions in the lab studies have provided more chances to increase those tests' validity (Bayne, 2011).

The *in vitro* tests can help in the current work by evaluating the effects of ILs on the physical and mechanical properties of the RBC materials. They have the potential to achieve the main aims and objectives of the present study to answer placed questions. That can provide more evidence and data that would explain the effects of The ILs on RBCs to help the dentists to make their decision. The Academy of Dental Materials have also recommended them as appropriate tests to be used to evaluate RBC materials' properties (Ferracane *et al.*, 2017; Ilie *et al.*, 2017). However, no clinical trial has been reported to investigate the effects of ILs on the physical and mechanical

properties, and the literature has not mentioned any other clinical studies conducted investigating these materials.

2.10 Summary

- Through the literature review, different methods of reducing the manipulation difficulty of RBC materials have been presented;
- Instrument lubricants are used in dental practice to decrease the stickiness of RBC material to dental instruments;
- The literature review indicates that all studies on ILs were *in vitro*. There is a lack of clear evidence of the effect of the clinical use of ILs on RBC restorations, suggesting the need for more evidence;
- There are no agreed guidelines for clinicians in the UK on using IL;
- The reasons for using ILs and how they are used have not been thoroughly investigated or determined;
- Dentists' preferences regarding the type of RBC, placement techniques, types of the ILs, and ways of applying these materials have not been explored.

For this reason, a survey would be appropriate to find further evidence on these points to help researchers have a clear idea regarding this topic, especially among UK dentists. Furthermore, a better understanding could be developed of the use of instrument lubricants during the placement of RBC, and their effects on the properties of the placed RBC restorations. The thesis thus aims to assess the knowledge of dental practitioners in the UK regarding the types of ILs that they use and how they use them, and to evaluate the effects of lubricants on the physical and mechanical properties of placed RBC restorations via laboratory investigations. Ultimately, the aim is to determine the most appropriate techniques and materials to use with lubricants during RC restoration placement.

Chapter 3: Aims and Programme of Work

3.1 Aims

The aim of the current study is to investigate the use of instrument lubricants (ILs) by dentists and the effects of their use on the stability and longevity of RBC restorations. To achieve the main aim, secondary aims and objectives have been determined:

1) Investigate UK dentists' opinions regarding use ILs during the placement of the RBC restoration in their daily practice and why and how they use them.
2) Investigate the effects of ILs on the physical and mechanical properties of the RBC materials.

3.2 Objectives

To achieve the stated aims, it was necessary to establish the following objectives:

1) To investigate whether UK dentists use ILs and, if so, which ones? How they are used and why do they use them?
2) To investigate does the use of ILs lead to differences in the degree of conversions and water uptake (WU) of RBCs?
3) To investigate after treatment with ILs, are there any obvious colour changes at the interfaces between increments?
4) To investigate does the use of ILs lead to differences in the hardness of composites?
5) To investigate does the use of ILs lead to differences in the DTS of composites?
6) To investigate does the use of ILs lead to differences in bond strength between increments?
7) To investigate do the mechanical and physical properties of RBCs treated with ILs significantly change after artificial ageing in an aqueous environment?

3.3 Hypotheses

All the following hypotheses were tested:

1) The questionnaire will show the UK dentists use the ILs to ease the manipulation RBCs;
2) The questionnaire will show UK dentists use different types of ILs;
3) The questionnaire will show UK dentists use different techniques to apply the ILs during the manipulation RBCs;
4) Applying different ILs categories between the RBCs increments will show changes in the RBCs at the interface area;
5) The physical and mechanical properties of tested RBCs will be affected differently by using different ILs categories to manipulate the RBCs;
6) The different categories of ILs will affect the adhesive strength between increments of RBC materials differently.
7) The artificial ageing in an aqueous environment for long extend time intervals will aggravate the effects of the ILs on the treated RBCs.

3.4 Programme of work

1) Literature review phase;
2) Questionnaire-based survey phase;
3) Laboratory investigations phase;

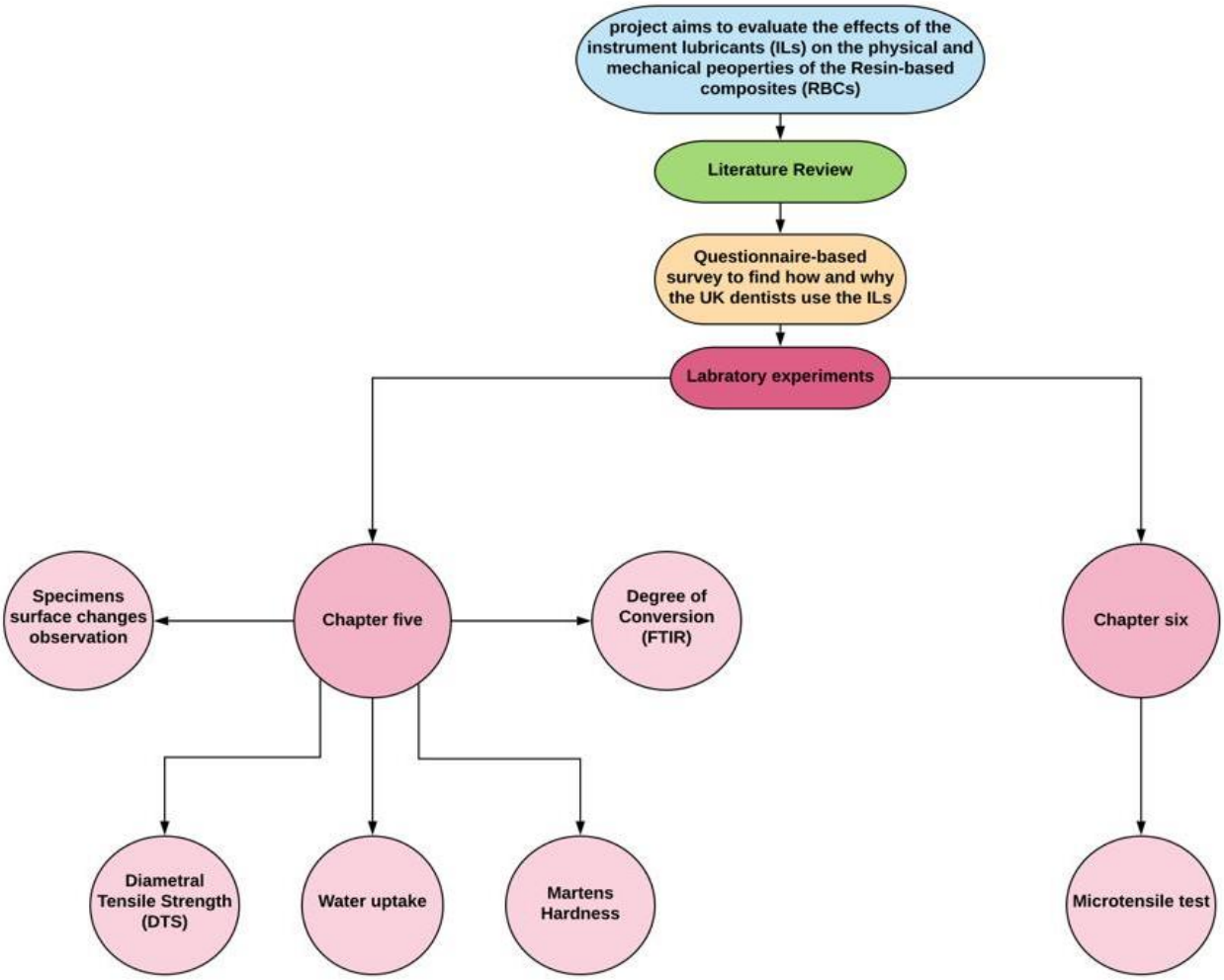


Figure 3-1: Programme of Work (project phases)

Chapter 4: The Questionnaire-Based Survey

4.1 Introduction

There is currently no literature on the use of instrument lubricants in dentistry in the UK. For this reason, a well-designed survey would be important with valid questions around the use of ILs by dentists to determine the current status of use in the UK. Survey designed to gather data on UK dentists' perspectives of the ILs in their practice, as well as their attitude to the placement needs of RBC restorations. Therefore, a self-completion postal questionnaire was designed to investigate whether ILs were being used by UK dentists, and why or why not this was so. The questionnaire covered the following aims:

1) To investigate how and why UK dentists use ILs when placing RBC restorations;
2) To explore UK dentists' opinions on using ILs to manipulate RBC materials.

Also, it is useful to know the type of ILs in use and how they are applied by UK dentists to further support investigations of the effect of their use in laboratory-based experiments, based on current clinical practice.

4.2 Materials and methods

4.2.1 Study design

The self-administered, paper-based questionnaire survey employed in this thesis was sent to UK dentists who were randomly selected and stratified to ensure wide distribution around the UK. In summary, the questionnaire was comprised of 15 multiple-choice questions covering various key themes, to investigate whether and how UK dentists use instrument lubricants whilst placing RBC restorations.

4.2.2 Questionnaire development

The survey instrument was created for the purposes of this study and was therefore not based on an existing published questionnaire. To conceptualise the study topic, a literature review of ILs was conducted in advance and thus a carefully defined concept of IL use in dentistry was attained. The literature review also facilitated a clear and evidence-based approach to questionnaire design, administration, and delivery. The question themes were arranged and piloted so as to be in a continuous, logical and homogeneous sequence, as follows:

- Demographic information
- Types of resin-based composite materials used by dentists
- Techniques used to place RBC restorations
- Types of instrument lubricants used and their application when placing RBC restorations
- Perceived reasons for using or not using instrument lubricants
- Practitioners' knowledge of the effects of lubricants on the properties of RBC restorations

The majority of questions used in the present questionnaire were closed-end questions with limited free text comments encouraged if required. They were written to meet study aims and objectives and cover all the areas that need to be investigated in this topic. The previous sequence arranged the questions to take the respondents through the survey topic smoothly. Also, that can make the links between those parts sensible. Also, at the end of each question's choices, the respondents can add any more information they would like to add.

4.2.3 Refining and piloting the questionnaire

Fifteen questions were constructed and structured into three sections: demographic information; the most commonly used RBC types in the dentists' practice; and, the most commonly used IL when restoring RBCs. The first draft of the questionnaire was revised and refined by the principal researcher (SA), and this draft was then reviewed by the supervisory team and edited further. It then passed through other steps to investigate the clarity and validity of the questions in relation to the standard methods identified in the literature (McColl et al., 2001). This draft was then informally assessed by colleagues from outside the dental field to check the simplicity of the questions' content. The next stage involved four registered dentists and expert researchers from outside the research team. These four were invited and interviewed separately to discuss the clarity, layout, arrangement and sequence of questions, and their comments and suggestions were collected on a feedback form for consideration in the next draft. The final version of the questionnaire was then piloted with twelve registered dentists, who were asked to record their feedback in writing in a section placed at the end of the questionnaire. Some of their comments and suggestions were used to revise further and improve the quality and clarity of the questions before a review of the final version (1.5) by the investigators. All previous steps of the questionnaire refining and piloting for this version has taken within six months of its use in the survey.

4.2.4 Participant selection

This study sought to recruit registered dentists in the UK. Potential participants were selected randomly by selecting the participants from the UK dentists lists by using a randomising tool with commercially available spreadsheet software (Microsoft Excel version 16.59). Also, they were stratified by selecting them from different regions in UK to represent a diverse targeted population. The sample size of each area was

depending on proportions of each one by considering the number of registered dentists in each area. So, the main part was selected from the England and the second part was divided between Wales and Scotland. Participants from England were identified from the Care Quality Commission (CQC) directory, which is updated monthly with all registered health practitioners; participants from Wales and Scotland were identified from the National Health Service's (NHS) service provider directories, as shown in Figure 4-1. The targeted sample size and survey designing were determined depending on some factors. For instance, the main aim in the current survey was to have an idea about what going on in the daily practice of the UK dentists, and how, why are they using ILs in their daily practice. That was not to investigate or evaluate the incidence and prevalence in the targeted population.

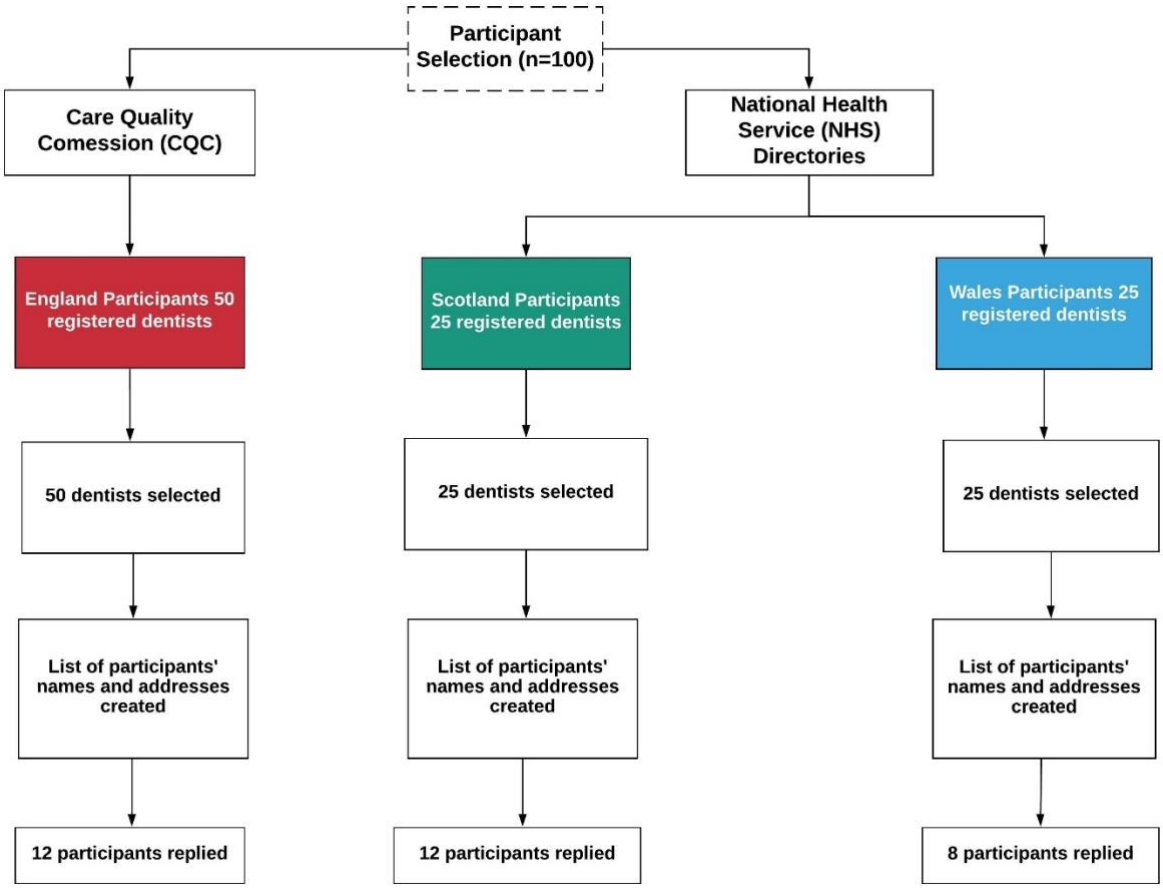


Figure 4-1: Process of participant identification and the numbers targeted

4.2.5 Administration of the questionnaire

Before starting, the university ethics approval for survey was granted and the permission was given to continue. The survey was administered by the principal investigator (SA). All participant information such as names, practice address, and other contact information was collected in a table to manage the sending and receiving of survey documents. One hundred questionnaires were printed with a coloured cover page, and an introductory letter was also included explaining the aims and objectives of the survey. This letter was printed on university-headed paper and addressed with the full name of the dental practice. Prepaid return envelopes size C5 with Royal Mail first class stamps were prepared and attached to the survey documents and placed in

C4 envelopes ready for sending. A participant log was maintained to monitor response rates and facilitate reminders to return a completed questionnaire. After four weeks, a reminder copy with all the documents was sent to the participants who had not completed the questionnaire until that point. The survey was closed six weeks after sending the reminder copies. All the data were then collected, analysed, and presented in tables ready for the next step.

4.3 Results

At the close of the data gathering stage, thirty-two questionnaires had been returned from the one hundred sent, leading to a response rate of 32%. Data were complete except for one respondent who did not include their demographic information, despite completing the remaining parts of the questionnaire. Most of the respondents were male (74%), and as a sample set they held a wide variety of qualifications, with most holding a bachelor's degree (48%). Other qualifications are as described in Table 4-1. The training of the dentists who participated in this study and the mode length of time from qualification are also shown in Table 4-1.

		Response (n=31)	Proportion response (%)
Gender	Male	23	74
	Female	8	26
Qualifications	Bachelor	15	48
	Diploma	5	16
	Master	4	13
	Doctorate	3	10
	Fellowship	4	13
Years of experience	> 10 years	25	81
	4–10 years	4	13
	1–3 years	2	7
Place of clinical training	UK	23	74
	Outside UK	8	26

Table 4-1: Demographic information of the respondents

The responses showed that different types of RBC were used in these dental practices. The distribution of all types of RBCs used are summarised in Table 4-2, and most of the respondents used nanohybrids (53%). The respondents have shown they were familiar at last with some of the RBC types they used them in their practice, as shown in table 4-2. They gave different reasons for their choice of one type of RBC material over others, but the main reason for using a specific type of RBC was ease of manipulation. Some found the material to be easier to finish and polish, and they also appreciated its aesthetic results for colour matching. Lower cost and the ease of availability of some RBCs in their dental clinics were other reasons given for using certain types of RBC in their practice. The results of this section of the questionnaire are summarised and presented in Table 4-2.

		Response (n=32)			Proportion (%)
		With other types	Alone	Total	
RBC Types	Nanohybrid RBC	7	10	17	53
	Hybrid RBC	11	3	14	44
	Microfilled RBC	6	3	9	28
	Bulk-fill RBC	7	1	8	25
	Macrofilled RBC	1	0	1	3
Reasons for selecting RBC types	Easy to manipulate		22		69
	Easy to finish and polish		15		47
	Aesthetic/colour matching		12		38
	The only option available		6		19
	Gives a better adaptation		5		16
	Lower cost		3		9
	↑ strength-wear resistance		3		9
	Less stickiness		2		6

Table 4-2: Distribution of RBC types and the reasons for using them

Approximately half of the respondents (47%) reported using instrument lubricants for various reasons, summarised in Table 4-3. The most popular or frequently used IL was two-step composite bonding agent, followed by one-step composite bonding agent. Modelling resins were also used by one-third of respondents. This section of the questionnaire is summarised in Table 4-4.

		Response(n=32)	Proportion response (%)
Using ILs	Yes	15	47
	No	17	53
		Response (n=15)	
Reasons for using ILs	Makes material manipulation easier and more controllable	14	93
	Gives more aesthetic result	9	60
	Reduces the placement time for direct restoration of RBCs	3	20
		Response (n=17)	
Reasons for not using IL	Would affect the mechanical properties of the materials	7	41
	Unnecessary	6	35
	No evidence to support its use in clinical practice	3	18
	Wastes time	2	12

Table 4-3: The rationale for the use or non-use of instrument lubricants

		Response (n=15)	Proportion response (%)
Most used IL types	Composite bonding agent	10	67
	Resin modelling materials	5	33
Most used bonding agents	Two-step composite bonding system	6	40
	One-step composite bonding system	5	33
	I don't use these materials as instrument lubricants	4	27
Reasons for preference	I was taught to use it	6	40
	Other colleagues use it	4	27
	Has no negative effect on RBC properties	4	27
	Gives a better result than other instrument lubricants	2	13
	Several studies support using this lubricant	1	7
	None	1	7

Table 4-4: Types of instrument lubricant employed and the rationale for their use

Respondents indicated that they used incremental and/or bulk-fill techniques to place RBC restorations, distributed as presented in Table 4-5. Most of the respondents used both techniques independently of each other, with the incremental technique being used more often. IL was used on top of the last increment by 40% of the sample; the remainder either applied the ILs with every increment or only the first (Table 4-5). The main methods of applying the IL to the RBC were by direct application onto the RBC with a microbrush or by wiping the surface of the instrument with a lubricant agent. Other methods are shown in Table 4-5.

		Response (n=32)	Proportion Response (%)
Placement techniques of RBCs	Incremental only	14	44
	Bulk-fill only	1	3
	Incremental & bulk-fill	17	53
		Response (n=15)	**Proportion Response (%)**
Techniques of applications	With the last increment	6	40
	With every increment	5	33
	With the first increment	3	20
	On occasion	1	7
Methods of application	Direct application with a microbrush	7	47
	Wiping instrument surface with a lubricant	6	40
	Dipping the instrument tip into a lubricant	2	13

Table 4-5: Techniques and methods for applying instrument lubricant

About half of the respondents indicated they used IL, and they used different methods and techniques to manipulate the materials. Some used the IL to make the placement of the direct RBC restoration easier and more manageable. Their opinions regarding

the use of IL were diverse, as summarised in Table 4-6, but the majority (59%) were neutral regarding this matter.

		Response (n=32)	Proportion response (%)
Respondents' opinions of the use of ILs	Neutral	19	59
	Recommend	8	25
	Not recommend	4	13
	Strongly recommend	1	3

Table 4-6: Respondents' opinions of the use of instrument lubricant

4.4 Discussion

This self-administered, paper-based questionnaire was distributed to UK-registered dentists through their professional practice addresses. Prospective participants were randomly selected after being stratified to cover different areas of the targeted population. The choice of this type of questionnaire was based on being able to reach a broad and representative group of participants because the sample can be smaller and more manageable to represent the larger surveyed population. So, the selected participants can aid in the analysis of bigger groups. that can save a lot of time and money by using the representative sampling. So, that can capture an accurate picture of a larger targeted group through statistical analysis and data assessment (Edwards, 2010; Gorrell *et al.*, 2011; Sinclair *et al.*, 2012; Rowley, 2014; Song;Son and Oh, 2015; Daikeler;Bošnjak and Lozar Manfreda, 2019). In the present survey, the sample size was small and affected by some factors that were mentioned in previous section 4.2.4 to explain how the targeted sample was determined. However, the collected data still can represent some important data that would cover the present survey aims and objectives by answering the study questions.

The previous studies have found that the self-administered, paper-based questionnaire as data collection method has an advantage over other methods like the face-to-face method. The technique also reduces costs, time, and effort. Moreover, people may prefer to participate in a postal survey in comparison to other modes (Horowitz and Sedlacek, 1974; Lozar Manfreda *et al.*, 2008; Sinclair *et al.*, 2012; Song;Son and Oh, 2015; Daikeler;Bošnjak and Lozar Manfreda, 2019) due to it being more convenient for the participants, reducing bias from the interviewer, and being able to cover a broad geographical area of participants in one survey. However, the possibility of a lower response rate with this method of survey is high, and this can affect the outcome and generalisability of the survey due to potential response bias (Hochstim, 1967; Sudman, 1967; Horowitz and Sedlacek, 1974; Johnson and Wislar, 2012; Rowley, 2014).

The response rate of a survey is a helpful element which facilitates valid inferences to be drawn about the targeted population (Rattray and Jones, 2007). The response rate of 32% in the current study is within the range of previously conducted surveys in dentistry, which is reported in the literature as 16–100%. Also, the literature anticipates an average postal-based survey response rate of 40% or less, and the current survey is within this rate. The expected response rate of the current study was between 40-60%. However, the literature has mentioned there was low response rate from the previous studies in the medical and dental studies. Also, the postal-based survey has considered as one of the low response rate surveys and the returns mail can be missed which make the response rate low (Tan and Burke, 1997; McColl *et al.*, 2001; Watt *et al.*, 2002; Rowley, 2014; Holmes, 2016).

There are many methods of increasing the rate response of questionnaires and mail-based surveys as highlighted in the literature, to improve the response rate and reduce

the bias possibility of the collected data. Of these methods, the current study used an introductory letter on official paper, printing on coloured paper, provision of a prepaid return envelope, and reminder letters. Previous studies have also found that the use of incentives or rewards might increase the response rate (Fanning, 2005; Robertson;Walkom and McGettigan, 2005; Vangeest;Johnson and Welch, 2007; Dykema *et al.*, 2011). In the current study, the targeted population of UK dentists predominantly have a high income, and so offering small incentives or rewards was unlikely to achieve any great increase in the return rate (Clark;Khan and Gupta, 2001). Another reason for not employing incentives was that some ethics committees consider them unacceptable due to potentially exerting pressure on the respondents to participate (Edwards, 2010). Also, project funding would not provide vouchers and incentives of sufficient value to be practical and useful in the current situation. Despite the use of several methods to increase the response rate, in comparison to other targeted populations, the relatively low response rate from the selected health service providers cannot be ignored (Tan and Burke, 1997; Vangeest;Johnson and Welch, 2007). In considering this, as the survey was to determine whether dentists used ILs, what types of IL were in use and how they were being used an accurate population estimate of proportions doing so was considered to be less important and the survey provides important and valid data.

The response rates of many types of survey have declined over the past decades (Sivo *et al.*, 2006; Baruch and Holtom, 2008; Fincham, 2008; Lisa R Carley-Baxter, 2009). This limitation of low response rate could be related to different factors. The researcher has the potential to control those factors like questionnaire design, mailing and follow-up reminders, and questionnaire management. Some of those factors beyond the researcher control, like previous personal experience of the respondents, link the

survey to the institution that conducted the study and some individual personality traits (Morris;Cantrill and Weiss, 2001). In some studies that discussed some reasons for the low response rate, they found the main reason that has been mentioned the respondents were too busy, or there were no clear reasons stated (Morris;Cantrill and Weiss, 2001; Sivo *et al.*, 2006; Baruch and Holtom, 2008).

The literature has shown that the response rate is an important indicator of the sample quality. From the present study, even within the limitation of low response rate coming from a small sample size that can limit the power of the collected data. However, the collected data still can show that about half of respondents used the ILs in their daily practice. Also, they had a different opinion regarding ILs, and they used other techniques and methods to apply those materials when they manipulated RBC materials. So, these data do show that ILs are used by some of registered dentists in the UK to manipulate the RBC materials. Also, they had a different opinion regarding ILs and used different techniques and methods to apply those materials when they manipulated RBC materials.

The respondents' group was representing opinion part of UK registered dentists that were part of the targeted population not only representing response rate. They have given an important opinions and data regarding the use of ILs. Their opinion and experience were a helpful start point to understand what happened in their daily practice as UK registered dentists. The collected data that obtained from those participants has clarified an important aspect and helped to go further to investigate this matter. In considering this, that can provide important, valid, and importantly novel data.

Having a wide variety of respondents may help to gather a broad view of opinions. In the current survey, most respondents were male, representing about three-quarters of those who responded; this is despite the fact that NHS statistical data for 2018/19 showed that the percentage of male to female dentists was roughly equal (NHS, 2020). According to the literature, the response pattern of males and females can fluctuate in survey-based studies, although some studies have shown the same pattern as the current survey (Yusuf *et al.*, 2015; Holmes;Burford and Vance, 2020). In the current study, it was not anticipated that the gender of the participants would make an essential difference to answering the survey. Both genders are considered as registered dentists and they practice in UK. They were certified and have the skills to place the RBC restoration in their daily practice. So, the participants in present survey either males or females still consider as UK registered dentists. Also, from the demographic information gathered, it can be noticed that most respondents had been practising for over ten years and had a bachelor's degree in dental surgery (BDS) (or other qualifications), as summarised in Table 4-1. Therefore, newly qualified participants represented a smaller section of the sample, of which most had higher qualifications and more up to date knowledge in dentistry. However, all of them still considered as registered dentists and they are placing RBC restorations in their daily practice. So, that will reflect the situation in the UK dentists' daily practice and validity of collected data.

The use of RBC materials has increased in the daily practice of dentists in recent decades, but the manipulation of these materials is an ongoing problem (Moraes *et al.*, 2009; Tuncer *et al.*, 2013; de Paula *et al.*, 2016; Patel *et al.*, 2017). The current survey has shown that this selection of dentists in the UK used different types of RBCs in their clinical practice, with the most popular types being nanohybrid, hybrid, and microfilled.

These types are reported to exhibit better aesthetics and reliable physical and mechanical properties compared to other materials (Alzraikat *et al.*, 2018), and this explains the participants' selection of these types in the current survey.

Different techniques for placing RBC materials were used by the participants in the current survey, especially incremental placement, which is supported by many previous studies. These studies suggest that the incremental technique is one of the more common for applying RBCs to cavities (Dunn, 2007; Shawkat *et al.*, 2009; Tiba *et al.*, 2013). Among the respondents who participated in the current survey, about half used an IL in their practice. In general, applying an IL during the placement of an RBC restoration can reduce the stickiness of these materials to the placement instruments (Tuncer *et al.*, 2013; Münchow *et al.*, 2016; Patel *et al.*, 2017). However, while the use of IL has been shown to affect some of the physical and mechanical properties of the tested materials, results from these previous studies cannot be automatically applied and generalised for all recently introduced RBC materials.

From the collected data within the limitations of the current survey, the surveyed dentists continue to be split to either support or reject IL materials. As shown in the responses from the current survey, most of the respondents reported neutral views regarding the use of ILs to manipulate RBC restorations, possibly due to the lack of available evidence. These participants used different types of ILs to manipulate RBC materials, with most using a bonding agent or modelling resins, and they applied them using various application methods, such as dipping and wiping the hand instruments with the lubricant or using a microbrush. The frequency of applying ILs also varied between respondents, with some applying them to each increment of RC placed, and some only applying them to the last increment to sculpt and shape the restoration. This shows that the ILs and application techniques selected by the respondents were

dependent on the dentists' preferences, and so more evidence is required from the literature to support one IL and technique over another.

4.5 Conclusion

Within the bounds of the current study, the key conclusions are:

- Approximately half of the respondents reported IL use when placing RBCs;
- Respondents used bonding agents and the modelling resins as an IL;
- The main reasons for using ILs was to reduce the stickiness of the RBC and improve manipulation of the RBC material;
- Some respondents suggested using an IL enhances the aesthetic outcome of the RBC restorations;
- Multiple methods of application were used to apply the ILs, including by microbrush or wiping the surface of an instrument;
- Most respondents applied the IL on the last increment when placing a RBC but some applied IL with each increment;
- About half of the respondents used ILs in their practice even though they were unsure of the benefits/adverse effects of ILs upon the material properties or longevity of restorations;
- The findings from the questionnaire suggest that no clear evidence or guidance is followed by dentists on the use of ILs to manipulate RBC restorations.

Chapter 5: Laboratory Investigation of the Effects of Instrument Lubricants on the Physical and Mechanical Properties of RBCs

5.1 Introduction

Although instrument lubricants are used to enhance the handling of RBC restorative materials, they affect the main components of these materials and thus the physical and mechanical properties. In this context, previous studies have investigated the effects of ILs on degree of conversion (DC), surface hardness, water uptake and diametral tensile strength (DTS). Most found that ILs diminished the physical and mechanical properties of treated materials, which then affect the stability and longevity of RBC restorations (Tuncer *et al.*, 2013; de Paula *et al.*, 2016; Patel *et al.*, 2017). Although these studies tested specific types of RBC and ILs, many new types of materials have recently been introduced in dental practice. To extend current knowledge in this regard, this chapter reports on the laboratory investigations which were undertaken of DC, HM, water uptake, DTS and interface area change observations. These parameters were selected to evaluate the effects of different classes of ILs on the physical and mechanical properties of different RBC materials. They have been selected to evaluate the RBC materials polymerisation efficiency, and the amount of the converted monomers to polymers. That is very important point in the present study to evaluate the effects of the ILs on the DC and polymerization process. Furthermore, how that would affect the amount of the water uptake of the RBC materials in parallel to another parameters like DC. Also, the evaluation of the strength of the treated RBC materials with ILs by using DTS can provide a data that can detect in general the effects of ILs on strength of treated RBC. That can help to have a broad idea before conducting more specific tests. Also, to test the possible effects of ILs on the hardness of the treated compered to non-treated materials, and if that has a relation to the other parameters. So, some data were collected from the survey-based

study reported in Chapter 4, in order to establish the design and methodology of the current chapter. All these data were used as guidance to design the laboratory investigations and make them more linked to the dentists' daily practice.

5.2 Materials

In this chapter, different types of RBC materials were evaluated, as summarised in Table 5-1. The effects of classes of ILs such as bonding agents, wetting resins and solvents, noted in Tables 5-2, 5-3 and 5-4, were investigated. All the materials were tested using established laboratory tests at various time intervals.

Material	Contents	Manufacturer
Filtek Supreme XTE Resin-based composite (A2), LED cured with a minimum intensity 400 mW/cm^2 in the 400–500 nm range	Bis-GMA, UDMA, TEGDMA, Bis-EMA, with PEGDMA replacing part of TEGDMA. Nanoparticles are 72.5% by weight (55.6% by volume, initiators and silane	3M ESPE Dental Products 2510 Conway Avenue St. Paul, MN 55144-1000 USA
Harmonize Nanohybrid Universal Composite (A2), LED cured with a minimum intensity 600 mw in the 400–520nm	Bis-GMA, Bis-EMA, TEGDMA. 81% fill ratio by weight and 64.5% fill ratio by volume. Smallest primary particle size ~5 nm	Kerr Corporation 1717 West Collins Avenue Orange, CA 92867 USA

Table 5-1: RBC materials components

Material	Contents	Manufacturer
Scotchbond Universal Adhesive (2 step)	MDP phosphate monomer, dimethacrylate resins, HEMA, Vitrebond copolymer, filler, initiators, silane, water, ethanol	3M Deutschland GmbH Dental Products Carl-Schurz-Str. 141453 Neuss-Germany
Optibond Solo Plus Total-etch Bottle (2 step)	HEMA, 2-hydroxy-1,3-propanediyl, bismethacrylate, filler, initiators, silane, water, ethyl alcohol	Kerr Corporation 1717 West Collins Avenue Orange, CA 92867 USA

Table 5-2: Bonding agent components and their manufacturers

Material	Contents	Manufacturer
Brush & Sculpt	Multi-functional acrylic resin and 0.04-micron silica filler. 36% by weight and 24% by volume. Contains 1,4-Butandiol dimethacrylate, BIS-GMA and diurethane dimethacrylate	Deltamed GmbH Raiffeisenstrasse 8a 61169 Friedberg Germany
Modelling resin: PREMISE INDIRECT	Uncured methacrylate ester monomers, Photoinitiators and stabilizing additives, an unfilled universal wetting agent used for sculpturing	Kerr Corporation 1717 West Collins Avenue Orange, CA 92867 USA
Signum Liquid	Dimethacrylate, multifunctional methacrylic acid ester, silane, photo-initiators Triacrylateines oxyethylierten Trimethylpropans Diphenyl(2,4,6-trimethylbenzoyl)phosphinoxid Solids content: 10.8 %	Kulzer GmbH Leipziger Straße 2 63450 Hanau, Germany

Table 5-3: Wetting resin components and their manufacturers

Material	Manufacturer
Acetone	Fisher Scientific UK Limited, Loughborough, UK
Ethanol (absolute, >= 99.8%)	Fisher Scientific UK Limited, Loughborough, UK
Isopropyl (Propan-2-ol, >=99.8%)	Fisher Scientific UK Limited, Loughborough, UK
Distilled water	Dental Materials lab, School of Dental Sciences, Newcastle, UK

Table 5-4: Manufacturer information for organic solvents and sources of distilled water

5.3 Methods

5.3.1 Depth of cure

The depth of cure (DoC) for both RBC types was tested, with an LED LCU (Elipar™ DeepCure-S, 3M ESPE, 3M Deutschland GmbH, Germany) being used to polymerise the RBC specimens. The wavelength range of this LED LCU is between 430–480 nm and the wavelength peaks at 444–452 nm. The irradiance of the LCU was measured for 40 seconds using Bluephase Meter II (Ivoclar Vivadent Limited, UK) at 1,020 mW/cm^2, and the LCU tip was protected with a plastic sleeve, as shown in Figure 5-1. The test was performed following ISO 4049 for evaluating the DoC of the LCU for both RBC types. A brass mould with a rectangular slot was used, the dimensions of which were 25 mm length x 2.5 mm x 2.5 mm (Figure 5-2 and Figure 5-3). The microscope slide was placed first followed by the Mylar strip (Kent Dental, Gillingham, UK) and then the mould. To reduce the air bubbles, the mould slot was overfilled with RBC and the material compressed digitally by applying constant finger pressure with the Mylar strip so that it was flush with the brass-mould surface. A second brass mould was then pressed against the first and then both moulds secured with a rubber band (Figure 5-4a). The LCU tip guide was placed in direct contact with the mylar strip against the top side of the slot and the materials were exposed for 40 seconds (Figure 5-4b). All the Mylar strips were then removed and the mould parts separated.

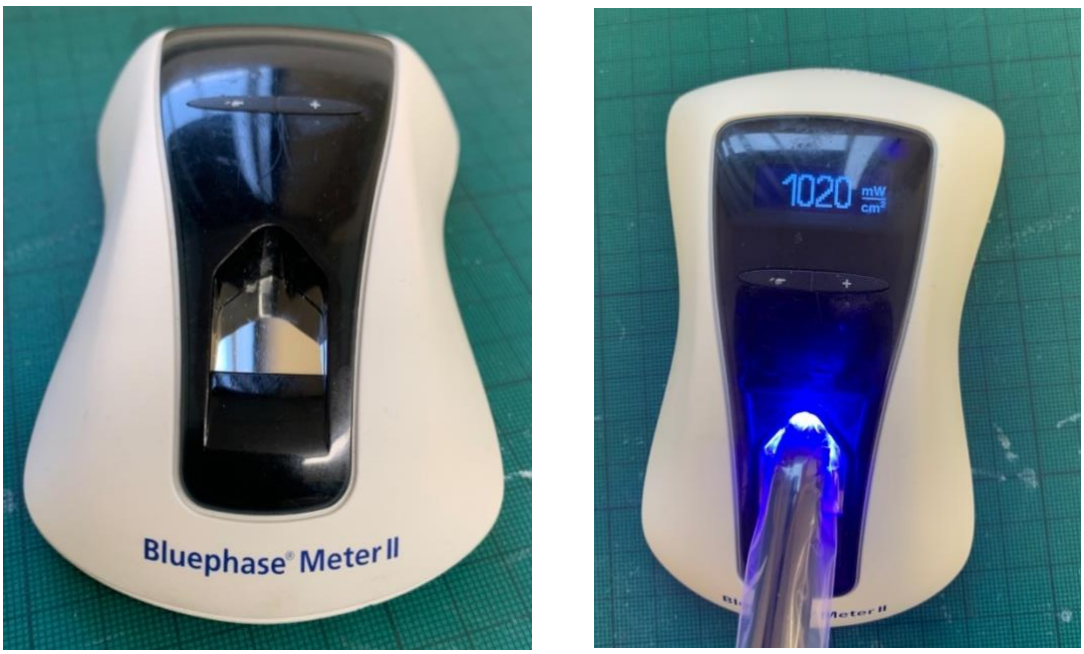


Figure 5-1: Bluephase Meter II and irradiance measuring of the LED LCU

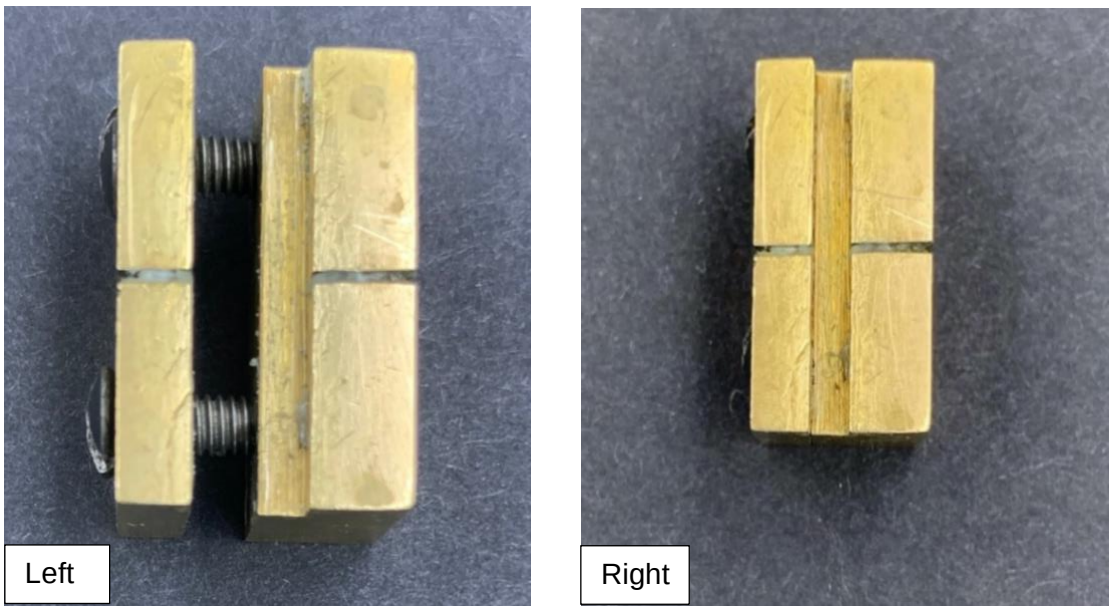


Figure 5-2: Left: Separated mould parts; Right: Assembled mould and a view of the mould slot

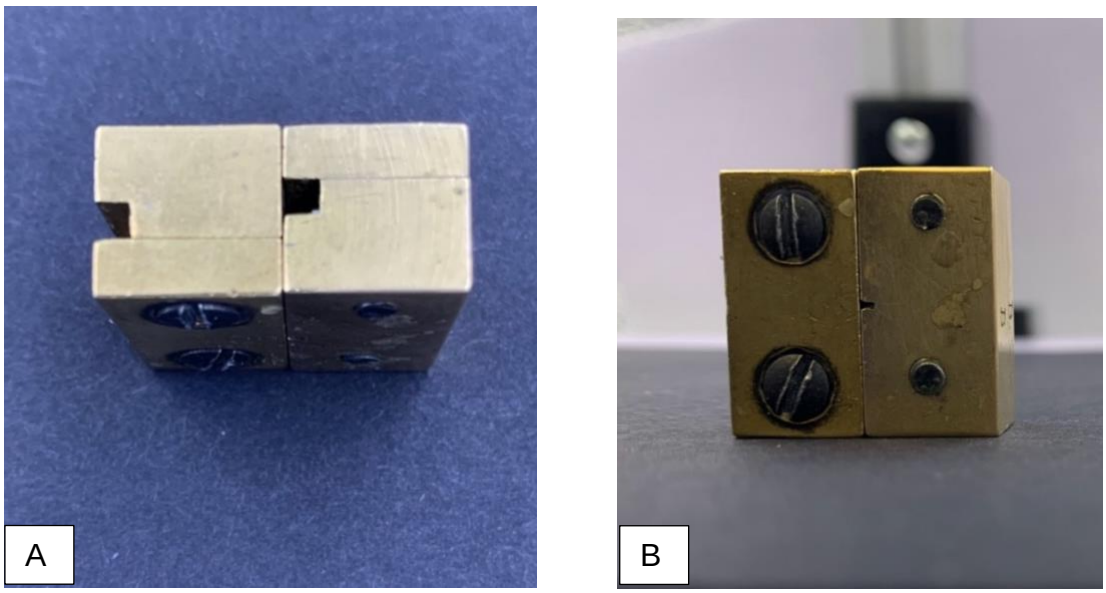
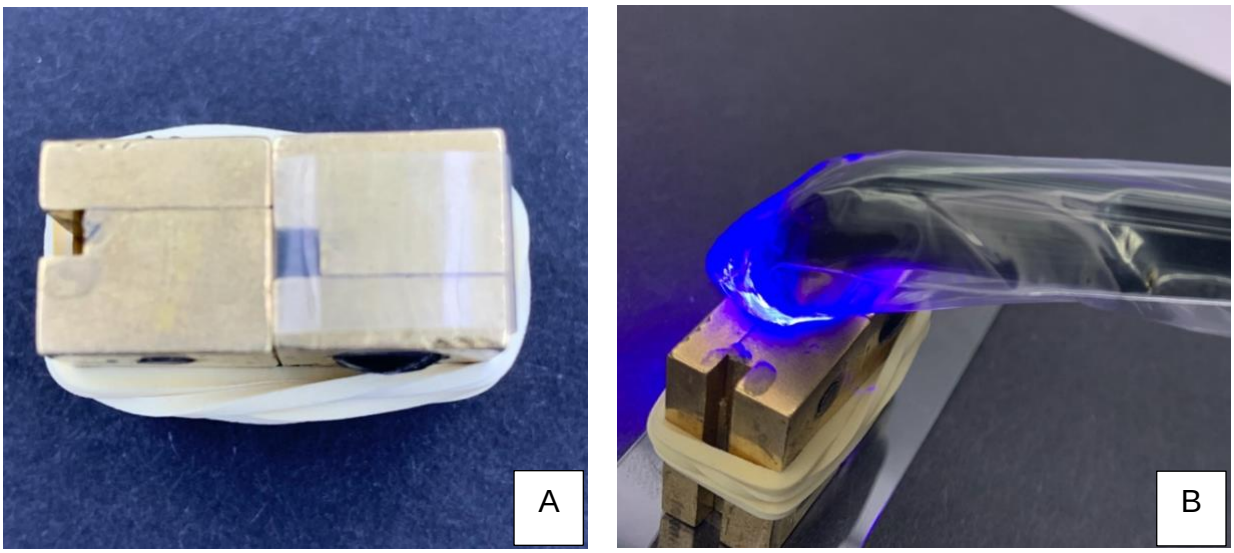


Figure 5-3: Top (A) and lateral (B) views of the DoC mould



A. Top view of the mould with the Mylar strip and slot pressed using the back of another mould, and held with a rubber band

B. Position of the LCU on the top of the mould at the slot opening

Figure 5-4: (A) The DoC mould ready for curing the specimen and (B) the LCU position.

A plastic spatula was then used to remove the uncured RBC material. The polymerised part of the materials was measured with a digital calliper, accuracy ±0.1 mm (carbon fibre composite digital caliper, Esslinger & Co., Saint Paul MN, USA). Then full length of cured RBC after uncured part was removed divided by two as mentioned in ISO standard to determine the DoC All these procedures were repeated five times for each RBC type.

