THE EFFECTS OF BLEACHING PRODUCTS ON THE COLOUR STABILITY OF ION-RELEASING RESTORATIVES

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THE EFFECTS OF BLEACHING PRODUCTS ON THE COLOUR STABILITY OF ION-RELEASING RESTORATIVES

ABSTRACT

The aim of this study is to evaluate the effects of bleaching products on the colour stability of ion-releasing restorative materials (IRMs). The evaluated IRMs included bioactive composite Activa (AB), giomer Beautifil II LS (BI), bulk-fill alkasite Cention-N (CN), encapsulated Riva light-cured resin-reinforced high viscosity glass ionomer cement (RV) and a conventional nanohybrid composite resin Luna (LN). Customised cylindrical acrylic mould was used to fabricate 45 disc-shaped specimens of each material. The samples were then finished and polished, and immersed in artificial saliva for two weeks. The baseline colour of all samples was measured with a spectrophotometer using the CIEL*a*b* colour space. The specimens were then randomly distributed into three groups of 15. Group A specimens were immersed in artificial saliva (AS) for seven days, Group B specimens were bleached with the take-home bleaching product Pola Night (PN) daily for seven days, and Group C specimens were bleached with the in-office bleaching product Pola Rapid (PR) on day seven, according to manufacturer's instructions. The colour of the specimens was measured again at 24 hours (T1), two weeks (T2) and one month (T3) after bleaching. The normality of data was first determined using the Shapiro-Wilk test. Then, the colour differences between IRMs and bleaching products were analysed using one-way ANOVA. Their interaction was analysed using two-way ANOVA. Repeated measures ANOVA was done to determine the differences between T1, T2 and T3. All statistical analyses were done with a significance level of $p<0.05$. The data of all parameters were as follows: ΔL* ranged from (-0.02 ± 1.23) to (4.94 ± 1.37); Δa* varied from (-0.31 ± 0.12) to (0.65 ± 0.13); Δb* differed from (-3.08± 1.24) to (0.31 ± 0.86); ΔE* ranged from (0.94 ± 0.48) to (5.99 ± 1.33). Colour change was mainly ascribed to the increase in L* and decrease in b* values. Results of statistical analysis showed that the colour change in RV was significantly affected by PN (3.34 ± 0.72) and PR (3.55 ± 0.78), compared to the unbleached samples (1.38 ± 0.68). PN and PR produced similar colour changes. At T1, the highest colour change was seen in RV bleached with PN and PR. At T3, AB showed the greatest colour change in AS (5.99 ± 1.33), PN (3.80 ± 1.41) and PR (4.02 ± 1.31). The colour of BI and LN were the least affected by bleaching. Between time intervals, there were no differences in the colour change for all IRMs, except for AB which significantly increased from T1 to T3. In conclusion, the effects of bleaching products on the colour stability of IRMs were material dependent in which RV was significantly affected. There were no differences in the colour change produced by PR and PN. The post-bleaching colour of most IRMs remained stable over time, except for AB. This implied that bleaching product should be cautiously applied to avoid undesirable effects on restorative materials.

Keywords: Ion-releasing restoratives, tooth bleaching agents, spectrophotometry, colour stability

KESAN PRODUK PEMUTIHAN TERHADAP KESTABILAN WARNA BAHAN RESTORATIF PELEPASAN ION

ABSTRAK

Tujuan kajian ini adalah untuk menilai kesan produk pemutihan terhadap kestabilan warna bahan restoratif pelepasan ion (IRMs). IRMs yang dinilai termasuk komposit bioaktif Activa (AB), giomer Beautifil II LS (BI), pengisian pukal bahan yang beralkali Cention-N (CN), simen glass ionomer yang diperkuatkan dengan resin Riva (RV) dan resin komposit nanohibrid konvensional Luna (LN). Acuan akrilik silinder digunakan untuk menghasilkan 45 spesimen berbentuk cakera bagi setiap bahan. Kemudian., sampel kemudian dicahaya-pulih dan direndam dalam air liur tiruan selama dua minggu. Pengukuran warna asas untuk semua sampel diukur dengan spektrofotometer menggunakan ruang warna CIEL*a*b*. Spesimen kemudian dibahagikan secara rawak kepada tiga kumpulan masing-masing 15. Spesimen Kumpulan A direndam dalam air liur tiruan (AS) selama tujuh hari, spesimen Kumpulan B diputihkan dengan produk pemutihan ambil-pulang Pola Night (PN) setiap hari selama tujuh hari, dan spesimen Kumpulan C diputihkan dengan produk pemutihan dalam-pejabat Pola Rapid (PR) pada hari ketujuh mengikut arahan pengeluar. Warna spesimen diukur semula 24 jam (T1), dua minggu (T2) dan sebulan (T3) selepas pemutihan. Normaliti data ditentukan menggunakan ujian Shapiro-Wilk. Selepas itu, perbezaan warna antara IRMs dan produk pemutihan dianalisis menggunakan ANOVA satu hala. Interaksi mereka dianalisis menggunakan ANOVA dua hala. ANOVA pengukuran berulang dilakukan untuk menentukan perbezaan antara T1, T2 dan T3. Semua analisis statistik dilakukan pada paras keertian $p < 0.05$. Data semua parameter adalah seperti berikut: ΔL^* berkisar dari (-0.02 ± 1.23) hingga (4.94 ± 1.37); Δa^* berbeza dari (-0.31 ± 0.12) hingga (0.65 ± 0.13); Δb^* berbeza dari (-3.08 ± 1.24) hingga (0.31 ± 0.86); ΔE^* berkisar dari (0.94 ± 0.48) hingga (5.99 ± 1.33). Perubahan warna terutamanya disebabkan oleh peningkatan nilai L* dan penurunan nilai b*. Hasil analisis statistik menunjukkan bahawa perubahan warna dalam RV secara signifikan dipengaruhi oleh PN (3.34 ± 0.72) dan PR (3.55 ± 0.78), berbanding dengan sampel yang tidak diputihkan (1.38 ± 0.68). PN dan PR menghasilkan perubahan warna yang serupa. Pada T1, RV yang diputihkan dengan PN dan PR menunjukkan perubahan warna tertinggi berbanding IRMs lain. Pada T3, AB menunjukkan perubahan warna terbesar dalam AS (5.99 ± 1.33), PN (3.80 ± 1.41) dan PR (4.02 ± 1.31). Warna BI dan LN paling kurang dijejaskan oleh pemutihan. Antara selang masa, tidak terdapat perbezaan dalam perubahan warna untuk semua IRMs, kecuali untuk AB yang meningkat secara signifikan dari T1 ke T3. Kesimpulannya, kesan produk peluntur terhadap kestabilan warna IRM bergantung pada bahan yang digunakan di mana RV terjejas dengan ketara. Tiada perbezaan dalam perubahan warna yang dihasilkan oleh PR dan PN. Warna selepas pelunturan kebanyakan IRM kekal stabil dari masa ke masa, kecuali AB. Ini menunjukkan bahawa produk peluntur harus digunakan dengan berhati-hati untuk mengelakkan kesan yang tidak diingini pada bahan restoratif

Kata kunci: Bahan restoratif pelepasan ion, produk pemutihan gigi, spektrofotometri, kestabilan warna

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TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

LIST OF SYMBOLS AND ABBREVIATIONS

°C	:	Degree Celcius
a*	:	Colour Along The Red And Green Dimension
AB	:	Activa Bioactive Restorative
ANOVA	:	Analysis Of