5.3.2 Specimen preparation for the changes observation, water uptake and diametral tensile strength experiments

Each class of ILs was divided into groups representing the different lubricants and a control group prepared without ILs (Figure 5-5). The pilot experiments were conducted to calculate means and standard deviations. Twelve specimens for DTS, six for water uptake and three for the changes observation were prepared and tested. The control groups were prepared to compare it to the experimental groups. Effect sizes was measured between groups to determine the magnitude of the experimental effect. The power and the number of specimens determined at the level of significance at (5%) and the power (80%) were calculated (Minitab 18.1, Minitab, Inc., United States). After the power and sample size were confirmed, six cylindrical specimens were prepared for water uptake, twelve for DTS, and three for the changes observation at increments interface area for each type of lubricant (Figure 5-6a,b,c). The experiments specimens' preparation steps, number of increments and time intervals were illustrated in Table 5-5, Figure 5-6, 5-7 and 5-8

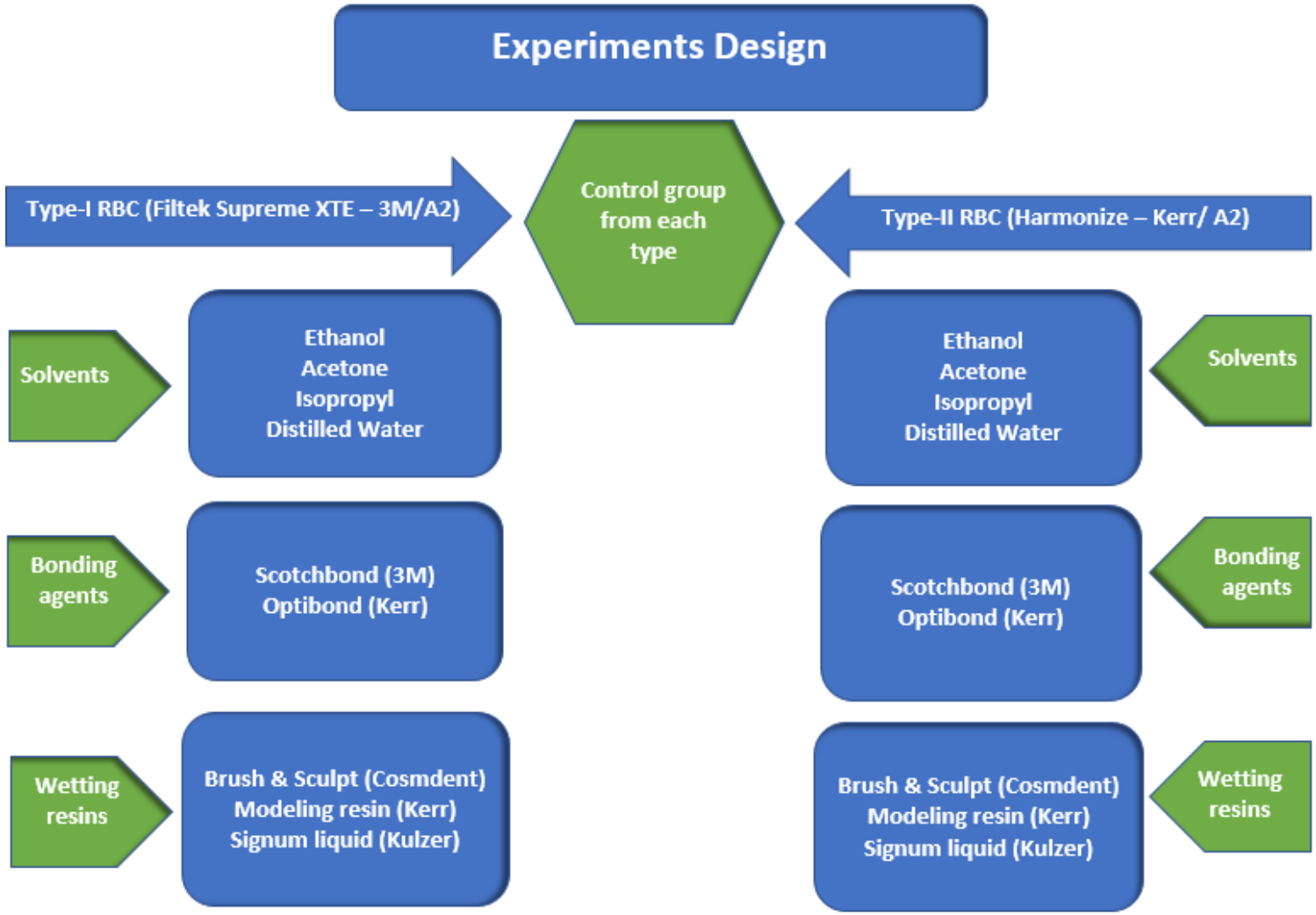


Figure 5-5: Experimental design and the experimental group

Experiments	1st Step	2nd Step	3rd Step	4th Step	Final Shape	Testing Machines
The changes observation at interface area 3 (specimens/group)	1st increment 3mm	Curing with LED LCU for 40 Second	2nd increment 3mm	Curing with LED LCU for 40 Second	5 mm x 6 mm	Canon professional camera
Diametral Tensile strength 12 (specimens/group)						Universal Testing machine
Water uptake 6 (specimens/group)					Cylindrical shape specimen Time intervals (Baseline-90 days)	Electronic Balance Instron
Degree of conversion & Martens hardness 5 (specimens/group) Setup one & two	1st Increment 3mm	Curing with LED LCU for 40 Second	NA	NA	10 mm x 3 mm Disc shape specimens Time intervals (Baseline/1-day)	Fourier Transform Infrared Spectroscopy (FTIR) Zwick Roell, Universal Hardness Tester

Table 5-5: The specifications and steps in the specimen preparation

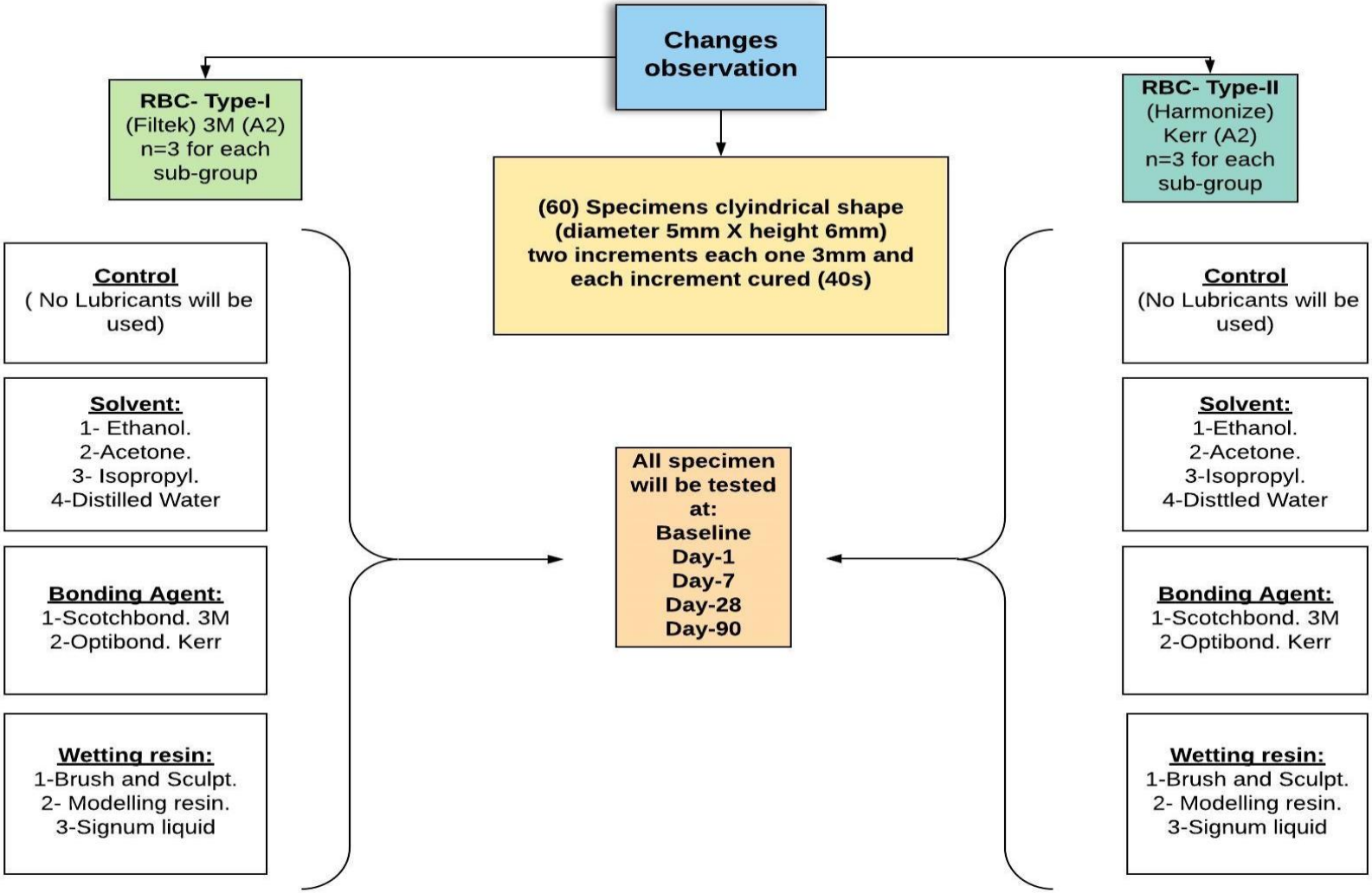


Figure 5-6: Specimen dimensions and key data for the observation of changes at the interface between the incrementsSpecimen dimensions and key data for the observation of changes at the interface between the increments

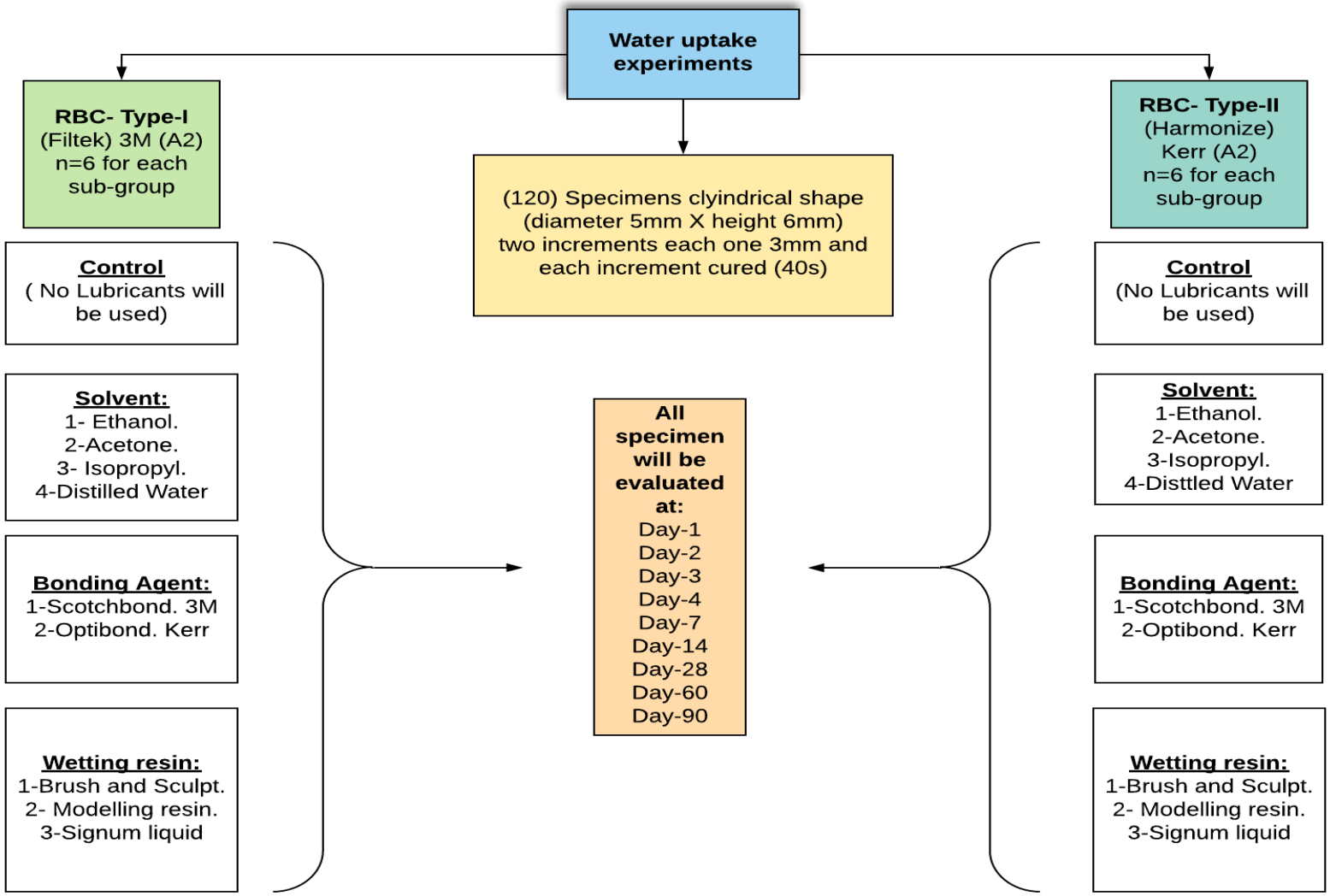


Figure 5-7: Specimen dimensions and key data for the water uptake experiments

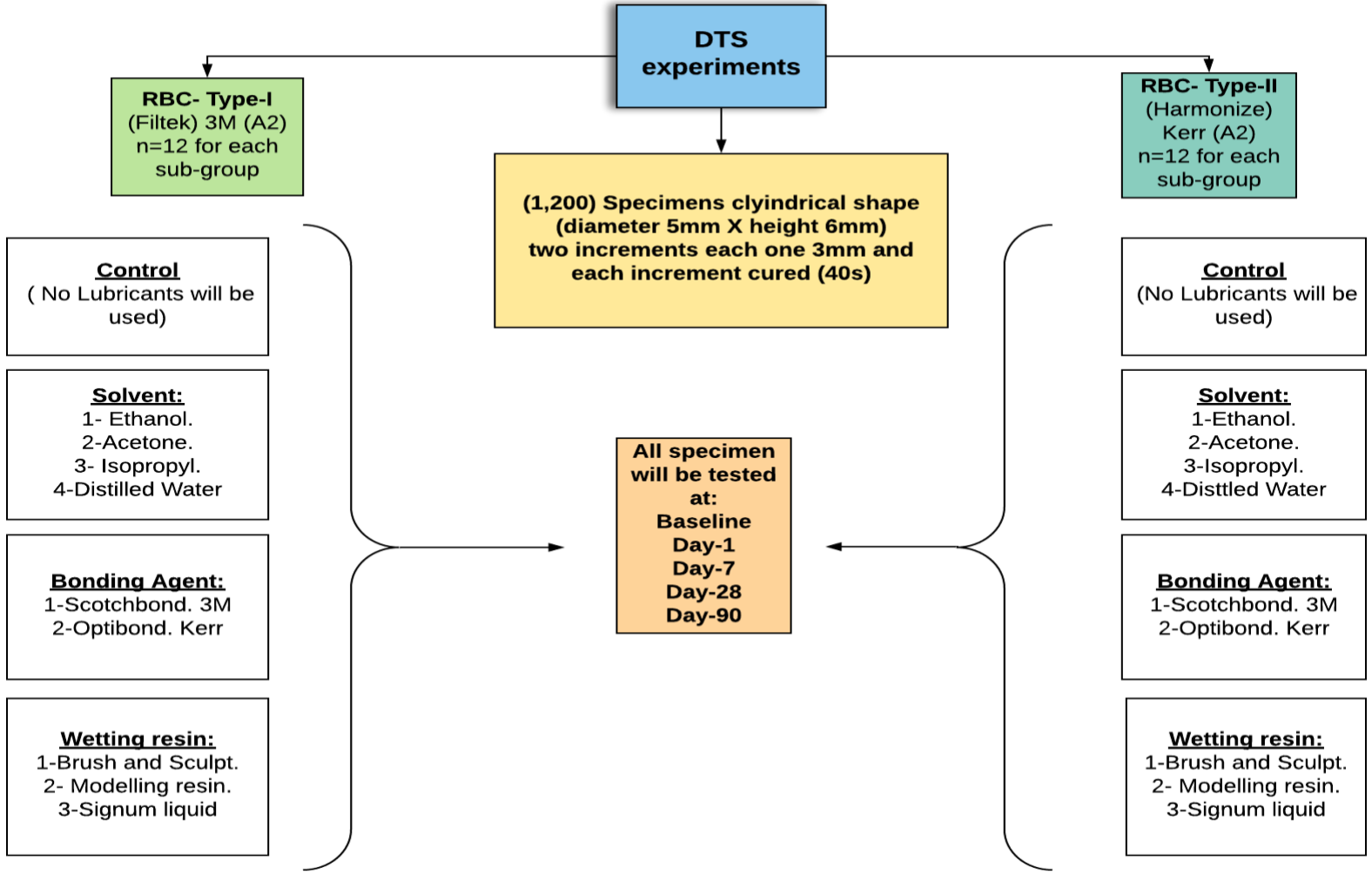


Figure 5-8: Specimen dimensions and key data for the diametral tensile strength experiments

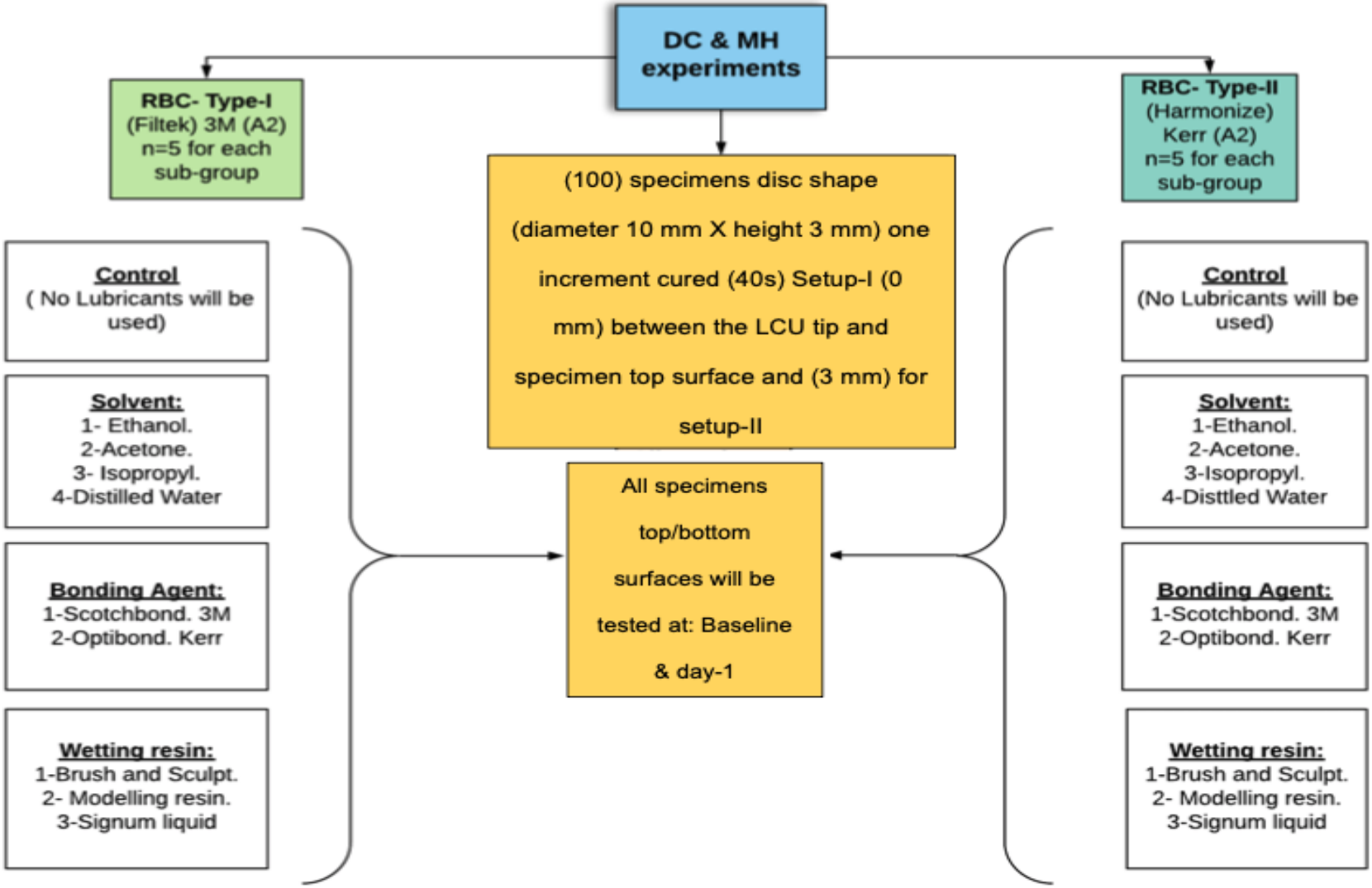


Figure 5-9: Specimen dimensions and key data for the degree of conversion and hardness experiments

During the preparation of the specimens, the lab curtains were closed, and the lab lights turned down to minimise the light received by each specimen. All the specimens were prepared with stainless steel moulds, with dimensions of 5 mm±1 in diameter and 6±1 mm in depth, in two increments of 3 mm each, as in Figure 5-11 (centre and right). One operator performed all the manipulation and curing of the specimens. During placement, a glass slide and Mylar matrix strip were placed beneath the mould to support the material. A stainless steel plugger (Dentsply Ltd, Weybridge Surrey, UK) with tip dimensions of 2.25 mm diameter and 3 mm height was used to place the material into the mould as shown in Figure 5-12 (left). A fresh plugger was dipped into the lubricant up to a depth of 3mm and allowed to drip for two seconds before being used on the top of the RBC materials as illustrated in Figure 5-12 (right). The motion of the plugger tip was moved constantly from mould rims side to side within same pattern for all specimens treated surfaces to cover it with the ILs. The plugger used to manipulate RBC at the top until all materials become even. Then this increment was polymerised for 40 seconds with a LED light cure unit (Elipar™ DeepCure-S, 3M ESPE, 3M Deutschland GmbH, Germany). The tip of LCU light-guide was covered with a plastic protective sleeve (Dentsply Detrey GmbH, Konstanz, Germany), and then the LCU was held as close to the mould as possible by using a prefabricated base (Figure 5-13), mechanical arm and reference points on the curing light to standardise its position to the mould. The second increment was added until the RBC filled the mould up to the orifice, after which a Mylar strip was placed over the surface and digital pressure applied with a microscope slide. The latter can help to ensure that the top surface is uniform with the orifice level to maintain a constant height across the specimens. The glass slide was removed and the second increment polymerised for 40 seconds. Finally, the Mylar strip and any excess material were removed from the specimens' margins, before the specimens themselves were removed from the mould.

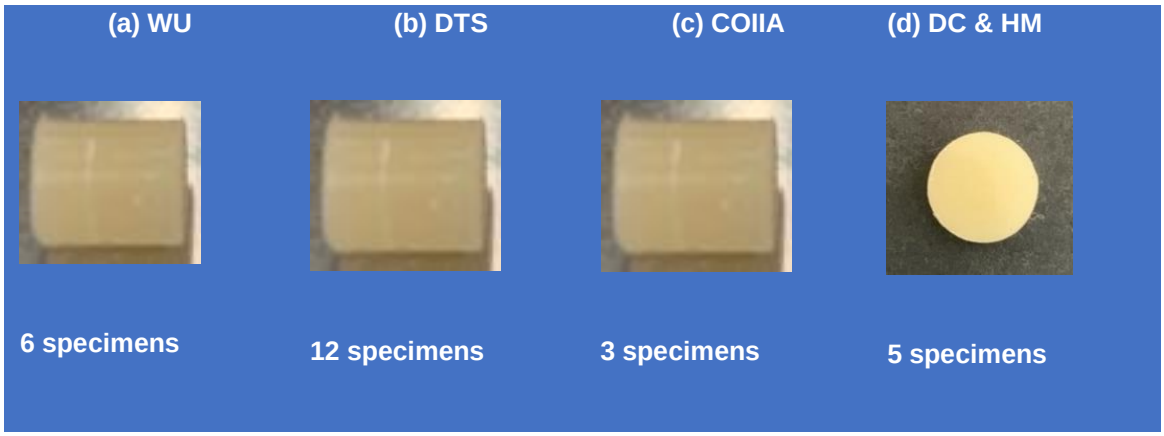


Figure 5-10: Final specimen shapes and number for each experiment

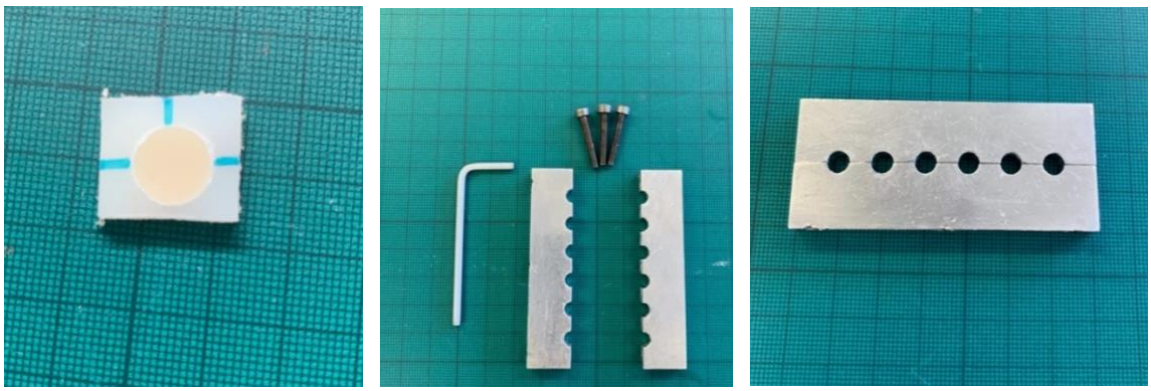
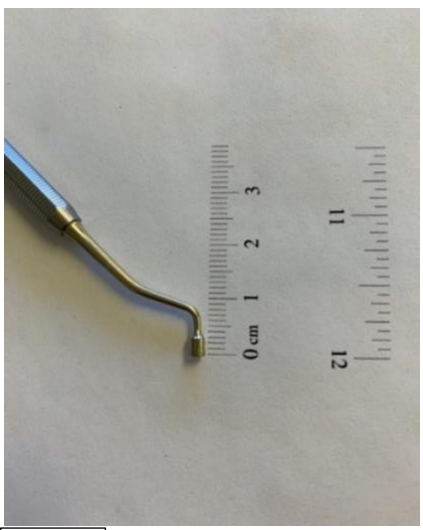


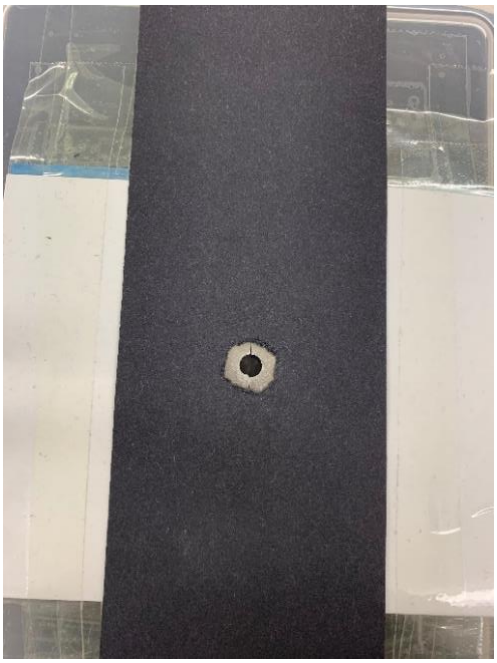
Figure 5-11: Left: polytetrafluoroethylene (PTFE) mould for the DC and HM specimen preparation; Centre and Right: stainless steel attachable mould for COIIA, DTS and WU specimens' preparation



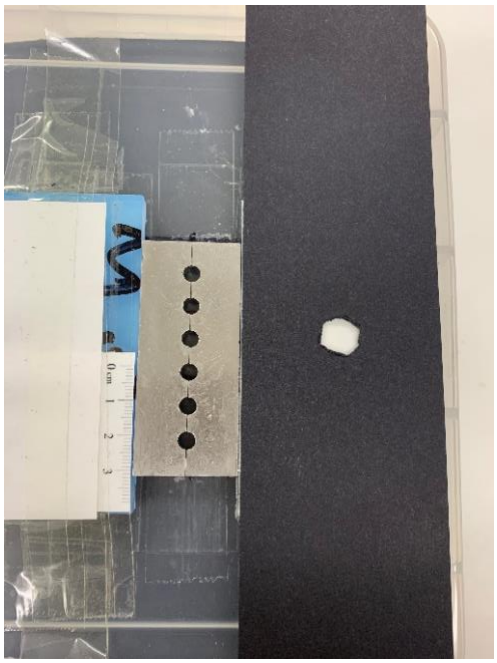
Left

Right

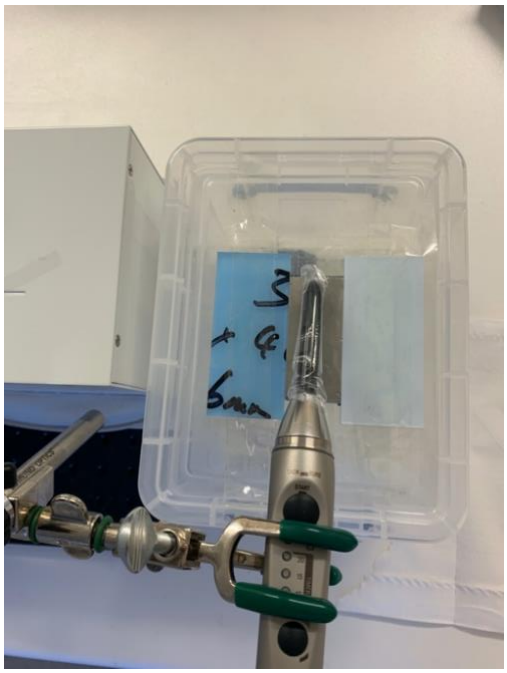
Figure 5-12: Left: The dimensions of the plugger tip; Right: The plugger tip adjusted with a reference point, the mould margin



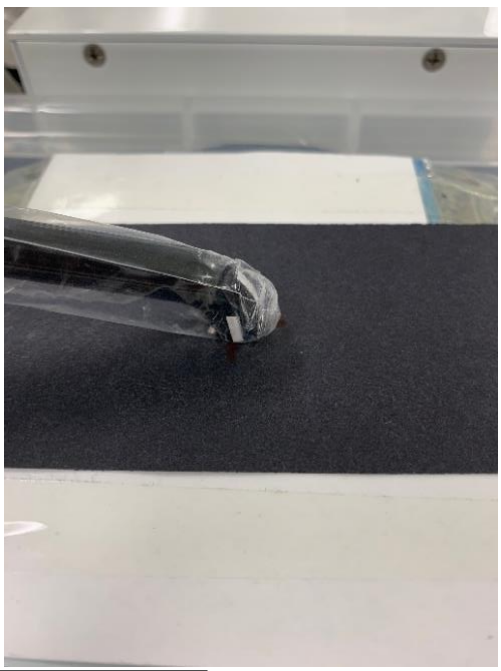
Top left



Top right



Bottom left



Bottom right

Figure 5-13: Top (left & right) , and bottom left: The base fabricated to standardise RBC curing and the reference points on the mould, base and LCU tip; Bottom right: How the LCU was attached to the mechanical arm to standardise its position

5.3.3 Specimen preparation for tests of degree of conversion and Martens hardness

two setups were used, one with the LCU tip placed directly onto the specimen top surface (termed 0 mm) and one with the LCU tip 3mm from the top surface of specimen, as presented in Figure 5-14 and 5-15. The pilot tests were performed as well as the power calculations for the number of specimens. The level of significance was looking at (5%) and the power (80%) (Minitab 18.1, Minitab, Inc., United States). Each group included five disc-shaped specimens as shown in (Figures 5-6d) prepared for each type of lubricant, as illustrated in Figure 5-5. The experiments specimens' preparation steps, number of increments and time intervals were clarified in Table 5-5 and Figure 5-9.

As described in the previous section, during the tests, ambient and electric lighting were controlled and one operator performed all specimen manipulation and curing. All the specimens were prepared using polytetrafluoroethylene (PTFE) moulds of 10 mm in diameter and 3 mm depth for the DC and hardness test specimens. The lubricated surface of the specimen was marked for identification during the test by placing indelible ink marks on the outer margins of the mould as shown in Figure 5-11 (left). A glass slide and Mylar strip were placed beneath the mould to support the material during placement. A plugger with tip dimensions 2.25 mm x 3 mm height was used to place the material into the mould without using IL. Another plugger was dipped into each respective lubricant for 1 s (up to a 3 mm marked line on the instrument) and left to drip for 2 s prior to use. This plugger was used to shape the material at the top until it was even. When the mould space was filled with RBC, the Mylar strip was placed over the mould and pressure applied with the microscope glass to ensure the surface was as smooth as possible. This process not only helps the material to maintain firm contact with the FTIR lens, it also makes the scanning of the specimen surfaces more

effective. It is also necessary to have a flat and smooth surface when the HM indenter takes readings from specimen surfaces.

Two setups were prepared for the testing. For setup one, the specimen after the mould filled up with RBC and lubricated with ILs on the surface. The Mylar strip and glass slide were placed on the lubricated side as described previously. Also, before curing the specimen, the mould and glass slides from each side were held together as one part and flipped to make the lubricated surface the bottom side of the specimen, which is labelled as setup one lubricated bottom surface. In this configuration, flipping the mould after filled with RBC and before curing the specimen to simulate the lubricated bottom surface of the upper increment at the interface area of two RBC increments, as shown in Figure 5-14. At the bottom of this figure, the part with a white colour is imaginary increment only to explain the idea of both setups and why the mould was flipped in setup one. The glass slide was then removed from the top side, labelled as setup one non-lubricated top surface. A Mylar strip was kept in place in preparation to cure the specimen from the top side. The LCU tip was covered with a protective sleeve. Then it was held as close to the specimen as possible on the non-lubricated top surface, as shown in Figure 5-14, by using the prefabricated base, mechanical arm and reference points on the curing light to standardise the position of the light cure unit in relation to the mould, as illustrated in Figure 5-16. The material was polymerised for 40 s with an LED LCU. Then the Mylar strip and excess material were removed. The final specimen shape was as shown in Figure 5-11 (left).

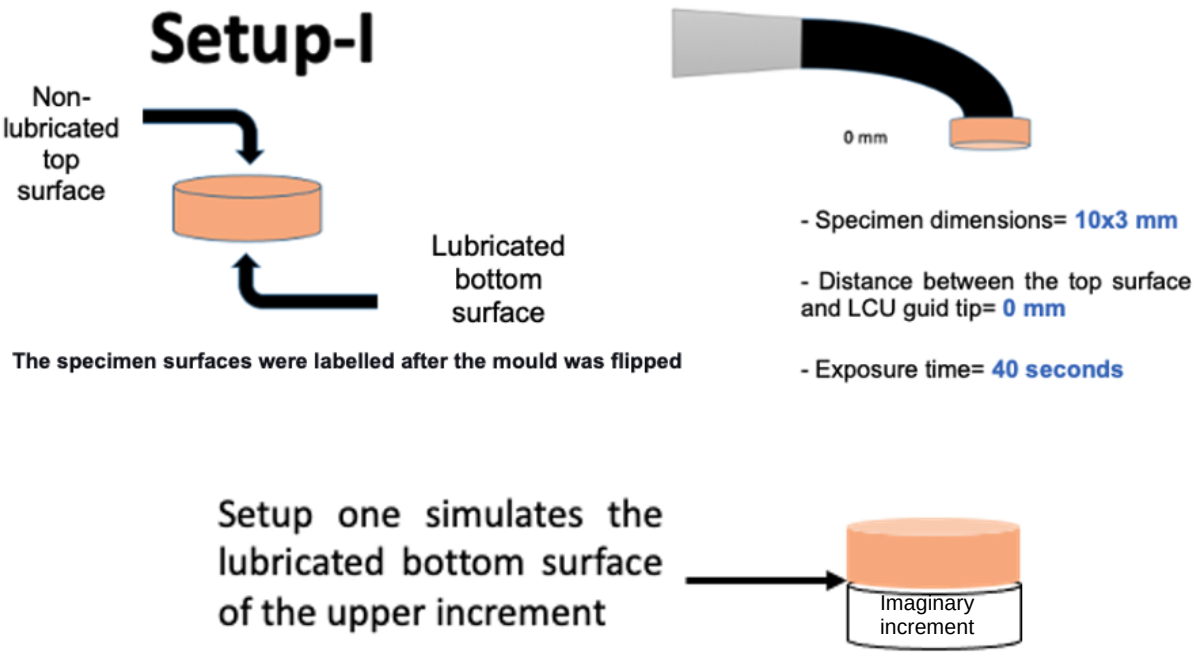


Figure 5-14: Setup one, showing the degree of conversion and Martens hardness tests on the specimen top non-lubricated surface and bottom lubricated surface

For setup two, the steps for placing the RBC materials in the mould were identical to setup one but the mould in setup two was not flipped. The treated top surface with ILs kept in position until the specimen cured. However, the curing light tip was adjusted to be 3 mm away from the specimen top surface to simulate the distance between the lubricated area and LCU tip when top increment placed in the real situation. This surface labelled as setup two lubricated top surface of the specimen as shown in Figure 5-15, using the mechanical arm shown in Figure 5-16 (right) to standardise the distance between the specimen and LCU tip. This setup simulated the top surface of the lower increment at the interface area between two increments as shown in the bottom part of Figure 5-15. The material was polymerised for 40 s with an LED LCU, and then the Mylar strip and excess material from around the specimen margins were removed.

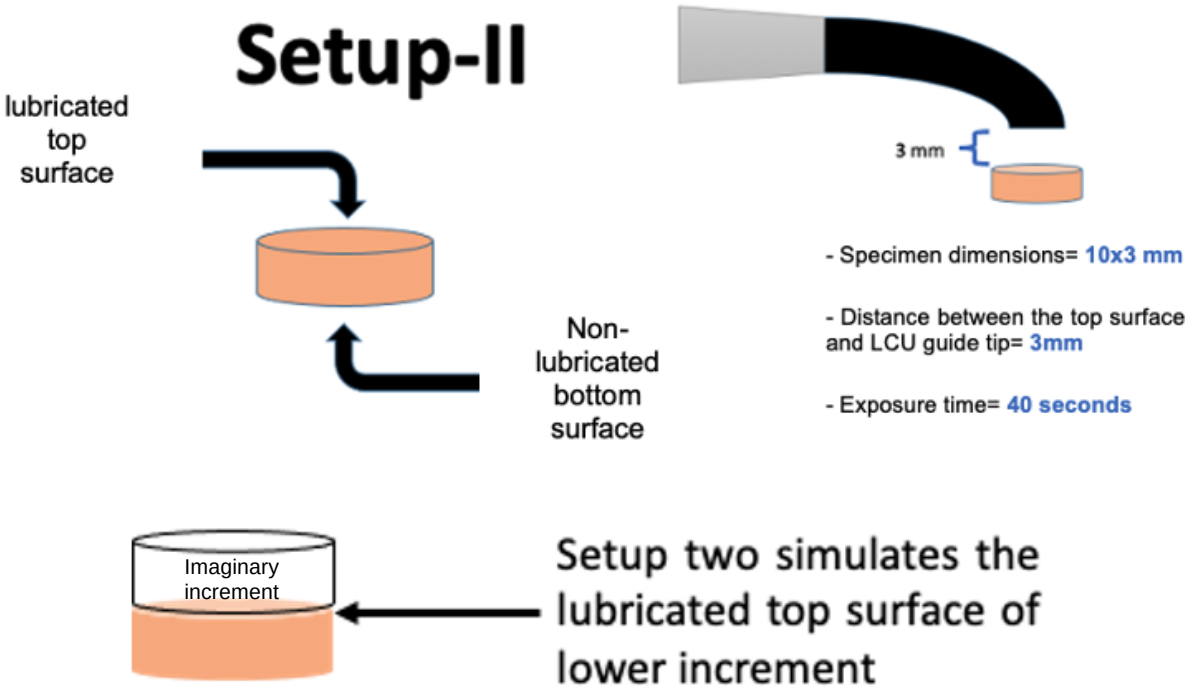
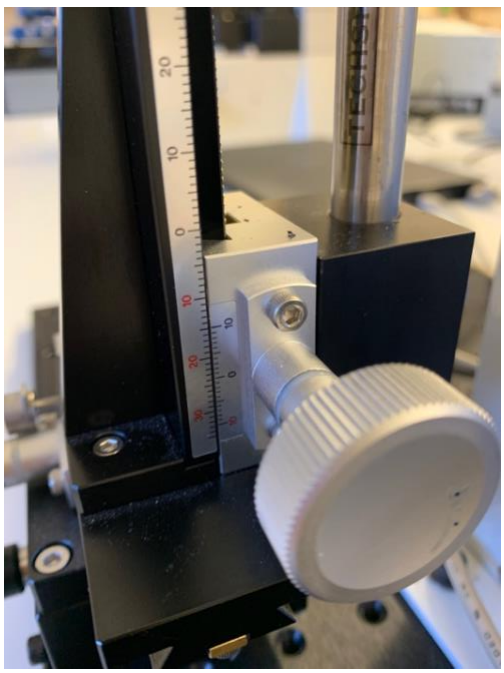
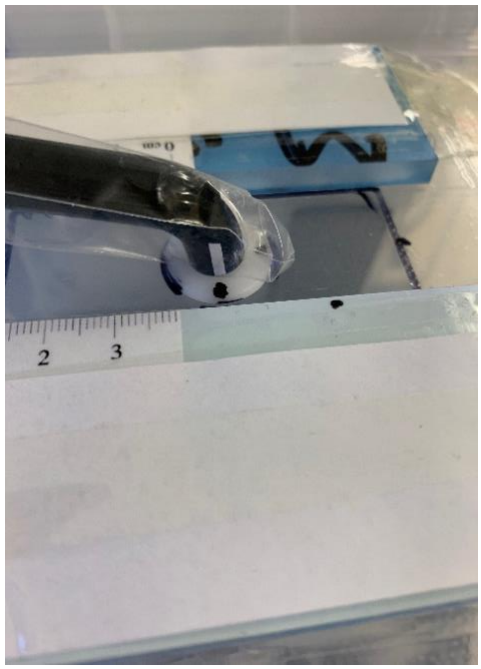


Figure 5-15: Setup two, showing the degree of conversion and Martens hardness tests on the specimen top lubricated surface and bottom non-lubricated surface



Left



Right

Figure 5-16: Right: The base fabricated to standardise the curing of the resin-based composite and the reference points on the mould, base and LCU tip to standerdise the position of the mould and LCU tip throughout all specimens; Left: The wheel used to adjust the height of the LCU tip in relation to specimen top surface

5.3.4 Observation of changes at increment interfaces due to ILs application

This experiment was designed to evaluate changes in the interface area of the RBC specimens due to the use of different ILs. A setup was, therefore, prepared to photograph the specimens within standardised measures (Canon E05, Tamron lens AF 90mm F/2.8, Macro Ø55 272E). A height adjustable tripod (Figure 5-17) was used to maintain the camera level in relation to the tested specimens, ensuring that the distance between the specimens and camera lens was equal across all time intervals. A professional photo light box was used (dimensions 40*40*40 cm, Heorryn, Yi ZHE - EU, Shang Hai, China) to control the amount of light reflecting on the specimen while the photos were taken. The box was equipped with 126 dimmable LED lights adjusted to a specific amount of light for all photo sets. A black background with slots was prepared to stabilise all the specimens in position (Figure 5-18).

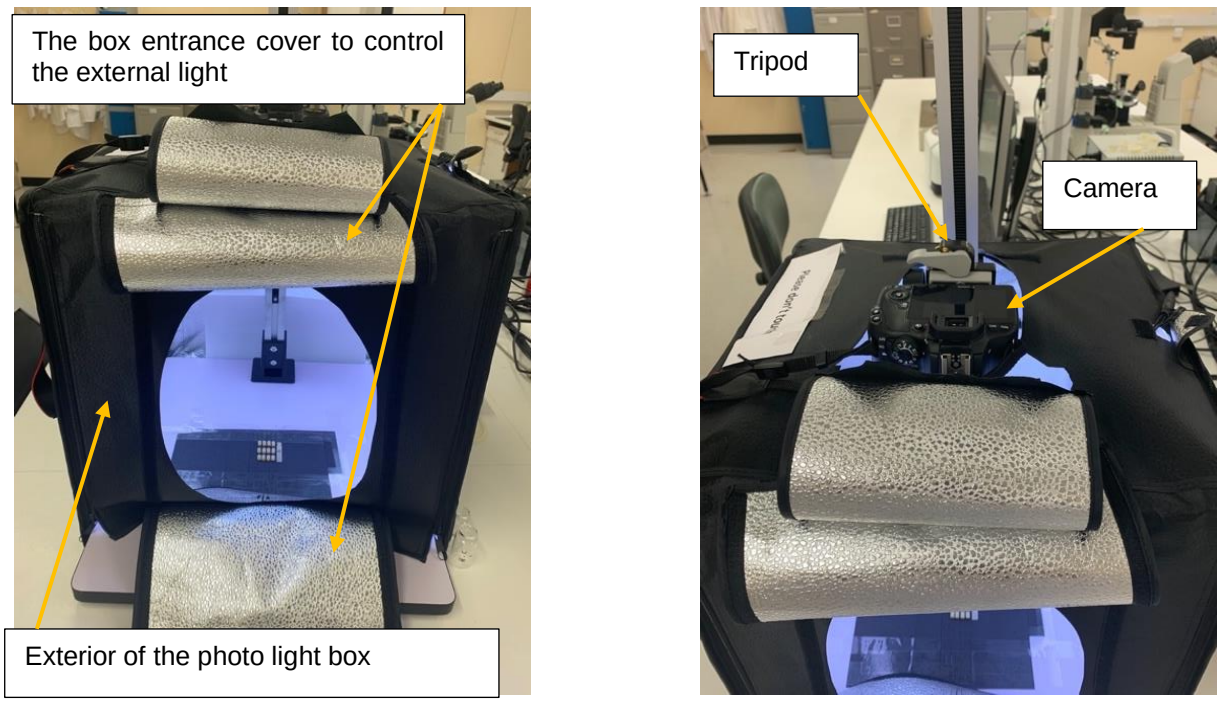


Figure 5-17: The exterior of the professional photo light box and tripod

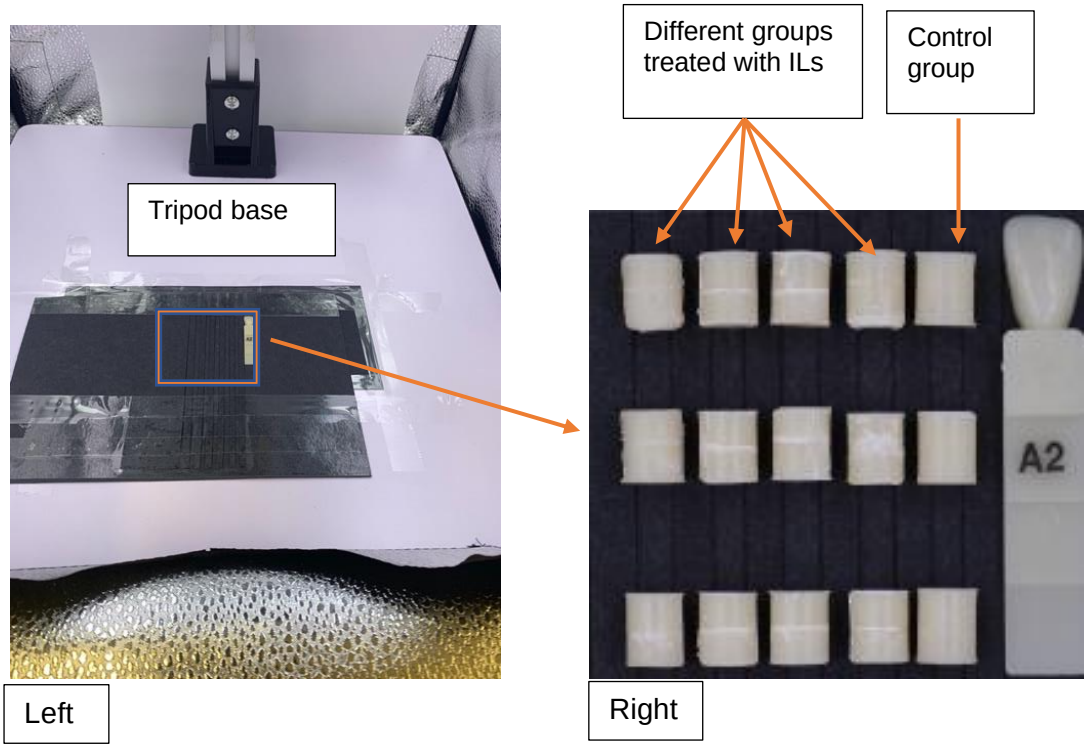


Figure 5-18: Left: Interior of the professional photo light box and tripod; Right: Arrangement of the groups of specimens in the photo light box

All the specimens treated with various ILs were stored in distilled water (DW) 10 mL after the baseline (without water storage) photos were taken in separate containers one for each specimen and placed in an incubator at 37±1 °C to simulate the temperature of the oral cavity. When the specimens were arranged in the box for photographing at each time interval, they were placed beside the control group for comparison. The RBC A2 colour shade guide (Filtek ™ Z250, 3M, USA) was placed next to the control specimens as an internal reference. Three specimens in each group were aligned in a column and the other groups for each IL class were placed in the same way next to the control group (Figure 5-18). The camera's default settings were applied, and the camera set to auto throughout the experiment. Photos were taken at different time intervals: baseline, day 1, 7, 14, 28 and 60.

After all the photo-sets had been taken, the colour changes at interface area on the specimens were compared and analysed, especially with regard to the interface between the two increment levels as this was the area where the ILs were applied. The

optical density of two areas of each specimen was measured for comparison with the controls. The area, place and size were standardised on each surface strip (Figure 5-19) using a tool in ImageJ software (1.52q, Wayne Rasband, National institutes of Health, USA). The first strip was in the middle of the specimen at the level of the interface of the increments, while the second was on the same surface but higher in position and away from the interface area.

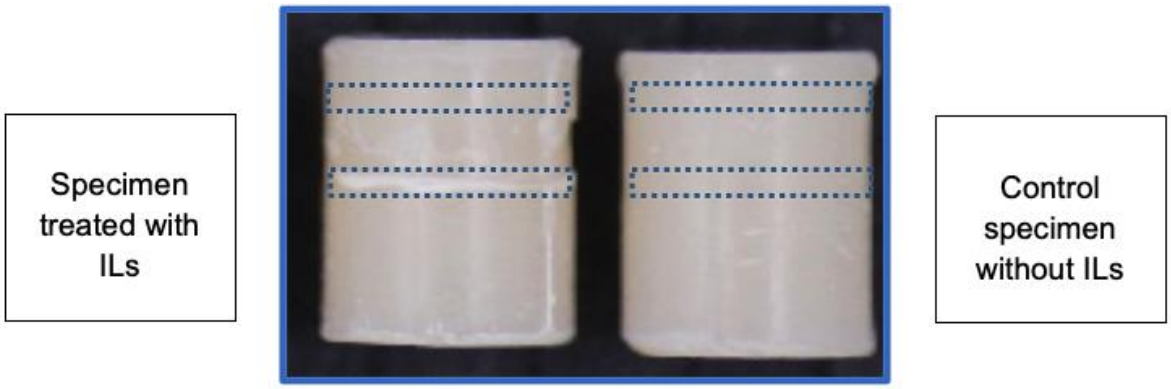


Figure 5-19: Location of the tested strips on the surface of the experimental and control specimens

The optical density of each specimen group was analysed digitally using software calibrated to 8-bit greyscale to use this scale, and to measure the mean number of grey pixels. The calculated mean represented the sum of the grey values of all the pixels in the selected strips divided by the number of pixels and reported in calibrated units (optical density). This mean of greyscale values was measured within a standardised strip marked on each specimen using the macro tool within the software. This tool determines a specific area, as shown in Figure 5-19. The dimensions of the area were within 100 x 15 width and length and were defined by the number of pixels. The shape and size were saved so that they could be applied to all the specimens for each time interval tested. All the values of the middle and top strips on the same surface were collected. The ratios of both strips were calculated (Microsoft Excel

version 16.43.1) by dividing the value of the middle strip by value of the top strip for each specimen.

5.3.5 Degree of conversion test

Five specimens in this experiment were evaluated immediately after preparation and after 24 hours dry storage at 37°C (GALLENKAMP Hot Box Oven with Fan - Size 1, UK) to make sure the most of monomers have chance to convert to polymers. Each specimen was stored in a bottle (Cole-Parmer, Neots, UK), and all bottles were placed in a lightproof container covered with aluminium foil. One scan was performed for each surface of each specimen for both setups one and two. The ATR- FTIR Spectroscopy (PerkinElmer, Inc., Waltham, MA 02451 USA) was used to perform the scan, which was also preceded by a background scan to prepare the device for the actual scanning. The resolution was 4 cm^{-1} and the accumulation was adjusted to 16 scans. The mid infra-red (MIR) region was used and the range was 4000–550cm^{-1}. The surface of the specimens was placed in direct, centred contact with the diamond crystal, and the swivel pressure clamp was located on the specimen's other surface. This helped apply the maximum pressure level allowed by the instrument until the ATR-FTIR started to detect the specimen surface and to give the desired curve shape. After this, scans were performed once for the lubricated and non-lubricated surfaces to collect the DC value from each surface. At the end of the scanning stage, each scanned curve was processed by baseline correction, and the data saved and exported to Excel in preparation for the next stage of data processing. The FTIR scans for uncured and cured specimens of all tested RBCs groups of setups one and two were collected. The aromatic and aliphatic peaks were used to calculate the DC as shown in Figure 2-18 in section 2.9.1. Then the percentage of the DC was calculated by using the equation (Eq.1) that was mentioned in section 2.9.1 to calculate the DC of all groups.

5.3.6 Martens hardness test

Both the lubricated and non-lubricated surfaces of setups one and two as shown in Figure 5-14 and Figure 5-15 in section 5.3.3 were evaluated for hardness by taking three readings on the same surface of the same specimen. After the specimens were cured and the Mylar strips removed, they were stored for 24 h in dry storage in an incubator at 37±1 °C as that was explained in section 5.3.2. Each specimen was stored in a bottle (Cole-Parmer, Neots, UK), and all bottles placed in a lightproof container covered with aluminium foil. The surface of the specimen to be tested was placed facing the indenter of the hardness machine (Zwick Roell, Herefordshire, UK), and the specimen surfaces were flat on the supporting stage of the machine. The test was set at 200 g load and this was maintained on the surface for 20 sec once the maximum load was reached. The load was reset to zero before taking a new reading and this was repeated three times for each surface. The data were collected and saved to be analysed.

5.3.7 Water uptake test

All specimens, six specimens for each group were weighed on an electric analytical balance (Mettler AE 240, Leicester, UK), and were in line with the expected readability of 0.01 mg with ±0.03 mg variability. They were then transferred to the desiccator and maintained at 37±1 °C. After 24 h, they were weighed again and this cycle was repeated until a constant mass was obtained, i.e. until the mass loss of each specimen was not more than ± 0.1 mg in each 24 h period. The specimens were evaluated at different experimental time intervals (baseline, one day, one week, one month and three months), and stored in DW. Every specimen was placed separately into a small glass storage bottle with a sealed, stoppered lid containing 10 mL of DW to make sure that the specimens covered totally with DW and to adapt the ISO 4049 immersion

steps, and then stored in an incubator at 37±1 °C, to replicate the temperature of the oral cavity environment. At the time of measuring, the specimen was removed with tweezers from the individual storage bottles and allowed to drip on a rack for a minute until visibly dry to remove most of the excess water on the surface. The specimens were then held with the tweezers and waved gently in the air for 15 sec to remove excess water prior to weighing. After the specimen had been weighed, it was replaced in the bottle with a fresh 10 ml of DW. All the specimens were returned to the incubator until the next evaluation period. The amount of water uptake for each specimen was evaluated gravimetrically, with all the specimens being weighed at the baseline and again after immersion in DW using the electrical analytical balance. The percentage of weight change over time was then calculated for each specimen by using the following equation:

$$\text{percentage of weight change} = \left(\frac{\text{Weight after immersion in DW} - \text{Weight before immersion in DW}}{\text{Weight before immersion in DW}}\right) \times 100 \quad (\text{Eq. 2})$$

5.3.8 Diametral tensile strength test

DTS testing was performed by using a universal test machine (model 5567, Instron, High Wycombe, UK), at baseline and again after immersion in 10 ml DW for one day, one week, one month and three months. Specimens were stored in the incubator at 37±1 °C, and every specimen was placed in a storage bottle. All bottles were stored in a lightproof container covered with aluminium foil. Each specimen was taken from the storage bottle with a tweezer and placed on the table until it was visibly dry. The specimen was waved in the air for 15 sec to ensure all excess water was removed. The cylindrical specimen's dimensions were measured and entered into the DTS test software, before being tested under a compressive load of 5kN at a crosshead speed

of 1 mm/min. The tested specimens were placed on their side to direct the applied compressive load perpendicularly to the specimen's longitudinal axis, until the point at which the specimen fractured, as illustrated in Figure 5-20. This applied load was measured in Newtons, while the DTS was calculated in MPa using the following equation:

$$DTS = 2F/\pi dh \qquad \text{(Eq.3)}$$

Where, π = 3.1416 was a constant, d = specimen diameter and h= specimen height. Data were saved and exported to Excel for the next stage of data processing. All steps were repeated for all the samples being tested.

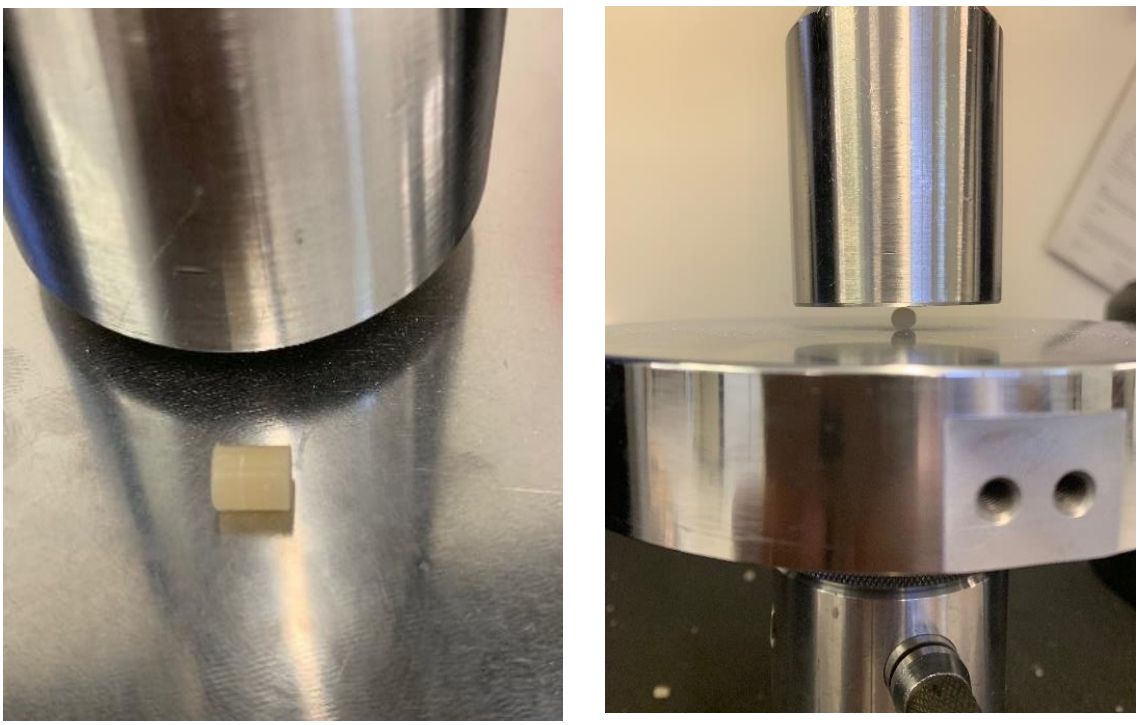


Figure 5-20: Position of the DTS specimens under compression

5.3.9 Statistical analysis

All values obtained and the relation between the groups was analysed via descriptive statistics. The ANCOVA-Multivariate test (SPSS 25 for windows, IBM SPSS Inc., USA) was used to identify statistically significant differences. A *p* value of 5% or lower was taken to be significant between the controls and experimental groups treated with instrument lubricants at different time intervals. Pairwise comparisons were made using Tukey to control the overall significance level at 5%.

5.4 Results

5.4.1 Depth of cure for Filtek and Harmonize RBCs

The DoC test results showed that the mean and standard deviation of the Filtek RBC height was 6.08 mm (SD=0.29) and the DoC 3.04 mm. The mean and standard deviation of the Harmonize RBC height was 5.98 mm (SD=0.17) and the DoC 2.99 mm. The t-test (*p*=0.176) of both tested RBCs showed no significant difference.

5.4.2 Specimen changes observation at increment interface area

All experimental groups of different ILs classes were analysed visually and by calculating the ratio of the optical density (OD) across the experimental time intervals. Statistical analysis was performed, and the mean of the OD and standard deviation values of all time intervals were calculated. In comparison to the control group of two RBCs, certain groups treated with ILs showed changes in RBC colour, with an opaque strip being formed at the level of interface area between the two increments of the specimen. Although the effects of ILs were varied between the experimental groups, there seemed to be almost the same pattern for each IL class in both tested RBCs, as presented in Figures 5-21 and 5-22. The abbreviations of the substances for both figures are explained in Table 5-6.

Keys	Words	Keys	Words
F	Filtek	**E**	Ethanol
H	Harmonize	**I**	Isopropyl
C	Control	**DW**	Distilled Water
A	Acetone	**S**	Scotchbond
O	Optibond	**B**	Brush & Sculpt
M	Modelling Resin	**SL**	Signum Liquid
BA	Bonding Agents	**WR**	Wetting Resins

Table 5-6: Key to abbreviations used in the photo observation sets

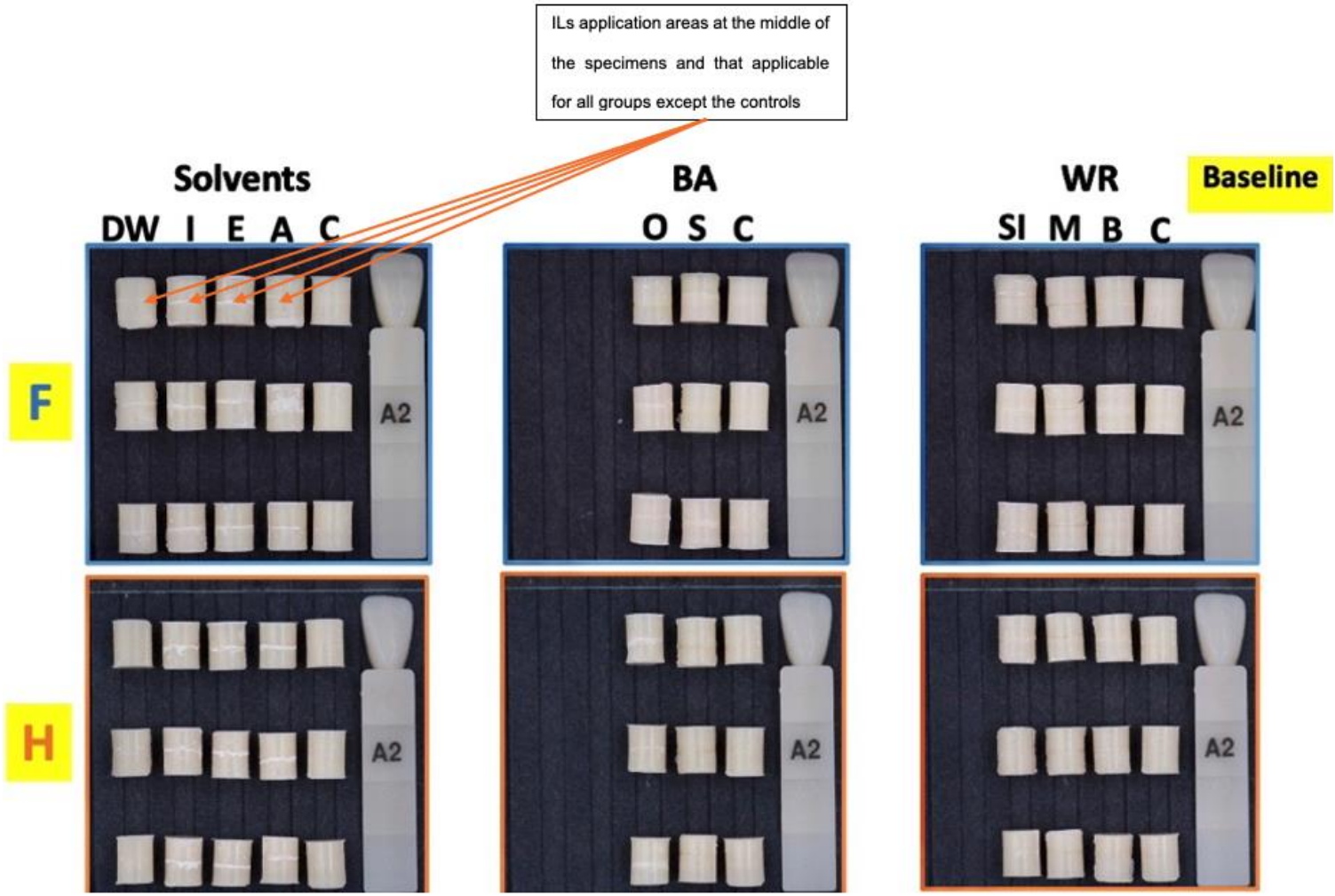


Figure 5-21: All experimental groups, showing the surface changes at baseline

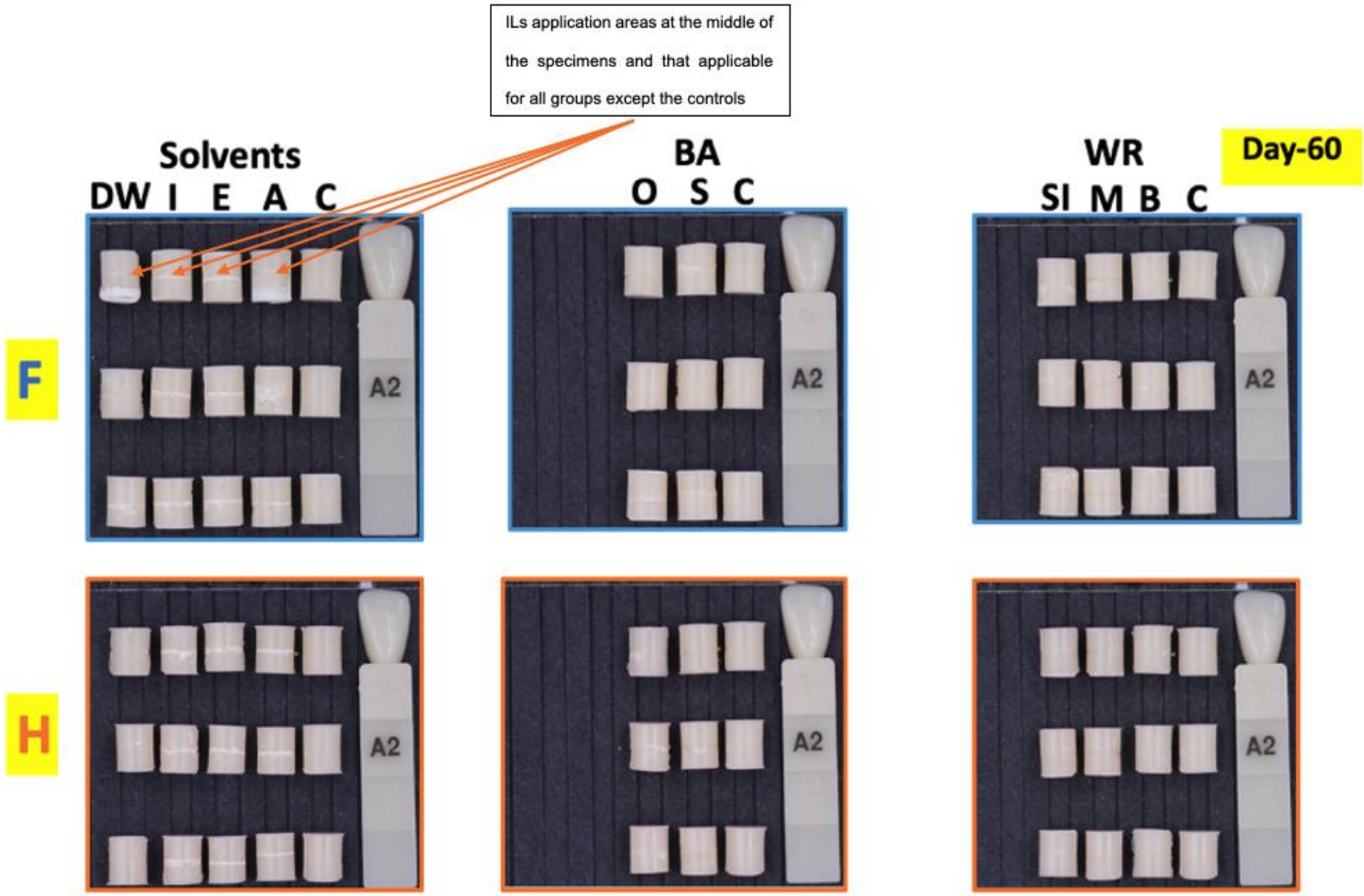


Figure 5-22: All experimental groups, showing the surface changes after 60 days

Regarding the solvents, visual observations identified surface changes in the interface area between the increments. Almost all solvent groups were affected by the use of ILs on both RBC types in comparison to the control. The distilled water and Filtek group had a mild change but not as much as other organic solvents, and with Harmonize there were no noticeable effects as shown in Figure 5-23.

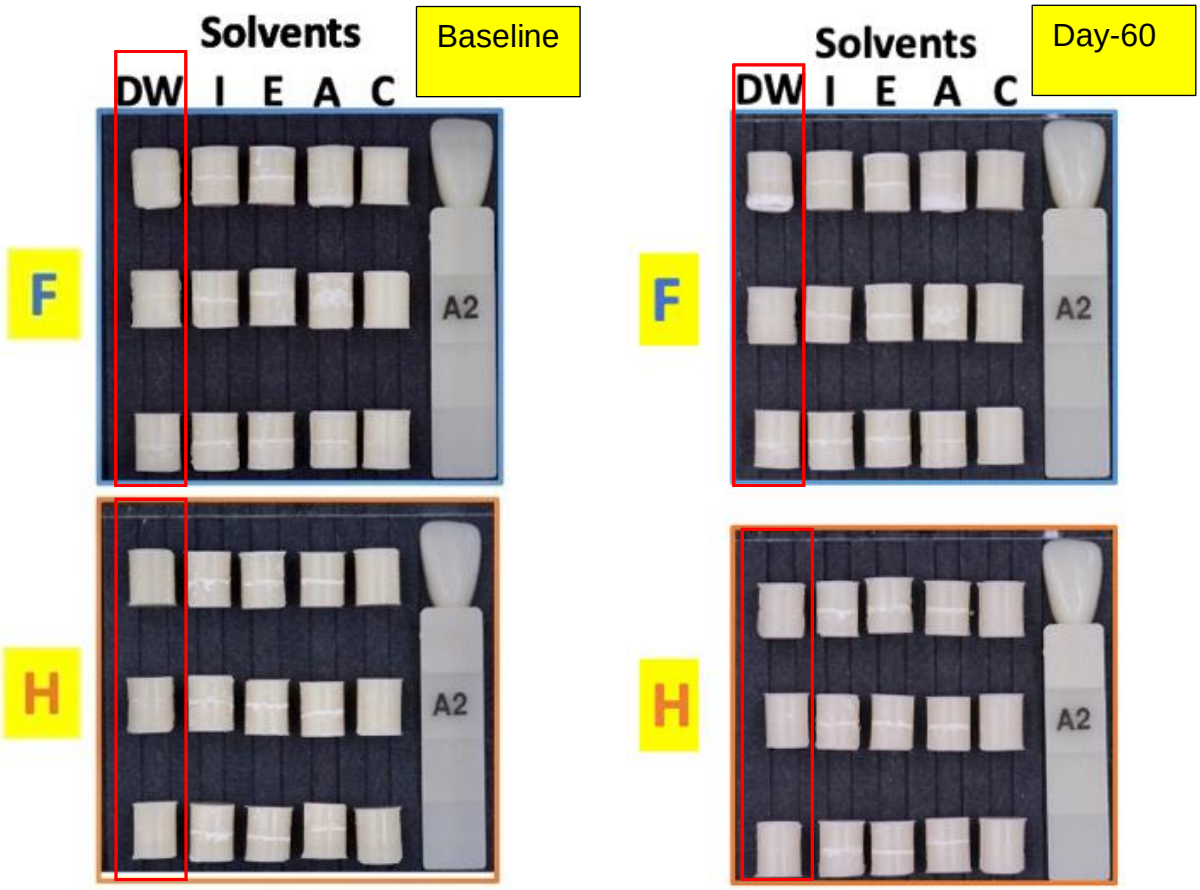


Figure 5-23: The solvent groups that have shown most effects throughout all time intervals of both RBCs. The DW groups were less effected in comparison to the other groups

Values for OD fluctuated across the experimental time intervals and then became stable after 28 days. The OD of the Filtek groups had statistically significant differences across the solvent groups (p<.001). Ethanol and isopropyl had the highest effects, and then acetone, with DW having the least effect, as seen in Figure 5-24. These findings match the visual observations for these groups, although the Harmonize RBC solvents

class showed differences between the experimental groups (p<.001). The effects of isopropyl, acetone and ethanol were statistically significantly different in comparison to the control group. However, the DW showed no differences compared to the control. These results are summarised in Figure 5-25

For the bonding agent experimental groups, Filtek showed no significant visual changes to specimen surfaces at the interface between the increments. Compared with the solvents groups, opaque lines did not form as much during the experimental time intervals. Harmonize was no different with Scotchbond but some slight opaque lines could be observed with the Optibond group at baseline. These lines diminished after a while during the experimental time intervals. Most of the changes occurred in the beginning and then became steady after 28 days. Both bonding agents showed no statistically significant differences (Filtek, p=0.544; Harmonize, p=0.062) across all experimental time intervals, as can be seen in Figures 5-26 and 5-27

Regarding the wetting agents, visual observation of both RBC types showed no major changes in comparison to the control group, and no prominent opaque lines appeared as in the previous classes. Some samples developed a transparent strip within the colour shade of the RBCs but were not significant. All changes fluctuated across the experimental time intervals but became steady after 28 days. Also, neither Filtek (p=0.132) nor Harmonize (p=0.542) showed statistically significant differences for all the ILs in the wetting agent experimental groups, as shown in Figures 5-28 and 5-29.

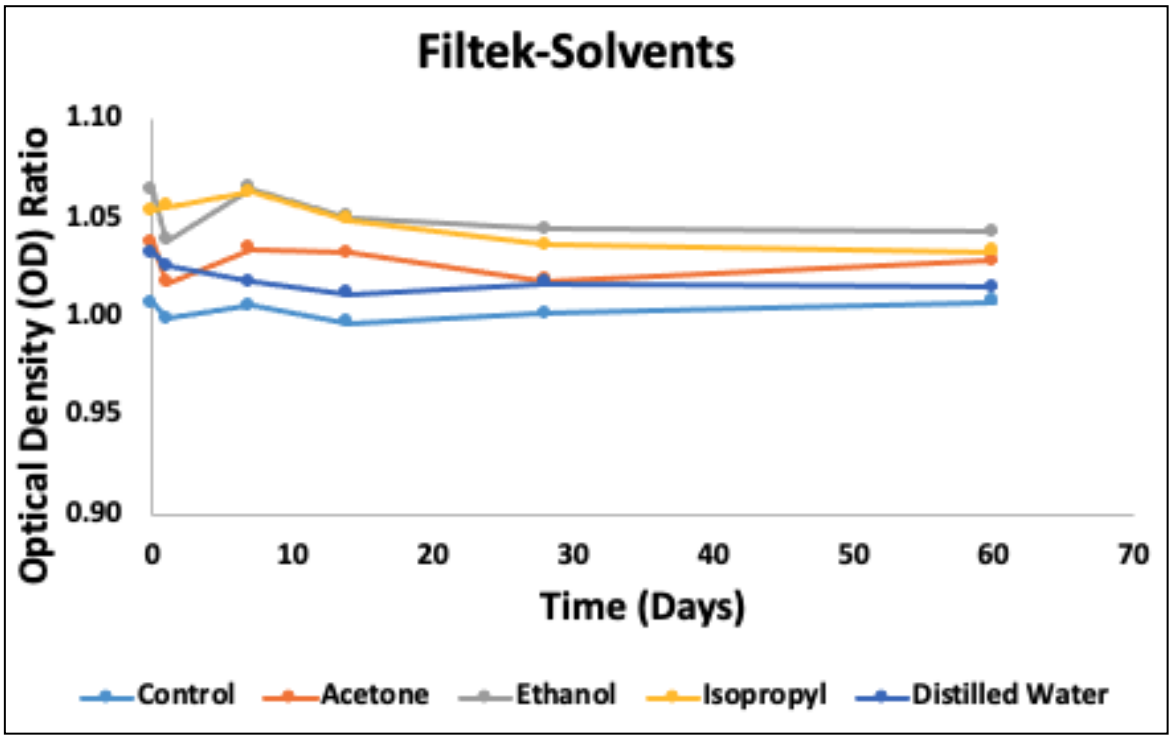


Figure 5-24: Optical density ratio of solvents over time for Filtek

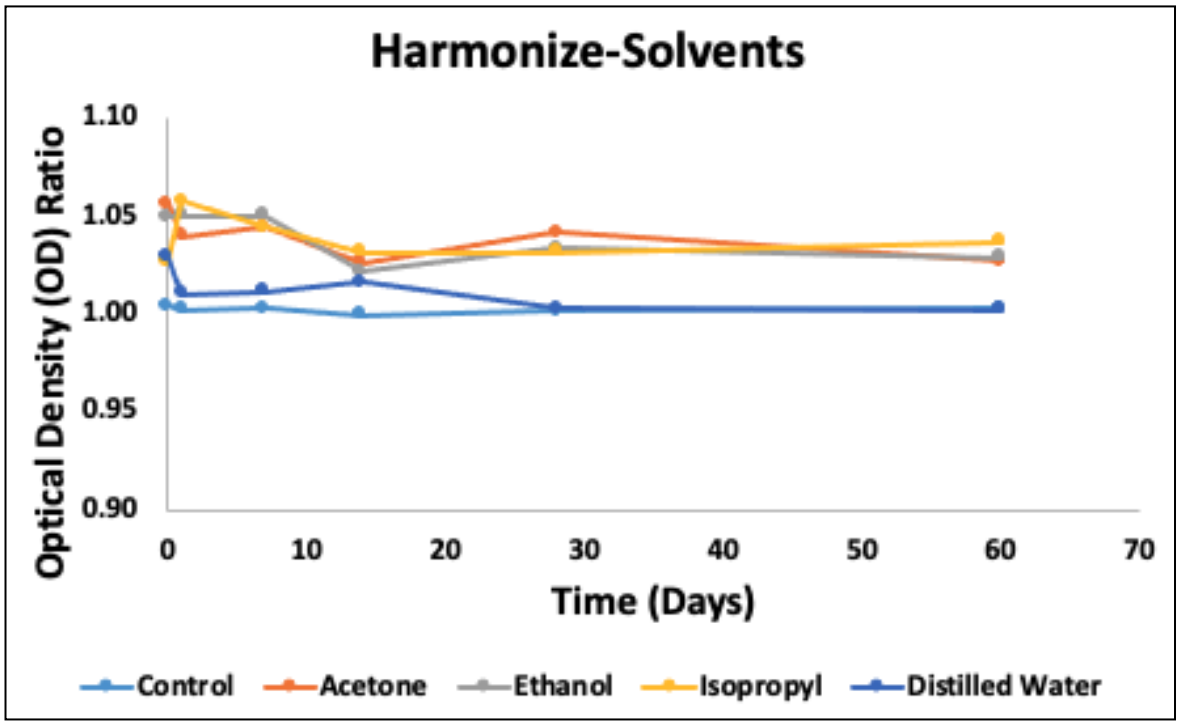


Figure 5-25: Optical density ratio of solvents over time for Harmonize

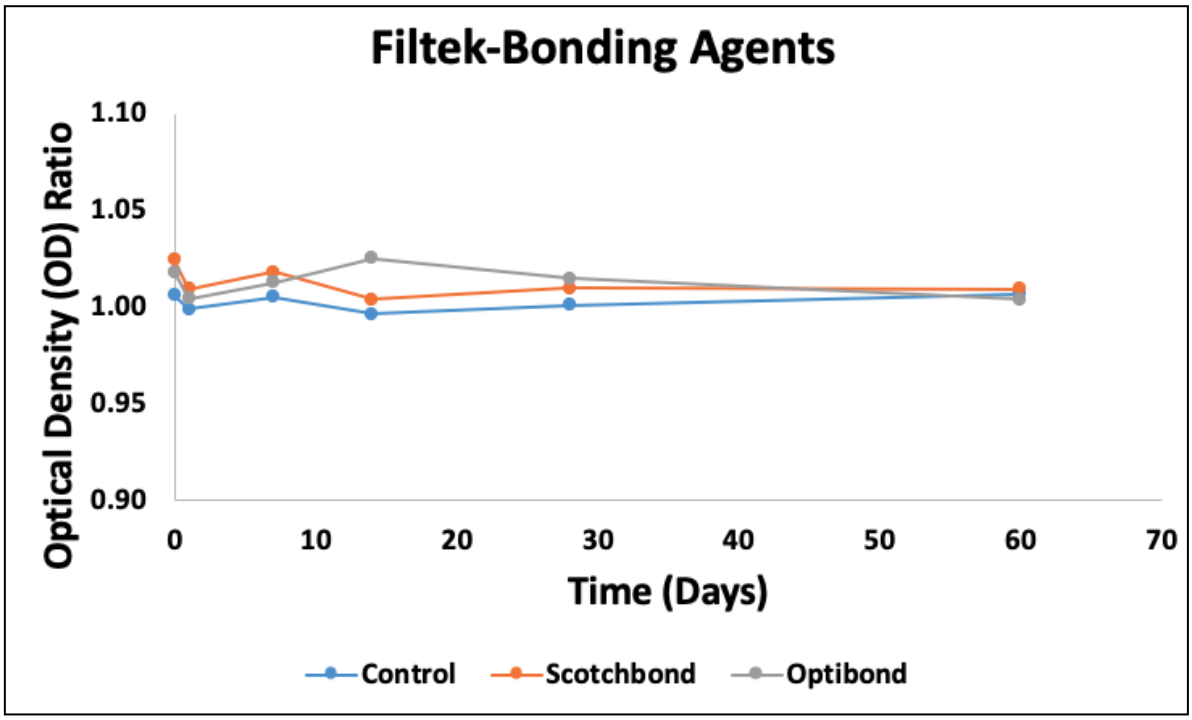


Figure 5-26: Optical density ratio of the bonding agents over time for Filtek

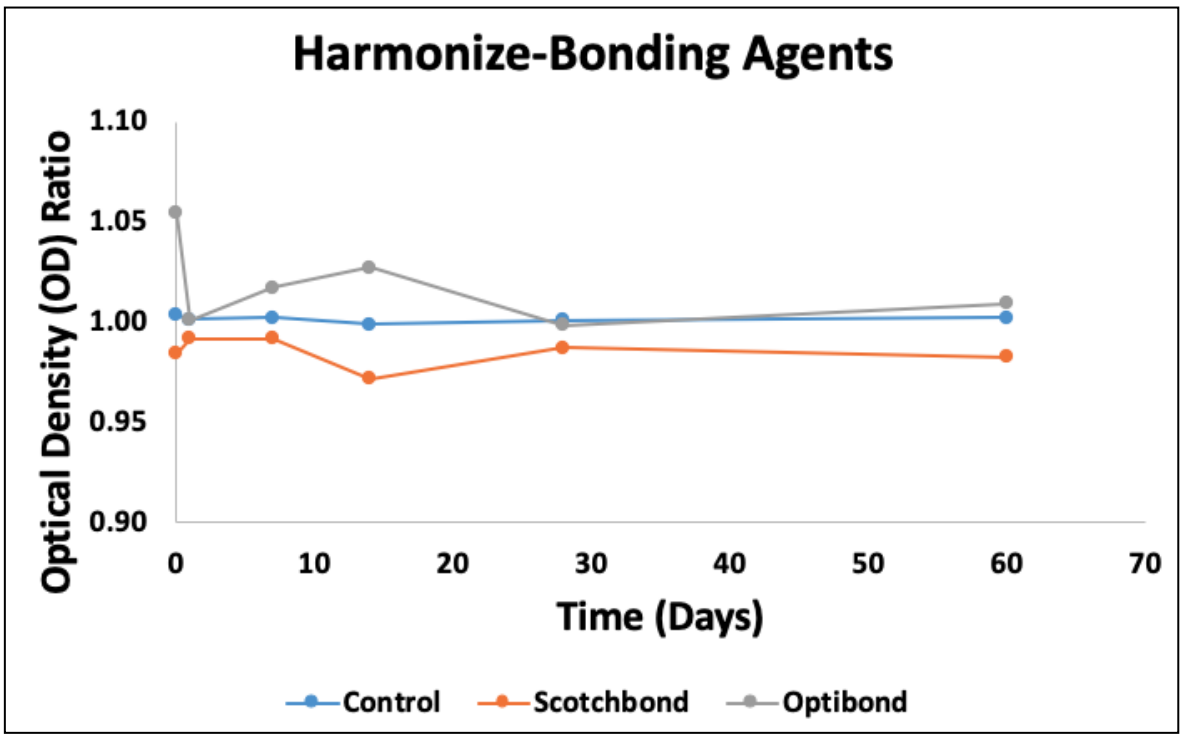


Figure 5-27: Optical density ratio of bonding agents over time for Harmonize

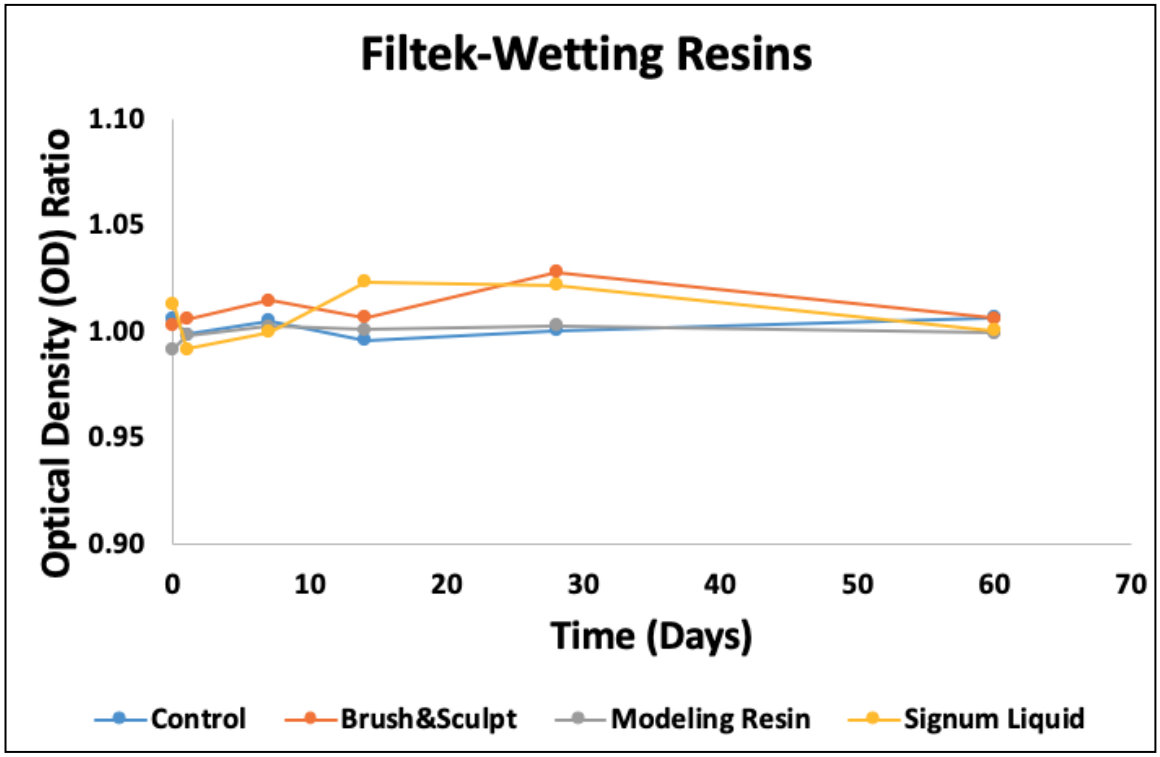


Figure 5-28: Optical density ratio of wetting resins over time for Filtek and Harmonize

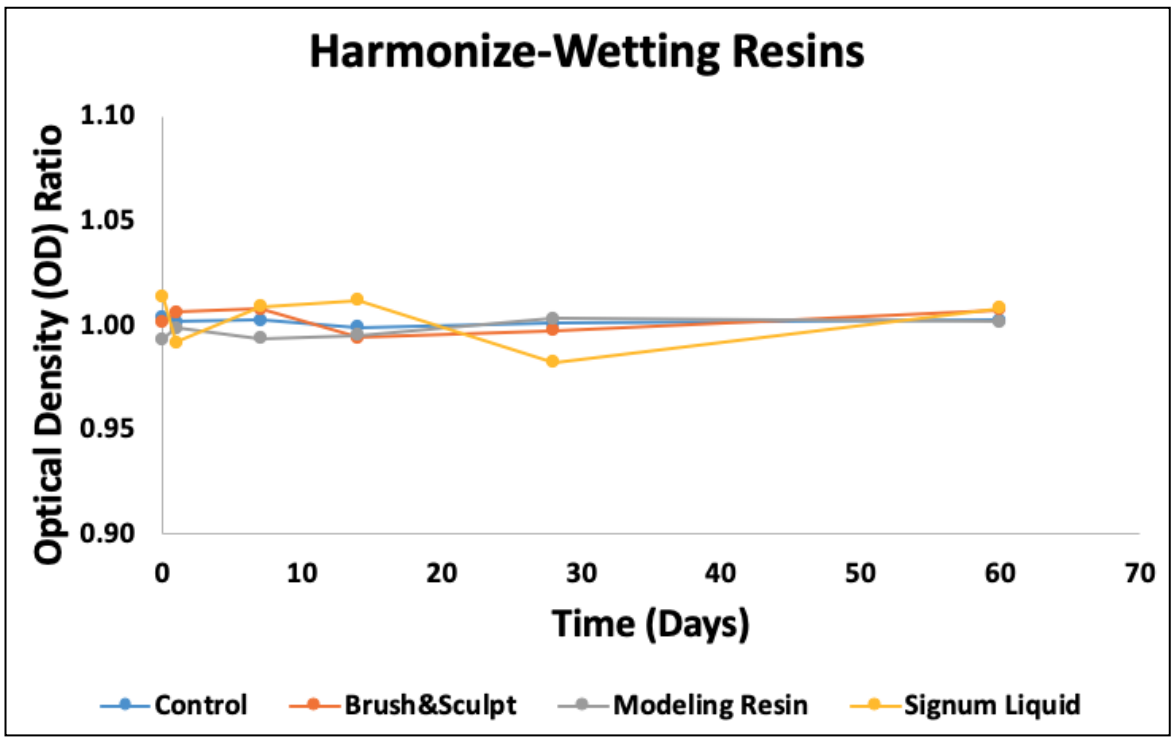


Figure 5-29: Optical density ratio of wetting resins over time for Filtek and Harmonize

Classes	ILs groups	Filtek	Harmonize
Solvents	Acetone	(p<.001) *	(p=0.03) *
	Ethanol	(p<.001) *	(p<001) *
	Isopropyl	(p<.001) *	(p<001) *
	Distilled water	(p=0.02) *	NS
BA	Scotchbond	NS	NS
	Optibond	NS	NS
WR	Brush & Sculpt	NS	NS
	Modelling Resin	NS	NS
	Signum Liquid	NS	NS

Table 5-7: Significant differences in optical density between the experimental and control groups

* Statistically significant different *p*-values in comparison to the control and (NS) means not significant.

5.4.3 Degree of conversion

The plots of DC for the tested groups collected and presented in Figure 5-30 and Figure 5-31. The mean DC of the lubricated and non-lubricated surfaces for setup one and two and standard deviation values for baseline and day one were calculated. Regarding the solvents used in setup one, no differences were detected on the non-lubricated top surface for all IL classes for both types of RBC. The results of setup one show that there were significant differences, specifically on lubricated bottom surface that was treated with different solvents (p<.001). For Filtek, distilled water was the only solvent that significantly reduced (p=0.02) the DC in comparison to the control group. However, on the lubricated bottom surface of Harmonize, none of the solvents reduced the DC significantly; some, such as the ethanol and isopropyl, increased the DC on this surface compared to the control. The results of this class of ILs show the solvents negatively affected Filtek on the lubricated bottom surface but did not affect Harmonize on the same surface, as indicated in Figure 5-32

Regarding the bonding agents in setup one, the results for this group revealed that both bonding agents did not negatively affect the DC for either RBCs on the lubricated bottom surface. These bonding agents increased the DC on surface when it was placed, as summarised in Figure 5-33.

The test groups for wetting resins in setup one, the lubricated bottom surface of Filtek showed a significant difference (p<.001). Signum Liquid was the only wetting resin that significantly reduced the DC of Filtek, while the other IL increased the DC on this surface. For Harmonize, there were no significant negative effects on the lubricated bottom surface with any wetting resin, as all these increased the DC on this surface in

comparison to the control. Within the wetting resin experimental group, only Filtek was negatively affected by wetting resins class, as shown in Figure 5-34

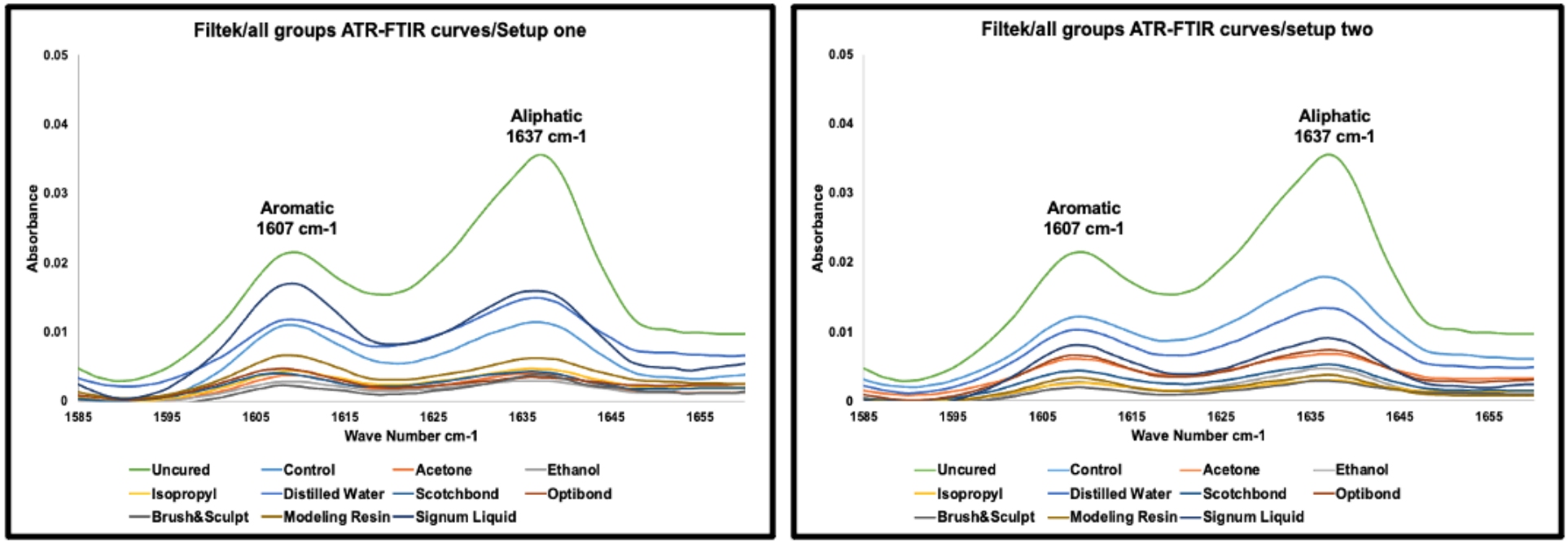


Figure 5-30: The FTIR scans for uncured and cured specimens of all tested Filtek RBC groups for setup one and two. Also, the aromatic and aliphatic peaks that were used to calculate the DC

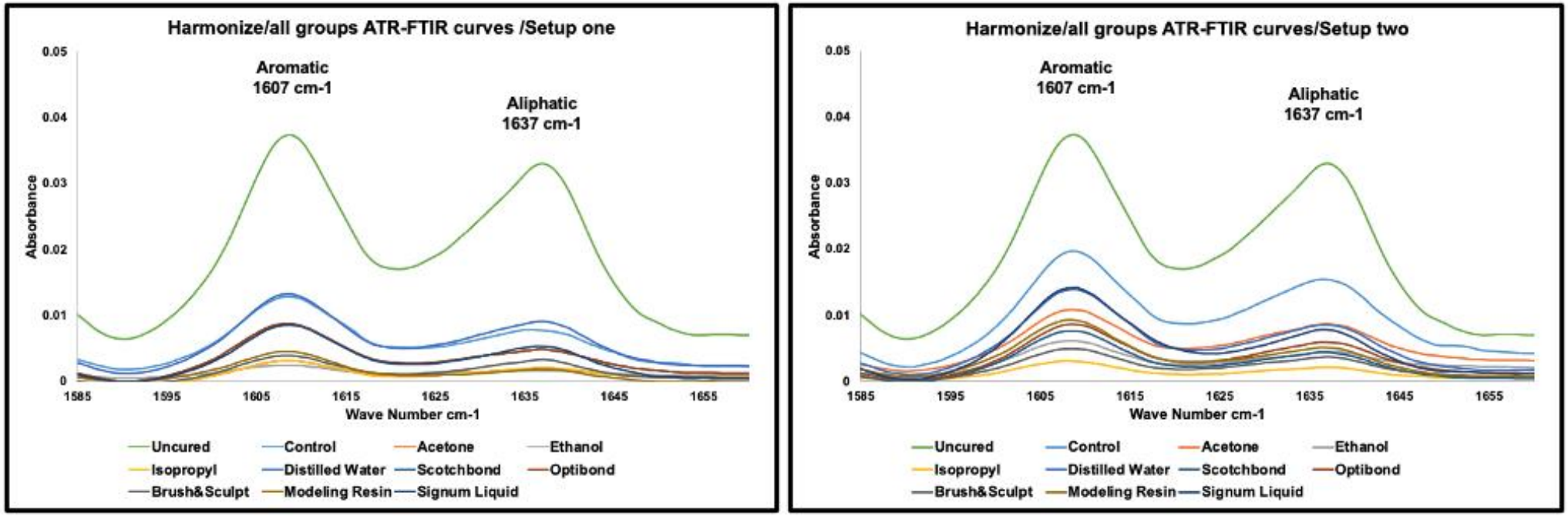


Figure 5-31: The FTIR scans for uncured and cured specimens of all tested Harmonize RBC groups for setup one and two. Also, the aromatic and aliphatic peaks that were used to calculate the DC.

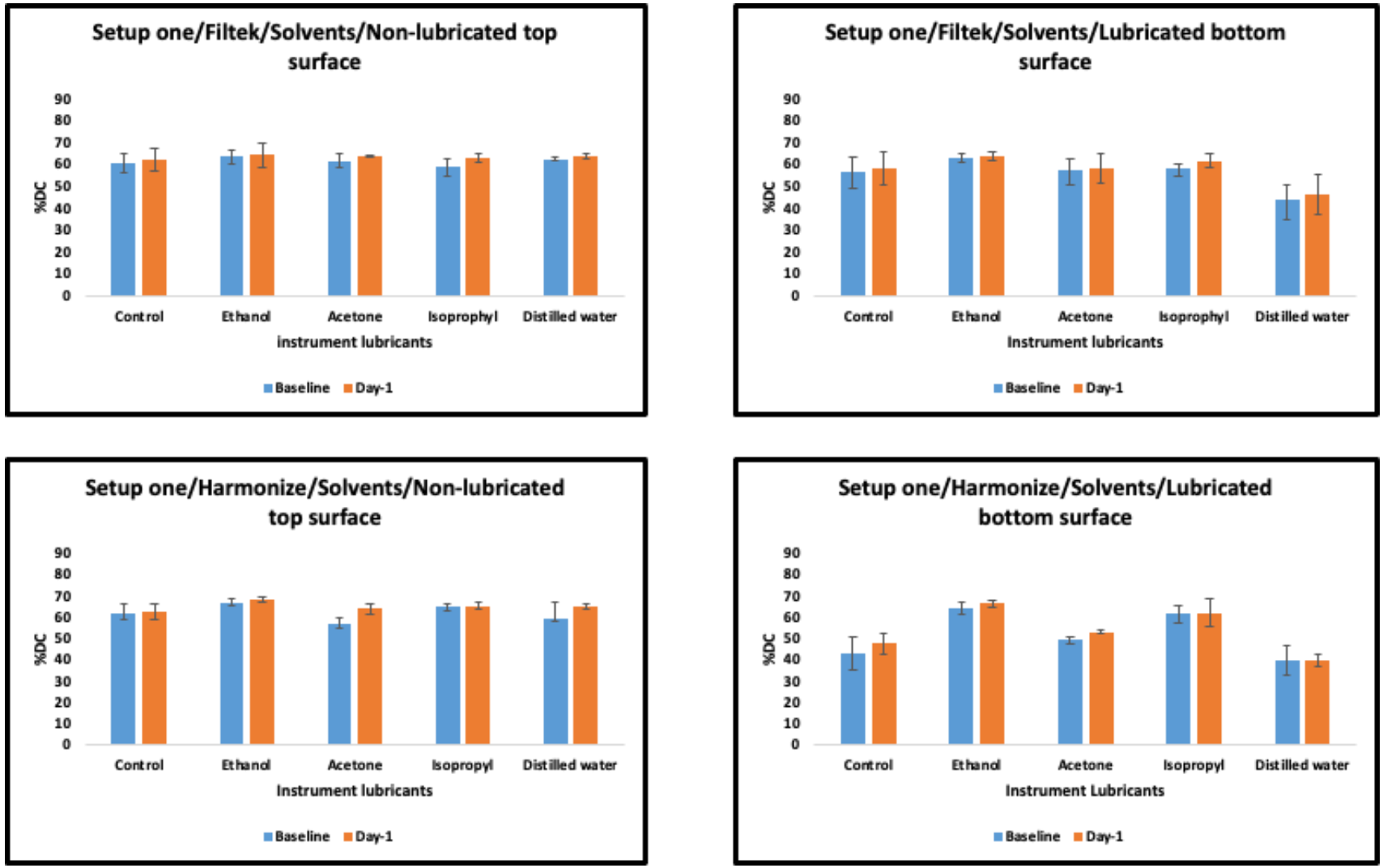


Figure 5-32: Mean and standard deviation values for the degree of conversion for Filtek and Harmonize for non-lubricated top surface and solvents-lubricated bottom surface/setup one

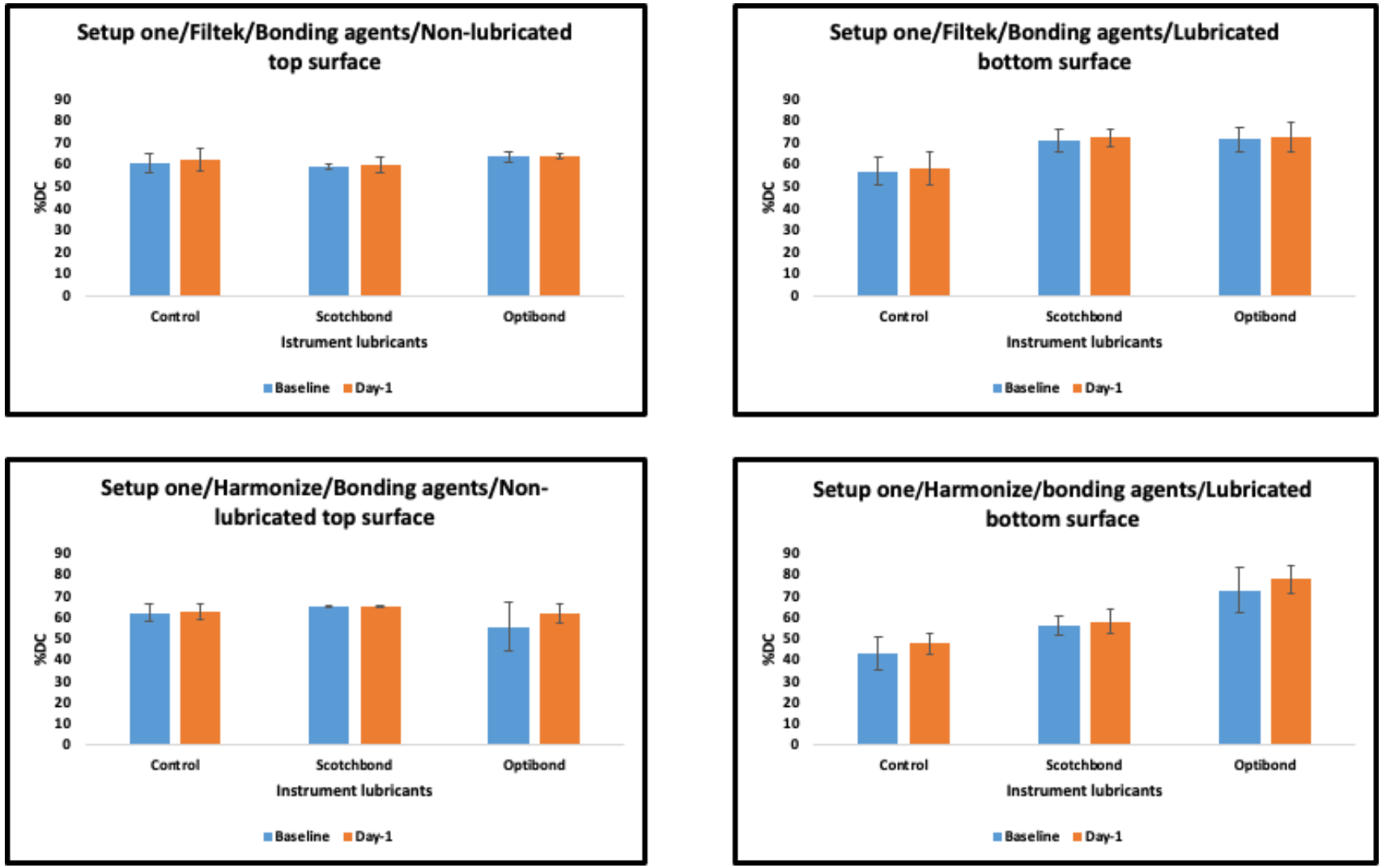
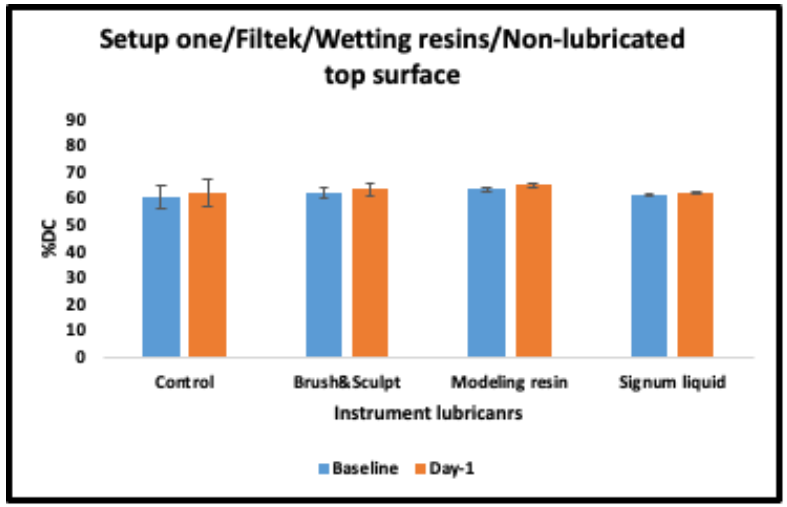
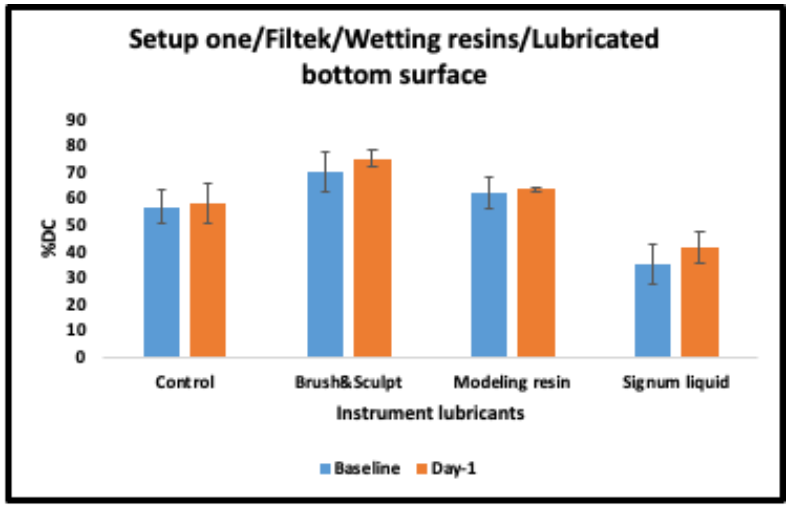
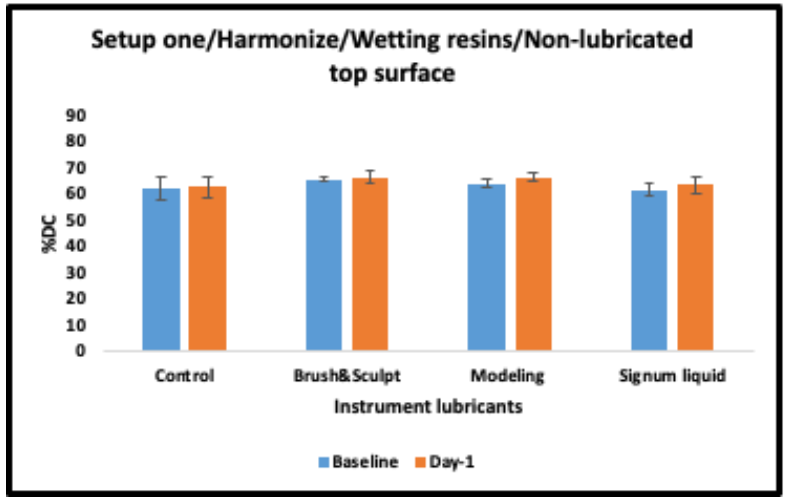


Figure 5-33: Mean and standard deviation values for the degree of conversion for Filtek and Harmonize for non-lubricated top surface and bonding agents-lubricated bottom surface/setup one







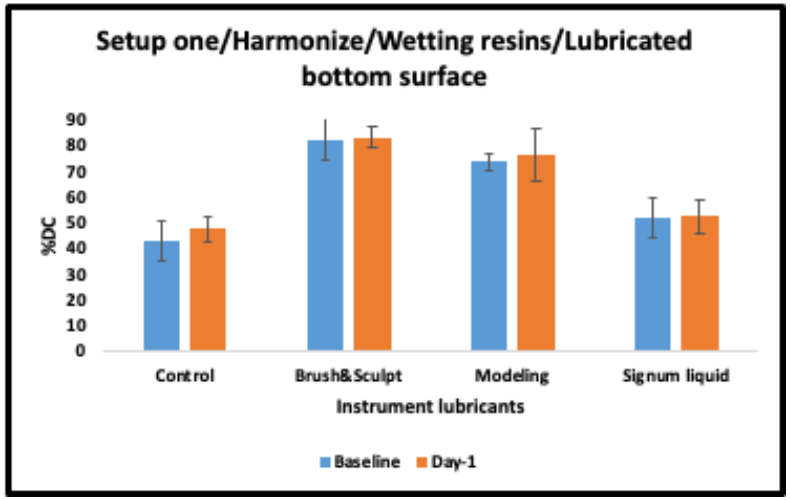
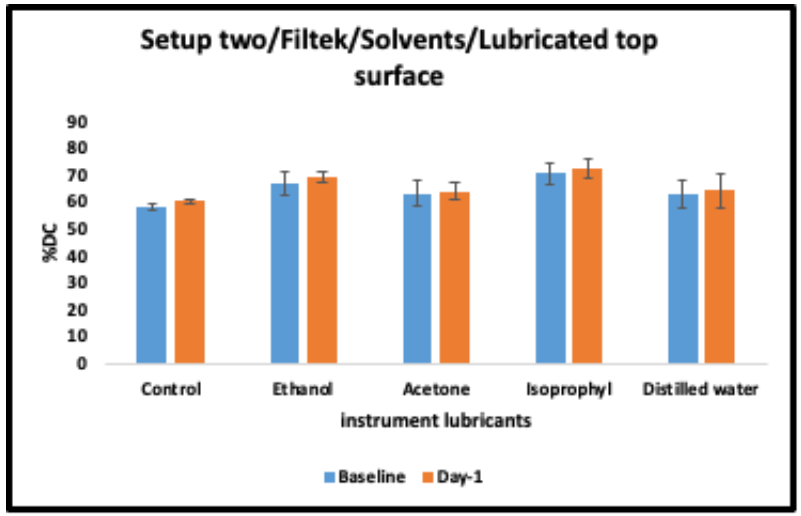


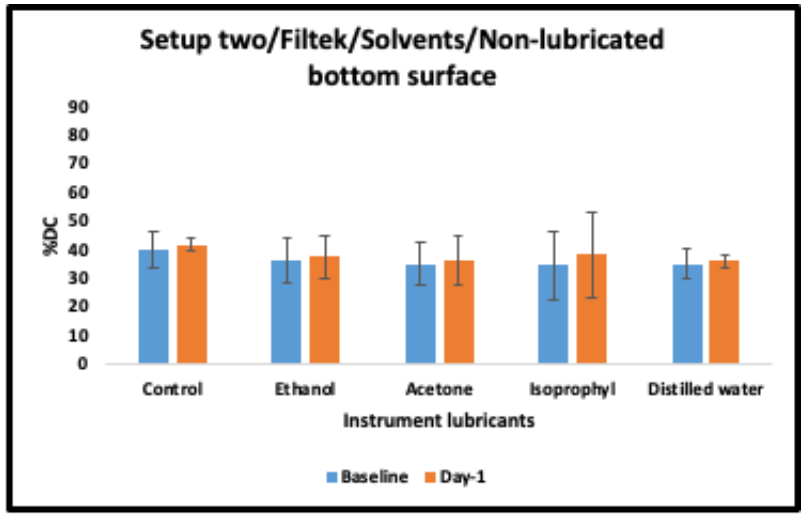
Figure 5-34: Mean and standard deviation values for the degree of conversion for Filtek and Harmonize for non-lubricated top surface and wetting resins-lubricated bottom surface/setup one

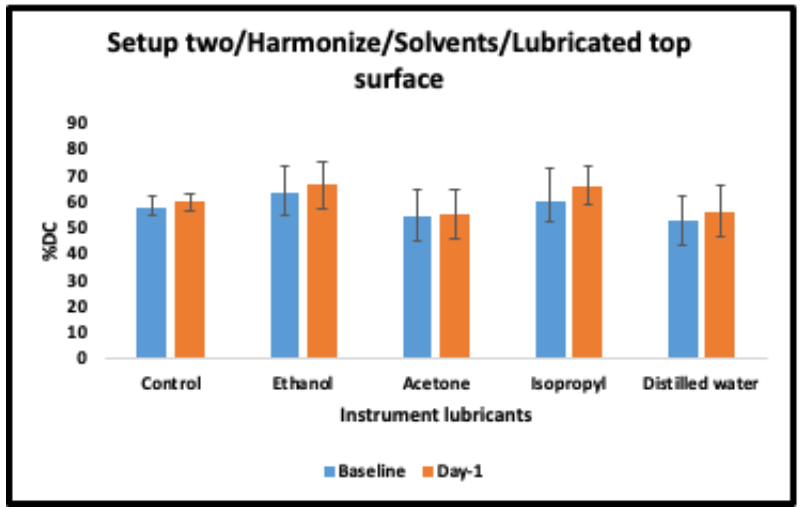
The next section focuses on the results from setup two. Regarding the solvents' experiments, significant differences were found between the experimental groups (p<.001). On Filtek's lubricated top surface, organic solvents such as ethanol and isopropyl mostly increased the DC compared to the control, and none of the solvents significantly reduced the DC of this surface. The non-lubricated bottom surface of Filtek showed no difference across all groups. For the Harmonize groups, no effects were found from the use of solvents and none reduced the DC on lubricated top surface, and nor were there any changes on the non-lubricated bottom surface, as presented Figure 5-32. The bottom surface which is non-lubricated surface of both RBCs at depth of 6mm showed low DC values.

Regarding the bonding agents in setup two, the respective Scotchbond and Optibond two-step bonding agents were used on lubricated surface of setup two. The Filtek groups showed no negative effect from either bonding agents and both increased the DC on this surface. Also, the DC for the Optibond group increased on non-lubricated bottom surface in comparison to the control. The Scotchbond and Optibond groups for Harmonize also did not negatively affect the DC on the lubricated top surface. Both increased the DC on lubricated and non-lubricated surface, as summarised in Figure 5-33.

The wetting resin experiments results for Filtek showed no reduction in the DC of lubricated top surface with IL; Brush & Sculpt and Modelling resin increased the DC on this surface. Non-lubricated bottom surface of the same resin type had no significant changes due to the changes that occurred on the lubricated surface. While the same results were obtained for Harmonize's on lubricated top surface, the DC was increased on the non-lubricated bottom surface of the same specimen was lubricated with Modelling resin on the top surface. All these results are summarised in Figure 5-34.







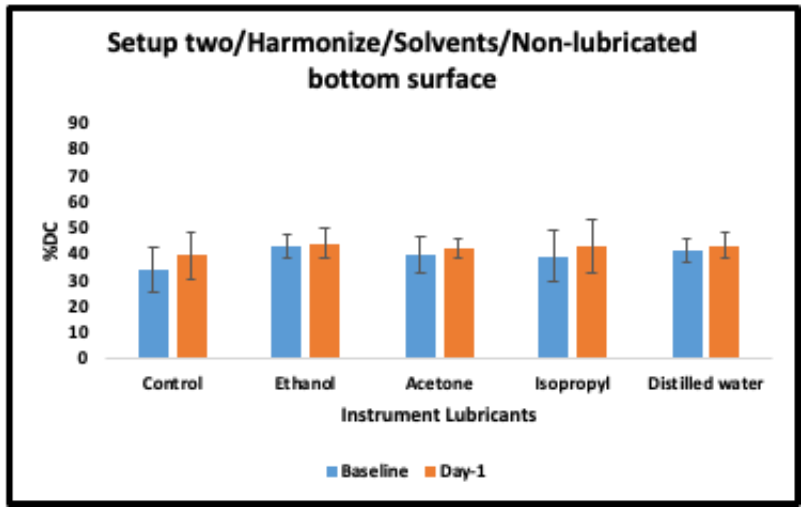
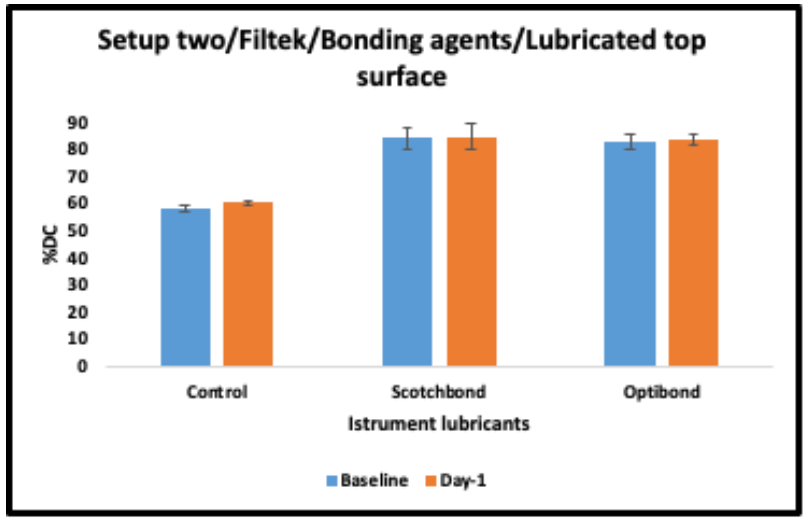
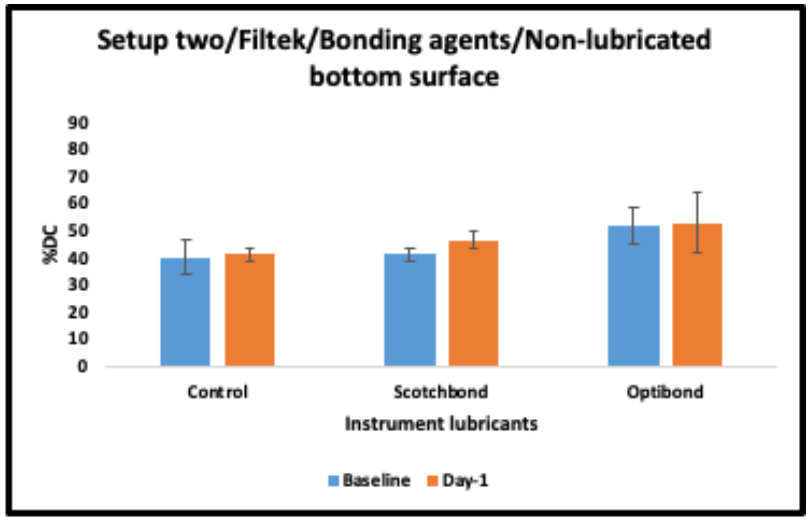
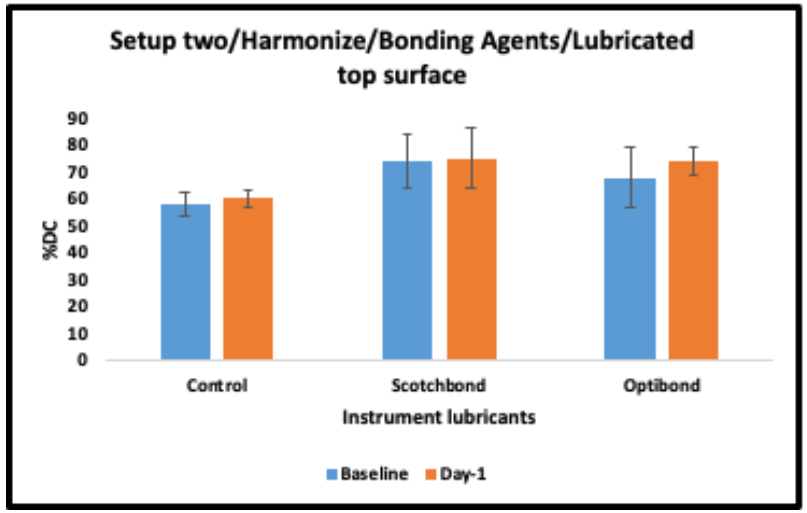


Figure 5-35: Mean and standard deviation values for the degree of conversion for Filtek and Harmonize for solvents-lubricated top surface and non-lubricated bottom surface/setup two







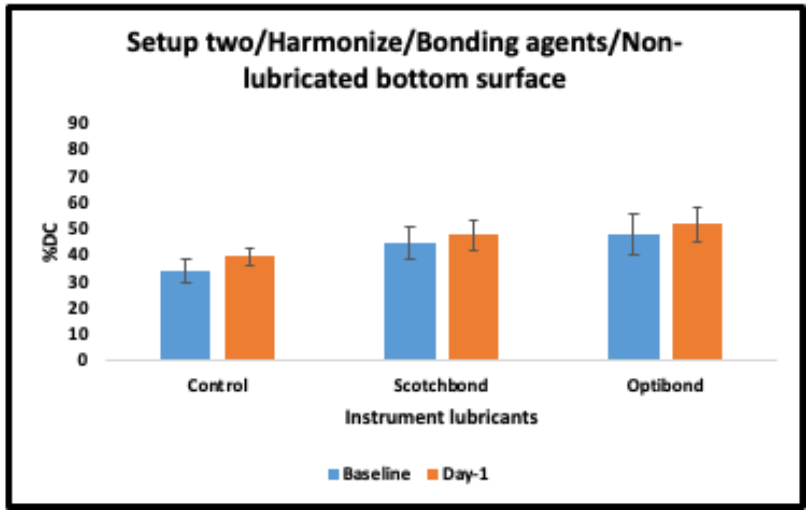
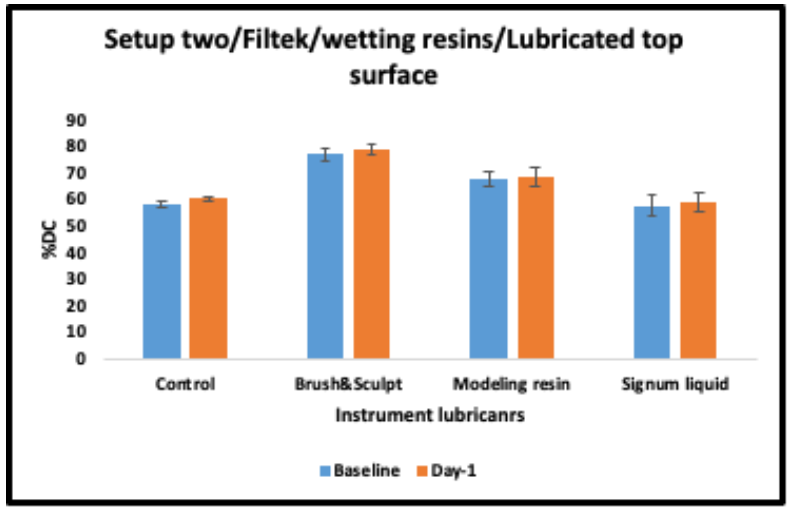
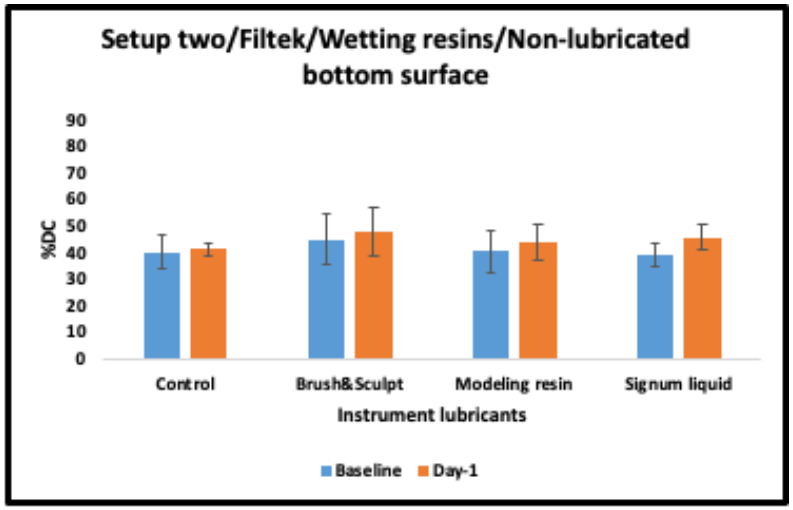
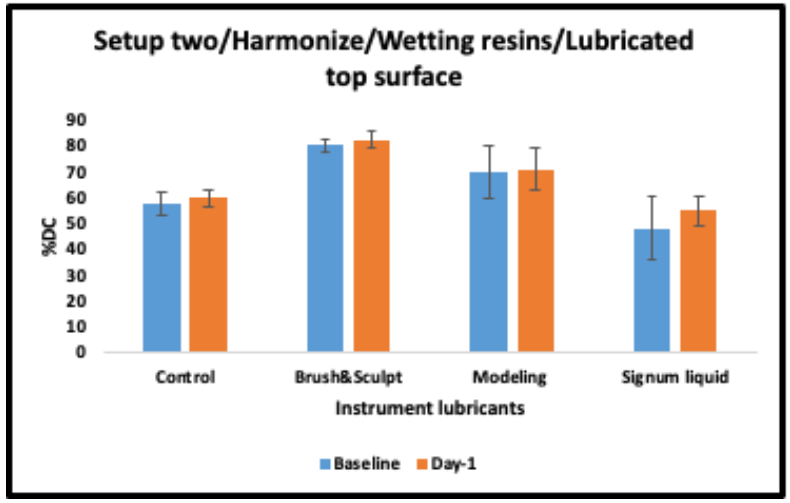


Figure 5-36: Mean and standard deviation values for the degree of conversion for Filtek and Harmonize for bonding agents-lubricated top surface and non-lubricated bottom surface/setup two







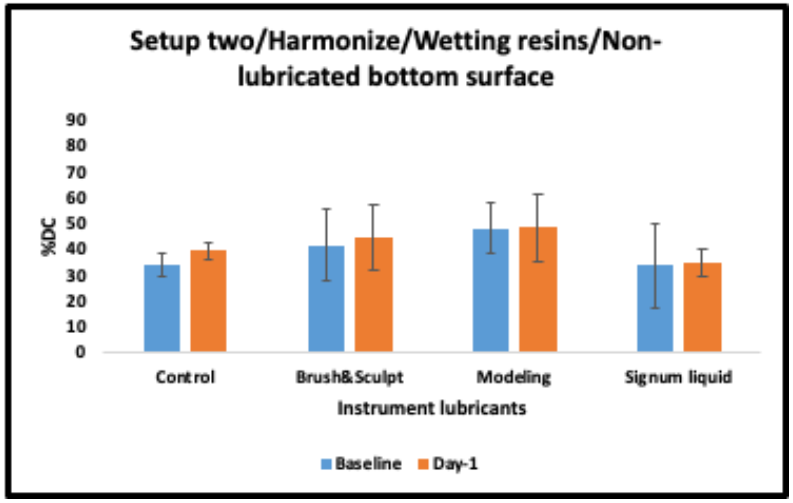


Figure 5-37: Mean and standard deviation values for the degree of conversion for Filtek and Harmonize for wetting resins-lubricated top surface and non-lubricated bottom surface/setup two

The results of both setups show that Filtek was more affected by the ILs. Distilled water from the solvents group (Figure 5-32) and the Signum liquid (Figure 5-37) from the wetting resins class both negatively reduced the DC on the setup one lubricated bottom. The remaining ILs either had no effect or increased the DC in comparison to the control. No negative effects were detected on all surfaces of Harmonize RBC, as summarised in Table 5-8.

Classes	**ILs groups**	**Filtek**				**Harmonize**			
		Surfaces				Surfaces			
		Setup one		Setup two		Setup one		Setup two	
		NL	L	L	NL	NL	L	L	NL
Solvents	Ethanol	NS	NS	NS	NS	NS	NS	NS	NS
	Acetone	NS	NS	NS	NS	NS	NS	NS	NS
	Isopropyl	NS	NS	NS	NS	NS	NS	NS	NS
	DW	NS	(*p*=0.02) *	NS	NS	NS	NS	NS	NS
BA	Scotchbond	NS	NS	NS	NS	NS	NS	NS	NS
	Optibond	NS	NS	NS	NS	NS	NS	NS	NS
WR	S & B	NS	NS	NS	NS	NS	NS	NS	NS
	MR	NS	NS	NS	NS	NS	NS	NS	NS
	Sig	NS	(*p*<.001) *	NS	NS	NS	NS	NS	NS

Table 5-8: Significant DC reduction in the lubricated (L) and non-lubricated (NL) surfaces of the experimental groups in both setups of the RBCs

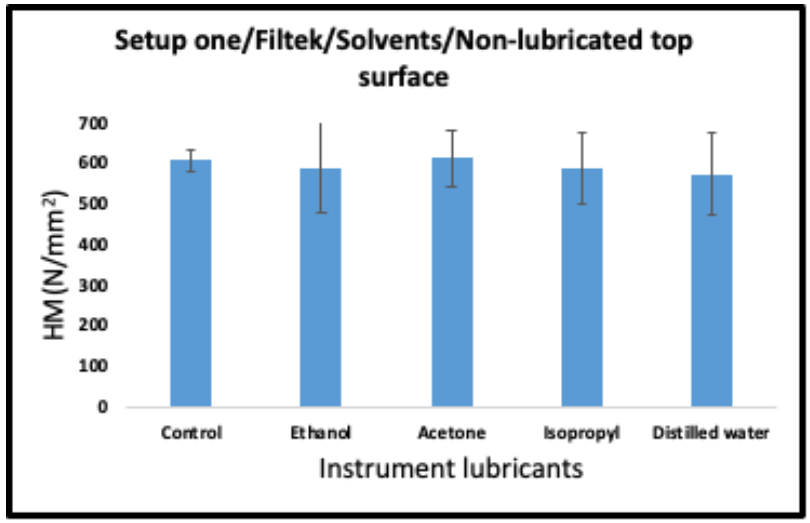
* Statistically significant different *p*-values in comparison to the control and (NS) means not significant.

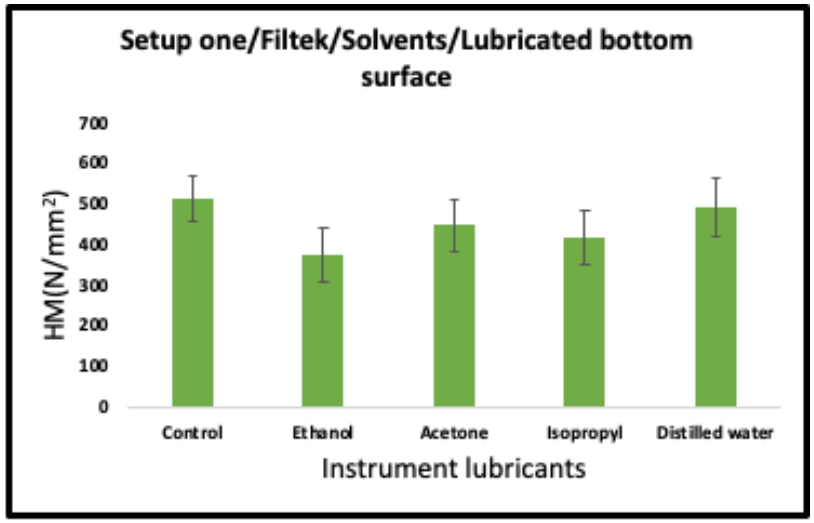
5.4.4 Martens hardness

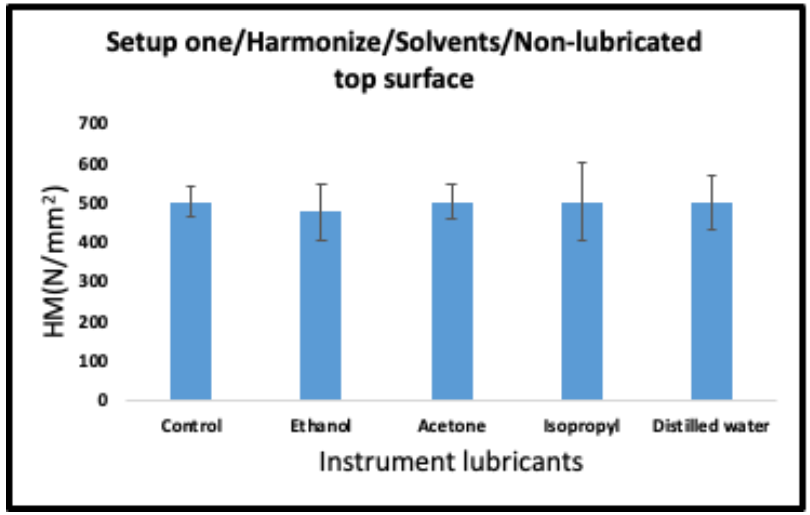
The results showed there were significant differences between the experimental groups in this class, with all groups showing no change or a significant difference on the non-lubricated top for Filtek (p=0.0796) and Harmonize (p=0.935). The Filtek groups revealed a statistically significant difference for lubricated bottom surface (p=0.021). Ethanol significantly reduced the hardness value on this surface (p=0.022). However, the Harmonize solvents tests showed no significant changes on the lubricated bottom surface (p=0.927) from using the solvents as ILs on this surface. According to the results of the solvents tests for setup one, only Filtek was affected by using solvents as ILs on lubricated bottom surface as Harmonize was unaffected, as shown in Figure 5-38.

Regarding the bonding agents in setup one, the tests of ILs revealed no statistically significant difference between the experimental and control groups, as neither RBC showed changes to non-lubricated top surface. No significant differences were detected on lubricated bottom surface for Filtek (p=0.090) and Harmonize (p=0.521) in comparison to the controls. In general, the bonding agent-treated surfaces of both RBCs showed no significant reductions in HM values, as presented in Figure 5-39.

The results of the wetting resins tests in setup one for non-lubricated top surface showed no changes for Filtek (p=0.349) or Harmonize (p=0.922). The wetting resins group displayed a statistically significant difference (p=0.014) on the lubricated bottom surface of Filtek. Brush and Sculpt reduced the HM values of Filtek's lubricated bottom surface (p=0.016), but for Harmonize there was no significant difference (p=0.084) on the same surface treated with wetting resins. These results generally show that Filtek was more affected by this class of ILs, as summarised in Figure 5-40.







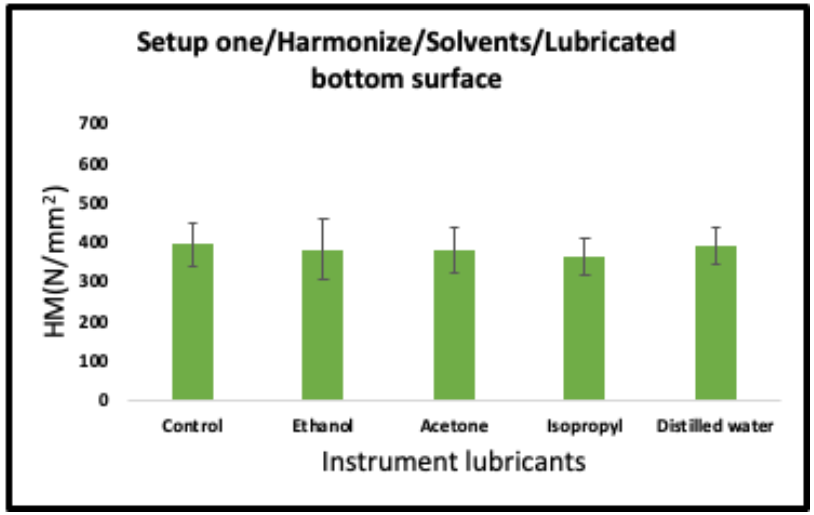


Figure 5-38: Mean and standard deviation values for Martens hardness tests (Filtek & Harmonize) on non-lubricated top surface and solvent-lubricated bottom surface in setup one

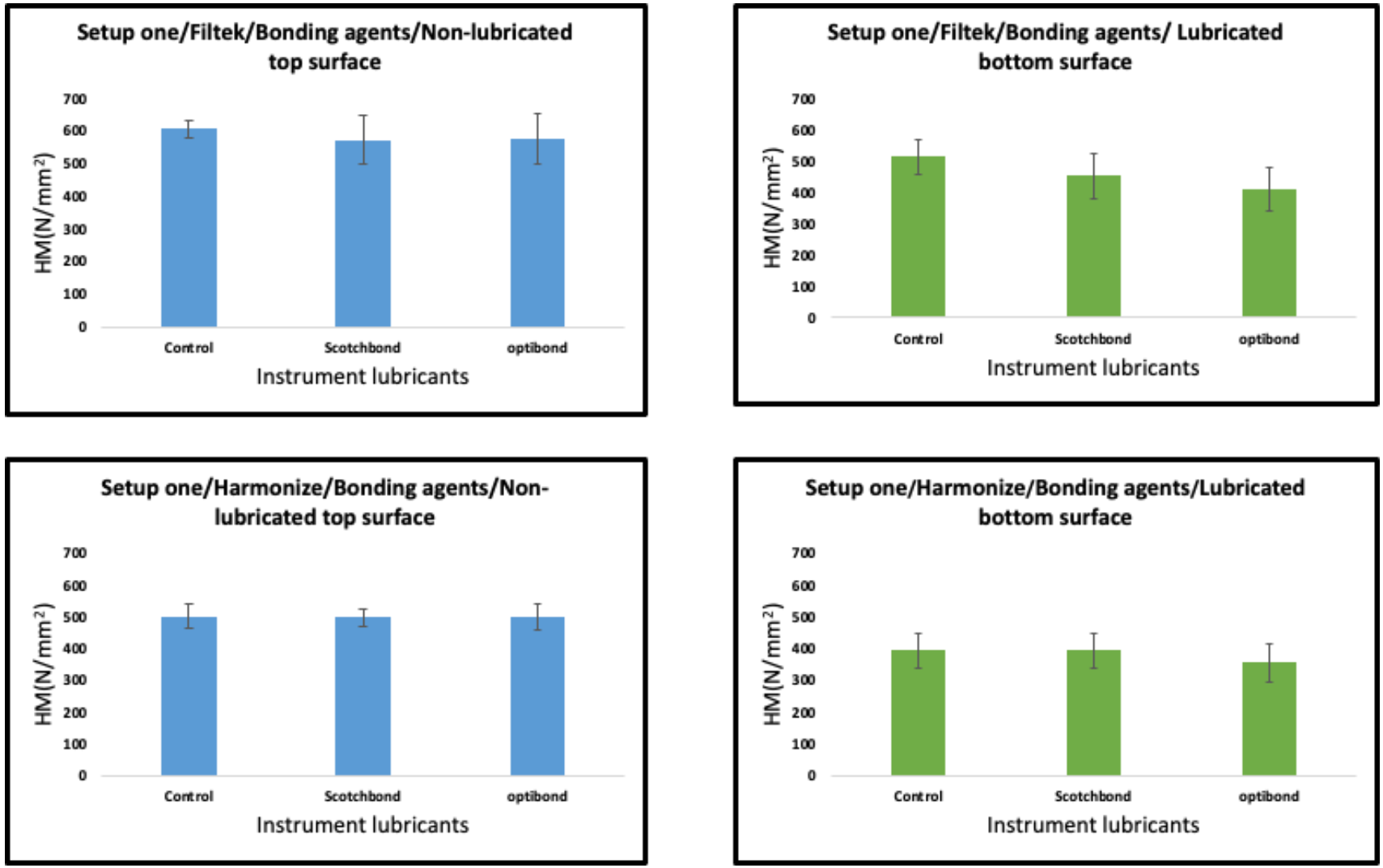
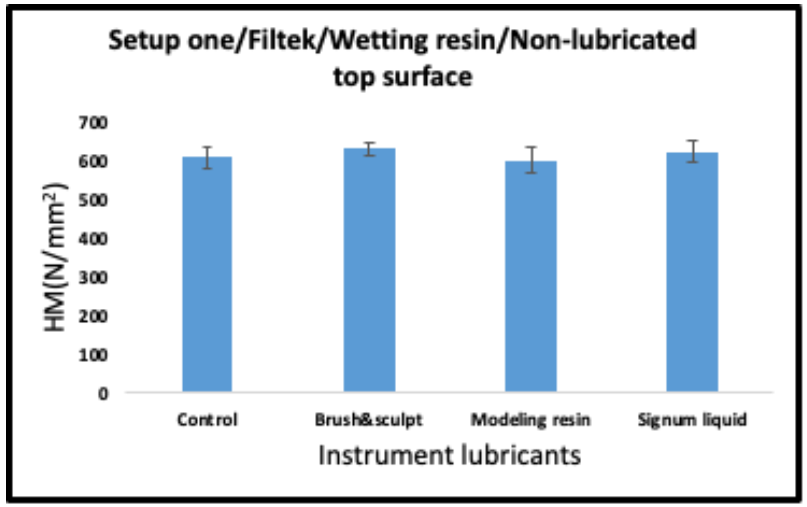
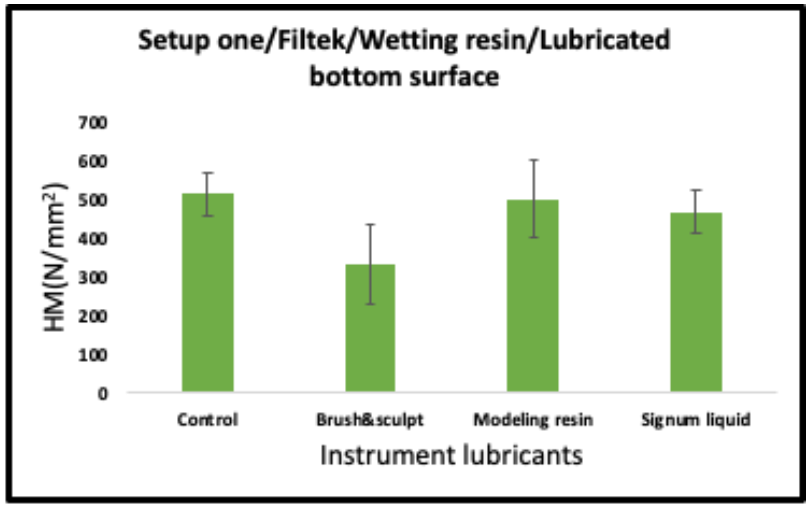
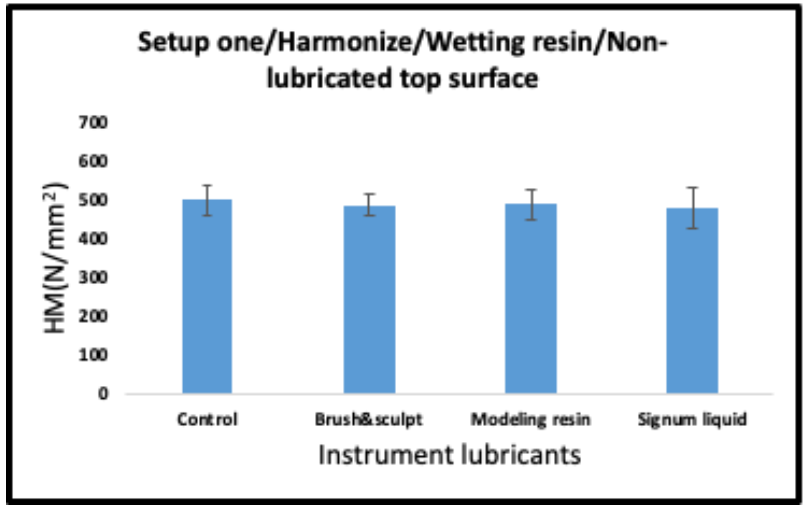


Figure 5-39: Mean and standard deviation values for Martens hardness tests (Filtek & Harmonize) for non-lubricated top surface and bonding agent-lubricated bottom surface in setup one







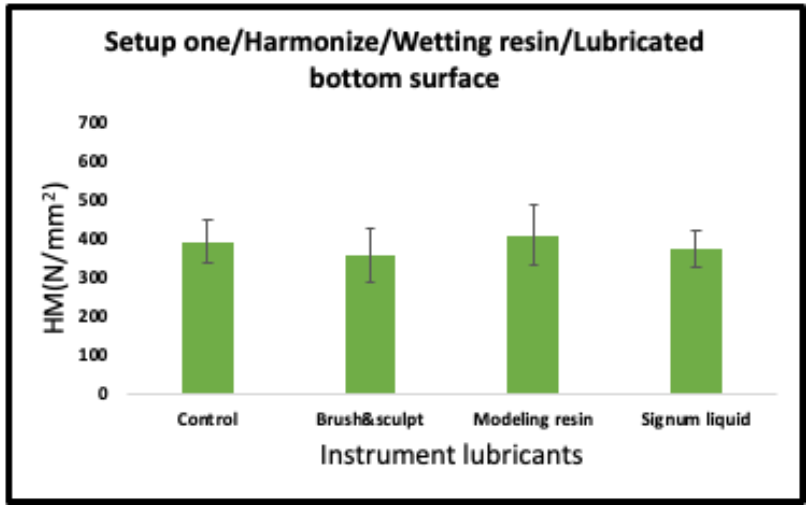
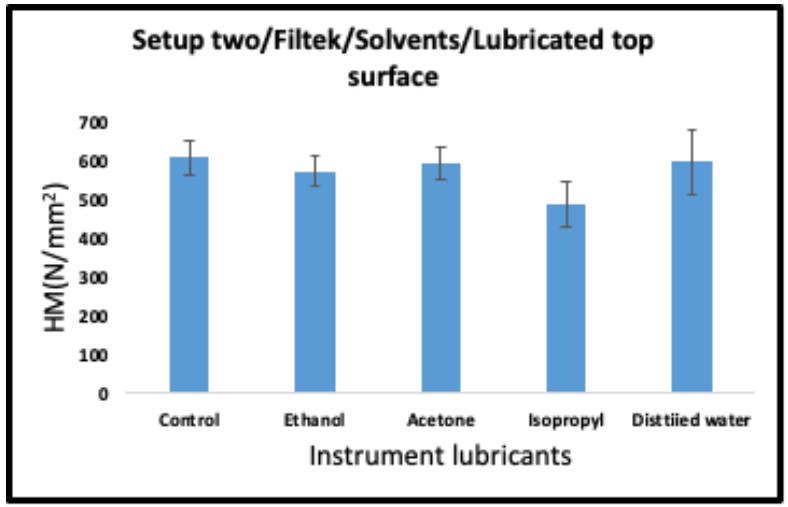


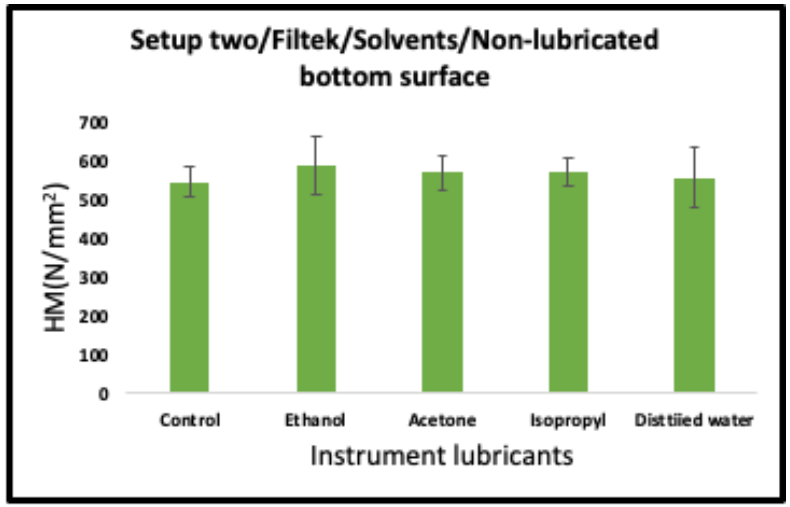
Figure 5-40: Mean and standard deviation values for tests of Martens hardness (Filtek & Harmonize) for non-lubricated top surface and wetting resin-lubricated bottom surface in setup one

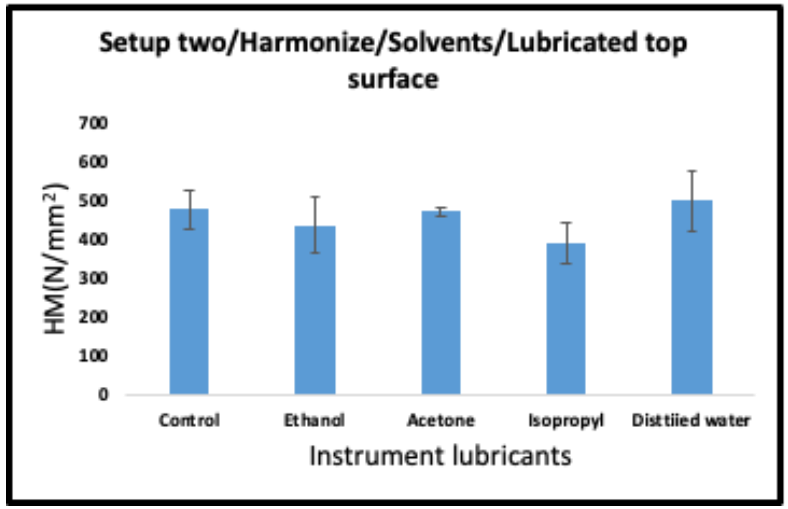
In setup two, the results of all the experimental groups using solvents as ILs, for both RBCs, showed statistically significant differences (p=0.016). The values for Filtek's lubricated top surface treated with isopropyl showed a significant reduction in the HM values (p=0.018). However, for the same surface, Harmonize had no statistically significant difference when the ILs were applied (p=0.059). The non-lubricated surfaces for both RBCs showed no statistically significant difference resulting from the ILs. For both RBC groups manipulated with ILs, only Filtek was affected when isopropyl was applied and the HM value was statistically significantly reduced, as shown in Figure 5-41.

Regarding the bonding agent tests in setup two, both the Filtek (p=0.001) and Harmonize (p<.001) groups showed there were statistically significant differences for the lubricated top surface when using Scotchbond and Optibond. However, while Filtek presented changes to the non-lubricated bottom surface and the HM value increased, there were no changes to the non-lubricated bottom surface of Harmonize. Both RBCs showed a significant reduction in HM values on the lubricated top surface, but only Filtek's HM values for the non-lubricated bottom surface only increased when Optibond was used on top surface, in contrast to the control, as presented in Figure 5-42.

The wetting resin results for setup two for both RBCs on the lubricated top surface showed differences between the groups (p>.001). All the ILs had statistically significantly reductions of the HM values of this surface for both RBCs in comparison to the control. However, no significant changes were detected on the non-lubricated bottom surface for either RBC. The lubricated top surface for all the groups was negatively affected, regardless of the different ILs, as summarised in Figure 5-43.







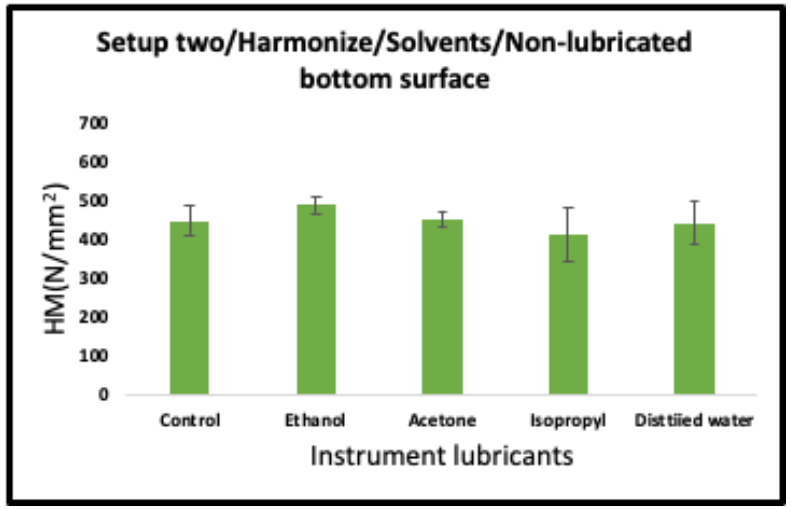
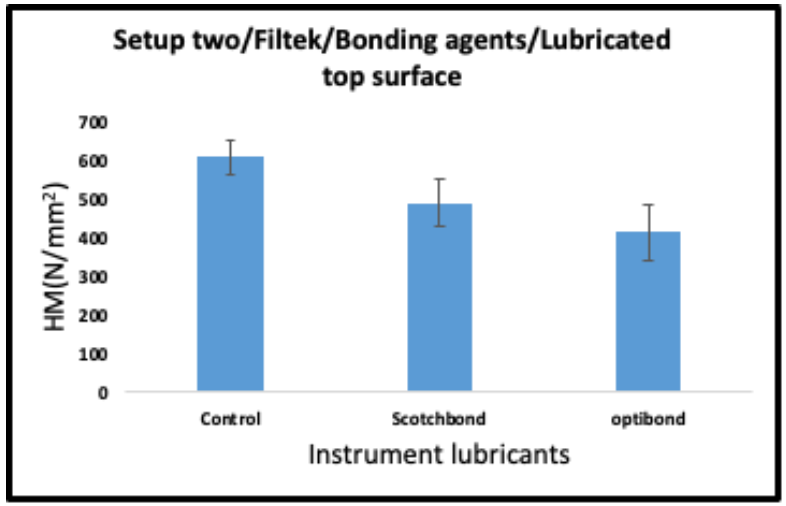
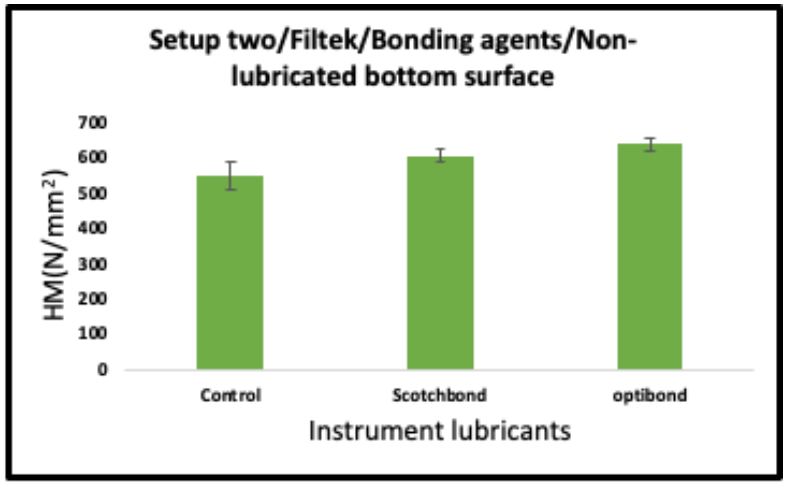
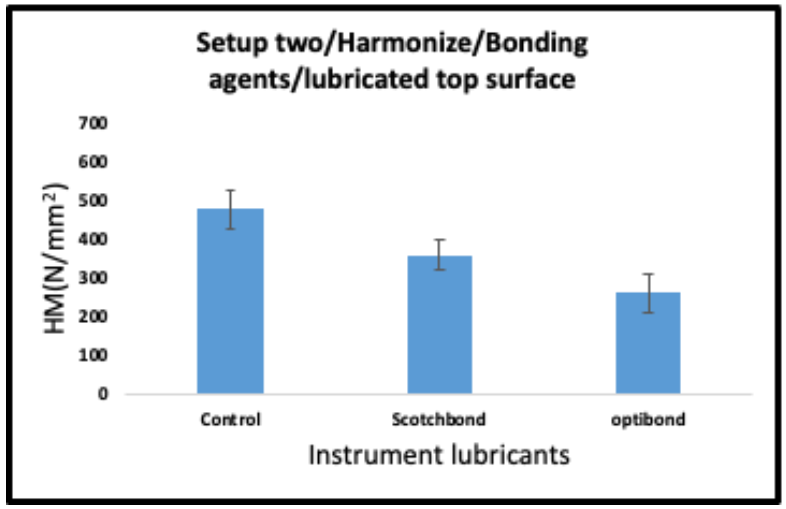


Figure 5-41: Mean and standard deviation values for Martens hardness tests (Filtek & Harmonize) for solvent-lubricated top surface and non-lubricated bottom surface in setup two







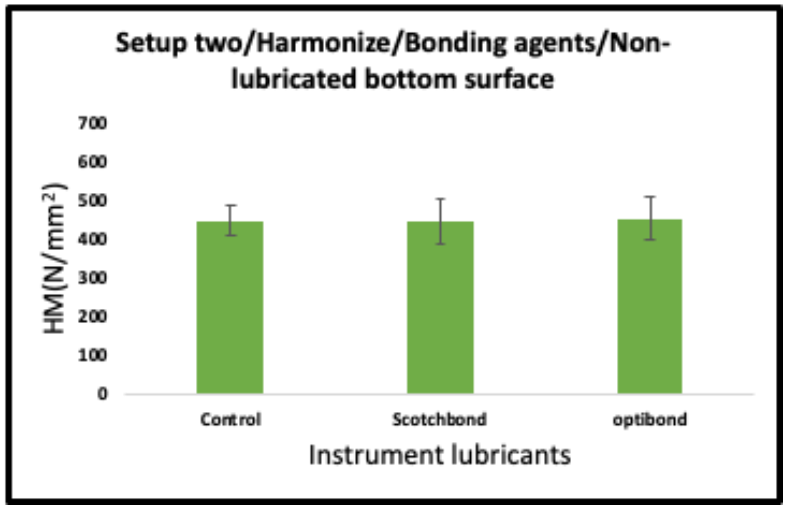
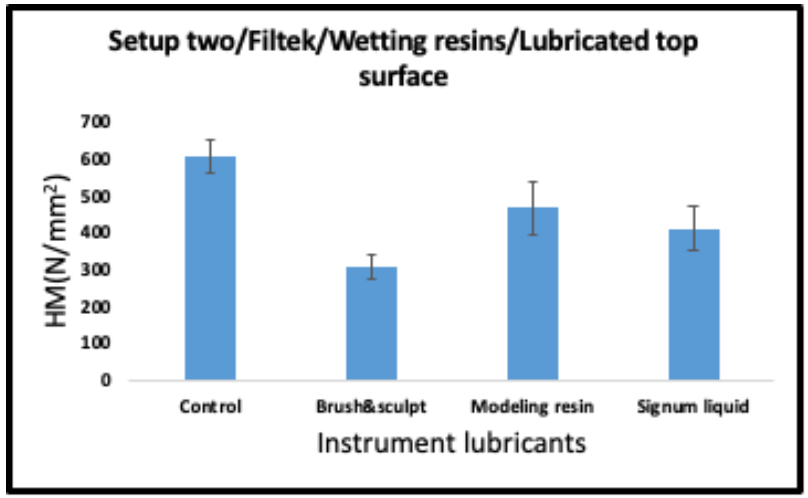
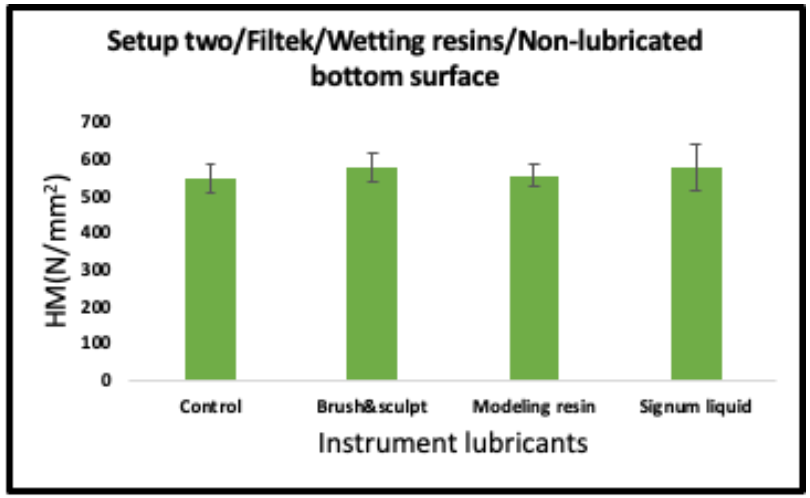
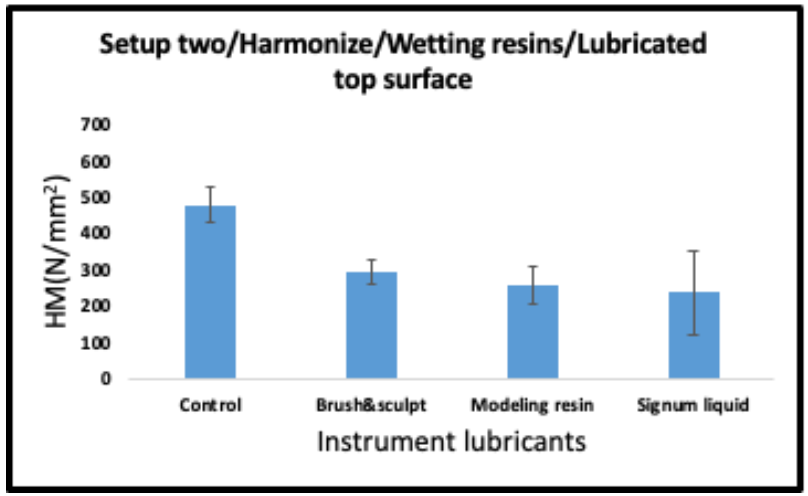


Figure 5-42: Mean and standard deviation values for Martens hardness tests (Filtek & Harmonize) for bonding agent-lubricated top surface and non-lubricated bottom surface in setup two







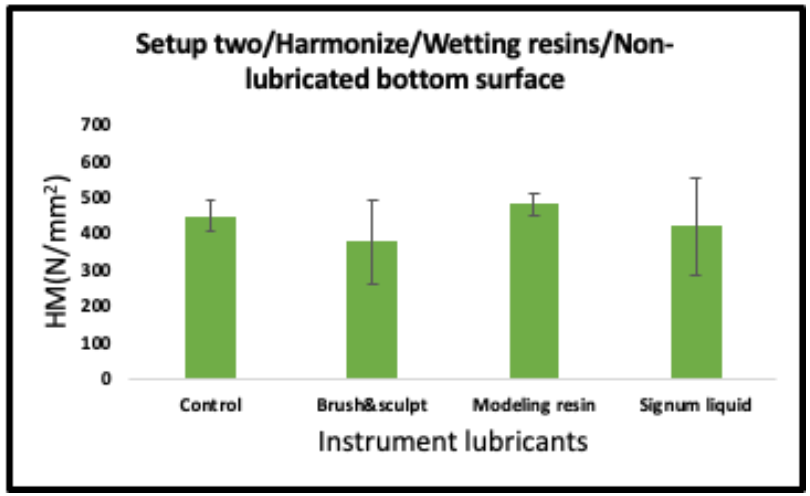


Figure 5-43: Mean and standard deviation values for Martens hardness tests (Filtek & Harmonize) for wetting resin-lubricated top surface and non-lubricated bottom surface in setup two

The results of both setups show that Filtek was more affected by the ILs. Ethanol from the solvents group (Figure 5-38) and the Brush and Sculpt (Figure 5-40) from the wetting resins class both negatively reduced the HM on the setup one lubricated bottom surface. All bonding agents and wetting resins had negatively affected the HM in comparison to the control in setup two as shown in Figure 5-42 and 5-43. Negative effects were not detected in setup one of the Harmonize RBC groups, only in the setup two the lubricated surfaces were affected by applying the bonding agents and wetting resins ILs as summarised in Table 5-8.

Classes	ILs groups	Filtek				Harmonize			
		Surfaces				Surfaces			
		Setup one		Setup two		Setup one		Setup two	
		NL	L	L	NL	NL	L	L	NL
Solvents	Ethanol	NS	(*p*=0.022) *	NS	NS	NS	NS	NS	NS
	Acetone	NS	NS	NS	NS	NS	NS	NS	NS
	Isopropyl	NS	NS	(*p*=0.018) *	NS	NS	NS	NS	NS
	DW	NS	NS	NS	NS	NS	NS	NS	NS
BA	Scotchbond	NS	NS	(*p*=0.021) *	NS	NS	NS	(*p*=0.004) *	NS
	Optibond	NS	NS	(*p*=0.001) *	NS	NS	NS	(*p*<.001) *	NS
WR	S & B	NS	(*p*=0.016) *	(*p*=0.016) *	NS	NS	NS	(*p*<.001) *	NS
	MR	NS	NS	(*p*<.001) *	NS	NS	NS	(*p*<.001) *	NS
	Sig	NS	NS	(*p*<.001) *	NS	NS	NS	(*p*=0.004) *	NS

Table 5-9: Significant HM reduction in the lubricated (L) and non-lubricated (NL) surfaces of the experimental groups in both setups of the RBCs

*Statistically significant different *p*-values in comparison to the control and (NS) means not significant.

5.4.5 Water uptake test

Water uptake data showed a statistically significant difference between the experimental groups (p<.001). The ethanol-treated specimens were statistically significantly higher in comparison to the control group. However, in the Harmonize solvents group, acetone, isopropyl, and distilled water tests showed higher statistical differences against the control. The results of both RBCs showed that Harmonize was more affected by the solvent ILs (Figure 5-44).

In the bonding agents groups, there were significant differences between experimental groups (p<.001). Optibond was statistically significantly higher, as was Scotchbond within the Filtek groups; both increased the water uptake of this RBC. For Harmonize, there were differences between the groups, and only Scotchbond was statistically significantly higher than the control. The findings on bonding agents indicate that Filtek was most affected by both bonding agent groups, as shown in Figure 5-45.

Third test group on wetting resins, the experimental groups for Filtek showed a significant difference between the groups (p<.001), and Modelling resin was statistically significantly higher than the control. With Harmonize, Signum liquid and Modelling resin showed higher statistical differences against the control (p<.001). Harmonize seemed to be affected by most of the wetting resins, while Filtek was only affected by Modelling resin, as presented in Figure 5-46.

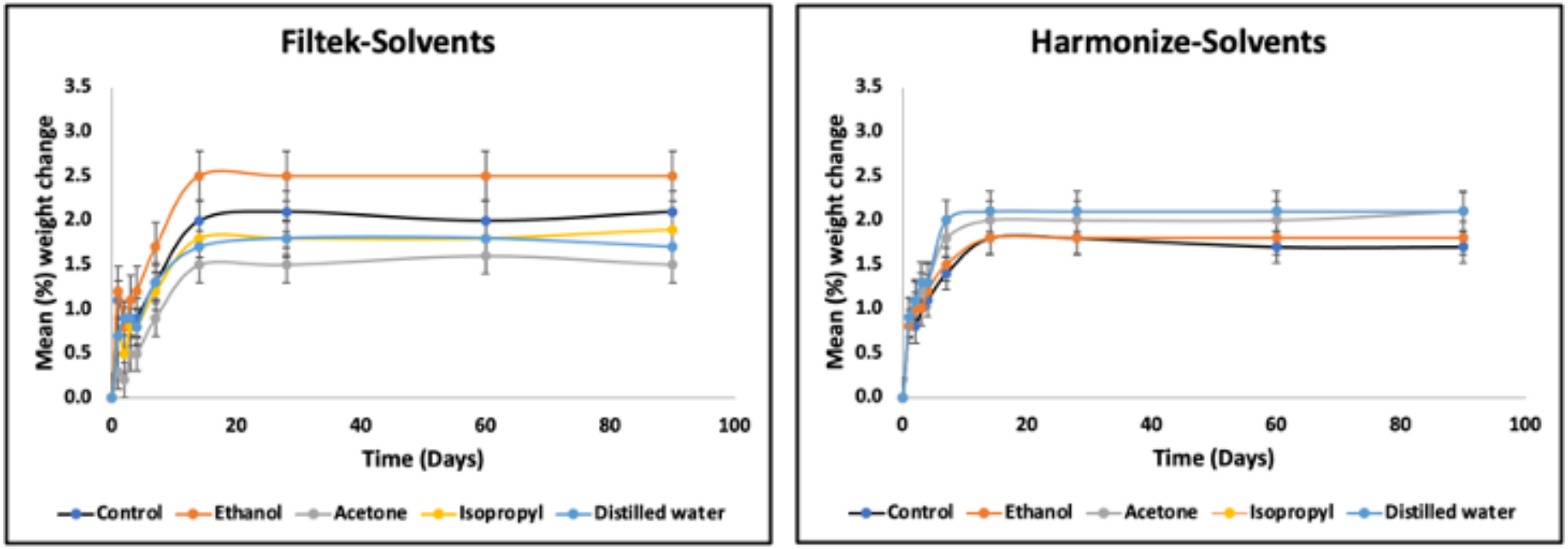


Figure 5-44: Solvent group % means for weight change and standard deviation for water uptake for Filtek and Harmonize stored in distilled water <90 days

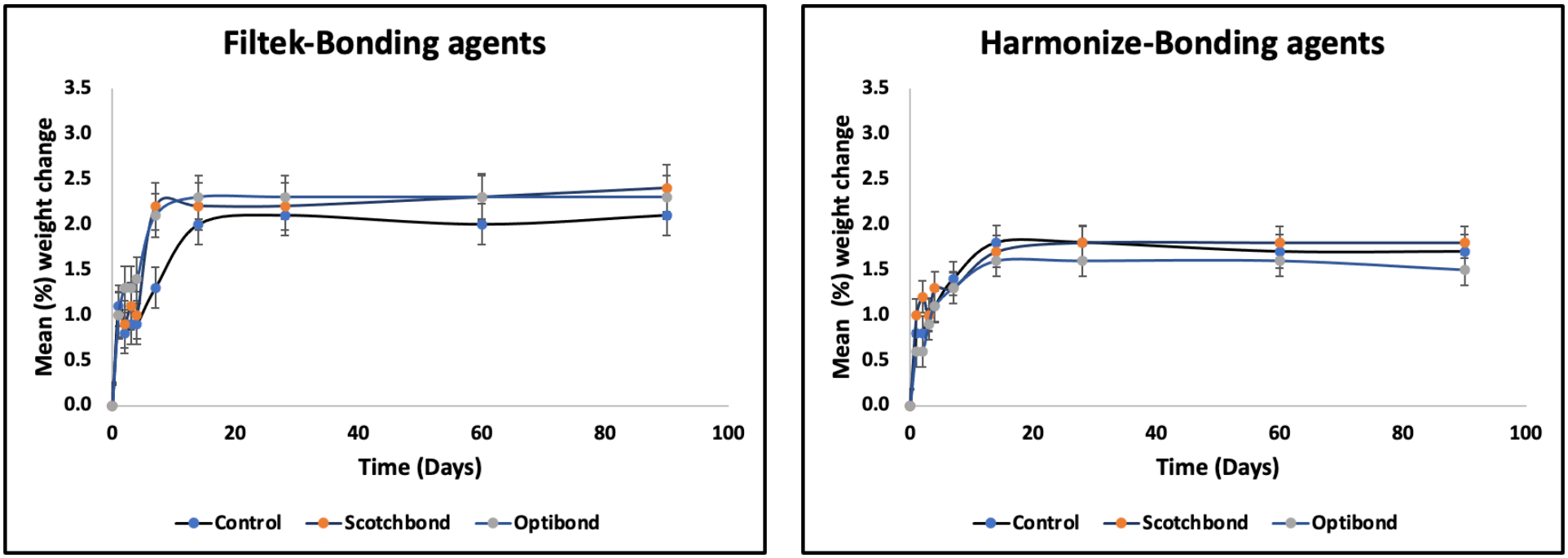
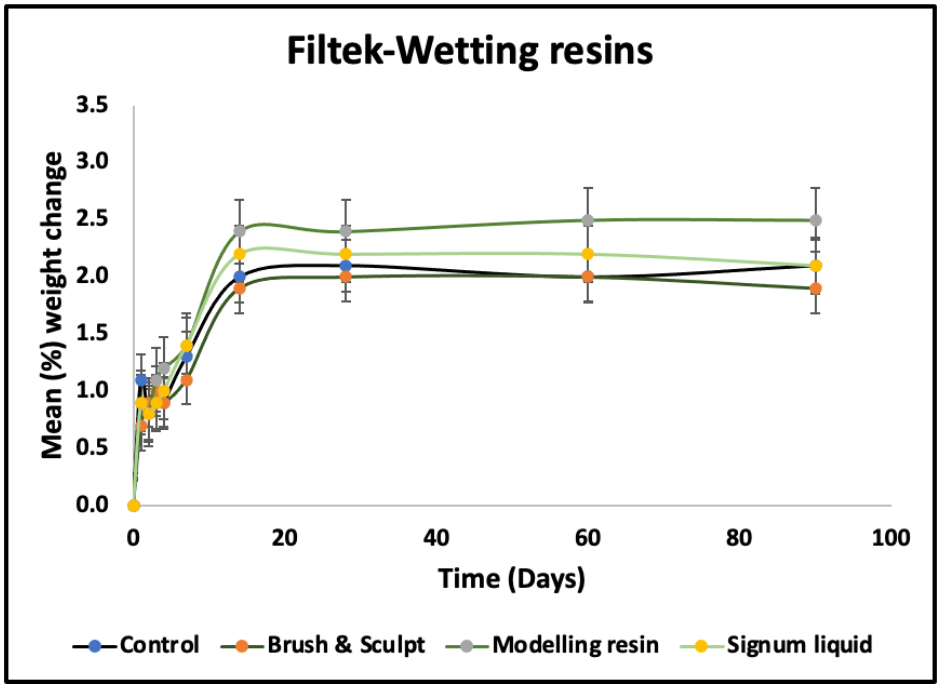


Figure 5-45: Bonding agent group means of % weight change and standard deviation for the water uptake in Filtek and Harmonize, stored in distilled water <90 days



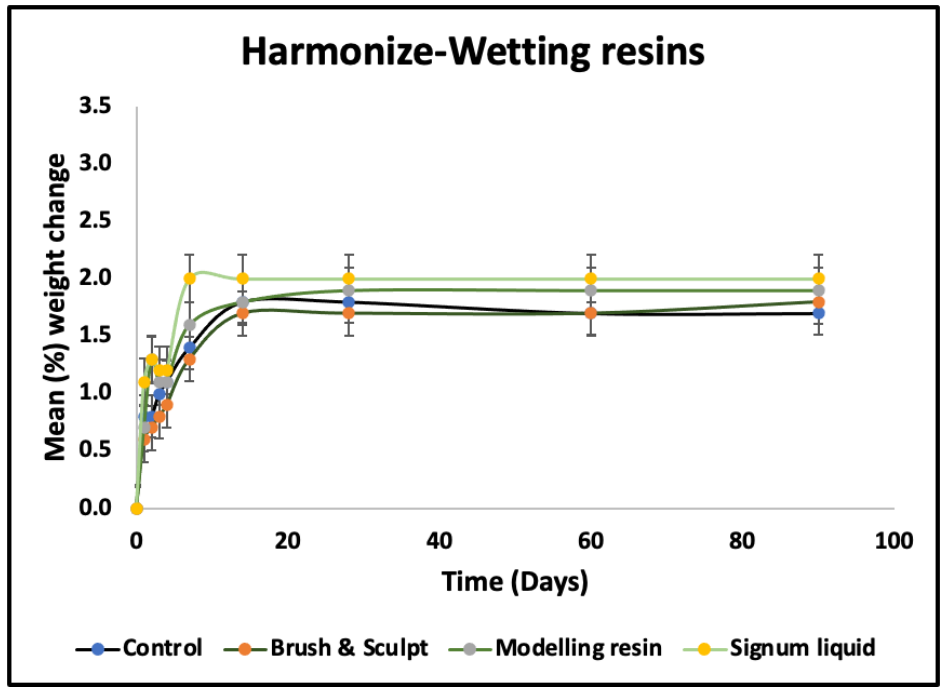


Figure 5-46: Wetting resin group mean % weight change and standard deviation of water uptake for Filtek and Harmonize, stored in distilled water <90 days

In brief, as shown in Table 5-10, water uptake was increased by applying the ILs. The values of all IL classes fluctuated from the baseline until day 14 and then their patterns remained steady across the time intervals. Filtek was most affected by the use of bonding agents, and by ethanol from the solvents tests and Modelling resin from the wetting resins. Harmonize was affected by different solvents such as distilled water, isopropyl and acetone, and several wetting resins increased water uptake in this RBC, such as Modelling resin and Signum liquid. Harmonize was only affected by one type of bonding agent, which was the Scotchbond. The only IL that did not affect the water uptake of either RBC was the Brush and Sculpt in the wetting resin class.

Classes	ILs groups	Filtek	Harmonize
Solvents	Ethanol	(p<.001) *	NS
	Acetone	NS	(p<.001) *
	Isopropyl	NS	(p<.001) *
	DW	NS	(p<.001) *
BA	Scotchbond	(p<.001) *	(p=0.004) *
	Optibond	(p<.001) *	NS
WR	S & B	NS	NS
	MR	(p<.001) *	(p<.001) *
	Sig	NS	(p<.001) *

Table 5-10: Significant increase in water uptake between the experimental and control groups

* Statistically significant different *p*-values in comparison to the control and (NS) means not significant.

5.4.6 Diametral tensile strength

In the solvents group, DTS data showed there was a statistically significant difference between the experimental groups (p<.001). All the solvents significantly reduced the DTS of Filtek, including isopropyl, ethanol, distilled water and acetone, in comparison to the control group. For Harmonize, none of the solvents reduced the DTS values significantly (p=0.067) against the control. Filtek was the most affected and the DTS was reduced by solvents, as shown in Figure 5-47.

The bonding agent tests showed there were differences between the groups (p<.001). Filtek was statistically significantly affected by both Scotchbond and Optibond, but in Harmonize none of the bonding agents affected the DTS (p=0.201) in comparison to the control. Filtek was thus most affected by the IL substances in this class, as presented in Figure 5-48.

Regarding the wetting resin, there was a difference between the Filtek experimental groups (p<.001). The Modelling resin group was statistically significantly lower than the control. For Harmonize, Modelling resin was the only IL wetting resin which statistically significantly affected and reduced the DTS (p=0.009). The effects of wetting resins were similar for both RBCs tested, as introduced in Table Figure 5-49.

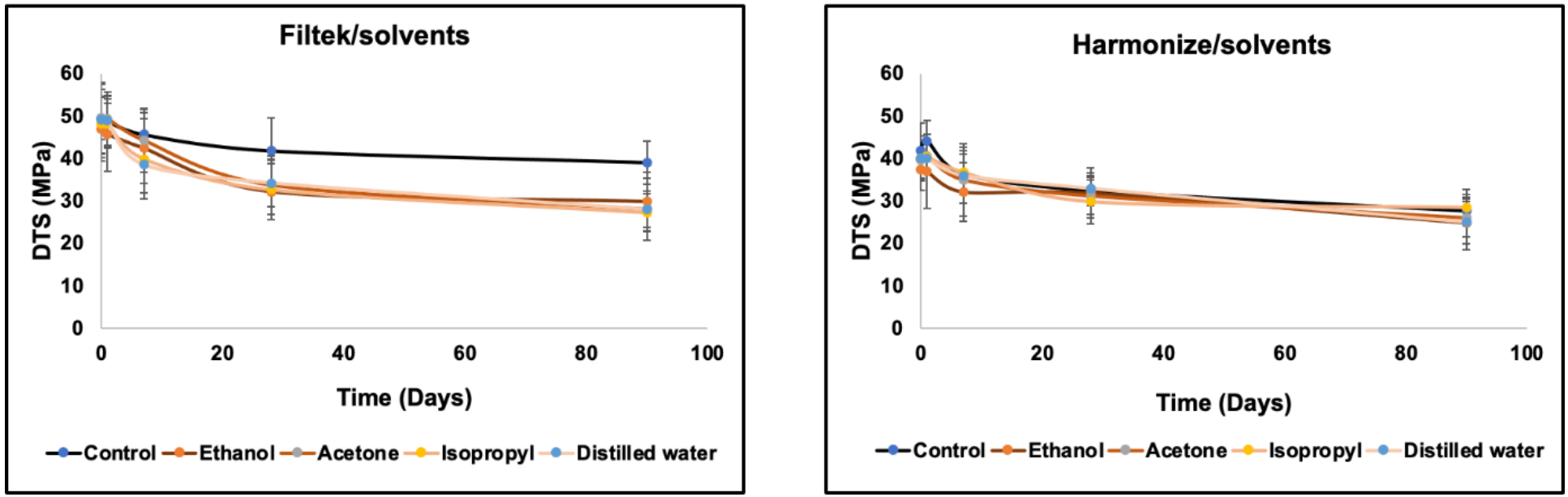
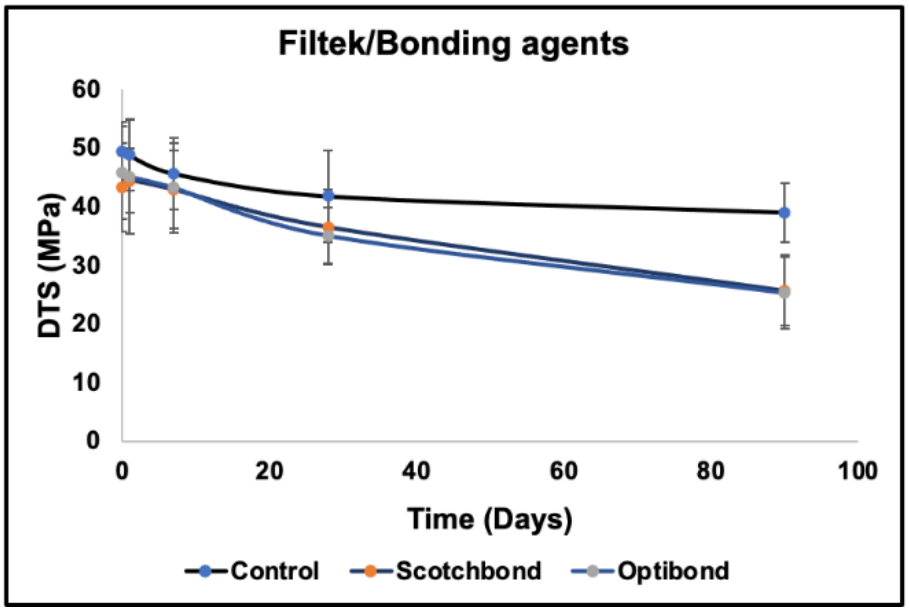


Figure 5-47: Solvents groups mean (MPa) DTS and standard deviation values for Filtek and Harmonize, stored in distilled water <90 days



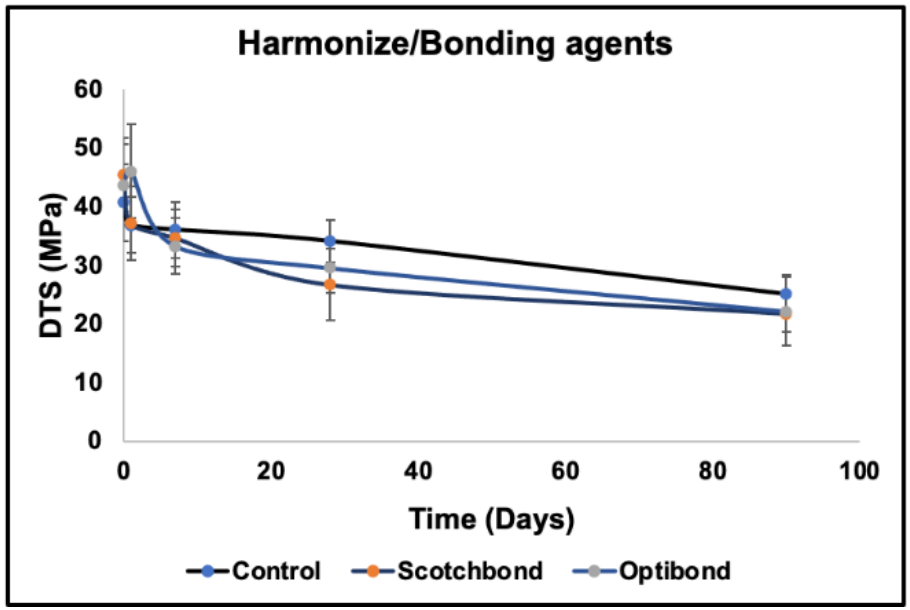
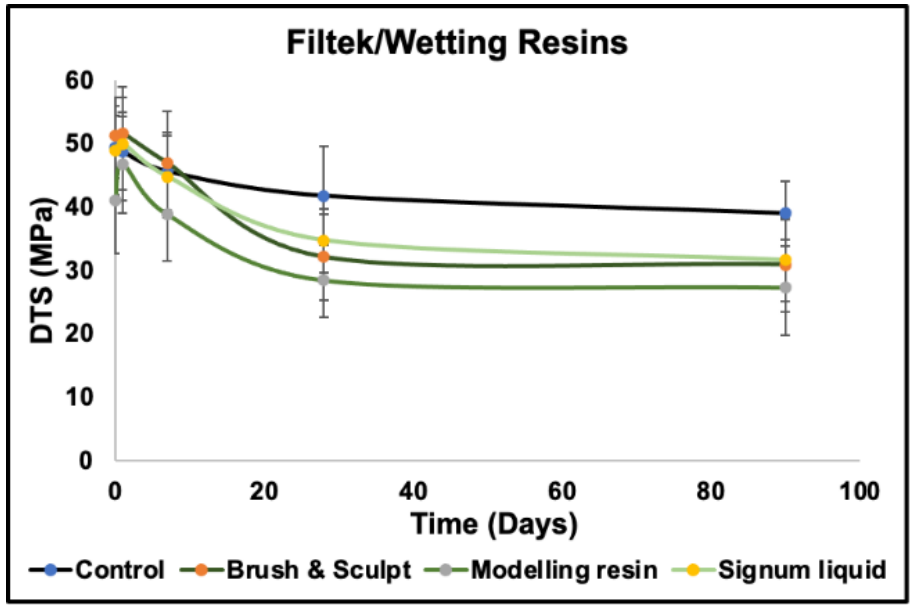


Figure 5-48: Bonding agent group mean (MPa) and standard deviation DTS for Filtek and Harmonize, stored in distilled water <90 days



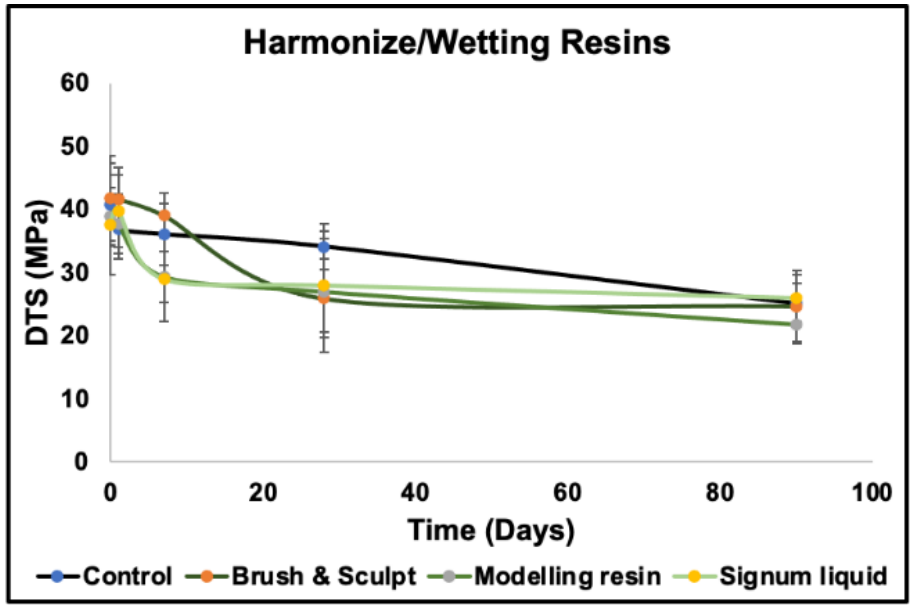


Figure 5-49: Wetting resin group mean (MPa) DTS and standard deviation values for Filtek and Harmonize, stored in distilled water <90 days

In general, the results of all classes of IL showed that the effects of these materials were different. Filtek was more affected by the use of ILs, including all the solvents, and the DTS was reduced as shown in Figure 5-47. Harmonize was not affected by any of the solvents. With the bonding agents, the same scenario was repeated as for the solvents for both types of RBC (Figure 5-48). The Modelling resin was the only IL that affected and reduced the DTS values of both RBCs (Figure 5-49). The results of all the experiments are summarised in Table 5-11 to show the effects of different ILs on both RBCs

Classes	ILs groups	Filtek	Harmonize
Solvents	Ethanol	(p<.001) *	NS
	Acetone	(p<.001) *	NS
	Isopropyl	(p<.001) *	NS
	DW	(p<.001) *	NS
BA	Scotchbond	(p<.001) *	NS
	Optibond	(p<.001) *	NS
WR	S & B	NS	NS
	MR	(p<.001) *	(p=0.009) *
	Sig	NS	NS

Table 5-11: Significant reduction in DTS between the experimental and control groups

* Statistically significant different p-values in comparison to the control and (NS) means not significant.

Table 5-12: Summary of experimental *p*-values showing the effects of the ILs on both RBCs.

ILs	Filtek					Harmonize				
	COIA	DC	HM	DTS	WU	SSCO	DC	MH	DTS	WU
Ethanol	(*P*<.001) *	NS	I (*P*=0.022) *	(*P*<.001) *	(*P*<.001) *	(*P*=0.03) *	NS	NS	NS	NS
Acetone	(*P*<.001) *	NS	NS	(*P*<.001) *	NS	(*P*<.001) *	NS	NS	NS	(*P*<.001) *
Isopropyl	(*P*<.001) *	NS	II (*P*=0.018) *	(*P*<.001) *	NS	(*P*<.001) *	NS	NS	NS	(*P*<.001) *
DW	(*P*=0.02) *	I (*P*=0.002) *	NS	(*P*<.001) *	NS	NS	NS	NS	NS	(*P*<.001) *
Scotchbond	NS	NS	II (*P*=0.021) *	(*P*<.001) *	(*P*<.001) *	NS	NS	II (*P*=0.004) *	NS	(*P*=0.004) *
Optibond	NS	NS	II (*P*=0.001) *	(*P*<.001) *	(*P*<.001) *	NS	NS	II (*P*<.001) *	NS	NS
Brush & Sculpt	NS	NS	I&II (*P*=0.016) *	NS	NS	NS	NS	II (*P*<.001) *	NS	NS
Modeling Resins	NS	NS	II (*P*<.001) *	(*P*<.001) *	(*P*<.001) *	NS	NS	II (*P*<.001) *	(*P*=0.009) *	(*P*<.001) *
Signum Liquid	NS	I (*P*<.001) *	II (*P*<.001) *	NS	NS	NS	NS	II (*P*=0.004) *	NS	(*P*<.001) *

* Represents the significant adverse effects of ILs in either the increase or decrease in the RBCs groups of the different tests and (NS) when there is no statistically significant different. (I) represents the lubricated bottom surfaces in setup one and (II) top surface in setup two for both DC and HM experiments.

5.5 Discussion

5.5.1 Specimen observation changes at increment interface

In the solvents test group, the different solvents showed significant changes in specimen interface between the increments. Their most noticeable effects were at the interface of the RBC treated with ILs, where the RBC materials in this area revealed an opaque strip surrounding the specimen. This was because IL had been applied on top of the first increment and manipulated with a plugger, which distributed solvent on the top of the RBC and some skipped onto the rims. The RBCs used in this study have Bis-GMA monomer which, in general, has a high viscosity due to its considerable molecular weight and other monomers added to reduce viscosity, such as TEGDMA (Leprince *et al.*, 2013). The ILs applied to the RBC reduce the viscosity even more due to the presence of organic solvents. All these factors can reduce the density of the polymeric layer of the treated RBC, increase the diffusion of the atmospheric oxygen to the RBC layer, and increase the thickness of the oxygen inhibition layer (Finger;Lee and Podszun, 1996; Studer *et al.*, 2003; Gauthier *et al.*, 2005). This in turn can affect the colour stability of the polymerised RBC and produce opaque strips between the increments (Yap *et al.*, 2001; Patel *et al.*, 2017). The strip changes fluctuated across the experimental time intervals; some appeared obviously opaque at the baseline and then after being stored in distilled water for 28 days they became lighter. This can be explained by there was lower DC in the inhibited layer which led to increase the water uptake in this area and forming the opaque strips. Then this layer started to dissolve in DW throughout the time intervals. In turn, the viscosity reduction and modification of the amount of oxygen inhibition layer then affects the RBC components and produces an opaque layer that can dissolve in DW over extended time intervals.

From the findings of the present study, the solvents had the most obvious changes on RBC colour at the level of the interface area between the increments. The RBC properties are fundamentally dependent on the stability of the organic and inorganic components of these materials (Ferracane, 2011). Substances like organic solvents can cause critical changes in the vital structure of the RBC, such as the organic matrix. Many changes can occur due to the presence of solvents between RBC increments, and most of these changes occur as the solvent can diffuse into the resin network and separate the chains in the RBC. This diffusion creates microvoids and enhances solvent uptake into the body of the RBC restoration (Dunn, 2007; Sideridou;Karabela and Vouvoudi, 2008). The result of this is a reduction in the RBC's physical and mechanical properties and colour changes, and potential chemical degradation (Cho and Dickens, 2004; Sideridou;Karabela and Bikiaris, 2007; Sideridou;Achilias and Karabela, 2007; Cadenaro *et al.*, 2009; Decky *et al.*, 2009; Martos *et al.*, 2019).

Both RBCs tested in the current study changed significantly after solvents were applied between the increments. The behaviour of these RBC types in relation to different solvents was not the same in the present study. Filtek was the most affected, and all solvents showed statistically significant differences against the control groups. In contrast, while Harmonize was affected by most of the solvents, it showed no significant changes with distilled water. That can have a link to the amount of monomer converted to polymers and crosslink density. Also, the relation and bonding between the organic and inorganic components of the RBCs. The differences in the components of these RBCs plays a significant role in these results (Leprince et al., 2013; Aydinoglu and Yoruc, 2017). Harmonize is superior from the aspect of filler volume loading, which makes a difference in fluid uptake between the increments (Rahim *et al.*, 2012; Leprince *et al.*, 2013), and so fewer changes are seen in this RBC type.

In recent decades, many bonding systems have been introduced of different compositions, using various application techniques (Van Meerbeek *et al.*, 2011). In the survey conducted at the beginning of the current project (reported in Chapter Four), the sampled UK dentists mostly used bonding agents as ILs. The two-step bonding system was generally selected by dentists for manipulating RBC restorations, and for this reason both tested bonding agents were two-step systems. Each bonding agent was produced by the manufacturer of the evaluated RBC materials, Filtek and Harmonize, and moreover each agent was investigated with both RBCs to determine if this would make a significant difference in the treated materials.

The current study's findings have shown that Filtek was not significantly affected when treated with either bonding system, and Harmonize showed the same behaviour across the set time intervals. A previous study presented findings which showed that some bonding systems do not affect the treated RBC materials (Münchow *et al.*, 2016). However, Harmonize was visually affected at baseline and OD values indicated changes when Optibond was used. These changes declined over time due to the presence of solvents such as water and ethyl alcohol that are part of Optibond's formula. These solvents, as explained previously, can cause changes in the treated area which in turn increase water uptake and affect the formation of the RBC's matrix (Sideridou;Karabela and Bikiaris, 2007). These solvents may become diluted when the specimen is stored in distilled water, such that the opaque strips on the surface become less prominent.

Regarding the wetting resins, although those used in the current study were resin-based, some of their components were not specified in detail by the manufacturer. Brush and Sculpt contains fillers but the manufacturers of the other types used in this study did not clearly state if they have any fillers. Wetting resins were the second most

common option for the manipulation of RBC materials chosen by the surveyed UK dentists. WR was applied at the interface between two increments of the specimen. Modelling resin was used in a previous study by Tuncer et al. (2013) as an IL to evaluate its effects on hardness and colour stability, and some adverse effects on colour stability were found due to the internal changes in the RBC components, and the different components of the Modelling resin and RBC matrices. However, these changes can be removed by finishing and polishing placed restorations (Tuncer *et al.*, 2013).

The current study has shown that there was no significant change in colour stability. No noticeable effect was seen either visually or in the intensity of OD in the evaluated area compared to the control. Unlike some of the other IL treatment groups, especially the solvents, none of the WR experimental groups showed an opaque strip. Although changes at the level of the interface area between increments were not opaque, there was a demarcation showing the borders of each increment area. These demarcations appeared due to the difference in the RBC and Modelling resin matrices. The absence of opaque strips at the interface area result of the absence of solvents from the components of WR.

5.5.2 Degree of conversion

Many external and internal factors can affect the stability of the physical and mechanical properties and the longevity of RBC restorations. One essential internal property of RBCs is the degree of conversion of monomers to polymers. The DC percentage is very important because it is correlated to many other essential material characteristics, and depends on converting the monomers' carbon double bond to polymers with multiple single bonds (Lovell *et al.*, 1999). This conversion helps

enhance the buildup of the degree of cross-linking between polymer chains. However, although the DC of some polymers can be similar, they are not the same in terms of cross-link density (Vouvoudi;Baxevani and Sideridou, 2016). Nevertheless, both are required to improve the stability of RBC materials and the physical and mechanical properties of RBC restorations (Leprince *et al.*, 2013; Al-Zain *et al.*, 2017).

The DC values of the different tested lubricated surfaces in setup one and setup two showed differences in the values collected in the current study. Many factors have a significant effect on the polymerisation process and changes in the DC percentage. These relate to the components of the RBC materials, such as the type of monomer, viscosity, and filler loading, as well as external factors related to the light-curing unit (LCU). Changing the distance between the tip of the LCU and the cured surfaces of the RBC has been confirmed in the literature as causing a change in the polymerisation process.

The amount of DC can be reduced when the distance between the LCU tip and cured surface is increased (Ferracane and Greener, 1984; Asmussen and Peutzfeldt, 2001; Finan *et al.*, 2013; Palagummi *et al.*, 2019). The distance between the light tip and cured restoration mentioned both in the literature and clinically can reach up to 10 mm (Price *et al.*, 2020). However, a previous study suggested that the distance between the LCU tip and cured surface to cure a 2mm increment is not more than 3mm away from the surface (Rode;Kawano and Turbino, 2007). Other studies have found that such effects occur only when the distance is more than 4mm (Lindberg;Peutzfeldt and van Dijken, 2004; da Silva *et al.*, 2008; Mohammed and Ario, 2015). This is supported by Price et al. (2000), who noted that the intensity of the LCU reduced about 55% when the distance between the tip of LCU and the cured surface is 6mm (Price;Murphy and Dérand, 2000). For instance, from the present study data, those surfaces that were

non-lubricated surfaces (bottom surfaces), they were away from the LCU tip about 6mm as presented in setup two. These surfaces have shown lower amount of DC for both types of RBC as shown in Figure 5-35, 5-36 and 5-37. Also, this point was mentioned in the literature as one of the external factors that would reduce the DC. Also, the curing parameters, irradiation mode and temperature have a role in changing the monomer polymerisation process. The literature has shown that all these factors have an influence on the percentage of the DC of RBC materials, either by increasing or decreasing the polymerisation process (Leprince *et al.*, 2013; Lempel *et al.*, 2019).

In the current study, the DC on the surfaces treated with different instrument lubricants was measured then compared to the RBC control groups to investigate the effects of the IL on the physical and mechanical properties of the RBC materials. The two setups were used to simulate the surfaces between the two increments, considering the position and distance of the LCU guide tip in relation to the experimental groups. The values of setup one on lubricated and non-lubricated surfaces for the DC percentage of both types of RBC ranged between 35–83 %; for setup two, the range on surfaces was 34–85%. The collected values of both RBCs can be compared to the values achieved in previous studies, regardless of the curing protocol and different surfaces of the specimens (Gajewski *et al.*, 2012; Finan *et al.*, 2013; Vale *et al.*, 2014; de Paula *et al.*, 2016; Yang *et al.*, 2019).

Throughout solvents groups, both lubricated surfaces of setup one (bottom lubricated surface) and setup two (top lubricated surface) showed significant changes in the percentage of DC. Using ethanol and isopropyl increased the DC of the setup two lubricated top surface of Filtek and setup one lubricated bottom of Harmonize. The presence of such solvents can reduce the viscosity of RBC materials and enhance the mobility of free radicals (de Paula *et al.*, 2016; Fugolin *et al.*, 2019). Moreover, the LCU

guide tip used with setup two was 3 mm away from the top of the specimen, and there were no materials between the top surface of the specimen and LCU tip to reduce the amount of potential light that passes through the RBC (Emami;Sjödahl and Söderholm, 2005). Also, the literature mentioned that the DC could increase due to an increase in the heat of the LCU tip. That can raise the polymerized RBC temperature and increase the radicals' mobility (Hofmann;Hugo and Klaiber, 2002). The temperature can be increased from 8.2-12 °C when LED LCU with an irradiance of 320 mW/cm^2 is used for 40 secs . Also, that can increase the DC by about 6–10% If the LCU tip temperature rises 22–35 °C (Price *et al.*, 2011b). However, the LED LCU used in the current study has an irradiance of 1,010 mW/cm^2 and the exposure time was 40 secs. Therefore, raising the temperature of the cured resin reduces the viscosity and increases free radical mobility, which increases the DC in the polymerised RBC (Daronch;Rueggeberg and De Goes, 2005; Leprince *et al.*, 2013).

The components of these RBCs, either resin matrix or filler load, were responsible for whether the uncured resin was contaminated by DW. The filler loading by volume of Filtek is lower than that of Harmonize, and the amount of filler loading has been shown to affect water uptake in RBC materials (Biradar and Arvind, 2012; Patel *et al.*, 2017). Water uptake increases the risk of foreign material interrupting the polymerisation process, as does the vapor pressure of water at room temperature, about 17.5 mmHg, compared to other organics such as ethanol (43.9 mm Hg), isopropyl (40 mm Hg) and acetone (185.5 mm Hg). This will keep more DW on the uncured RBC for a longer time in comparison to other organic solvents, hinder the conversion of monomers to polymers, and prevent their participation in chain growth due to the contamination of the cured RBC with residual DW (Nair;Hickel and Ilie, 2017; Fugolin *et al.*, 2019).

Regarding tests of bonding agents as ILs, this class has been investigated in the literature (Tjan and Glancy, 1998; Gorge Perdigao, 2006; Barcellos *et al.*, 2008; Tuncer *et al.*, 2013; de Paula *et al.*, 2016; Patel *et al.*, 2017). In the survey conducted for this study (see Chapter 4), bonding agents were mostly used by the sampled UK dentists as IL to reduce the tacking of RBCs to placement instruments, despite bonding systems being produced to enhance the bonding strength between the tooth structures and RBCs. These dentists were trying to reduce the surface tension of the RBC materials by applying the bonding agents as ILs, either on the surface of the materials or the placement instruments (Münchow *et al.*, 2016). Two different types of bonding agents from different manufacturers were investigated in the current study, to determine if using bonding agents and RBCs from the same company, for the purposes of ease of manipulation, would affect the physical and mechanical properties of an RBC restoration.

Applying bonding agents on both lubricated surfaces in setup one and two of Filtek showed that the bonding agents increased the DC on these surfaces. This result is comparable to the results of a previous study that used resin adhesive as an IL between RBCs layers (Münchow *et al.*, 2016). Also, the DC increased on the setup two non-lubricate bottom surface of the Optibond specimen compared to the control group. The presence of multiple monomers in the main components of bonding agents with a low amount of filler can enhance the DC (Van Landuyt, 2007). The presence in the bonding agents of silane functional groups creates bonding between the inorganic and organic parts, which react with the unreacted functional groups of the resin monomers and increase the DC (da Cruz *et al.*, 2019). Also, using solvents like ethanol can dissolve the organic resin monomers, potentially reducing the viscosity and increasing the mobility of the free radicals (de Paula *et al.*, 2016; Fugolin *et al.*, 2019).

This can explain why the DC increased on the setup two lubricated top surface and non-lubricated bottom surface of the Optibond group. The variety of different components present in these bonding agents can also play an important role, but the limitations of the information from the manufacturers make it difficult to anticipate their effects.

The presence of low viscosity monomers as essential components of the cured RBC can increase DC. That was mentioned in the literature and different studies showed that would increase the DC. The main reason for this it was the increasing the chance of the free radical mobility and increase the converting the monomers to polymers (Floyd and Dickens, 2006; Beun *et al.*, 2009; Leprince *et al.*, 2013; Cornelio *et al.*, 2014; de Paula *et al.*, 2016). Also, other components in the bonding agents, as mentioned above, reasonable justification for an increase in the DC on the lubricated surface and non-lubricated surface of both experimental RBCs. Reducing the viscosity may lead to a delay in the gelation and vitrification stages, so this will extend the polymerisation process through the specimen (Leprince *et al.*, 2013). The use of Optibond on setup two lubricated top surface have increased the free radicals and reduced the RBCs' viscosity. Furthermore, the homogeneity of the monomers in the bonding agent and RBC material, produced by the same manufacturer, can increase the DC on the setup two non-lubricated bottom surface of Harmonize.

The bonding agents used as ILs on the lubricated surfaces in both setups introduced the same effect by increasing the DC for both RBC types. The ILs, which are between increments, can increase the water uptake from the surrounding liquids in the oral cavity (de Paula *et al.*, 2016; Patel *et al.*, 2017). The presence of HEMA in the bonding agent can affect the DC of the RBCs and also increase water uptake (Zanchi *et al.*, 2013). HEMA is a hydrophilic monomer with a hydroxyl group at the end of its chemical

structure and can take in more water to the RBC matrix. Also, a high amount of HEMA reduces the vapour pressure of the solvents and will impact negatively on the solvents evaporation (Pashley *et al.*, 1998). For both RBCs, that can lead to contaminate the RBC with external substances and reduce the DC and increase the leaching of unconverted monomers. Also, that can promote debonding of the RBC's internal components, such as the monomers in the resin matrix and inorganic fillers due to an increase the water uptake. Furthermore, the HEMA tends to produce linear polymers that will have a weak polymer and affect the physical and mechanical properties of the RBCs.

Regarding the wetting resin substances tested in the current study, these were investigated to determine their effects on the physical and mechanical properties of the different RBCs. The selection of these substances was informed by the results of the questionnaire-based survey which proceeded the laboratory investigations. Also, the literature reports on certain materials used in previous studies (Gorge Perdigao, 2006; Tuncer *et al.*, 2013). All the selected materials were resin-based wetting resin, according to information provided by the manufacturers.

Most of the wetting resins used in this study contain dimethacrylate and multifunctional methacrylic acid ester. Their effects on the setup one lubricated bottom surface showed no effects, except for Signum Liquid, which reduced the DC in this group. The reason for the reduced DC in this group compared to the control may have been due to the presence of Signum on the bottom of the specimen and contamination of the uncured RBC by this IL's internal components; it may have also caused changes in the RBC's colour, (de Paula *et al.*, 2016). However, none of the wetting resins showed any reduction in the DC for Filtek on the setup two lubricated top surface. While Brush and Sculpt and Modelling resin increased the DC on the setup two lubricated top surface,

Signum Liquid had no effect, and therefore wetting resins have different effects depending on their internal components. The place of application of the ILs also influences the amount of DC as an external factor.

The ILs used on the setup two lubricated top surface were either unfilled or low-filled resins with low viscosity, which can raise free radical mobility and increase the DC of the cured resin (Daronch;Rueggeberg and De Goes, 2005; Leprince *et al.*, 2013). Also, the LCU's tip guide position was 3 mm away from the top surface, and there were no materials between the tip and the cured RBC surface. The light beam directed onto that surface was not interrupted or reflected by the inorganic fillers of the RBC materials (Turssi;Ferracane and Vogel, 2005; Ferracane, 2011; Finan *et al.*, 2013; de Paula *et al.*, 2016). The amount of heat produced by the LCU during the exposure time of 40 secs can also enhance free radical mobility and lead to a higher DC (Hofmann;Hugo and Klaiber, 2002; Price *et al.*, 2011b). Thus, the reason for the increased DC with the Brush and Sculpt and modelling resin groups was due to their internal components and irradiance intensity reaching the exposed surface

The Harmonize groups revealed different effects from the wetting resins on the surface values of the DC, largely due to the different internal components of these ILs. Also, the components of the treated RBC can cause a change in the DC due to the internal composition of the RBCs themselves (Ferracane, 2013; de Paula *et al.*, 2016). The multiple functional monomers of the Brush and Sculpt can led to an increase in the DC values on both setups lubricated surfaces of Harmonize. However, Modelling resin increased the DC only on setup two lubricated and non-lubricated surfaces because the unfilled monomer in this IL increases free radical mobility and the DC of the treated surface.

5.5.3 Martens hardness tests

The stability of the physical and mechanical properties of restorative materials is essential to the longevity of RBC restorations. These are usually subjected to complex conditions in the oral cavity which affect the longevity, strength and wear resistance of the RBC restorations (Fischer *et al.*, 2010). The hardness of such materials was measured in the current study to evaluate the surface changes that occurred in the RBC restorations due to different ILs. Also, hardness measurements can indirectly be used to evaluate the bottom/top ratio and determine the efficiency of the polymerisation process of the RBC materials (Ferracane, 1985; Hampe *et al.*, 2018). However, this method cannot give an actual quantitative value due to other influencing factors such as cross-linking. Even so, hardness measurements can still be used to evaluate the liner correlation between hardness and the DC in tested materials (Ferracane, 1985; Leprince *et al.*, 2012).

Many factors can affect the hardness values of the tested RBC materials. Some are internal factors such as the type of organic matrix, inorganic fillers, photoinitiators, and viscosity, while others are external factors, including the LCU irradiance intensity, radiant exposure, LCU tip position, and temperature. All these have a noticeable effect on the physical and mechanical properties of the RBC, especially surface hardness and wear resistance (Leprince *et al.*, 2013; Kelic *et al.*, 2016). In conjunction with these factors, the type of resin matrix, volume of filler loading, and filler size are other main causes of noticeable changes in the material properties of RBCs (Van Landuyt *et al.*, 2007; Leprince *et al.*, 2013; Ayub *et al.*, 2014; Strnad *et al.*, 2015; Alzraikat *et al.*, 2018; Yang *et al.*, 2019). Different hardness tests have been used to evaluate the surface hardness of RBC materials, such as Knoop, Vickers and Martens hardness tests (Shahdad *et al.*, 2007; Albino *et al.*, 2011; René *et al.*, 2015; Al-Zain *et al.*, 2017). In

the current study, Martens hardness was used to evaluate the effect of ILs on the treated surfaces' ability to avoid the disadvantages seen in other tests. This reflected the actual effects of specific ILs on the tested RBCs' surfaces. In this test, the depth of an indentation was measured following force applied for 30 secs from the diamond indenter onto the specimen surface. The indenter displacement represents the elastic displacement of the specimen surface and plastic depth of the indenter impression. Viscoelastic behaviour has many forms and is conveniently measured via creep and recovery phenomena. Thus, the Martens test eliminates the influence of the viscoelastic recovery of the RBC materials, whereas other tests' values can be affected by this property and by visual judgment (Shahdad *et al.*, 2007; Fischer *et al.*, 2010; Broitman, 2017; Ilie *et al.*, 2017).

The values collected from the specimen surfaces treated with different IL classes showed statistically significant differences compared to the control. The Martens hardness values of surfaces in setup one ranged between 304–631 HM for both types of RBC. The highest values were collected from the non-lubricated top surface and the lowest on the lubricated bottom surface. For setup two, surfaces showed values between 238–638 HM, with the lowest value being collected from the lubricated top surface and the highest from non-lubricated bottom surface. From this, it can be noted that the application of the ILs, regardless of whether the surface was top or bottom, had a negative effect on surface hardness. These values are within the range for comparable RBC values compared to previous studies using Martens hardness to evaluate the surface of different RBC materials (Shahdad *et al.*, 2007; Hampe *et al.*, 2018).

Regarding the solvents testing, applying different IL classes on both lubricated surfaces of the two different types of RBC materials revealed different effects. Filtek

was only affected by ethanol, which reduced the surface hardness of setup one lubricated bottom surface compared to the control group. However, none of the solvents used to the same surface of Harmonize caused statistically significant changes on this surface. The setup two lubricated top surface of Filtek was affected by using isopropyl alcohol, but no solvents affected Harmonize on the same surface. The use of organic solvents like ethanol can increase the DC of the RBC but cannot produce a high cross-linking density at the same time (Cadenaro *et al.*, 2009; de Paula *et al.*, 2016). This can significantly decrease the surface hardness values of the treated RBC materials. Also, filler size and their loading volume can affect the values when the material has a higher volume. The presence of these inorganic fillers increases the physical and mechanical properties, and they can reduce water uptake and its effects on the properties of the cured RBC restorations (Biradar and Arvind, 2012; Patel *et al.*, 2017). This could be the reason why both lubricated surfaces in both setups of Harmonize RBC with higher inorganic fillers loading were unaffected, even when the setup two lubricated top surface was treated with IL solvents, as the filler loading volume in Harmonize (64.5%) higher in comparison to Filtek (55.6%). The presence of more fillers in general can distribute the load through the tested specimens and increase the resistance to the applied load (Chan *et al.*, 2010; Ferracane, 2011; Rastelli *et al.*, 2012; Leprince *et al.*, 2013; Tuncer *et al.*, 2013; Alzraikat *et al.*, 2018; Bayraktar *et al.*, 2021).

All the tests of bonding agents as ILs indicated statistically significant effects on both lubricated surfaces of setups. Both these surfaces for Filtek and Harmonize had reduced surface hardness from the use of Scotchbond and Optibond. The presence of low-filled multifunctional monomers and organic solvents in the bonding agents can reduced the viscosity, increase free radical mobility, and raise the DC in the RBCs

(Asmussen and Peutzfeldt, 1998; Cadenaro *et al.*, 2009; Palagummi *et al.*, 2019). However, the presence of HEMA and contamination of the uncured resin with other components in the bonding agents can reduce the cross-linking density of the RBC materials and thus the surfaces' hardness values (Navarra *et al.*, 2012; Zanchi *et al.*, 2013). As mentioned before, the DC of the RBC materials is not the only factor that can enhance surface hardness, as the cross-linking density of the material and filler loading volume are important factors which improve the resistance of the material's surface (de Paula *et al.*, 2016).

The hardness values of setup two non-lubricated bottom surface for both RBC materials were higher than the setup two lubricated top surface, and even the setup two non-lubricated surface was the bottom of the specimens. Applying the IL on top surface increased free radical mobility, which may delay the vitrification stage (Leprince *et al.*, 2013). This delay can enhance how the light passes through the specimens and increase the DC and cross-linking on the bottom surface of setup two. The same effect have been produced when Optibond was applied on surface setup two lubricated top surface of Harmonize, both of which are produced by the same manufacturer; the homogeneity between the bonding agent and RBC monomers can be considered a contributing factor to the increase in the DC of the material during the polymerisation process (de Paula *et al.*, 2016).

With wetting resins, when placing RBC restorations, the literature has reported how solvents, bonding agents and wetting resins are used to reduce the sticking of materials to the placement instruments (Tuncer *et al.*, 2013; de Paula *et al.*, 2016; Patel *et al.*, 2017). In the current study, the survey conducted with UK dentists before the laboratory experiments showed that wetting resins were the second most used IL

after bonding agents. These dentists used wetting resins to manipulate and sculpt RBC materials and avoid tacking the materials to the placement instruments.

All the wetting resins used in the current study had either no or low filler loading. The only loaded wetting resin was Brush and Sculpt, loaded with 0.04-micron silica filler at 24% by volume. The other types were either unfilled or the filler loading could not be determined as the manufacturer does not define the main components, for example Signum liquid. Most of these materials have an opaque appearance when dispensed either on the surface of materials or placement instruments. The only IL which shared a common manufacturer as an RBC was the Modelling resin product, produced by the same manufacturer as Harmonize.

The wetting resins tests showed a statistically significant reduction of surface hardness for lubricated surfaces of both setups. Brush and Sculpt on Filtek caused the most reduction in Martens hardness values on both lubricated surfaces; this type was the only wetting resin with fillers. However, the opacity of this material may have played a role in reducing the DC and cross-linking density (Bakkal *et al.*, 2019). Also, Modelling resin and Signum had the same effects on setup two lubricated top surface but no significant effects on setup one lubricated bottom surface. The presence of these lubricants on unfilled resin-based materials have diminished the surface hardness values of the tested materials. As the ILs placed on the tested surface were low-filled or unfilled, this have decreased the hardness values collected because the amount of filler loading plays a major role in the material's resistance to the indenter on the universal testing machine (Ferracane, 2011; Leprince *et al.*, 2013; Tuncer *et al.*, 2013; Alzraikat *et al.*, 2018; Cangul *et al.*, 2020). Furthermore, other components of the IL could have contaminated the components of uncured RBC materials, interrupt and reduce the DC and cross-linking of the treated materials, and reduce the surface

hardness values (Martins *et al.*, 2015); however, this is hard to determine in every instance without the full details of the manufacturer's products. Thus, different reasons can cause reductions in the hardness values of treated surfaces, including the opacity of the used IL, a low- or unfilled IL, and other components of ILs that can contaminate and affect the DC and cross-linking of the treated uncured RBC.

5.5.4 Water uptake testing

All the RBCs and resin-based ILs used in the current project included organic and inorganic elements, which play essential roles in controlling the physical and mechanical properties and longevity of RBC restorations. The presence of the organic matrices of these materials can increase water uptake on exposure to fluids, affecting many important physical and mechanical properties and reducing the longevity of the RBC restorations. Greater water uptake may increase material discolouration, reduce wear-resistance and deteriorate the mechanical and physical properties of RBC restorations (Sideridou;Karabela and Bikiaris, 2007; Gonçalves *et al.*, 2009; Park *et al.*, 2011; Leprince *et al.*, 2013; Cornelio *et al.*, 2014).

The polymerisation process of the RBC depends on the conversion of the monomers of the organic matrix to polymers. The hydrophilicity of monomers in the RBCs used in the present project can increase the water uptake. The percentage of conversion of monomers does not reach 100%, and some unreacted monomers will leach into surrounding fluids (Peutzfeldt, 1997; Leprince *et al.*, 2013). This allows water to be absorbed and causes swelling stresses, and degraded bonds between the organic matrix and fillers. Consequently, the material's physical and mechanical properties will decrease and longevity will be affected (Cramer;Stansbury and Bowman, 2011; Lavigueur;Christine and Zhu, 2011; Wang;Zheng and Zheng, 2011; Mansouri and Zidan, 2018).

Regarding solvents, in the present study, both RBCs were Bis-GMA-based and nanofilled. When treated with solvents, they showed increases in the percentage weight change of water uptake compared to the control group. The water uptake pattern of each type depended on the filler loading volume (Sideridou;Karabela and Bikiaris, 2007; Aydinoglu and Yoruc, 2017). Harmonize has the highest filler load by volume (64.5%) while Filtek has (55.6%). The control group for Harmonize showed less water uptake than the same group for Filtek, suggesting that the difference in filler loading may explain some of the differences in water sorption.

Filtek was affected by using ethanol as an IL, and water uptake increased significantly compared to the control; it was also higher than all groups of both types of RBC. Previous studies have shown that ethanol increases water uptake and diffusion coefficients (Sideridou;Karabela and Bikiaris, 2007; Decky *et al.*, 2009), which is dependent on the presence of ethanol and the hydroxyl group of the Bis-GMA RBCs, as well as the cross-linking, amount of unreacted monomer, and porosity between the polymer networks (Sideridou;Karabela and Bikiaris, 2007; Cadenaro *et al.*, 2009).

Harmonize RBC showed higher water uptake when manipulated with IL solvents like distilled water, acetone, and isopropyl compared to the control group. These solvents plasticised the RBC and reduced the bonding between the organic and inorganic parts, which increases the chance of fluid uptake in the presence of the hydroxyl group of Bis-GMA (Sideridou;Achilias and Karabela, 2007). Also, the contamination of the uncured materials by using these solvents as ILs can produce more unreacted monomers, and again increase the water uptake of this RBC (Loos *et al.*, 2008; Martins *et al.*, 2015; de Paula *et al.*, 2016; Patel *et al.*, 2017; Carvalho *et al.*, 2019). The adding Bis-EMD as one of the components of Filtek increased the conversion of this type of RBC and the aromatic groups of the Bis-EMA (Cornelio *et al.*, 2014), leading to a

decrease in the amount of leached unreacted monomers and less water uptake. This conclusion explains why Filtek was only affected by ethanol, while Harmonize was affected by multiple solvents.

For the bonding agents, the details provided by the manufacturers showed a similarity in some components of the two types of bonding agents but also some differences. Water uptake was increased with the Filtek groups when this type was treated with either bonding agent. The presence of HEMA in both bonding agent types can increased the water uptake. Previous studies have discussed that HEMA which are more susceptible to water uptake (Zanchi *et al.*, 2013; Tauscher *et al.*, 2017). The hydrophilicity of this material due to the presence of the hydroxyl group in their chemical structure could therefore increase the water sorption of the RBC. Similarly, the presence of other substances such as solvents like water/ethanol may contaminate placed resin materials and increase water uptake due to microporosity. The presence of methacryloyloxydecyl dihydrogen phosphate (MDP) monomer in Scotchbond can increased the water uptake, as this type of functional monomer has been shown to have an adverse effect on the DC. It can affect the CQ photoinitiator and hinder the conversion of monomers to polymers (Matsui *et al.*, 2015; Hanabusa *et al.*, 2016; Carrilho *et al.*, 2019), and then increase the number of unreacted monomers, the leaching of monomers, and water uptake (Rahim *et al.*, 2012).

Only Scotchbond-treated specimens showed a significant increase in water uptake for Harmonize as no effects were seen in the Optibond group. The presence of hydrophilic monomers and the bonding agent solvent increased the possibility of water uptake. Moreover, the functional monomer of the MDP of Scotchbond can reduce the conversion of monomers to polymers, negatively affecting the RBC by increasing the water uptake and reducing the physical and mechanical properties. However,

Optibond is about 15 % loaded with size 0.4-microns and has no MDP, and so the higher filler load volume could be the reason for lower water uptake in this group. Since the RBC and the bonding agent shared a manufacturer, this may have had an impact, but the filler load of Harmonize resin and Optibond can play a significant role in the current situation.

For the wetting resins tests, Filtek's water uptake increased significantly when Modelling resin was used to manipulate this RBC. Although the components of the Modelling resin were not given in detail by the manufacturer, they did state that it is an unfilled and resin-based material. Placing a layer of Modelling resin between the increments would thus increase the pattern of water uptake. Increasing the unfilled amount in this area can cause more diffusion of the water between the increments and increase the water uptake (Biradar and Arvind, 2012; Rahim *et al.*, 2012; Patel *et al.*, 2017).

The use of ILs like Modelling resin and Signum significantly increased water uptake when applied between increments of Harmonize. Both these ILs are unfilled and resin-based materials that can increase the water uptake pattern between increments of RBC, as explained previously. Lower filler load volume can also provide a suitable area for increased water uptake (Biradar and Arvind, 2012), and over a long time interval degrade the RBC's organic matrix and the bonding between the organic and inorganic elements (Liebermann *et al.*, 2017). Contamination of RBC materials by different IL components can also reduce the DC of resin-based materials by interrupting the conversion of monomers to polymers. The ILs themselves have different components and some are unknown, potentially hindering the conversion process and affecting the density of the cross-linking. This can diminish the properties and longevity of RBC restorations (de Paula *et al.*, 2016).

5.5.5 Diametral tensile strength testing

In the current study, the experimental groups were treated with different ILs to reduce the sticking of the RBC materials to dental instruments, and then compared to controls prepared without ILs. The results for each RBC showed different behaviours due to the different components of the matrix monomers and the filler load by volume of Filtek and Harmonize. The DTS values of the Filtek RBC at the baseline interval of all experimental groups were higher than Harmonize RBC. The values of the DC of the Filtek were higher than the Harmonize as shown in the previous section. Filtek has more matrix monomers like UDMA and PEGDMA that have been shown to produce higher conversion from monomers to polymers. (Zhang *et al.*, 2005; Barszczewska-Rybarek, 2009; Gonçalves *et al.*, 2009). That is because those monomers have low viscosity and increase the mobility of free radicals. Also, the Harmonize contains more filler compared to the Filtek RBC. That can make a difference in the DC by restricting the movement of the free radicals and reducing the amount of monomers' conversion to polymers. Also, that can reduce the amount of light going through the RBC by reflecting the LCU light (Leprince *et al.*, 2013). All these different reasons can affect the RBC properties and the DTS values of the tested materials. However, from the current study results, the DTS results of Harmonize groups treated with ILs throughout the time intervals was less effected when compared to the control group. The presence of more fillers can make a difference in the amount of the water uptake, which can reduce the amount of water uptake throughout time intervals. So, the negative effects on DTS values of Harmonize groups due to the use of ILs was lower than the Filtek groups throughout the time intervals compared to the control groups.

The values for Filtek ranged between 22–51 MPa and for Harmonize 22–45 MPa, while the literature has reported variation in DTS values for hybrid and nanocomposites of

between 38–96 MPa (Alzraikat *et al.*, 2018). It can be noted that the values in the literature are higher than in the current study, especially the minimum values. Also, the reported values of DTS collected from RBC treated with ILs, ranging between 32–53 MPa, are higher than the current study's values (Patel *et al.*, 2017). The presence of different ILs between increments of RBC materials can play a role in this by increasing water uptake and decreasing the DTS. Also, the specimens in this study were stored in distilled water for 90 days, and so it is important to consider this factor in the process of aggravating the reduction of DTS values in the experimental groups (Sideridou;Karabela and Bikiaris, 2007; Rahim *et al.*, 2012; Patel *et al.*, 2017).

With the tests of solvents, all the ILs used in the current study produced statistically significant differences in comparison to the control groups for Filtek, which was most affected by the use of solvents. The reduction of DTS in all stored experimental groups across the time intervals was expected and confirms results from previous studies (Sunbul;Silikas and Watts, 2016; Patel *et al.*, 2017). The solvents can increase the RBCs solubility and affect the connection between their main components and lead to increase water uptake. This enhances debonding between the matrix and fillers, as shown in the literature (Sideridou *et al.*, 2004; Sideridou;Achilias and Karabela, 2007). The presence of solvents like ethanol, acetone and isopropyl in between the increments of the RBC materials can produce microporosities, which increase the possibility that the formation areas will become fracture points. Also, the contamination of uncured increments with water reduces the DC and cross-linking density and decrease the DTS of the RBC materials (Loos *et al.*, 2008; Martins *et al.*, 2015; Carvalho *et al.*, 2019). On the other hand, Harmonize showed no significant reduction in DTS values, which related to the higher filler loading volume compared to Filtek. Higher loading is an essential part of enhancing RBC materials not only by increasing

their strength and wear resistance, but also by reducing water uptake and its negative consequences for the strength and longevity of RBC restorations (Sideridou;Achilias and Karabela, 2007; Aydinoglu and Yoruc, 2017).

With DTS testing in the bonding agents groups, statistically significant reductions were seen for both bonding agents for the same RBC across all time intervals of storage in distilled water compared to the control. This due to Filtek's resin matrix and HEMA being part of the bonding agents, which may increase water uptake. Also, other components of the bonding agents can contaminate the uncured resin and affect the DC of the RBC materials by producing microporosities (Loos *et al.*, 2008; de Paula *et al.*, 2016; Carvalho *et al.*, 2019). These factors reduced the values of the DTS for all time intervals of the study.

Harmonize treated with both bonding agents as ILs showed no statistically significant differences compared to the control. The high filler loading in this type of RBC may have a primary role in this scenario and reduced the effects of the ILs on the properties of Harmonize (Della Bona *et al.*, 2008). The values for the bonding agent groups at baseline were higher than the control, possibly because of the presence of the unfilled resin in the bonding agents. This resin can enhance the mobility of the free radicals and increase DC, and thus increase the DTS (Leprince *et al.*, 2013). However, the values started to diminish across the time intervals and the values of the bonding agent groups became lower than the control, although not significantly. This reduction in values is caused by the increased amount of unfilled resin between the increments, which increases the water uptake and hydrolysis of the bonding between the organic and inorganic components of the RBC materials (Patel *et al.*, 2017; Tauscher *et al.*, 2017).

The behaviour of both RBCs was different with each bonding agent used as an IL. Regardless of the manufacturer and type of bonding agent used, the main components of the RBC materials seem to effect changes in the DTS and other physical and mechanical properties.

With the DTS testing of the wetting resins, the Brush and Sculpt used with both RBC types showed higher values at the baseline than the control groups. The type of resin matrix of this wetting resin and its fillers could be the reason for the high DTS values compared to the control at baseline (Leprince *et al.*, 2010). The low load resin matrix of the IL can be a good source of free radicals by delaying the gelation stage and vitrification (Leprince *et al.*, 2013). In turn, this reduces the viscosity of the RBC materials and increases the DC of the RBC. Fillers, even in a low amount, may increase the DTS values by increasing the amount of support for the resin matrix of the RBC materials. Signum Liquid has unfilled resin containing dimethacrylate and multifunctional methacrylic acid ester, but there were no manufacturer details regarding the presence of fillers.

The Modelling resin was the only wetting resin to show a statistically significant reduction in the DTS values across the time intervals for both Filtek and Harmonize. The only justification for this behaviour is that this type of IL is an unfilled resin, which can be a good area to increase water uptake, especially between the increments of the RBC materials (Patel *et al.*, 2017; Tauscher *et al.*, 2017). The increased water uptake aggravates the stress on the bonding between the organic and inorganic components of the RBC material, leading to hydrolysis, weakening of the material, and reduced physical and mechanical properties (Cramer;Stansbury and Bowman, 2011; Mansouri and Zidan, 2018). Although the Modelling resin used in the current study was

manufactured by the same manufacturer as Harmonize, this made no difference and was statistically significantly different compared to the control over time.

Drawing from the above discussion of the experimental work, there are links between the tests in some situations. For example, the increased water uptake in the bonding agent groups for Filtek also decreased the DTS values. In other words, the bonding agents had an adverse effect on both water uptake and the strength of the tested RBCs. Certain components of both bonding agents, like HEMA, and of the solvents, like ethanol, can increase the water uptake in the areas treated area with. The literature has shown that the substances like those can affect the amount of water uptake (Van Landuyt *et al.*, 2007; Sideridou;Karabela and Vouvoudi, 2008; Sideridou and Karabela, 2011; Zanchi *et al.*, 2013). Also, the ageing of the treated RBC groups in the current project compared to the control groups showed significant differences in water uptake and DTS. Consequently, the presence of these substances in the bonding agents and their use as part of the ILs can increase water uptake in, and reduce the strength of, the treated RBC. This leads to degradation of the bond between the organic and inorganic components and diminishes the strength of the RBC (Patel *et al.*, 2017).

The DC increased in most of the experimental groups for both RBCs. Only DW and Signum Liquid negatively affected the degree of conversion of Filtek. In general, increasing the DC of RBC can improve the physical and mechanical properties of the polymerised materials, and reduce the possibility of water uptake in the RBCs when exposed to fluid (Leprince *et al.*, 2013). Low cross-linking may influence the properties of RBC materials, even if there is a high DC. From this, it can be concluded that the DC is not the only essential factor in achieving better physical and mechanical RBC properties, as the cross-linking network formation also plays a major role. In some of the experimental work in this project, an increase in the DC does not necessarily reflect

the level of formation in the cross-linking network. For example, the presence of HEMA in the ILs can enhance polymerisation but only in the linear chain because it contains mono-methacrylate and not improve cross-linking intensity in that area (Van Landuyt *et al.*, 2007; Araújo-Neto *et al.*, 2018). In this way, the physical and mechanical properties of the treated RBC reduced in comparison to the control groups. Filler volume loading also played a vital role in this respect alongside DC in creating different values across the tests.

All the experiments in the current project tested three classes of ILs. Different types of bonding agents and wetting resins were selected according to the UK dentists' survey discussed in Chapter 4. Solvents were not selected in this survey but were included in the current project because of their use in previous studies testing the effects of ILs on RBCs (Sneed and Draughn, 1980; Tjan and Glancy, 1998; Gorge Perdigao, 2006; Dunn, 2007; de Paula *et al.*, 2016; Patel *et al.*, 2017). The presence of organic solvents in the bonding agents made it also essential to clarify and understand the specific effects of solvents on the tested RBCs. Since water is present in bonding agents, it was also important to determine its effects separately on both tested RBCs. In general, the tests were performed to determine the effects of these fluids when they contaminate uncured resin and the subsequent adverse effects on the physical and mechanical properties of the two tested RBCs.

Several important factors were considered when selecting the bonding agent ILs. One of these was the type of bonding system, and the two-step systems selected for this study were chosen based on the survey responses of the UK dentists. Another factor was the manufacturer of the bonding agents, and these were selected so as to be the same as the tested RBCs. Each type of bonding agent was tested on its co-manufactured RBC to test if there is any benefit from using a bonding system from the

same company. In the literature, this point has not been thoroughly discussed, with one study mentioning if this point makes a difference (Tjan and Glancy, 1998), and another selecting RBCs and bonding agent systems from the same manufacturer to standardise the products used (Patel *et al.*, 2017). In the current project, using bonding agents from the same or different manufacturers does not seem to have had a significant effect on the results, and indeed this is in line with the findings of Tjan and Glancy (1998). Generally, the main effect of using bonding agent on the tested surfaces was related to certain components within in, such as HEMA and solvents.

One of the present study's limitations, the specimen increment thickness was 3mm. The literature and manufacturing guidance suggested using 2mm increment thickness to increase the cured RBC physical and mechanical properties (Roopa;Usha and Vedhavathi, 2011; Narene, 2014). However, there are some studies that show that using 3mm would not significantly affect the RBCs' DoC and physical and mechanical properties (Mills;Jandt and Ashworth, 1999; Barreto;Gayosso and Ibarra, 2015; Rodriguez *et al.*, 2017; Hamouda and Almalki, 2020; Hoshino *et al.*, 2021). In the current study, the thickness of the increment was selected after considering the DoC of tested RBC materials, which were both found to be within 3mm. Additionally, the increments were cured using light at an intensity of 1,020 mW/cm^2 for 40 s. The use of LCU with higher intensity and longer exposure time can make the DoC higher, as shown in the literature (Mills;Jandt and Ashworth, 1999; David *et al.*, 2007; Hegde;Hegde and Malhan, 2008; Kassim *et al.*, 2012; Leprince *et al.*, 2013; Rodriguez *et al.*, 2017; Lempel *et al.*, 2019; Hamouda and Almalki, 2020). Consequently, even with this increment thickness greater than the thickness advised by the manufacturers, the data presented in this chapter allow the effect of the different ILs to be measured.

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Another limitation of the current project is the effects of the ILs were detected only at the surface of the treated specimen. The current study methodology involved applying the ILs on the outter layer of treated RBC. This technique would help standardise the amount and the area of the ILs application throughout all prepared specimens. However, this does mean that it was not possible to measure the effects of the ILs at deeper levels . Previous studies have considered the effects of different factors like LCU intensity on the DoC, DC and microhardness at different levels (Mills;Jandt and Ashworth, 1999; Hegde;Hegde and Malhan, 2008; Leprince *et al.*, 2012; Barreto;Gayosso and Ibarra, 2015; Rodriguez *et al.*, 2017; Hamouda and Almalki, 2020; Hoshino *et al.*, 2021). However, there were no previous studies investigating the effects of the ILs on physical and mechanical properties at different depth points compared to the control group. So, it should be acknowledged that the effects of ILs at the deepest levels have not been explored. While, investigating this matter can provide more valuable data to determine if that would affect different levels the present study focussed just on the effects of ILs on surfaces.

The DTS test was introduced to test materials under uniaxial compressive load but has some limitations. Compared to the axial tensile strength test, the DTS distribution of the load and the centre of the tensile strength can be affected by the surface of the specimens prepared (Penn;Craig and Tesk, 1987; Darvell, 1990; Sood;Ramarao and Carounanidy, 2015). The DTS was utilised to measure mechanical strength until the fracture point. This test was used in literature to detect the effect of the ILs in general on the strength of tested specimens (Patel *et al.*, 2017). The DTS test in the current project was a workable tool to compare the collected data to the previous data. Specimen with a simple cylindrical shape was used instead of a more complicated one in other test like tensile strength. This simple protocol was used in the literature to test

the effects of different factors on the strength of the tested RBCs (Casselli *et al.*, 2006; Della Bona *et al.*, 2008; Alrahlah, 2018; Medikasari, 2018). That would make the effects of ILs on the strength of the different treated RBCs more understandable. So, the obtained data from DTS helped evaluate and analyse the general impact of the ILs on the strength of treated RBCs. However, this test was not enough to determine the impact of ILs on the bonding strength at the interface area between the increments. So, the microtensile has been conducted in the next chapter and uses both chapters' data to analyse the impact of ILs on the treated RBC materials.

5.6 Conclusions

- The solvent tests showed the most significant, distinct opaque demarcations at the interface of the increments, but the wetting resins tests revealed no significant visible changes;
- Regarding the degree of conversion of the treated surfaces of the RBC materials, all the IL classes either increased or decreased the DC. This is linked to the components of both the RBC and the ILs in relation to the RBC surface and the tip of the light-cure unit;
- The selected ILs affected the hardness values of the treated surfaces, due to their components and the main components of the RBC materials;
- Different IL classes caused a significant increase in the water uptake of the RBCs. The internal components of both the RBCs and ILs played a major role in the values collected;
- The components of the ILs and their compatibility with the RBC can affect setting reactions, leading to changes in the physical and mechanical properties and longevity of RBC materials;

- The presence of the ILs between the treated RBC increments increased water uptake and diminished the diametral tensile strength values of both tested RBCs. The ILs increased water uptake and diminished the strength of the tested RBCs;

Overall, these laboratory investigations on the effects of ILs on the physical and mechanical properties of RBC materials had significant effects on the properties tested. That can increase their negative effects on the stability and longevity of the RBC restorations. The degree of effect is related to the main components of both the ILs and the treated RBCs.

Chapter 6: The Effects of Instrument Lubricants on Adhesive Strength between Resin-Based Composite Increments

6.1 Introduction

One essential factor for RBC restoration stability and longevity is the strength of the material, especially in posterior areas (Leprince *et al.*, 2013). The strength of different RBC materials has been tested under different circumstances, and diametral tensile strength (DTS) testing has been employed in previous studies and in the current project. However, from the limitations that were discussed in chapter 5, the use of the DTS test would not be the correct to test the effects of ILs between the RBC increments with a direct tensile load. Testing specific areas of the tested specimen needs to use the test that would help to focus the load on the tested site. So, instead of the DTS, the microtensile test is commonly used in dentistry to evaluate tensile bonding strength between different substrates. Tensile strength is essential as its lack can lead to the failure of the restorative materials. (Armstrong *et al.*, 2017; Ilie *et al.*, 2017), and so testing it is most appropriate for the evaluation of RBC properties (Münchow *et al.*, 2016). In the current study, microtensile testing was adapted the methodology with some modifications on the specimens and attachment apparatus design to evaluate the effects of ILs between the increments of RBC materials. Such testing focused on the tensile strength of the tested area. So, that can help to evaluate the effects of the ILs on the bonding strength in that specific area. Also, can improve the understanding of how use these materials impact strength and longevity of treated RBC.

6.2 Materials and methods

6.2.1 Specimen preparation

Filtek Supreme XTE RBC in shade A2 and three different classes of instrument lubricant, solvents, bonding agents, and wetting resins, were investigated. Each class was divided into groups representing the different types of lubricants and a control group prepared without IL. Pilot tests were conducted and the power calculations for the number of specimens were performed, and the level of significance was looking at (5%) and the power (80%) (Minitab 18.1, Minitab, Inc., United States). Two blocks were used to produce twenty specimens for each group representing one type of lubricant.

Daylight from windows and the lab lights were controlled during specimen preparation to limit the light received by each specimen. A mould was designed using CAD (Autodesk Inventor Professional 2018, Autodesk Inc., USA), and then printed out in photopolymer resins with a 3D printer (Form 3, Formlabs Inc, USA). The mould had four layers, each with a thickness of 2.5 mm, and a frame to keep all the layers in place when the RBC was inserted, as presented in Figure 6-1. The mould's dimensions were 10x10x10 mm, to achieve a cube-shaped block of RBC which was then sectioned into stick-shaped specimens with cross-sections of 1± 0.1 mm and lengths of 10± 0.1, as illustrated in Figure 6-2.

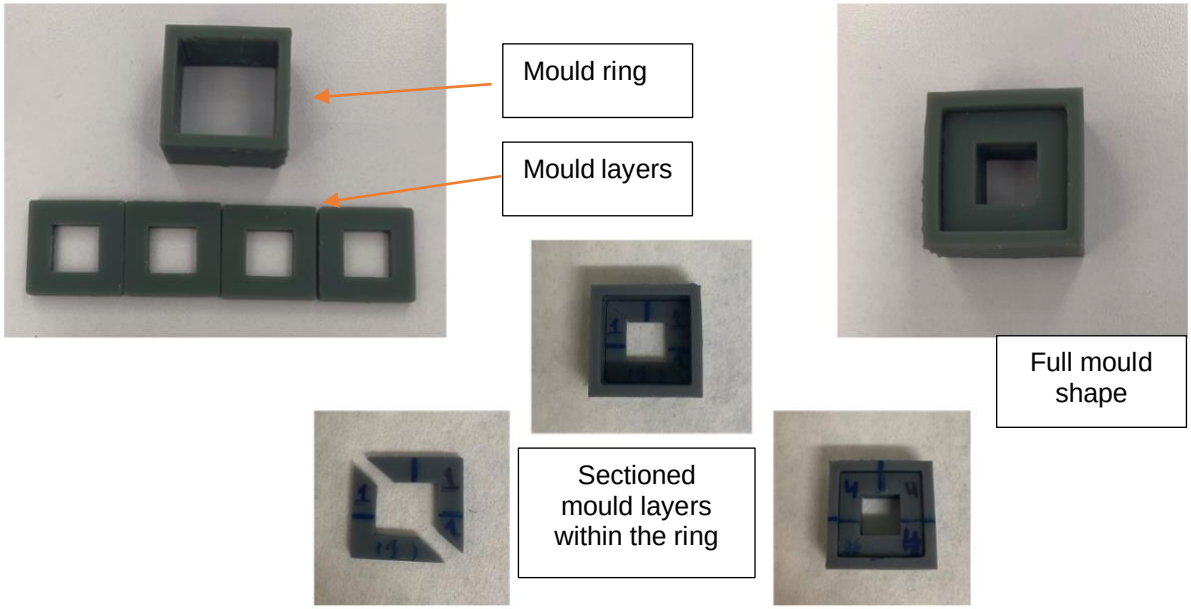


Figure 6-1: The final shape of the mould's layers and holding ring

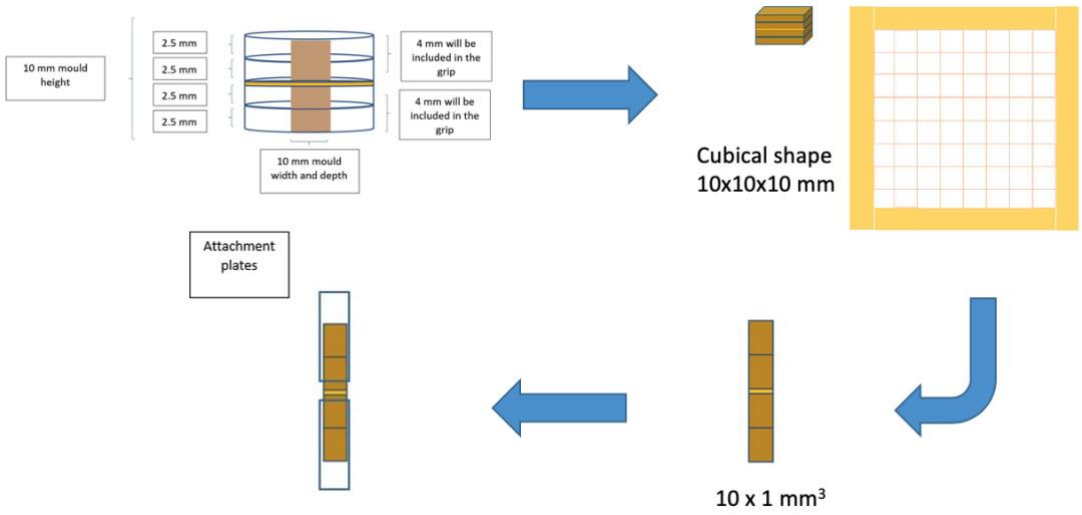


Figure 6-2: The design and preparation process for producing the blocks and specimens for the microtensile test

One operator performed all the specimens' manipulation and curing. A glass slide and Mylar strip were placed beneath the mould to support material during placement, and a Mylar strip was placed above to prevent the formation of an oxygen-inhibited layer. A plugger with tip dimensions 2.25 mm x 3 mm height was used to place the material

into the mould and no IL was used. Another plugger was used to apply the ILs, after being immersed into each respective lubricant for 1 sec and left to drip/drain for 2 secs. The ILs were only applied between the second and third increments to measure the effects of ILs on a specific area of the specimens. Each increment was polymerised for 40 secs with an LED LCU (Elipar™ DeepCure-S, 3M ESPE, 3M Deutschland GmbH, Germany). The irradiance of the LCU was measured for 40 secs using a Bluephase Meter II (Ivoclar Vivadent Limited, UK), and it was found to be 1,020 mW/cm^2. The tip of the LCU was covered with a protective sleeve and then the LCU held as close to the mould as possible. After removing the Mylar strip, the second to fourth increments were placed and cured, respectively. The build-up of the block layers is shown in Figure 6-3 for each layer during block preparation.

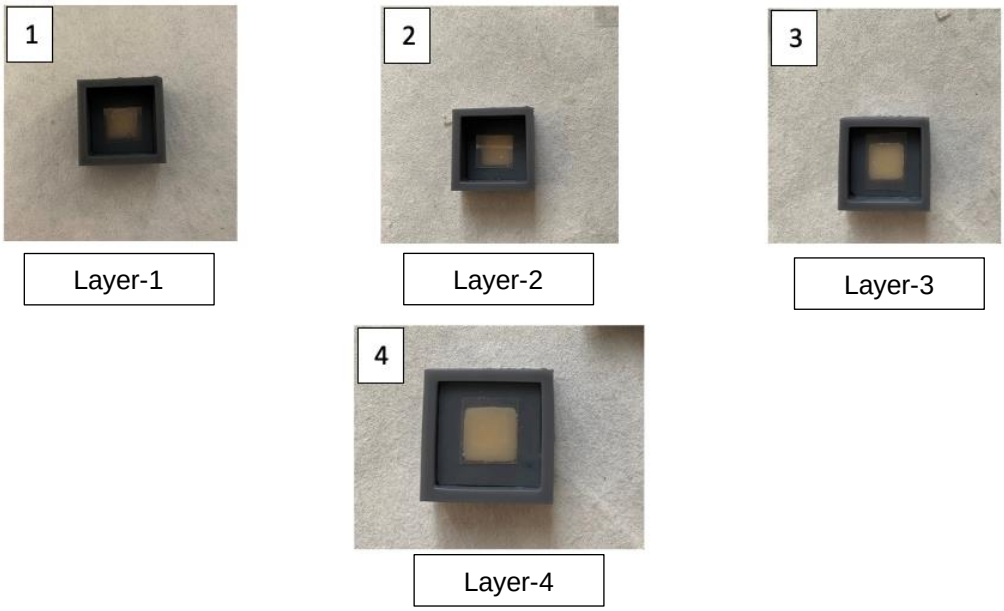
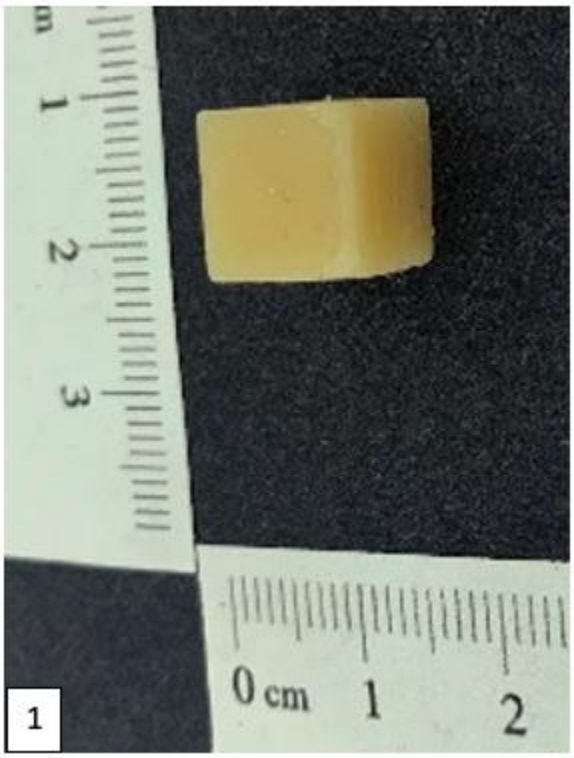


Figure 6-3: Steps in building up the block using the multiple layer mould; four layers (1-4) were filled with RBC and each cured with the LED LCU

The final block shape is presented in Figure 6-4(1). The prepared blocks from each group were tested at baseline and after one week. Blocks were placed separately into a small glass storage bottle with a sealed, stoppered lid in an incubator at 37±1°C. The

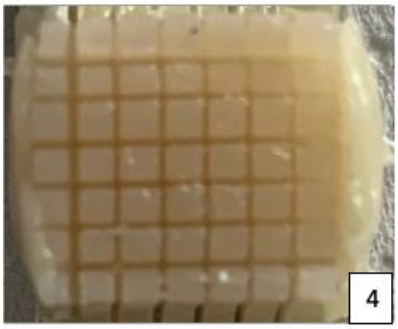
blocks were sectioned after 24 hours for the baseline groups and then tested. Day 7's blocks were stored in 10 ml of DW in glass bottles as previously described, until they were removed for sectioning and testing. Each block was firmly stabilised with sticky wax as shown in Figure 6-4(2) on the holding part of the mechanical arm of the low-speed saw. Each time, the mechanical handle of the saw moved within the required thickness of 1 mm. The saw was placed on the block surface to be perpendicular to the interface of the block layers, as shown in Figure 6-4(3), and then it followed both axes of the blocks to produce specimens with the required dimensions and shape. At least ten stick-shaped specimens from each block were produced, as presented in Figure 6-4(4–5), and all were measured with callipers and inspected under the microscope to ensure they were free of defects. The dimensions were double-checked to ensure the specimens were within the required cross-section and length (Figure 6-5). Specimens with no defects and of the required dimensions were then placed in DW in preparation for the microtensile test.

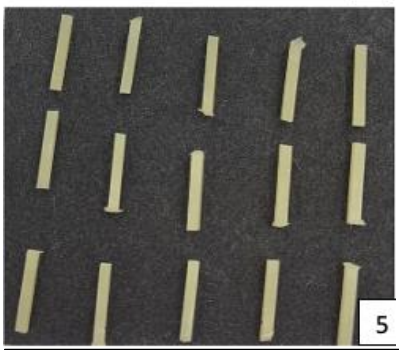


The built-up RBC block

Block attached to the saw arm to begin sectioning

The low-speed saw used to section the RBC block





The sectioned block and stick-shaped specimens

Figure 6-4: (1) The built-up RBC block; (2) Block attached to the saw arm to begin sectioning; (3) The low speed saw used to section the RBC block; (4) & (5) the sectioned block and stick-shaped specimens

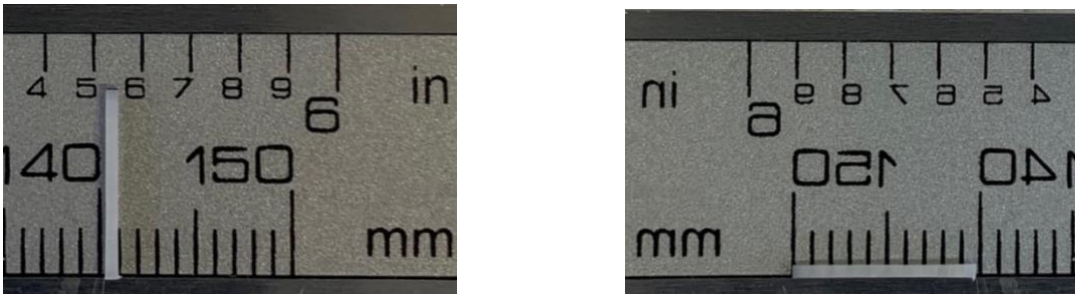
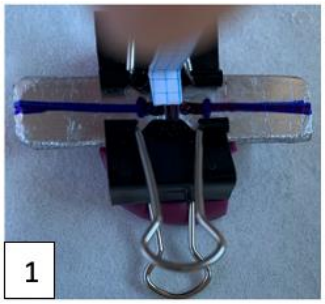


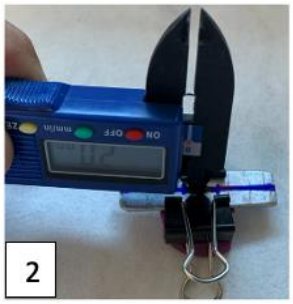
Figure 6-5: Size of the stick-shaped, sectioned RBC specimen

6.2.2 Microtensile testing

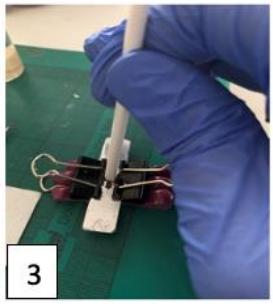
After sectioning, inspecting, and measuring 20 specimens, those for the baseline were tested after 24 hours. The specimens stored in DW for seven days were sectioned, inspected, and tested on the same day; they were removed from the storage container with a tweezer and placed on the attachment pieces prepared in advance from aluminium plates of dimensions 10 mm width x 20 mm length x 2 mm thickness (Metal Mania, Leicestershire, UK). Multiple reference points were used on the attachment apparatus parts to ensure that 4 mm of the specimens' ends were on each attachment part. Both parts of the attachment were secured to make one apparatus using paper clips on both sides. The space between the attachment parts was checked with different methods to ensure it was 2 mm, as illustrated in Figure 6-6. The attachment pieces and paper clip position were then secured using medium-bodied a monophase-regular set silicone impression materials (Aquasil ultra monophase, Dentsply, UK) to ensure the whole apparatus remained conjoined throughout the experiments. Excess impression material was removed using a sharp blade to avoid any interference when the specimen was attached to the Instron grips (Figures 6-7 and 6-8).



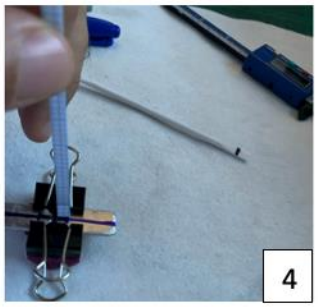
Graph paper to measure 2mm

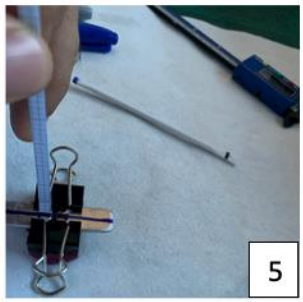


Digital caliper



Plastic tip with 2mm thickness





check and standardise 4 mm reference points on both attachment parts

Figure 6-6: Different tools used to check and standardise the 2 mm space between the attachment parts and the 4 mm reference points on both attachment parts

An adequate amount of the base of cyanoacrylate (Zapit, Dental Ventures of America, inc, USA) was applied to the two attachment pieces to ensure the glue was away from the 2 mm space between the attachment pieces. The specimen was then placed in an upright position using the reference marks for this purpose, as shown in Figure 6-7. The cyanoacrylate base was activated by spraying the accelerator on both spots separately. After the glue was set, the specimen glued to the attachment pieces was placed and secured in the grips of the Instron testing machine. After the attachment pieces were gripped tightly, the paper clips were removed to avoid any effects on the testing machine's reading. The specimens were stressed at crosshead speed 1 mm/min until the specimen fractured. At least 20 specimens from two blocks were tested and values collected for analysis. From previous studies and the pilot test, the means and SD were used to calculate the power and sample size at 80% and p-value

<0.05. The sample size was about 18 specimens/group. Therefore, 20 specimens, at last, were prepared without defects by checking them under the microscope to ensure this number would be enough. These steps are summarised in Figures 6-7 and 6-8.

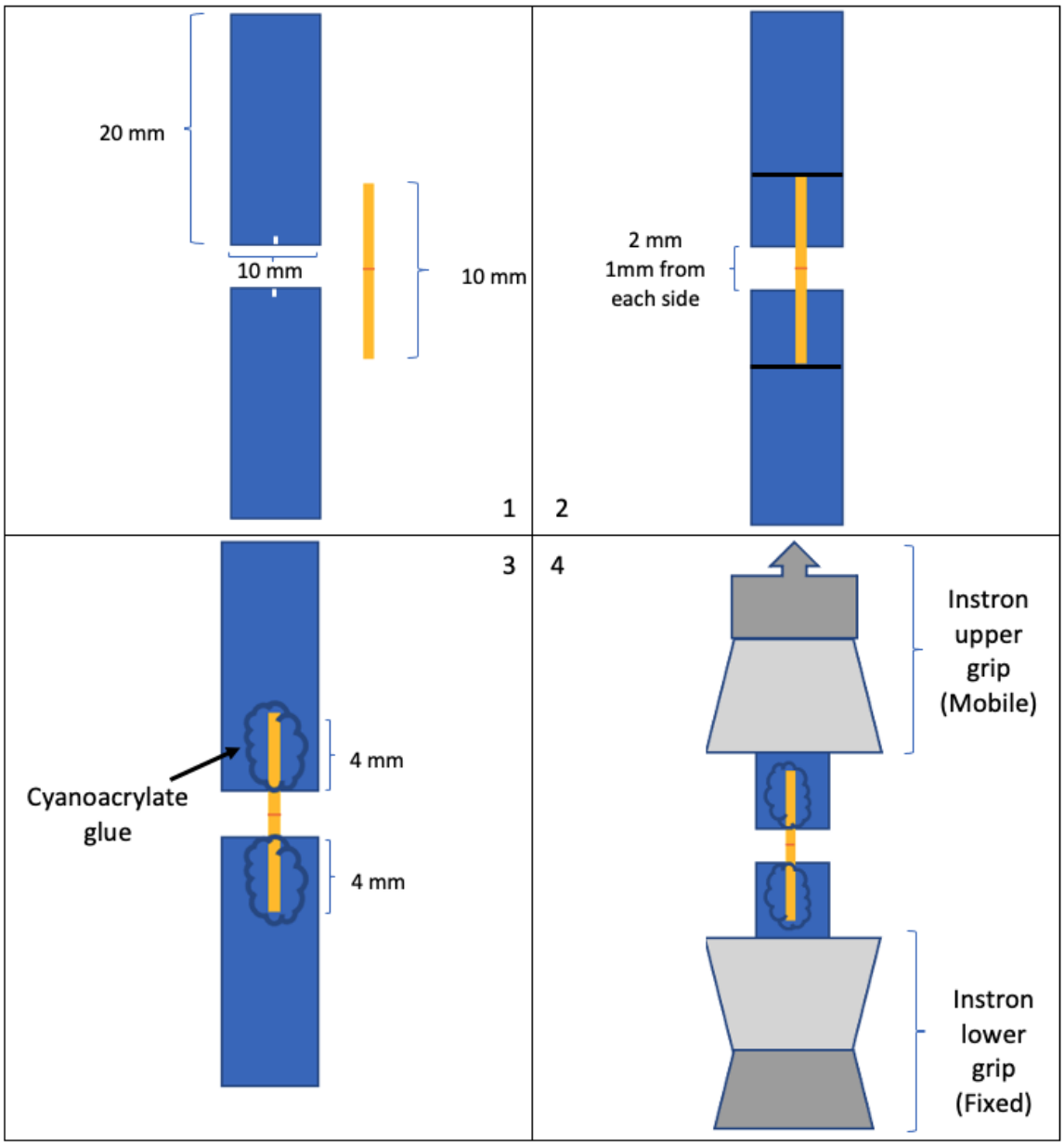


Figure 6-7: The reference points and the position of the tested specimens, and the area glued with cyanoacrylate glue

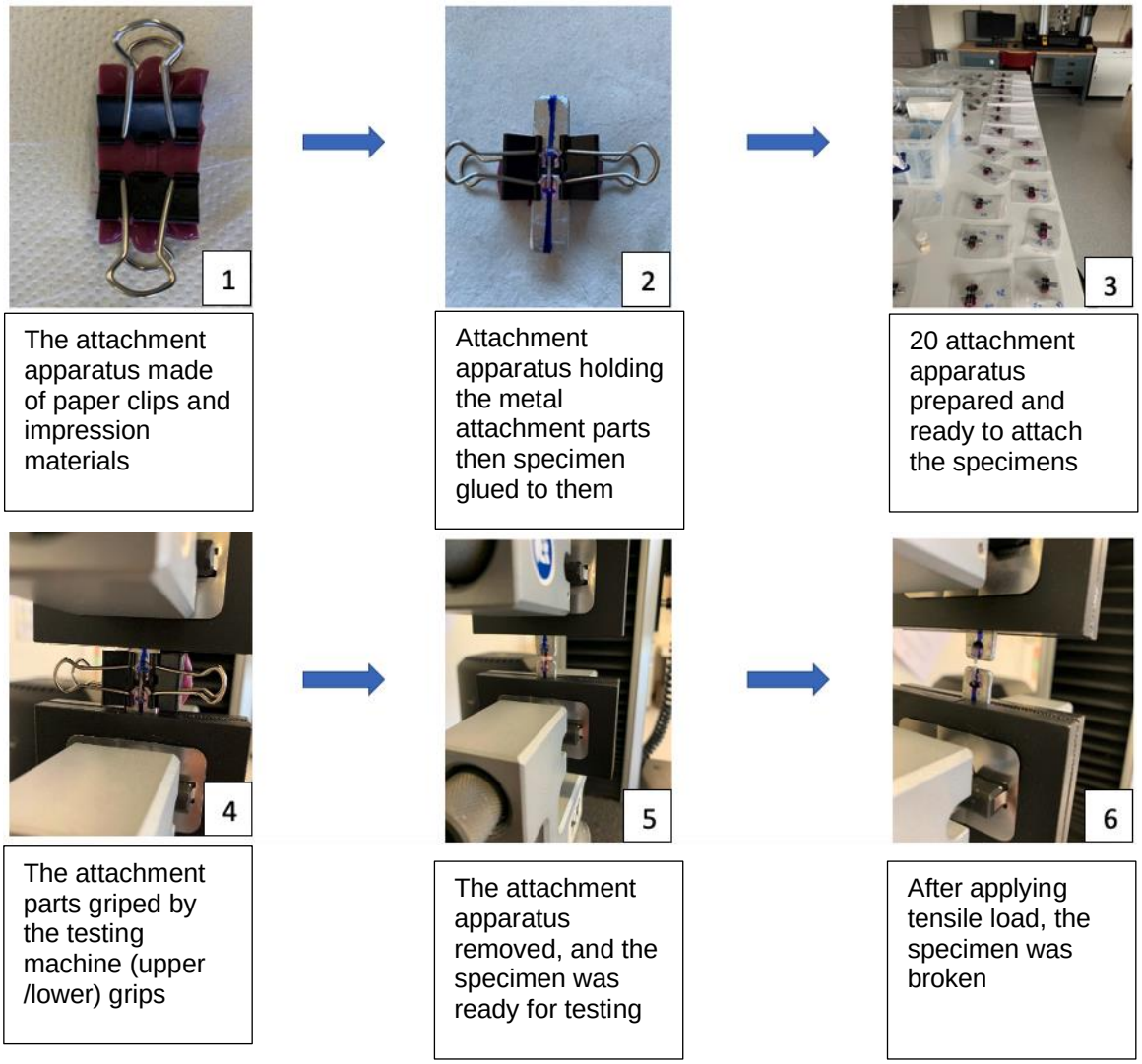


Figure 6-8: The attachment pieces and specimen testing steps

6.2.3 Statistical analysis

After obtaining all the values, the relationships between the groups were analysed using descriptive statistics. ANCOVA-univariate (SPSS 25 for Windows, IBM SPSS Inc., USA) was used to analyse the statistically significant differences ($p<0.05$) between the control and all the groups treated with ILs. Then, pairwise comparisons were made using the Tukey test to control the overall significance level at 5%.

6.3 Results

All experimental groups for the different IL classes were analysed for the experimental time intervals. The statistical analysis of the results was performed using the ANCOVA-univariate test. The *post hoc* test Tukey was used at the level of (p<0.05) to test the difference in the experimental groups. The mean of the microtensile strength and standard deviation values of all time intervals were calculated, summarised, and placed in figures to clarify the results of each IL class. In comparison to the RBC control group, some groups treated with ILs showed changes in the microtensile strength values, according to the type of IL used.

6.3.1 Solvents

The data collected from the solvents' groups showed statistically significant differences (p<.001) at baseline and after storage in DW for seven days. In comparison to the control group, all the solvent ILs reduced the microtensile strength values between the increments of Filtek. Isopropyl impacted these values the most, followed by ethanol and acetone. Distilled water caused a statistically significant reduction in comparison to the control, but its impact was less than the other above mentioned organic solvents. From these results, the solvents class produced noticeable effect on microtensile strength and it was more after storage for seven days, as presented in Figure 6-9.

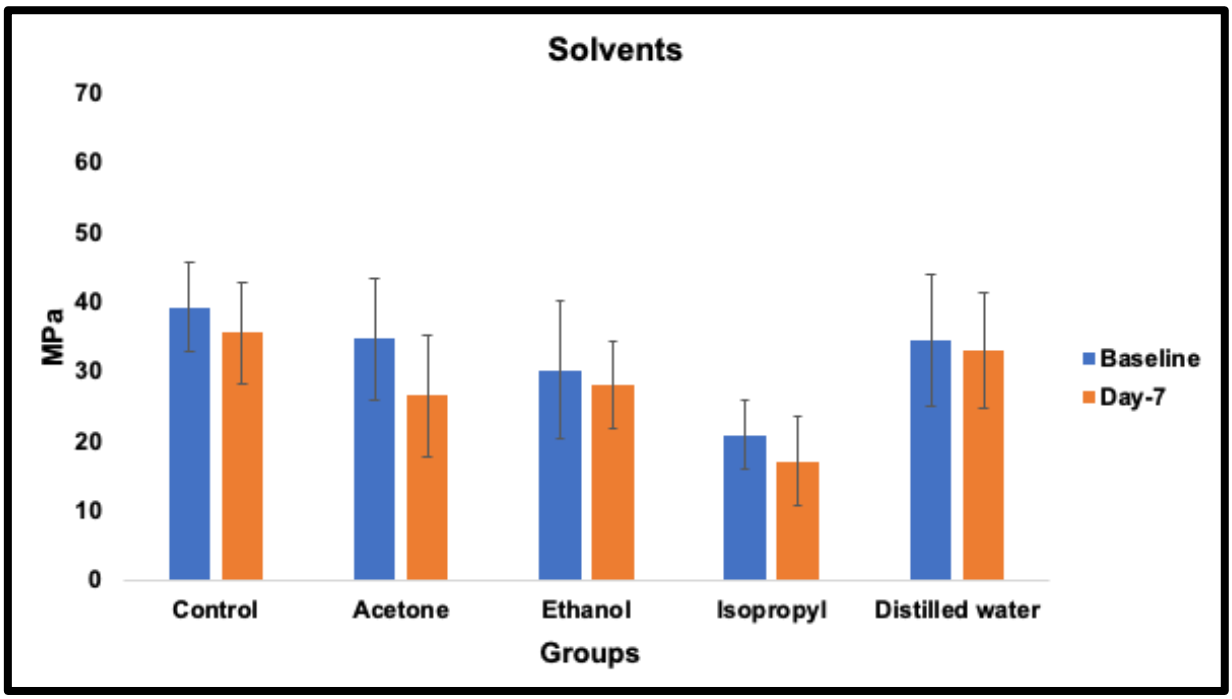


Figure 6-9: Mean and standard deviation values for the microtensile strength of Filtek RBC treated with solvents as ILs

6.3.2 Bonding agents

When bonding agent ILs were applied between the RBC increments, their effects produced statistically significant differences compared to the control (p<.001) for all time intervals. The Optibond bonding agent system registered the lowest value, followed by Scotchbond, but both of them reduced the microtensile strength and the effect was more after specimens storage in DW (Figure 6-10).

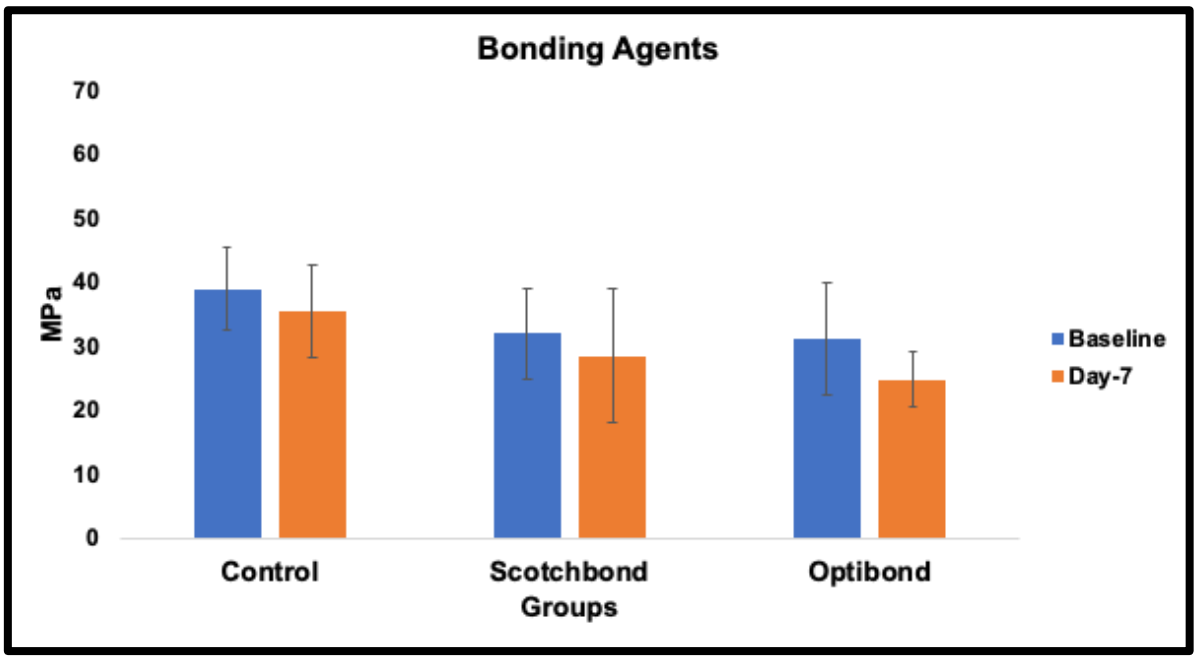


Figure 6-10: Mean and standard deviation values for the microtensile strength of Filtek RBC treated with bonding agents as ILs

6.3.3 Wetting resins

When wetting resins were used as ILs between Filtek increments, they showed statistically significant differences between the groups (p<.001) compared to the control group, as presented in Figure 6-11. However, none of the wetting resins reduced the microtensile strength of the tested groups. All treated groups with WR showed higher values in comparison to the control group that was prepared without ILs across the time intervals at baseline and day 7. The values of the tested specimens after storage in DW also were less than the baseline groups. Brush and Sculpt produced the highest values, followed by Signum and then the Modelling resin. The reduction in strength across the time intervals was higher in Brush and Sculpt and Signum, but not in the modelling resin.

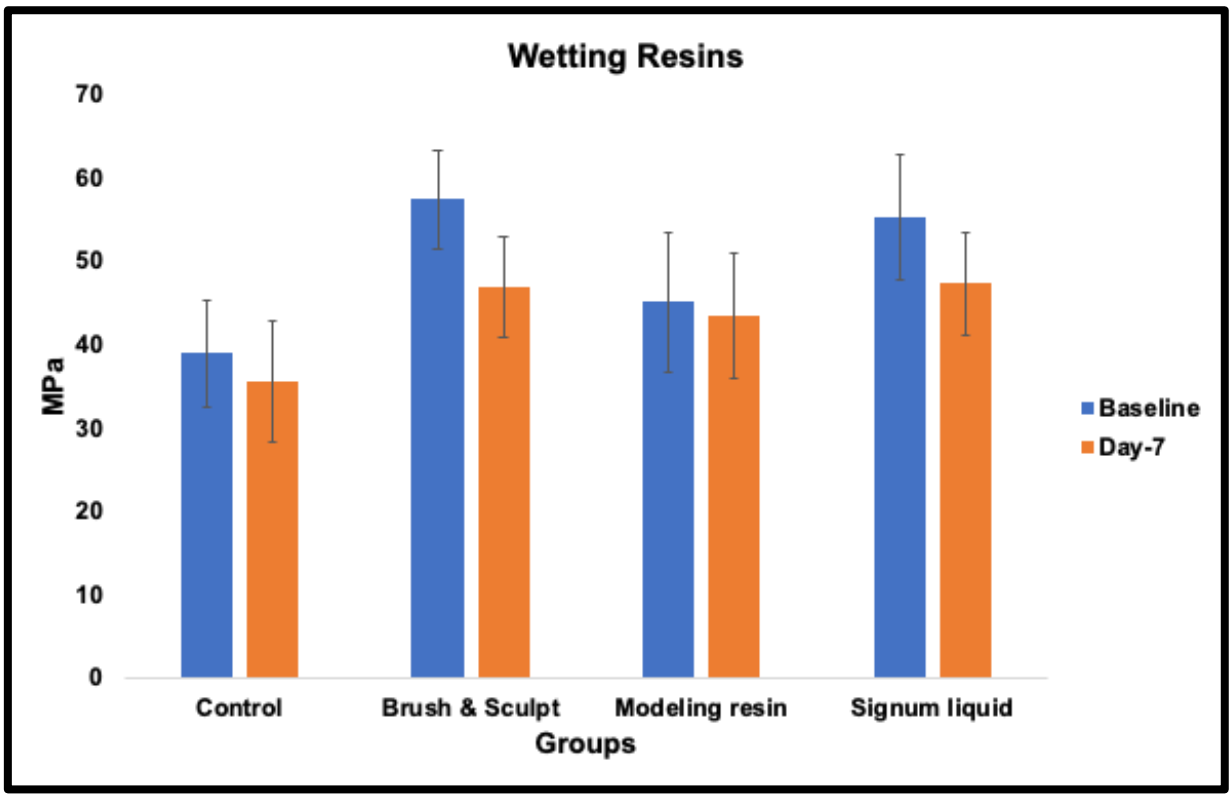


Figure 6-11: Mean and standard deviation values for the microtensile strength of Filtek RBC treated with wetting resins as ILs

From all the experimental results, the most significant effects could be noted in the solvents class, and both bonding agents also had a significant impact on the microtensile strength of the RBCs. However, the wetting resins improved the microtensile strength of the tested groups. The lowest values were seen with the Modelling resin across the time intervals, but it still had higher values than the control group. All groups were more effected after storage in DW for seven days. The results of groups showing significant reduction effects are summarised in Table 6-1.

Classes	ILs Groups	Filtek
Solvents	Acetone	(p<.001) *
	Ethanol	(p<.001) *
	Isopropyl	(p<.001) *
	DW	(p=0.047) *
Bonding Agents	Scotchbond	(p<.001) *
	Optibond	(p<.001) *
Wetting Resins	Brush & Sculpt	NS
	Modelling resin	NS
	Signum liquid	NS

Table 6-1: Significant reduction in microtensile strength across groups compared to the control

* Statistically significant different *p*-values in comparison to the control and (NS) means not significant

6.4 Discussion

Because of the COVID-19 pandemic situation, some of project's sections design were altered, particularly in chapter six. This chapter was adapted so that experiment could be completed within the available project timeline. So, only one type of RBC, not two, was studied and storage in an aqueous environment for one week rather than the more extended periods, were used. Through all that, all the decisions were taken to overcome the effects of the pandemic were after intensive pilot tests and discussion with supervisors. The main goal was to keep the experiment main aim without jeopardising the validity of findings.

The RBC materials are used to restore damaged tissues and they face high loads during mastication. Most of these applied loads are tensile, produced by converting compression loads inside the restoration to tensile loads (Armstrong *et al.*, 2017). This type of load can significantly endanger the functioning of RBC restorations if any intrinsic components of the RBC like the organic matrix, inorganic fillers, and coupling are modified (Leprince *et al.*, 2013). Also, the presence of other contributing factors such as saliva can aggravate the effects of the tensile loads on the placed RBCs, because the components of saliva have a high percentage of water and different organic substances, including enzymes, proteins and urea which degrade the RBC (Özcan and Koc-Dundar, 2014; Liebermann *et al.*, 2017). Therefore, in the current study, the microtensile bond strength test was used to evaluate the effects of using various ILs between RBC increments. The test was designed to simulate the moist environment in the oral cavity at different time intervals; specimens were stored in distilled water and placed in an incubator at 37±1°C to simulate the oral cavity temperature. To evaluate the effects of ILs for a more extended period, the specimens were stored in this way for different time intervals. However, due to the COVID-19

pandemic and the national UK lockdown, lab access was limited. For this reason, the decision was made to test the effect of the ILs on the specimens at baseline and after one week. The type of load and the test design in this experiment were considered in trying to achieve the aim of the study. This would therefore investigate the effects of the ILs between the RBC increments in simulation to oral cavity circumstances under tensile load.

Testing the effects of different types of ILs on the microtensile bonding strength between the increments of the RBCs is an original concept. The literature shows this test has mainly been used to evaluate the bonding strength of RBC to tooth structure or other substrates like ceramic and polymeric based materials (Armstrong *et al.*, 2017; Sano *et al.*, 2020). In the current experiments, the aim was to test the effect on the microtensile bonding strength of ILs applied on the interface between the second and third increments, in order to focus only on the load on the area treated with ILs during the build-up of layers. This ensured standardisation of the location of the treated area at the specimen's centre, so that the same area of all groups could be compared for changes between treated and untreated groups. This procedure was designed to mimic a test of the bonding strength between dentin and resin composite at one interface area, as described in the literature (Soares *et al.*, 2008; Ramos *et al.*, 2016; Armstrong *et al.*, 2017; Sano *et al.*, 2020).

Also, the shape and size of the tested specimens played a major role in the test design. Micro stick-shaped specimens were selected, as these are the most commonly used specimens reported in the literature for evaluating bonding strength (Soares *et al.*, 2008). The bonding strength values that can be collected from micro-specimens are higher than those from macro-specimens, because the applied tensile load is distributed on the larger area and the specimen's failure will occur at low value. This

makes the detection of significant differences between specimens more challenging (Sano *et al.*, 2020). Micro-specimens thus help to concentrate the tensile load on the targeted area and ensure the highest load is on the tested point (Gorge Perdigao, 2006; Özcan *et al.*, 2007; Ramos *et al.*, 2016; Armstrong *et al.*, 2017; Sano *et al.*, 2020). Thus, the present study took many points into consideration to test the effects of ILs between the RBC increments.

6.4.1 Solvents

Throughout all tested ILs classes, specimens treated with solvents as ILs had the greatest reduction in microtensile bond strength, as the presence of organic solvents plasticised and softened the RBC in the treated area (Moraes *et al.*, 2007; Shahdad;McCabe and Wassell, 2007; Malacarne-Zanon *et al.*, 2009). In turn, this affected the bonding area between the outer surface of the fillers and the organic matrix (Sideridou;Karabela and Bikiaris, 2007; Sideridou;Achilias and Karabela, 2007). Organic solvents can increase the DC of the treated RBCs by increasing free radical mobility (Cadenaro *et al.*, 2009). However, the solvents penetrate the organic matrix of the treated RBC, opening and increasing the spaces between the RBC chains (Moraes *et al.*, 2007). The type of monomer in the RBC can also play a major role in the formation and density of the cross-linking of the polymers. The monomers in Filtek, such as TEGDMA, UDMA and PEGDMA, help increase the crosslinking of the polymers. However, because the solubility parameters of TEGDMA are close to those of the organic solvents, their effectiveness on this monomer are greater, leading to a reduction in the formation of dense polymer chains (Neveen *et al.*, 2017).

The tests of DW as a solvent reduced the DC of the contaminated uncured surface, which impacts the polymerisation process and growth of polymer chains (Nair;Hickel

and Ilie, 2017; Fugolin *et al.*, 2019). Besides plasticising and softening the RBCs, in addition to reducing the DC, the solvents are an obvious reason for the reduced microtensile strength for both time intervals in this class.

In the current study, the substances used between the built-up RBC block increments as ILs increased water uptake. The presence of some of these materials between the RBC increments has been previously shown to increase the DW diffusion coefficient (Malacarne-Zanon *et al.*, 2009). This led to an increase in the internal stress between the main components of the RBC. The increased solubility caused by organic solvents can in turn increase the spaces between the polymer chains, as explained above, and lead to more water uptake (Sideridou;Karabela and Bikiaris, 2007; Sideridou;Achilias and Karabela, 2007; Cadenaro *et al.*, 2009; Malacarne-Zanon *et al.*, 2009; Neveen *et al.*, 2017; Patel *et al.*, 2017). Increased water uptake would reduce the tested materials' stability and longevity. The current study's microtensile strength values showed more reduction after the specimens were stored in DW, and so the presence of solvents that increase solubility and the diffusion coefficient can explain the reduction in the values for the solvents groups.

Unset RBC materials are sensitive to any contamination during the manipulation and polymerisation steps, and in this context the presence of solvents used as ILs will jeopardise the stability and longevity of the placed restorations (Eiriksson *et al.*, 2004; Koppolu *et al.*, 2012; Oskoee *et al.*, 2012; Deprá *et al.*, 2013; Martins *et al.*, 2015). The use of ILs between RBC increments in the present study can be considered a contamination of the main structure of the unset materials. The creation of microvoids spaces between the chains of the network provide a high chance of a greater volume of solvents being retained in the RBC matrix (Moraes *et al.*, 2007; Sideridou;Karabela and Bikiaris, 2007; Sideridou;Karabela and Vouvoudi, 2008). These void spaces can

also accelerate the early failure of the treated restorations (Martins *et al.*, 2015). In general, all the factors mentioned above are a possible source of the reduced strength of the tested RBC materials.

6.4.2 Bonding agents

The bonding agents used in the current study are produced by different manufacturers and were selected by design to determine if the one manufacturer's bonding agent would be better than another's. Using different manufacturers would also help to check if some of the tested RBCs' components reacted differently to the bonding agents and thus impacted the RBCs' physical and mechanical properties. In the literature, this matter has not been investigated and discussed thoroughly. One study suggested using bonding agent systems and RBCs from the same manufacturer to standardise the products (Patel *et al.*, 2017). In that study, the effects were dependent on the components of the tested RBCs and ILs. Another study mentioned that an adhesive system from the same brand as the RBC would be more suited for use as an IL (Tjan and Glancy, 1998). Thus, in the present study, using different types of bonding agents provided a broader view of their effects on the tested RBCs. Also, how the components of the bonding agents from the same or different manufacturers affected the polymerisation process in reacting with organic and inorganic RBCs components. That could explain if there is any benefit, or they would cause contamination to the main components of treated RBCs and interrupt the polymerization process (Tjan and Glancy, 1998; Leprince *et al.*, 2013; Patel *et al.*, 2017).

In both bonding agent groups, the values of the microtensile tests were significantly reduced, and the components of the bonding agents used in this study played a significant role in the effects on these values. Some substances like ethanol, water and

HEMA can impact the RBC polymerisation process and strength following manipulation (Cadenaro *et al.*, 2008; Cadenaro *et al.*, 2009; Patel *et al.*, 2017). When ILs are applied to the RBC, the solvents affect the strength of the bonding between the surface of the inorganic fillers and the organic matrix (Sideridou;Karabela and Bikiaris, 2007). These effects reduce the strength of the treated interface area between the RBC increments, affect the polymerised RBC materials' cross-linking chain density, and plasticise the manipulated surfaces, as explained previously (Moraes *et al.*, 2007; Patel *et al.*, 2017). Therefore, the use of bonding agents, which contain solvents as a main component, is a reason for decreased microtensile strength in the tested groups.

Both the bonding agents used in this study between the treated specimens' increments contained HEMA, a small monomer, as an essential component (Van Landuyt *et al.*, 2007). HEMA can reduce the DC and produce linear polymers which can reduce the microtensile strength in the treated area (Zanchi *et al.*, 2013). Also, it is soluble in water, ethanol, and acetone. It is hydrophilic, and the presence of HEMA within the polymer chain will increase water uptake in the internal structure of the RBC. This can increase swelling between the main components of the RBC and consequently induce the separation of the bonding between the organic and inorganic parts, to the detriment of the RBC materials' strength (Jacobsen and Söderholm, 1995; Van Landuyt *et al.*, 2007). A high amount of HEMA reduces the vapour pressure of the solvents and will impact the solvents' negative evaporation (Pashley *et al.*, 1998; Tay and Pashley, 2001; Van Landuyt *et al.*, 2007). The presence of solvents increases the diffusion coefficient, thus increasing the water uptake (Sideridou;Karabela and Bikiaris, 2007; Patel *et al.*, 2017). Thus, the presence of HEMA and solvents can reduce the microtensile bonding strength of the RBC manipulated with bonding agents as ILs, as shown in the experimental results.

6.4.3 Wetting resins

Wetting resins were selected as the second most commonly used IL after bonding agents by the UK dentists surveyed in this study. The Brush and Sculpt and Signum Liquid used in this study are dimethacrylate base monomers, but the components of the modelling resin were not identified by the manufacturer. Only Brush and Sculpt in the wetting resins tests has 24 % inorganic fillers by volume; no manufacturing information was available for the other substances. The use of different wetting resins can provide a clear idea of the effects of these ILs on the treated RBCs' physical and mechanical properties.

The specimen groups stored in DW for one week had reduced microtensile strength. Due to the wetting resin substances being used between the increments, water uptake increased. Also, these low- or unfilled wetting resin substances made the DW uptake greater than in the control groups (Patel *et al.*, 2017). Increased water uptake accelerated the hydrolysis process at the bonding area between the organic matrix and the inorganic fillers, leading to decreased RBC material strength (Rahim *et al.*, 2012). This feature was also apparent in the DTS test results, when the specimens were tested for longer time intervals. These values for the wetting resins groups were either equal to or higher than the control group. However, after three months, they showed lower values in comparison to the controls. In a future study, therefore, the time intervals for wetting resins groups may need to be extended in order to evaluate them using μTBS.

However, the effects of the solvents on Filtek in both DTS and μTBS tests were significant, showing that using the solvents, even to ease the manipulation of the RBC, still reduces the physical and mechanical properties of the treated materials. Also, the bonding agents groups had the same results in both tests, again indicating the

significant effects of this class on the treated RBCs. This confirms the negative effects of the components of bonding agent, like HEMA, as well as solvents, and their ability to reduce the strength of the treated RBC. It is also important to note that the μTBS test was able to detect the significant effects of the solvents and bonding agents at earlier time intervals in comparison to the DTS.

The discussion of the physical and mechanical results from the previous chapter can help to explain the findings from the present chapter. From the data collected from solvents and BA groups used as ILs on the lubricated surfaces. These ILs have shown increases in the DC of both setups in the previous experiment conducted to test ILs effects on the polymerisation process and convert the monomers to polymers. The Filtek had high DC (62-85%) throughout the solvents and BA groups except DW that decreased the DC on the bottom surface in setup one (46%). However, that would not be the only factor affecting the microtensile bonding strength between the RBC increments. The crosslink density of the polymer chains is an essential factor as well (Leprince *et al.*, 2013; de Paula *et al.*, 2016). The presence of organic solvents alone as ILs or part of the BA components, even they increase the DC, can affect the crosslinking by affecting the crosslink density by plasticizing the polymers and affecting the density of the chain, and that would reduce the microtensile strength.

There were some of the main components of the treated RBC or the used ILs that can impact the treated RBC more distractively. TEGDMA is one of the main components of the Filtek RBC and has a close solubility parameter to those of the organic solvents, which would make their effects on this monomer more significant and lead to a reduction in the formation of dense polymer chains (Neveen *et al.*, 2017). Also, the presence of HEMA can produce linear polymers, which provides polymers with less strength. The HEMA is a monofunctional monomer that can make a liner polymer

without complex crosslinking (Van Landuyt *et al.*, 2007; Zanchi *et al.*, 2013). So, these polymers can show low properties like low bonding strength when the microtensile bonding strength is tested in the present experiment.

The presence of external substances can increase the water uptake and that would lead to more effects on the stability and longevity of treated RBC restorations. The hydrophilic HEMA, which has a high tendency to water uptake, can increase the amount of water diffused into the treated RBC's organic matrix (Van Landuyt *et al.*, 2007; Patel *et al.*, 2017). Also, the organic solvents can increase the water uptake by plasticizing and softening the link between the organic and inorganic components of the treated RBC (Zanchi *et al.*, 2013). So, that can increase the spaces between those components. That increase the water uptake as presented in the water uptake experiment. Therefore, those ILs like organic solvents and BA can reduce the tensile strength between the treated RBC increments in the present experiment.

The volume of the fillers loading in the RBC can affect the water uptake, as shown in the WU experiment in Filtek and how that can make the physical and mechanical properties weaker. Also, the tested RBCs in the DTS experiments in the previous chapter have shown that the Filtek with lower fillers loading had a significantly impacted the DTS values. So, that can play a significant role in the amount of the effects that would be made by the fillers loading volume in RBCs (Leprince *et al.*, 2013). In the present study, this point can create a noticeable impact on the bonding strength values between the RBC increments as well. The presence of the solvents as ILs or part of the main components of the BA can aggravate the reduction of the microtensile. That can occur by effecting the relation between the organic and inorganic parts of the tested RBC and increase the water uptake throughout the time intervals in the present experiment.

The current study was designed to store RBC specimens for seven days in DW. This point can be considered a limitation of this *in vitro* experiment. The literature had guided the storage of the specimens in aqueous solutions before the specimens were tested under load at different intervals. The less than 1 month, as recommended by the Academy of Dental Materials, is a short-term storage period to evaluate the microtensile bonding strength between the tooth structure and RBC materials (Armstrong *et al.*, 2017; Sano *et al.*, 2020). In the current experiment, there were circumstances behind this decision to test the effects of ILs on the microtensile bonding strength at baseline and after seven days. The covid-19 pandemic and limited access to the laboratory made longer time intervals unmanageable for many tested groups. The literature used the effects of storage time on the monomer elution and water uptake within seven days (Effrosyni *et al.*, 2014). Also, the ISO 4049 protocol to test the water sorption was evaluated after storage in DW for one week (Sideridou *et al.*, 2004; Gajewski *et al.*, 2012; Park and Ferracane, 2014; Vale *et al.*, 2014). Moreover, the tested groups in the current study at seven days have significantly impacted the bonding strength compared to baseline. The collected data from both intervals in the current study shows the significant impacts of organic solvents, either as ILs or part of the BA main components, on the RBC materials. So, that has shown valuable data that can determine the effects of ILs on the bonding strength between the RBC increments.

Different aqueous solutions in the literature were used to store specimens before testing, like natural saliva, artificial saliva, and DW (Armstrong *et al.*, 2017). The type of aqueous solution in the current project like DW can be considered a limitation. Using DW to store specimens before testing can be considered is not like the oral cavity environment. However, natural saliva contains about 99% of the water from its main

components in the human mouth, as mentioned in the literature (Iorgulescu and Davila, 2009). So, many previous studies have used the protocol of storage in DW before testing the specimens under tensile stress (Cho and Dickens, 2004; Özcan *et al.*, 2007; de Andrade *et al.*, 2010; Zanchi *et al.*, 2013; Dao Luong *et al.*, 2016; Münchow *et al.*, 2016; Ramos *et al.*, 2016; Sano *et al.*, 2020). The current study findings after the specimens stored in DW have significantly impacted the bonding strength compared to the baseline groups. One of the main targets of using the DW instead of other types of aqueous solutions was to test the tendency of the water uptake in the area of application of the ILs between the RBC increments. That would help show its impact on the bonding strength between RBC increments within the tested intervals. The current study's collected data can show how DW presence in trial to simulate the oral circumstances has aggravated the effects of using the ILs compared to the control group.

Scanning the fracture area of the specimens was recommended in the literature to provide more data and details that would help analyse the fracture area (Armstrong *et al.*, 2017). The scan of the fracture area with scanning electron microscope (SEM) can show if there was any defect or voids that could cause a premature fracture at the interface area. Also, it can give a view of the RBC surface condition after it was manipulated with ILs. This part was not performed in the current study, which can consider a limitation of the current study. That is because the SEM would help analyse the effects of the ILs on the microtensile bonding strength of the tested specimens besides the microtensile bonding strength values. The fracture area scanning needs a specific instrument like SEM with suitable training for the principal researcher, which was unavailable at this stage of the experiment due to the COVID-19 restrictions. However, the collected data from the microtensile bonding strength test in the current

study can still be valid for detecting the effect of the ILs on the bonding strength at the interface area. Even though, scanning the fracture would provide more supportive data that can help a deeper understanding of ILs effects on the bonding strength between RBC increments.

6.5 Conclusions

Within the limitations of the present study, the findings from the results can be summarised as:

- Both solvents and bonding agents used as an IL between layers of the RBC reduced the strength of the tested groups. This was due to their effects on the main components of tested RBC by effecting the bonding between the organic matrix and inorganic fillers. Also, they can increase the water uptake and increase their impact on the internal components' relation across the time intervals;
- All the wetting resins used showed higher values against the control groups;
- Brush and Sculpt as an IL had the highest values at baseline and after being stored in DW for one week;
- Future tests should consider implementing a more extended time interval for testing μTBS than that used in the current study.

Chapter 7: General Discussion and Conclusion

7.1 General discussion

The concept of this project was developed in different stages, providing the benefit of being able to cover different essential aspects to meet the study objectives. Each phase had specific aims linked to the next phase, and thus all the phases were able to build on each other to the successful investigation of the effects of ILs on the physical and mechanical properties of RBCs. The phases are shown in Figure 7-1.

The literature review on the use of ILs in dental practice provided limited background information showing that certain substances like organic solvents, BA and WR have been used as ILs to ease the manipulation of the RBC materials (Tjan and Glancy, 1998; Gorge Perdigao, 2006; Dunn, 2007; da Silva *et al.*, 2008; Barcellos *et al.*, 2011; Tuncer *et al.*, 2013; de Paula *et al.*, 2016; Patel *et al.*, 2017). However, all the studies employed *in vitro* studies, and there were no clinical studies or systematic literature reviews which would help situate the current topic in critical depth. Also, the literature lacks agreed and established guidelines or recommendations to help dentists make evidence-based decisions on the use of ILs with RBCs. These factors indicate the importance of investigating different aspects of this area to understand key effects of using ILs. From this perspective, the identification of these gaps in knowledge highlights how a lack of essential information led to the current project's aims and objectives.

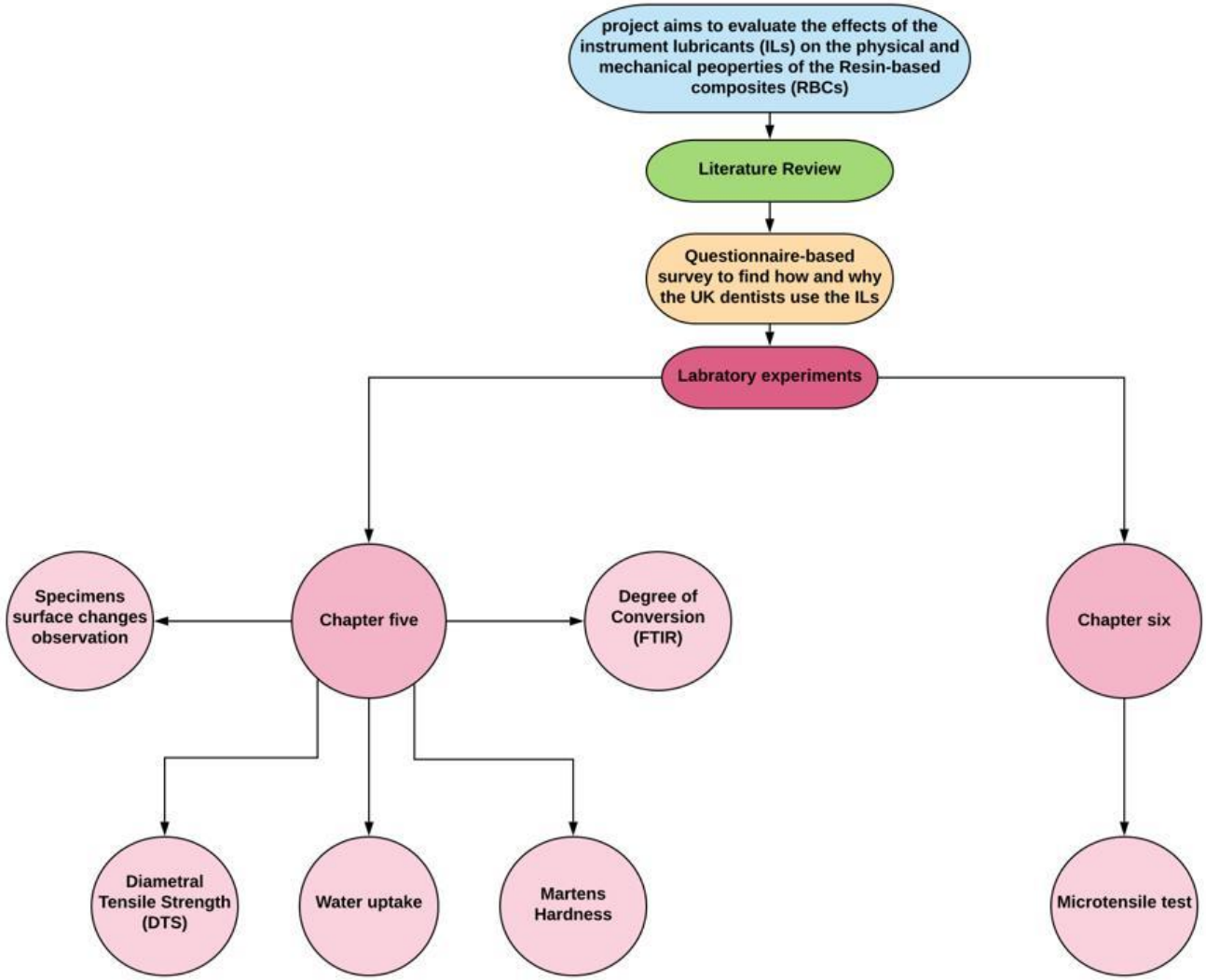


Figure 7-1: The sequence and content of each project phases

Survey-based studies are an effective tool in much research conducted in dentistry (Gilmour;Evans and Addy, 2007; Lynch *et al.*, 2010b; Stone *et al.*, 2014; Martins *et al.*, 2016; Blum;Younis and Wilson, 2017; Rosentritt *et al.*, 2019; Holmes;Burford and Vance, 2020), helping to collect data to clarify, explain, and answer different questions. In the current project, a survey was used to collect data on the dentists' practice and opinions of using ILs to ease RBC manipulation of RBC restorations, and these data became the basis of the laboratory investigations simulating the clinical situation. In effect, the survey data were not only reflective of clinical practice, they were more applicable to support the design of the laboratory investigations in clinical situations. Therefore, the connections between the different phases of the project added probably more like credibility to the collected data.

The perspective of UK dentists regarding the use ILs in their daily practice was an essential factor in understanding and analysing the latter. The literature review produced no studies which would shed light on important questions related to dentists' wishes, attitudes, and behaviour in this regard, even though it is important to know why and how they used ILs. It was therefore helpful to create a solid basis on which to design and perform both the current and further studies in this area.

Conducting a questionnaire-based survey was essential to the clarification of many aspects of the current study. The opinion of the UK dentists and the results of this survey showed that about half of the respondents used ILs in their daily practice to ease the manipulation of RBCs. Most IL application techniques are not determined in the literature, and so the survey in the current project can be a source for the design and implementation of other investigations of the effects of ILs on RBC physical and mechanical properties. Within some limitations, for example a relatively small sample, this survey succeeded in confirming the use of ILs by UK dentists and there appears

to be diversity in materials used and application of those materials. Some factors have affected the size of the sample including the funding of the entire survey project, as well as the timeline of project phases and experiments. The selected sample size can impact of the generalising the results of the survey for all registered UK dentists. However, the collected data helped draw some points from the results of this survey. About half of the participants dentists were using the ILs to ease the manipulation of uncured RBC materials. They used different types of ILs and different methods and techniques to apply those ILs. Also, the participants have shown that most of them were not against or with using ILs to manipulate uncured RBC, and they have mentioned different reasons to support their opinions.

In the laboratory investigations phase, these physical and mechanical properties were tested, because they play an essential role in the evaluation of the effects of ILs on a treated RBC. The literature review showed different *in vitro* tests can help researchers and developers evaluate RBC materials under different circumstances by testing various essential properties. For example, they can evaluate the materials' strength, hardness, water uptake, DC, and colour stability (Ferracane *et al.*, 2017; Ilie *et al.*, 2017). Such tests provide valuable data that helps develop the materials' physical and mechanical properties. That makes the materials better to withstand being in a mouth and more likely to be used. Also, the laboratory tests used in the current project helped to investigate the effects of external factors that can alter the main components of the RBC and affect its longevity as a restoration. These factors in turn affect the organic matrix, inorganic fillers, or coupling agents linking them (Leprince *et al.*, 2013; AlShaafi, 2017). Thus, the current project was designed to include laboratory investigations in support of the survey data. Thus, Using the established and accepted laboratory tests

give insights into materials' performance is sensible, primarily when that has relation to what the dentists use in their actual practice.

In this phase, physical and mechanical properties were tested. The key information collected from previous project phases was considered during the design of the experiments, which mimicked the clinical situation of the surveyed dentists' daily practice. Regardless of whether the dentists' decisions were based on evidence or experience, the collected data was generated from their practice and use of these materials for ease the manipulation of the treated materials. The types of ILs and application techniques tested were extracted from the survey data and the literature review. Moreover, specimen design considered the effects of ILs between RBC increments, to view their effects on RBC restorations, and for this reason different types of materials were applied when the specimens were prepared during the experimental work. The specimens were stored in DW and incubated at 37±1 °C to simulate the situation in the oral cavity and determine the effect of ILs in the presence of moisture. Overall, the laboratory investigations were designed and performed to evaluate the ILs' effects on certain RBC materials to determine the answers to the project's questions.

According to the results from the two tested RBCs and the multiple classes of ILs, it seems the effects of ILs on the treated RBCs' physical and mechanical properties negatively varied, as shown in Table 7-1. Some of the ILs increased one of the RBC's water uptake but did not significantly change that of the other. DTS values also reduced significantly across time intervals when some IL classes were used on one of the tested RBCs, but other ILs made no significant changes in the same conditions. Also, Martens hardness values were different for both tested RBCs and the effects of the ILs varied on the same type of RBC.

ILs	COIIA		DC		HM		WU		DTS		µTBS
	F	H	F	H	F	H	F	H	F	H	F
Acetone	(p<.001) *	(p=0.003) *	NS	NS	(p=0.022) *	NS	(p<.001) *	NS	(p<.001) *	NS	(p<.001) *
Ethanol	(p<.001) *	(p<.001) *	NS	NS	NS	NS	NS	(p<.001) *	(p<.001) *	NS	(p<.001) *
Isopropyl	(p<.001) *	(p<.001) *	NS	NS	(p=0.018) *	NS	NS	(p<.001) *	(p<.001) *	NS	(p<.001) *
DW	(p=0.02) *	NS	I (p=0.002) *	NS	NS	NS	NS	(p<.001) *	(p<.001) *	NS	(p=0.047) *
Scotchbond	NS	NS	NS	NS	II (p=0.021) *	II (p=0.004) *	(p<.001) *	(p=0.004) *	(p<.001) *	NS	(p<.001) *
Optibond	NS	NS	NS	NS	II (p=0.001) *	II (p<.001) *	(p<.001) *	NS	(p<.001) *	NS	(p<.001) *
B&S	NS	NS	NS	NS	I&II (p=0.016) *	II p<.001) *	NS	NS	NS	NS	NS
MR	NS	NS	NS	NS	II (p<.001) *	II (p<.001) *	(p<.001) *	(p=0.001) *	NS	(p=0.009) *	NS
SL	NS	NS	I (p<.001) *	NS	II (p<.001) *	II (p=0.004) *	NS	(p=0.001) *	NS	NS	NS

Table 7-1: Summary of the laboratory investigations of the effects of ILs on the physical and mechanical properties of both tested RBCs

* Represents the significant effects in either the increase or decrease in the values of the RBCs. (NS) represents when there are no adversely significant effects. (I) represents setup one lubricated bottom surface and (II) setup two lubricated top surface in both DC and HM experiments.

The diversity in the effects of the tested ILs on both RBC materials is most likely related to the RBCs' components and the ILs used to manipulate the tested groups. The monomer's organic matrix is the backbone of the RBC polymerisation process (Chan *et al.*, 2010; Ferracane, 2011; Lavigueur;Christine and Zhu, 2011); this process can be affected by external foreign substances that interrupt the conversion of monomers to polymers and in turn affect chain cross-linking density (Leprince *et al.*, 2013). Also, the load of various inorganic fillers by volume was different for the two RBCs, and could have caused different changes to appear due to the ILs (Alzraikat *et al.*, 2018). The coupling agent between the organic matrix and inorganic fillers could be degraded by the effects of the solvents in some ILs, or by the organic solvents used in the solvents tests (Park and Ferracane, 2014; Aydinoglu and Yoruc, 2017). Furthermore, some of these solvents, like ethanol, could have increased the diffusion coefficient of the water uptake and weakened the bond between the internal components of the RBC materials. That is because of the presence of ethanol and the hydroxyl group of the Bis-GMA RBCs, as well as the low cross-linking, more amount of unreacted monomer, and porosity between the polymer networks (Sideridou;Karabela and Bikiaris, 2007; Cadenaro *et al.*, 2009; Decky *et al.*, 2009; Rahim *et al.*, 2012; Patel *et al.*, 2017). Also, HEMA is an essential component in most bonding agent systems, and its hydrophilicity increases water uptake (Van Landuyt *et al.*, 2007). Additionally, the RBC's treated with different ILs were exposed to fluid for long time intervals, potentially aggravating the ILs' effects on the RBCs' physical and mechanical properties. This scenario explains the effects of the ILs and the RBC components on the polymerised treated RBCs' physical and mechanical properties.

Different laboratory experiments tested the strength of the treated RBCs. DTS was one test in this project to evaluate the effects of the ILs on the treated RBC. This test

identified the effect of the ILs on the specimens by indirectly testing the strength. However, this test was not enough to determine the effect at the interface area of the increments. The test has been described in the literature as having some limitations in controlling the centre of the tensile load (Darvell, 1990), because the compression load is converted to tensile load and the centre of this load can be affected by any specimen surface discrepancies. However, in the current project, μTBS values were obtained with a modified methodology by testing the actual effects of the ILs on the bonding strength between the RBC increments. This test is ranked in the literature as one of the most common to detect the tensile bonding strength differences directly in the tested interface area (Armstrong *et al.*, 2017). However, the effects of the ILs on the whole specimen strength were indirectly determined by using DTS.

From all these investigations, it seems the use of ILs to ease the manipulation of RBCs during the placement process led to adverse effects, especially on the RBCs' physical and mechanical properties. Consequently, a restoration's stability and longevity can be affected and reduced. The effects of the IL classes seen in the current study were varied and not constant, such as by reducing values in one RBC but causing no significant reduction in the other. Also, storing the tested experimental groups in DW for longer time intervals caused more reduction in the treated groups' values than in the control groups.

The findings in the current survey revealed that most of the ILs used by the surveyed UK dentists were bonding agents and wetting resins, just as in most other studies of ILs and their effects on the physical and mechanical properties of RBC (Tjan and Glancy, 1998; Gorge Perdigao, 2006; da Silva *et al.*, 2008; Tuncer *et al.*, 2013; de Paula *et al.*, 2016; Münchow *et al.*, 2016; Patel *et al.*, 2017). Some of these studies have suggested that using ILs can improve bonding strength between RBC increments

or not negatively affect the treated RBC (Tjan and Glancy, 1998; Gorge Perdigao, 2006; da Silva *et al.*, 2008; Münchow *et al.*, 2016). However, results of the laboratory investigations in the current project recommend stopping or limiting their use because the findings revealed different behaviour in the RBCs when treated with bonding agents and wetting resins. The Sculpt and Brush wetting resin was the only resin which had a minimal effect on HM values on both tested RBCs and no adverse effects on the DTS and μTBS of Filtek, as summarized in Table 7-1.

The components and structure of both the RBCs and the different ILs have changed the degree of the effect on the physical and mechanical properties of the RBCs. Resin organic matrices and the loading volume of the inorganic fillers played a significant role in generating adverse effects from the ILs on the treated RBC materials (Tuncer *et al.*, 2013; de Paula *et al.*, 2016; Patel *et al.*, 2017). Furthermore, the components of the ILs themselves have contaminated the uncured RBC materials and thus affected the polymerisation process, producing microvoids (Moraes *et al.*, 2007). In the current study, wetting resin showed better μTBS results but exposure to DW for longer time intervals could have affected the physical and mechanical properties of the RBC in the presence of ILs.

Thus, from all collected data, the use of ILs to ease the manipulation of uncured RBC materials can affect the physical and mechanical properties of placed RBC restorations. This matter should be considered during the dentists' daily practice during the RBC restorations placement procedures. Also, throughout the literature the adverse effects of using ILs on the RBCs physical and mechanical properties were pointed out (Tuncer *et al.*, 2013; de Paula *et al.*, 2016; Patel *et al.*, 2017; Cangul *et al.*, 2020). Besides that, the current project findings have shown the effects of ILs on the physical and mechanical properties of RBCs, especially on the water uptake, DTS, HM

and μTBS. So, making more focused efforts to educate the registered dentists regarding the negative aspect of using ILs to ease and enhance the aesthetic of placed RBC restorations. That can be done by sharing the current project findings with all dentists, and make sure they are accessible. Also, making some educational interviews and presentations to reach most of the targeted populations. Furthermore, using the latest findings as a solid basis to perform more research work to provide a broad database regarding the use of the ILs and their adverse effects on the manipulated uncured RBCs.

The limitations of the current project phases were discussed in the previous chapters. These limitations were manageable, and the collected data helped answer the project questions. For instance, even the collected data may not be generalised to all UK registered dentists because of a small sample size. Some of the collected data that provided answers to confirm that some UK dentists used the ILs in their daily practice. Also, using some data to mimic the clinical situation throughout the *in-vitro* tests in the current project depends on collected data from the survey-based study. Even though the literature has shown the *in-vitro* tests are not entirely related to the clinical situation because the clinical situation has some different circumstances (Bayne, 2011). The tests used in the current project still can provide valid data that can detect the effects of ILs on the physical and mechanical properties. This is especially true when the test methodology considers some factors dentists mention about their daily practice. These factors would help mimic the clinical situation to simulate the clinical circumstances as much as possible. That was achievable by implementing all collected data and linking all project phases to answer the project questions.

7.2 Conclusion

This work and its results regarding UK dentists' opinions of why and how they used ILs in their daily practice, the effects of these materials on the physical and mechanical properties of the manipulated RBC materials can be concluded as follows:

- About half of surveyed UK dentists (47%) used different ILs and apply them with different techniques to ease the manipulation of RBC materials. Most dentists had no definitive opinion to support or reject their use. This proved that the hypotheses one, two, and three (section 3.3) were correct.
- The effects of the ILs on the physical and mechanical properties evaluated by laboratory investigation produced varied results depending on the essential components of the ILs themselves and the tested RBCs. This approved that the hypotheses four and five (section 3.3) were correct.
- Storage in distilled water of the RBCs treated with ILs for longer time intervals can increase the water uptake and significantly affect the manipulated RBCs' properties. This proved that the hypothesis seven (section 3.3) was correct.
- Some tested ILs like organic solvents and BA decreased the microtensile strength of the RBC increments, and only the wetting resins increased the values between the increments. This approved that hypothesis six (section 3.3) were correct .;

Based on the above, it is recommended that the use of ILs to manipulate uncured RBC is limited or stopped.

7.3 Future work

- Use some of the collected data from the survey-based study to conduct a more specific qualitative study regarding the dental students' and the registered UK dentists' opinions. This could provide more clarity in understanding their background about using the ILs during their study and practice experience.
- Test the effects of different ILs on the monomers' matrices, such as non-dimethacrylate-based RBC to detect the effects of the ILs on various types of monomers and differently loaded organic fillers. This will help determine if the use of ILs has more impact on some types of monomers compared to others;
- Test the effects of the ILs on the microtensile strength of multiple types of RBC materials stored in DW for more extended time intervals. This will help show if the presence of the ILs in a wet environment for a more extended period can reduce the microtensile strength of the tested groups;
- Evaluate the microtensile specimen fracture pattern using a SEM, to provide a clear picture of the fracture area;
- Investigate the possible depth that can be affected by using ILs on treated RBC surfaces, to show the range of the ILs' effects at different depths on the treated surfaces.
- Investigate the effects of the ILs on RBC crosslink density by estimating the percentage hardness decrease (%HD). This would help to evaluate the crosslink density of cured RBCs, to see the effects of the ILs on the polymerisation process, not only from the aspect of DC but also from

crosslink density, which is an essential element affecting the physical and mechanical properties of RBCs.

- Investigate the possibility of new dental instrument with non-stick coating materials that can be used to reduce the sticking the uncured materials to the placement instruments without using ILs.

Appendices

Appendix 1: Flowchart of participants selected from the UK

Appendix 2: Letter and invitation to UK dentist participants

School of Dental Sciences
Framlington Place
Newcastle upon Tyne
NE2 4BW
United Kingdom

Saleh Alqahtani BDS, MSD, PhD candidate Dental Sciences
e-mail: E-mail/ S.alqahtani2@ncl.ac.uk
Tel: +44 (0) 191 208 8847

Date:

Re: Survey

To: The Dental Centre

Dear Colleagues,

I am working on a study to better understand the existing evidence on the effects of using instrument lubricants on the physical and mechanical properties of the resin-based composite restorations. As part of this project, I would like to invite you to take part by completing the enclosed questionnaire, to share your opinion and experiences of this topic.

All information provided will be anonymized, will be held in the strictest confidence, and not directly attributed to your personal information. If you are happy to participate, please follow the instructions enclosed that should guide you through the questionnaire booklet.

Thank you for taking the time to read this short introduction letter and please feel free to contact me regarding the study if you would find it helpful to do so.

Yours sincerely,

Saleh Alqahtani
PhD candidate Dental Sciences

Appendix 3: Used questionnaire in the survey

School of Dental Sciences

Effect of instrument lubricants on the physical and mechanical properties of resin-based composite (RBC) materials

Conducted by: Saleh Ali M Alqahtani
Dental Sciences PhD student

Supervised by:
Dr Giles McCracken **Dr Matthew German**

Dear respondent,

Firstly, thank you for agreeing to take part in this questionnaire-study regarding the use of instrument lubricants to aid the placement of direct resin-based composite (RBC) restorations in dental practice. The research will be conducted by Saleh Alqahtani a PhD student of Dental Sciences, at the Institute of Cellular Medicine, Newcastle University, UK. All information provided will be anonymous and will be held in the strictest confidence without using any personal information. The questionnaire should only take approximately 5 minutes to complete. Please make sure you answer all the following questions by Ticking your answer.

Demographic (Q1-Q4) Please tick only one

Q1: What is your gender?

1. Male. 1
2. Female. 2
3. Would prefer not to say 3

Q2: Would you please tell us about your latest qualification?

1. Bachelor. (e.g., BDS) 1
2. Master. (e.g., MSC, MClinDent) 2
3. Doctorate. (e.g., DDS, DMD, PhD) 3
4. Fellowship. (e.g., MFDS, FDS, FDS RCS) 4
5. Diploma 5
6. Other 6

Q3: How many years of experience do you have in clinical dental practice as a qualified dentist?

1. Less than one year. 1
2. 1 to 3 years 2
3. 4 to 10 years 3
4. > 10 years 4

Q4. Where did you get your clinical training?

1. In UK 1
2. Out of UK 2

Please turn over

Resin-based composite types (Q5-Q7) Please tick all that apply

Q5: What are the types of direct resin-based composite material you use mostly? Please tick all that apply

1. Macrofilled RBC. 1
2. Microfilled RBC. 2
3. Hybrid RBC. 3
4. Nanohybrid RBC. 4
5. Bulk-fill RBC 5
6. I don't know. 6
7. Other (Please specify): 7

Q6: What is the main reason behind your preference (this type or these types) of direct resin-based composite material more than others?

1. Easy to manipulate. 1
2. Easier to finish and polish. 2
3. Sticks less to the placement instruments. 3
4. It gives a better adaptation. 4
5. Gives a good aesthetic result by giving better color matching. 5
6. Less cost 6
7. The only option that is available in the clinic. 7
8. Other (Please specify): 8

Q7: When you are placing a direct resin-based composite restoration, which technique do you prefer to use?

1. Incremental technique only. 1
2. Bulk-fill technique only. 2
3. Both techniques. 3
4. Other (Please specify): 4

Please turn over

Instrument lubricants (Q8-Q15) please follow the instructions

Q8: While placing the direct resin-based composite restoration, do you use any instrument lubricants to manipulate these materials?

1. Yes | 1
2. No **(if no please go to question 8b)** | 2

Q8a: If yes for Q8, what reason supports your decision for using instrument lubricants in Q8 is: (You can choose more than one reason if you need).

1. Makes material manipulation easier and more controllable. | 1
2. Reduces the time to place the direct restoration of RBC materials. | 2
3. Gives more aesthetic result. | 3
4. I use it although I do not believe there to be an obvious benefit | 4
5. Other (Please specify): | 5

Q8b: If no for Q8, what reason supports your decision for <u>not</u> using the instrument lubricants in Q8 is: (You can choose more than one reason if you need).

1. There is no evidence to supports its use in clinical practice. | 1
2. It wastes time. | 2
3. It increases the air voids in the restorations. | 3
4. I'm afraid that would affect the mechanical properties of the materials. | 4
4. Other (Please specify): | 5

Q9: Would you please tell us which kind of instrument lubricants do you use?

1.Composite bonding agent. | 1
2. Resin modeling materials. (Unfilled resin) | 2
3. Absolute Ethanol. | 3
4. 70% Ethanol. | 4
5. Acetone. | 5
6. None. **(If none please skip to question 15)** | 6
7. Other (Please specify): | 7

Please turn over

Q10: Do you think using more than one type of lubricant would help you more to manipulate and to place the direct RBC material easily?

1. Yes, I think that would give a better result. 1
2. I think that would not give any different if we use more than one type. 2
3. That would negatively affect the RBC properties. 3
4. I don't know. 4
5. I prefer to use only one type as instrument lubricant. 5
6. Other (Please specify): 6

Q11: Depending on your answer in question number ten, why do you prefer to use this type of instrument lubricant over others?

1. It has no negative effect on RBC properties. 1
2. Other colleagues use it. 2
3. Several studies support using this lubricant. 3
4. It gives a better result than other instrument lubricants. 4
5. I was taught to use it. 5
6. Other (Please specify): 6

Q12: If you are using a composite bonding system as a lubricant which one do you use?

1. One-step composite bonding system. 1
2. Two-step composite bonding system. 2
3. Three-step composite bonding system. 3
4. I don't use these materials as instrument lubricants. 4

Q13: When you use the instrument lubricants during the restorations placement how often do you apply the lubricants?

1. With every increment. 1
2. With the first increment. 2
3. With the last increment. 3
4. Other (Please specify): 4

Q14: Which methods do you prefer to use for application of the instrument lubricants materials? Please tick all boxes that apply

1. Direct application onto the resin-based composite with a microbrush. 1
2. Dipping the instrument tip into the lubricant. 2
3. Wiping the surface of the instrument with a lubricant agents. 3
4. Other (Please specify): 4

Please turn over

*Next question for all participants to answer

Q15: Depending on your experience in dental practice, do you recommend that dental practitioners use instrument lubricants?

1. Strongly recommend.
2. Recommend.
3. Neutral.
4. Not recommend.
5. Strongly not recommend.

1
2
3
4
5

End of questionnaire

Please if you have anything else regarding the questionnaire or the topic feel free and write it down

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***Please return the questionnaire in the envelope provided.**

Appendix 4: Measuring optical density

The following figures show the steps for measuring the optical density ratio of each experimental groups using ImageJ software.

Step 1

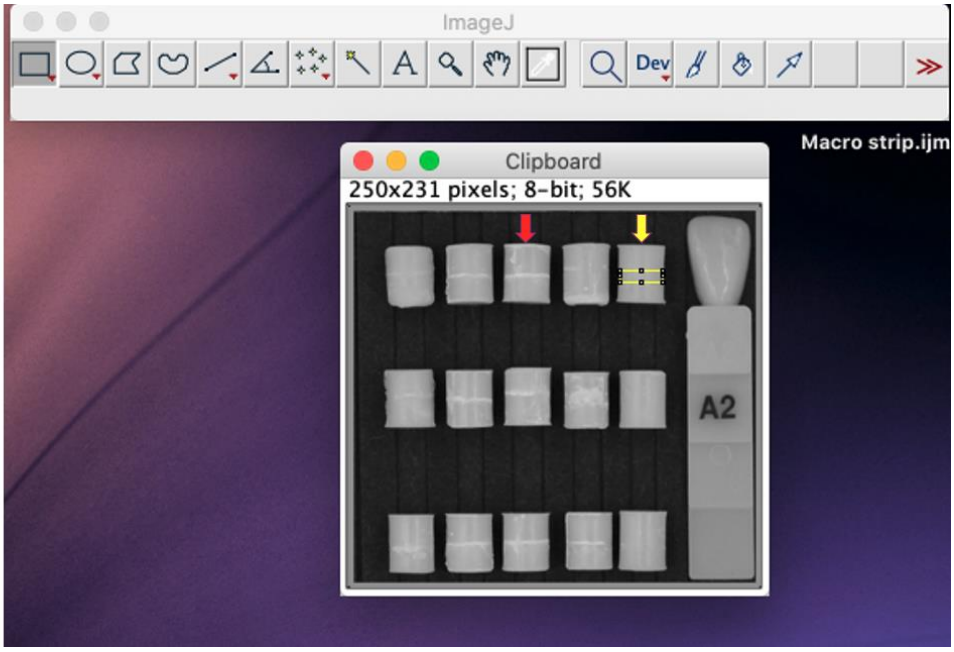
Determining the investigated area and box location

Ethanol top (A)
Control top (B)
Ethanol mid. (C)
Control mid (D)

* Control and Ethanol (have 3 specimens in column)

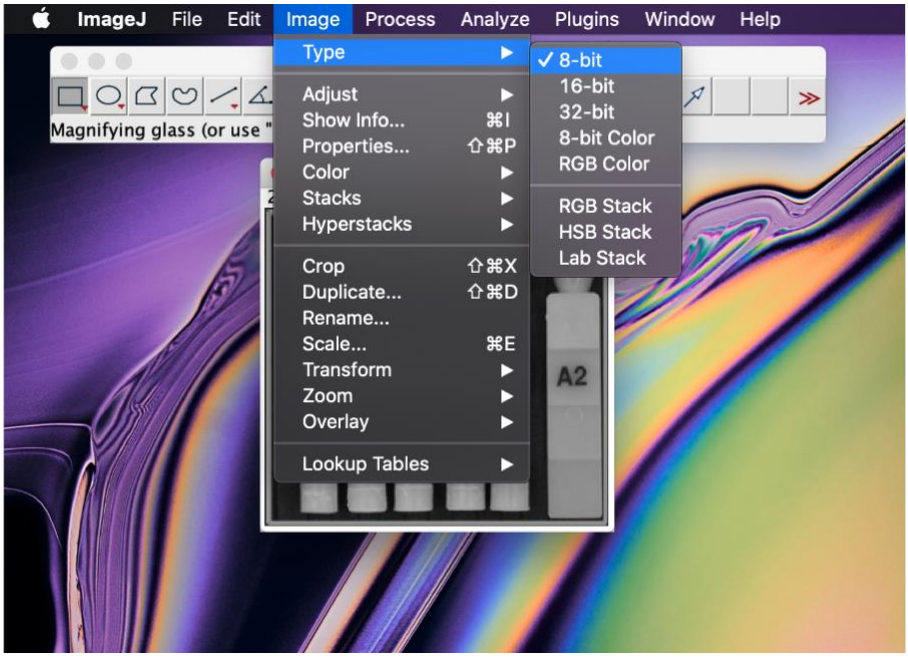
Control

Ethanol



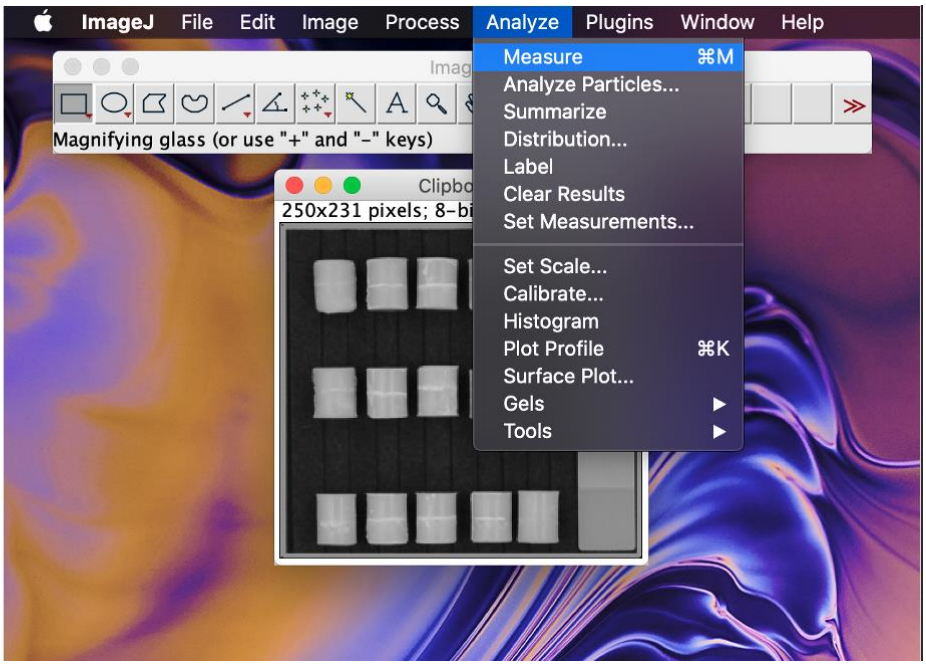
Step 2

After the software was calibrated to 8-bit the picture changed to this type to be in grayscale and measuring the different in the intensity in the determined area inside the box.



Step 3

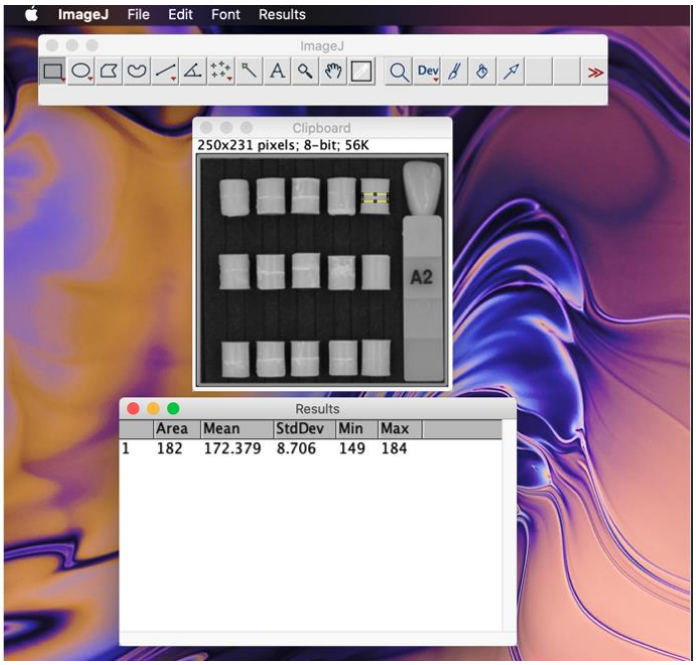
In this step the measure option was selected to measure the intensity in grayscale to get the mean value for each reading



Step 4

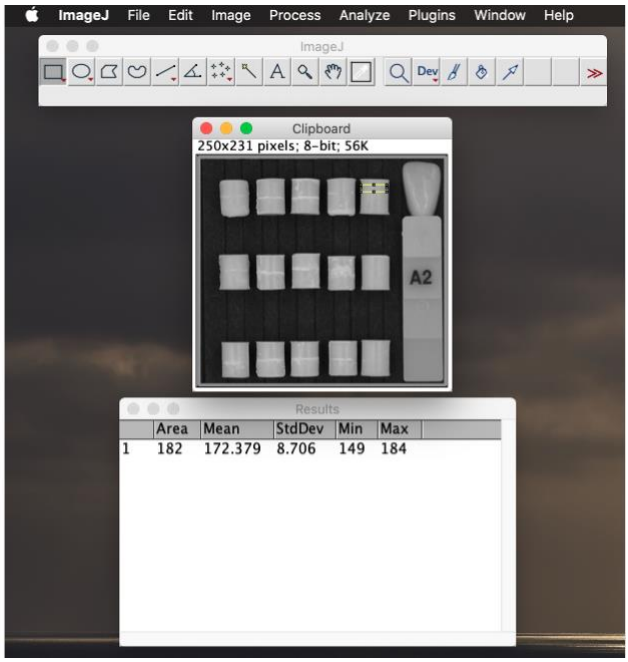
The box at the bottom showing the following:

- Area (the box size).
- Mean of the values that were collected from the determined area.
- STDEV of the collected values.
- The Min and Max values of each reading.
- The values mean and STDEV of all three specimens of each group were calculated for each area (A,B,C and D)



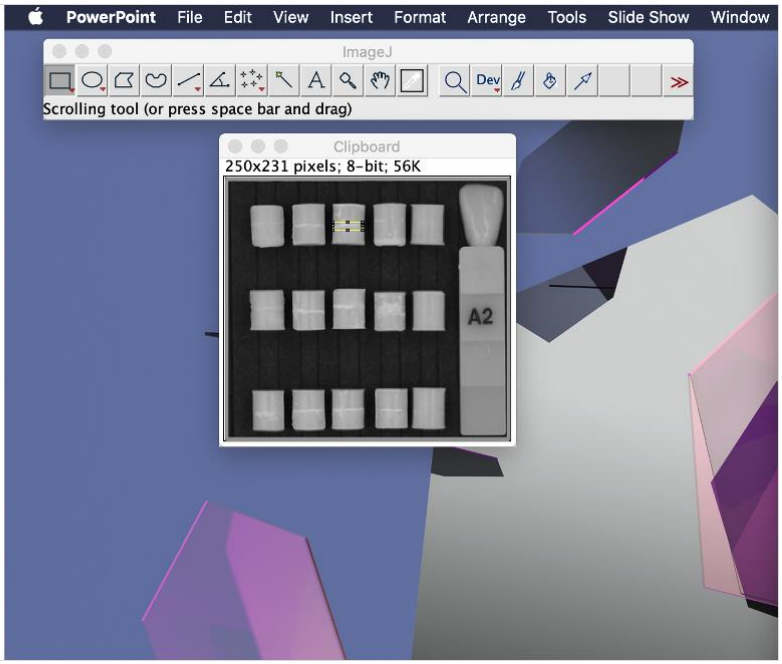
Step 5

The same procedure was repeated at the middle and top area of each specimen of the control specimens



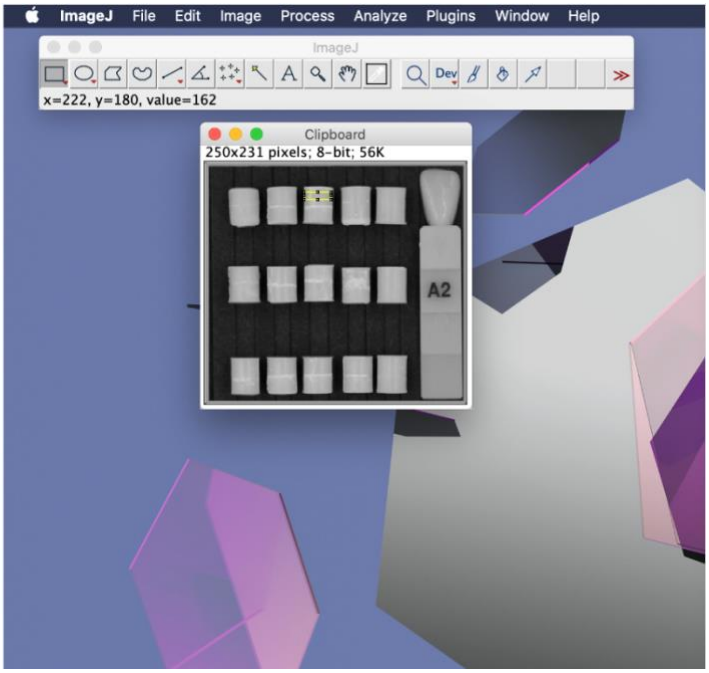
Step 6

The same procedure was repeated at the middle and top area of each specimen of the ethanol specimens.

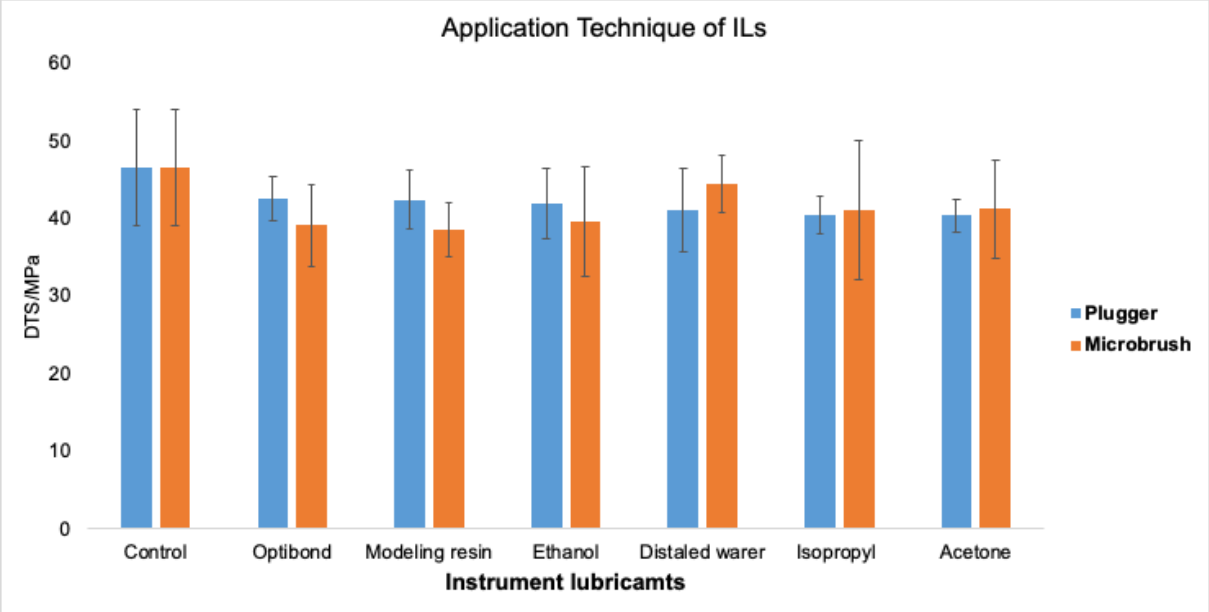


Step 7

The same procedure was repeated at the middle and top area of each specimen of the ethanol specimens.



Appendix 5: Application techniques of ILs



Appendix 6: The laboratory investigations conducted in chapter five values tables

ILs Classes	groups	Days					
		Baseline	Day-1	Day-7	Day-14	Day-28	Day-60
Solvents	Control	1.01	1.00	1.01	1.00	1.00	1.01
	Acetone	1.04	1.02	1.03	1.03	1.02	1.03
	Ethanol	1.06	1.04	1.06	1.05	1.04	1.04
	Isopropyl	1.05	1.06	1.06	1.05	1.04	1.03
	Distilled Water	1.03	1.03	1.02	1.01	1.02	1.01
BA	Scotchbond	1.02	1.01	1.02	1.00	1.01	1.01
	Optibond	1.02	1.00	1.01	1.03	1.02	1.00
WR	Brush & Sculpt	1.00	1.01	1.02	1.01	1.03	1.01
	Modeling Resin	0.99	1.00	1.00	1.00	1.00	1.00
	Signum Liquid	1.01	0.99	1.00	1.01	0.99	1.00

Optical density ratio values for all Filtek groups

ILs class	Groups	Days					
		Baseline	Day-1	Day-7	Day-14	Day-28	Day-60
Solvents	Control	1.00	1.00	1.00	1.00	1.00	1.00
	Acetone	1.06	1.04	1.04	1.03	1.04	1.03
	Ethanol	1.05	1.05	1.05	1.02	1.03	1.03
	Isopropyl	1.03	1.06	1.04	1.03	1.03	1.04
	Distilled Water	1.03	1.01	1.01	1.02	1.00	1.00
BA	Scotchbond	0.98	0.99	0.99	0.97	0.99	0.98
	Optibond	1.05	1.01	1.02	1.03	1.00	1.01
WR	Brush & Sculpt	1.00	1.01	1.01	0.99	1.00	1.01
	Modeling Resin	0.99	1.00	0.99	1.00	1.00	1.00
	Signum Liquid	1.01	0.99	1.01	1.01	0.98	1.01

Optical density ratio values for all Harmonize group

Resins	Groups	Solvents/Setup one surfaces			
		Non-lubricated top surface		Lubricated bottom surface	
		Baseline	Day-1	Baseline	Day-1
Filtek	Control	60(4.4)	62(5.3)	57(6.3)	58(7.6)
	Acetone	61(3.2)	62(1.4)	58(5.2)	58(6.6)
	Ethanol	62(2.5)	63(5.1)	63(2.6)	64(1.9)
	Isopropyl	60(3.1)	63(1.9)	58(2.1	62(3.2)
	Distilled water	62(2.1)	63(1.9)	44(7.1)	46(8.9)
Harmonize	Control	62(4.3)	63(3.6)	43(7.7)	48(4.6)
	Acetone	61(6.1)	63(2.2)	49(1.5)	53(1.9)
	Ethanol	63(5.2)	64(4.8)	64(2.5)	67(1.6)
	Isopropyl	62(4.2)	64(2.3)	61(4.1)	62(6.3)
	Distilled water	61(8.3)	62(3.1)	40(7.1)	40(3.1)

Mean percentage values and standard deviation scores for the degree of conversion for the solvents groups (Filtek & Harmonize) for non-lubricated top surface and lubricated bottom surface of setup one.

Resins	Groups	Bonding agents/Setup one surfaces			
		Non-lubricated top surface		Lubricated bottom surface	
		Baseline	Day-1	Baseline	Day-1
Filtek	Control	60(4.4)	62(5.3)	57(6.3)	58(7.6)
	Scotchbond	60(2.6)	61(3.9)	71(5.1)	72(3.8)
	Optibond	62(1.9)	63(1.4)	72(5.6)	72(5.9)
Harmonize	Control	62(4.3)	63(3.6)	43(7.7)	48(4.6)
	Scotchbond	61(8.8)	64(2.1)	56(4.5)	58(4.5)
	Optibond	61(9.9)	62(4.7)	73(4.7)	78(2.9)

Mean percentage values and standard deviation scores for the degree of conversion for the bonding agent groups (Filtek & Harmonize) for non-lubricated top surface and lubricated bottom surface of setup one.

Resins	Groups	Wetting resins/ Setup one surfaces			
		Non-lubricated top surface		Lubricated bottom surface	
		Baseline	Day-1	Baseline	Day-1
Filtek	Control	60(4.4)	62(5.3)	57(6.3)	58(7.6)
	Brush & Sculpt	62(1.7)	63(2.7)	70(7.5)	75(3.1)
	Modeling resin	62(1.5)	63(1.9)	62(5.6)	63(1.1)
	Signum liquid	62(1.4)	62(1.5)	38(5.9)	41(5.8)
Harmonize	Control	62(4.3)	63(3.6)	43(7.7)	48(4.6)
	Brush & Sculpt	61(10.1)	62(5.6)	82(1.1)	83(2.1)
	Modeling resin	62(2.5)	62(5.1)	74(2.4)	76(2.1)
	Signum liquid	61(2.3)	63(2.8)	52(3.1)	53(7.2)

Mean percentage values and standard deviation scores for the degree of conversion for the wetting resins groups (Filtek & Harmonize) for non-lubricated top surface and lubricated bottom surface of setup one.

Resins	Groups	Solvents/Setup two surfaces			
		Lubricated top surface		Non-lubricated bottom surface	
		Baseline	Day-1	Baseline	Day-1
Filtek	Control	58(1.1)	60(1.5)	40(6.4)	41(2.3)
	Acetone	63(4.9)	64(3.1)	34(7.7)	35(8.3)
	Ethanol	67(4.4)	69(2.1)	36(7.6)	37(7.3)
	Isopropyl	71(3.8)	73(3.4)	34(12)	38(15)
	Distilled water	63(5.1)	64(6.5)	35(5.3)	36(2.1)
Harmonize	Control	58(4.5)	60(3.3)	34(8.6)	39(8.8)
	Acetone	54(10)	55(9.5)	38(7.1)	40(3.5)
	Ethanol	64(10)	66(9.1)	43(4.5)	44(5.7)
	Isopropyl	60(12.7)	66(7.4)	39(9.6)	43(10)
	Distilled water	53(9.4)	56(10)	39(4.6)	41(4.9)

Mean percentage values and standard deviation scores for the degree of conversion for the solvents groups (Filtek & Harmonize) for lubricated top surface and non-lubricated bottom surface of setup two.

Resins	Groups	Bonding agents/ Setup two surfaces			
		Lubricated top surface		Non-lubricated bottom surface	
		Baseline	Day-1	Baseline	Day-1
Filtek	Control	59(1.9)	60(1.1)	40(6.4)	41(2.3)
	Scotchbond	84(3.9)	85(4.9)	41(2.3)	47(3.9)
	Optibond	83(2.5)	84(2.3)	52(6.6)	53(4.6)
Harmonize	Control	58(4.5)	60(3.2)	34(8.6)	39(8.6)
	Scotchbond	74(10)	75(11)	44(4.5)	48(4.5)
	Optibond	68(11)	74(4.3)	48(6.2)	52(5.5)

Mean percentage values and standard deviation scores for the degree of conversion for the bonding agents groups (Filtek & Harmonize) for lubricated top surface and non-lubricated bottom surface of setup two.

Resins	Groups	Wetting resins/ setup two surfaces			
		Lubricated top surface		Non-lubricated bottom surface	
		Baseline	Day-1	Baseline	Day-1
Filtek	Control	58(1.1)	60(1.5)	40(6.4)	41(2.3)
	Brush & Sculpt	77(2.3)	79(2.1)	45(9.2)	48(9.2)
	Modeling resin	67(2.9)	69(3.5)	41(7.9)	44(6.6)
	Signum liquid	58(4.1)	59(3.3)	39(4.6)	46(4.7)
Harmonize	Control	58(4.5)	60(3.3)	34(8.6)	39(8.8)
	Brush & Sculpt	80(2.4)	82(2.4)	42(13.9)	44(12.6)
	Modeling resin	70(10)	71(8.4)	48(9.7)	48(13.2)
	Signum liquid	48(12)	54(6.1)	34(16.2)	35(5.2)

Mean percentage values and standard deviation scores for the degree of conversion for the wetting resins groups (Filtek & Harmonize) for lubricated top surface and non-lubricated bottom surface of setup two.

Resins	Groups	Solvents/Setup one surfaces	
		Non-lubricated top surface	Lubricated bottom surface
Filtek	Control	608(27)	515(62)
	Acetone	614(68)	447(65)
	Ethanol	588(42)	372(67)
	Isopropyl	587(38)	418(66)
	Distilled water	608(74)	493(72)
Harmonize	Control	502(69)	399(54)
	Acetone	503(44)	380(58)
	Ethanol	476(71)	382(77)
	Isopropyl	502(18)	363(49)
	Distilled water	502(74)	393(72)

Mean (N/mm2) and standard deviation values for Martens hardness tests of solvents (Filtek & Harmonize) on non-lubricated top surface and lubricated to surface in setup one.

Resins	Groups	Bonding agents/Setup one surfaces	
		Non-lubricated top surface	Lubricated bottom surface
Filtek	Control	608(27)	515(62)
	Scotchbond	574(30)	453(73)
	Optibond	579(61)	411(68)
Harmonize	Control	502(69)	399(54)
	Scotchbond	498(28)	395(55)
	Optibond	501(40)	357(60)

Mean (N/mm^2) and standard deviation values for Martens hardness tests (Filtek & Harmonize) for non-lubricated top surface and bonding agent-lubricated bottom surface in setup one.

Resins	Groups	Wetting resins/Setup one surfaces	
		Non-lubricated top surface	Lubricated bottom surface
Filtek	Control	608(27)	515(62)
	Brush & Sculpt	631(16)	335(103)
	Modeling resin	602(34)	468(100)
	Signum liquid	623(28)	500(54)
Harmonize	Control	502(69)	399(54)
	Brush & Sculpt	488(27)	360(70)
	Modeling resin	492(40)	304(79)
	Signum liquid	552(85)	410(47)

Mean (N/mm2) and standard deviation values for Martens hardness tests of wetting resins (Filtek & Harmonize) for non-lubricated top surface and wetting resin-lubricated bottom surface in setup one.

Resins	Groups	solvents/Setup two surfaces	
		Lubricated top surface	Non-lubricated bottom surface
Filtek	Control	609(45)	547(63)
	Acetone	594(41)	570(44)
	Ethanol	574(37)	587(75)
	Isopropyl	487(58)	572(35)
	Distilled water	598(84)	557(76)
Harmonize	Control	479(49)	450(40)
	Acetone	473(13)	451(19)
	Ethanol	438(71)	490(22)
	Isopropyl	393(54)	414(67)
	Distilled water	502(78)	444(57)

Mean (N/mm^2) and standard deviation values for Martens hardness tests (Filtek & Harmonize) for solvent-lubricated top surface and non-lubricated bottom surface in setup two.

Resins	Groups	Bonding agents/Setup two surfaces	
		Lubricated top surface	Non-lubricated bottom surface
Filtek	Control	609(45)	547(63)
	Scotchbond	490(61)	605(20)
	Optibond	415(71)	638(19)
Harmonize	Control	479(49)	450(40)
	Scotchbond	360(39)	448(57)
	Optibond	262(50)	456(54)

Mean (N/mm^2) and standard deviation values for Martens hardness tests (Filtek & Harmonize) for bonding agent-lubricated top surface and non-lubricated bottom surface in setup two.

Resins	Groups	Wetting resins/Setup two surfaces	
		Lubricated top surface	Non-lubricated bottom surface
Filtek	Control	609(45)	547(63)
	Brush & Sculpt	309(32)	578(42)
	Modeling resin	412(71)	557(27)
	Signum liquid	468(60)	578(62)
Harmonize	Control	479(49)	450(40)
	Brush & Sculpt	296(35)	378(113)
	Modeling resin	238(117)	481(32)
	Signum liquid	260(52)	420(136)

Mean (N/mm^2) and standard deviation Martens hardness values (Filtek & Harmonize RBCs) for wetting resin-lubricated top surface and non-lubricated bottom surface in setup two.

Resins	Groups	Days								
		Day-1	Day-2	Day-3	Day-4	Day-7	Day-14	Day-28	Day-60	Day-90
Filtek	Control	1.1(0.24)	0.8(0.16)	0.9(0.11)	0.9(0.20)	1.3(0.18)	2.0(0.30)	2.1(0.18)	2.0(0.19)	2.1(0.18)
	Ethanol	1.2(0.50)	0.8(0.27)	1.1(0.12)	1.2(0.13)	1.7(0.18)	2.5(0.24)	2.5(0.26)	2.5(0.26)	2.5(0.30)
	Acetone	0.3(0.23)	0.2(0.25)	0.5(0.17)	0.5(0.12)	0.9(0.13)	1.5(0.24)	1.5(0.29)	1.6(0.25)	1.5(0.30)
	Isopropyl	0.7(0.31)	0.5(0.39)	0.8(0.33)	0.8(0.23)	1.2(0.29)	1.8(0.39)	1.8(0.39)	1.8(0.40)	1.9(0.39)
	Distilled water	0.7(0.30)	0.9(0.07)	0.9(0.10)	0.8(0.12)	1.3(0.16)	1.7(0.17)	1.8(0.31)	1.8(0.26)	1.7(0.35)
Harmonize	Control	0.8(0.07)	0.8(0.23)	1.0(0.20)	1.1(0.20)	1.4(0.17)	1.8(0.20)	1.8(0.17)	1.7(0.22)	1.7(0.23)
	Ethanol	0.8(0.33)	1.0(0.21)	1.0(0.21)	1.2(0.29)	1.5(0.26)	1.8(0.45)	1.8(0.45)	1.8(0.45)	1.8(0.45)
	Acetone	0.9(0.12)	1.1(0.17)	1.2(0.11)	1.3(0.05)	1.8(0.18)	2.0(0.50)	2.0(0.50)	2.0(0.51)	2.1(0.50)
	Isopropyl	0.9(0.15)	1.1(0.18)	1.3(0.12)	1.3(0.11)	2.0(0.16)	2.1(0.16)	2.1(0.15)	2.1(0.19)	2.1(0.20)
	Distilled water	0.9(0.15)	1.1(0.18)	1.3(0.12)	1.3(0.11)	2.0(0.16)	2.1(0.16)	2.1(0.15)	2.1(0.19)	2.1(0.20)

Solvent group % means for weight change and standard deviation for water uptake for Filtek and Harmonize stored in distilled water <90 days.

Resins	Groups	Days								
		Day-1	Day-2	Day-3	Day-4	Day-7	Day-14	Day-28	Day-60	Day-90
Filtek	Control	1.1(0.24)	0.8(0.16)	0.9(0.11)	0.9(0.20)	1.3(0.18)	2.0(0.30)	2.1(0.18)	2.1(0.19)	2.0(0.18)
	Scotchbond	1.0(0.21)	0.9(0.29)	1.1(0.40)	1.0(0.42)	2.2(0.39)	2.2(0.40)	2.2(0.40)	2.3(0.44)	2.4(0.35)
	Optibond	1.0(0.22)	1.3(0.32)	1.3(0.19)	1.4(0.20)	2.1(0.24)	2.3(0.29)	2.3(0.29)	2.3(0.28)	2.3(0.31)
Harmonize	Control	0.8(0.07)	0.8(0.23)	1.0(0.20)	1.1(0.20)	1.4(0.17)	1.8(0.20)	1.8(0.17)	1.7(0.22)	1.7(0.23)
	Scotchbond	1.0(0.11)	1.2(0.14)	1.0(0.24)	1.3(0.07)	1.3(0.07)	1.7(0.09)	1.8(0.15)	1.8(0.14)	1.8(0.26)
	Optibond	0.6(0.22)	0.6(0.14)	0.9(0.28)	1.1(0.12)	1.3(0.19)	1.6(0.18)	1.6(0.14)	1.6(0.13)	1.5(0.11)

Bonding agent group means of % weight change and standard deviation for the water uptake in Filtek and Harmonize, stored in distilled water <90 days.

Resins	Groups	Days								
		Day-1	Day-2	Day-3	Day-4	Day-7	Day-14	Day-28	Day-60	Day-90
Filtek	Control	1.1(0.24)	0.8(0.16)	0.9(0.11)	0.9(0.20)	1.3(0.18)	2.0(0.30)	2.1(0.18)	2.1(0.19)	2.0(0.18)
	Sculpt & Brush	0.7(0.31)	0.9(0.30)	1.0(0.23)	0.9(0.32)	1.1(0.13)	1.9(0.25)	2.0(0.21)	2.0(0.20)	1.9(0.19)
	Modeling resin	0.9(0.34)	0.8(0.21)	1.1(0.25)	1.2(0.19)	1.4(0.19)	2.4(0.40)	2.4(0.40)	2.5(0.50)	2.5(0.43)
	Signum liquid	0.9(0.24)	0.8(0.22)	0.9(0.32)	1.0(0.50)	1.4(0.45)	2.2(0.31)	2.2(0.31)	2.2(0.31)	2.1(0.30)
Harmonize	Control	0.8(0.07)	0.8(0.23)	1.0(0.20)	1.1(0.20)	1.4(0.17)	1.8(0.20)	1.8(0.17)	1.7(0.22)	1.7(0.23)
	Sculpt & Brush	0.6(0.35)	0.7(0.42)	0.8(0.32)	0.9(0.27)	1.3(0.33)	1.7(0.36)	1.7(0.36)	1.7(0.28)	1.8(0.39)
	Modeling resin	0.7(0.28)	1.3(0.15)	1.1(0.19)	1.1(0.16)	1.6(0.15)	1.8(0.20)	1.9(0.20)	1.9(0.21)	1.9(0.16)
	Signum liquid	1.1(0.24)	1.3(0.8)	1.2(0.16)	1.2(0.21)	2.0(0.19)	2.0(0.17)	2.0(0.14)	2.0(0.15)	2.0(0.15)

Wetting resin group mean % weight change and standard deviation of water uptake for Filtek and Harmonize, stored in distilled water <90 days.

Resins	Groups	Days				
		Baseline	Day-1	Day-7	Day-28	Day-90
Filtek	Control	50(5)	49(6)	46(6)	42(8)	39(5)
	Ethanol	47(8)	46(9)	43(8)	32(7)	27(7)
	Acetone	50(8)	49(6)	44(7)	34(7)	28(7)
	Isopropyl	48(8)	48(6)	40(9)	33(7)	27(4)
	Distilled water	49(8)	49(4)	39(7)	34(6)	28(4)
Harmonize	Control	40(7)	36(5)	36(5)	34(4)	25(3)
	Ethanol	37(5)	37(9)	32(7)	32(6)	25(5)
	Acetone	40(4)	41(3)	35(9)	31(4)	26(4)
	Isopropyl	40(5)	40(4)	37(5)	30(5)	28(4)
	Distilled water	40(5)	40(3)	36(7)	33(4)	25(6)

Solvents groups mean (MPa) DTS and standard deviation values for Filtek and Harmonize, stored in distilled water <90 days

Resins	Groups	Days				
		Baseline	Day-1	Day-7	Day-28	Day-90
Filtek	Control	50(5)	49(6)	46(6)	42(8)	39(5)
	Scotchbond	43(8)	45(5)	43(7)	37(6)	26(6)
	Optibond	45(8)	45(10)	43(8)	35(5)	25(6)
Harmonize	Control	41(7)	36(5)	36(5)	34(4)	25(3)
	Scotchbond	46(6)	37(6)	35(5)	27(6)	22(3)
	Optibond	44(7)	46(8)	34(5)	39(4)	23(6)

Bonding agent group mean (MPa) and standard deviation DTS for Filtek and Harmonize, stored in distilled water <90 days.

Resins	Groups	Days				
		Baseline	Day-1	Day-7	Day-28	Day-90
Filtek	Control	50(5)	49(6)	46(6)	41(8)	39(5)
	Sculpt & Brush	51(6)	52(6)	47(8)	32(7)	30(7)
	Modeling resin	39(9)	47(8)	41(7)	29(6)	27(8)
	Signum liquid	49(7)	50(6)	45(9)	35(5)	32(6)
Harmonize	Control	41(7)	36(5)	36(5)	34(4)	25(3)
	Sculpt & Brush	42(7)	42(5)	39(4)	25(6)	24(6)
	Modeling resin	39(5)	38(5)	29(4)	27(10)	22(3)
	Signum liquid	38(8)	40(6)	29(7)	28(7)	28(4)

Wetting resin group mean (MPa) DTS and standard deviation values for Filtek and Harmonize, stored in distilled water <90 days.

Appendix 7: Microtensile bonding strength

Solvents	Mean	
	Baseline	Day-7
Control	39 (6)	36 (7)
Acetone	35 (9)	27 (9)
Ethanol	30 (10)	28 (6)
Isopropyl	21 (5)	17 (6)
Distilled Water	35 (9)	33 (8)

Solvents groups mean (MPa) μTBS and standard deviation values for Filtek

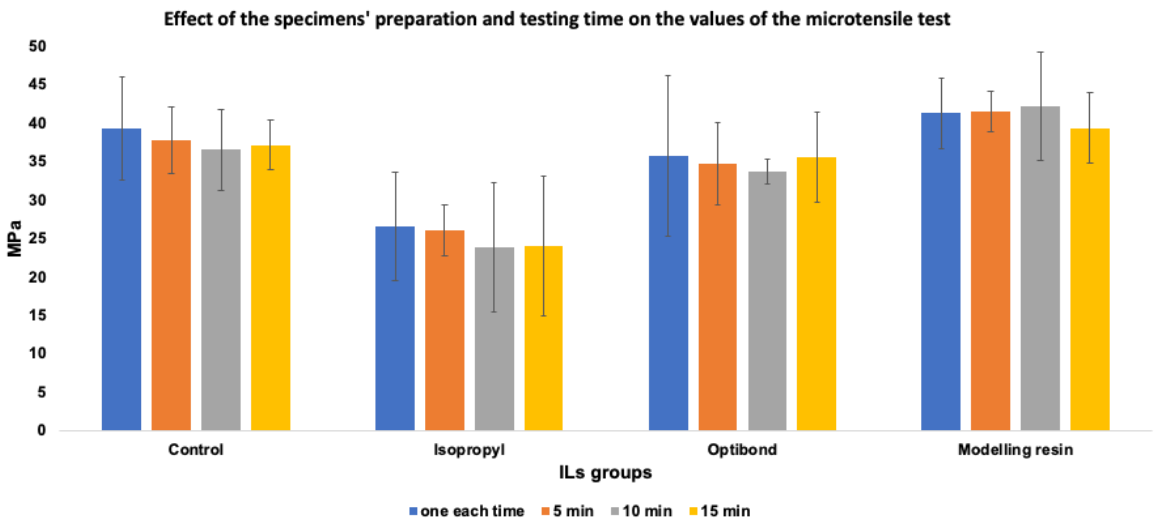
Bonding Agents	Mean	
	Baseline	Day-7
Control	39 (6)	36 (7)
Scotchbond	32 (7)	29 (10)
Optibond	31 (9)	25 (4)

Bonding agents groups mean (MPa) μTBS and standard deviation values for Filtek

Wetting Resins	Mean	
	Baseline	Day-7
Control	39 (6)	36 (7)
Brush & Sculpt	57 (6)	47 (6)
Modeling resin	45(8)	43 (8)
Signum liquid	55 (8)	47 (6)

Wetting resins groups mean (MPa) μTBS and standard deviation values for Filtek

Appendix 8: Effect of specimen preparation and testing time on the values of the microtensile test



Mean/STDEV				
Groups	one each time	5 min	10 min	15 min
Control	39 (7)	38 (4)	37 (5)	37 (3)
Isopropyl	27 (7)	26 (3)	24 (8)	24 (9)
Optibond	36 (11)	35 (5)	34 (2)	36 (6)
Modelling resin	41(5)	42 (3)	42 (7)	39 (5)

Appendix 9: IADR posters

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Investigation of the use of instrument lubricants to place composite restorations

S. A. Alqahtani, G. I. McCracken, M. J. German

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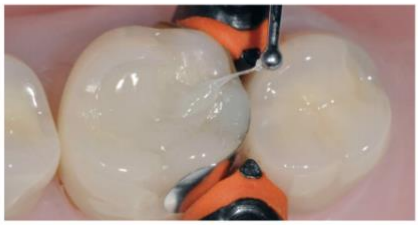
Aim

To investigate how and why UK dentists use instrument lubricants (ILs) when placing resin-based composite restorations (RBCRs).

Introduction

- Due to the tackiness of the RBC restorative materials to the placement instruments, previous studies have discussed different trials to reduce the manipulation difficulty of the RBC materials(1).
- The most used method in dental practice was the use of ILs, to decrease the tackiness of RBC material to the placement instruments(1).

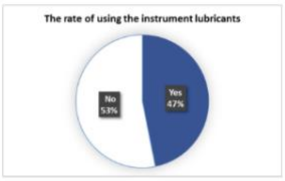


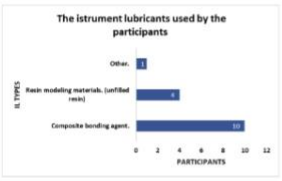
Materials and Methods

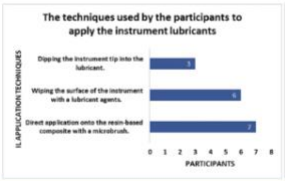
- Questionnaire-based survey of registered dentists in the UK was conducted.
- Paper based documentation was sent via their identified dental practice mail address.

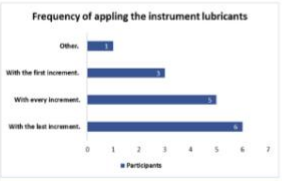
Results

- The response rate was 32%. One respondent did not provide demographic information but completed the remainder of the questionnaire.
- Most of them have practiced more than ten years and their clinical training was in the UK.
- Their qualifications were varied and the majority held Bachelor degree.









Discussion

- Response rate of the mailed questionnaire-based survey have been anticipated by the literature to be 40% or lesser (2). That can be comparable to the response rate that was founded in the current study.
- The survey was beneficial to know the opinion of the UK dentists regarding the manipulation of the RBC restorations with ILs.
- Also, it was an effective tool to find out the most ILs were used in their practice to manipulate the RBC restorations. Moreover, what were the methods and techniques of application they used to apply those materials.

Conclusion

Nearly half of the dentists used an instrument lubricants during the placement of resin-based composite restorations, with most using a bonding agent. The majority applied ILs either on the top of the last increment or with each increment placed. They used the microbrush or wiping the placement instrument surface to diminish the sticking of the RBC to the placement instruments.

References

1-(Patel et al., 2017)(Paula et al., 2016)(Tuncer et al., 2013)(Barcellos et al., 2011)(Dunn, 2007)(Gorge Perdigao, 2006)(Liebenberg, 1999)(Anthony et al., 1998)(Sneed WD, 1980).
2-(R.T. Tan, 1997) (David A. Asch et al., 1997) (S. Watt et al.,2002) (North East and North Cumbria Dental Workforce Survey, 2016)

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Effect of instrument lubricants on microtensile strength of resin-based composites

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Aim

To investigate the effect of using instrument lubricants (ILs) on the microtensile bond strength between increments of a resin-based composite (RBC).

Introduction

- Applying ILs between layers of RBC materials during their placement into a cavity has the potential to affect the mechanical properties and the stability of these restorative materials (1).
- The microtensile testing is a frequently used test to evaluate bonding between different substrates. Depending on the tensile strength, which is the most force would lead to failing most of the tested restorative materials (2).

Materials and Methods

One RBC (Filtek supreme XTE, 3M, USA) and three classes of IL were investigated: solvents; bonding agents; and wetting resins. Two RBC blocks were sectioned to produce at least 20 specimens (1mm X 1 mm cross-section) for testing in each group. All specimens were tested under tensile load.

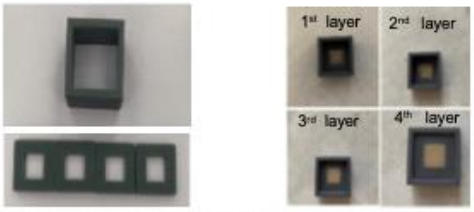

Figure-1: The mould layers and block building up. (each layer 2.5mm thickness)

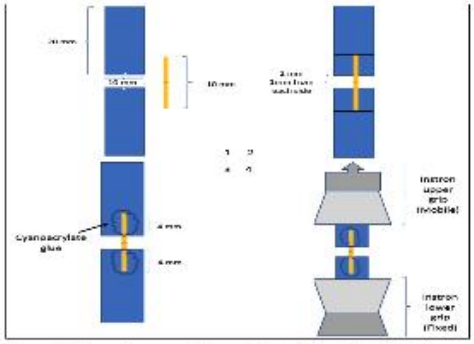

Figure-2: The reference points and the position of the tested specimens and the area that glued by cyanoacrylate glue.

Results

Both solvents and bonding agents had microtensile strengths that were statistically significantly lower compared to the control (P=0.001). They both reduced the microtensile strength at both baseline and after 7 days. The wetting resins did not reduce the microtensile strength.

Classes	ILs	24 hours (Mean/SD)	7 days (Mean/SD)
Control	None	39 (8)	36 (7)
Solvents	Acetone*	35 (9)	27 (9)
	Ethanol*	30 (10)	28 (6)
	Isopropyl*	21(5)	17 (6)
	Distilled water*	35 (9)	33 (8)
Bonding agents	Scotchbond*	32 (7)	29 (10)
	Optibond*	31 (9)	25 (4)
Wetting resins	Brush & Sculpt	57 (6)	47 (6)
	Modeling resin	45 (8)	43 (8)
	Signum liquid	55 (8)	47 (6)

*represents statistically significant differences in comparison to the control group.

Table-1: Filtek RBC treated with ILs microtensile strength values mean (MPa) and standard deviation of tested groups

Discussion

- The components of both ILs and RBCs can make a difference in collected values of tested RBCs (1).
- The HEMA can reduce the density of the cross-linking of the treated materials in this work through a reduction in the formation of the dense polymer chains (3,5).
- Some components of bonding agents like ethanol and water as solvents would impact the manipulated RBCs' polymerization process and strength (5).
- The presence of the ILs substances between the increments can increase the water uptake. Also, low filled or unfilled IL substances used in the present study can make the DW uptake more than control groups (1,4).

Conclusion

Only wetting resins maintained the microtensile bond strength between increments, while solvents and bonding agents reduced it

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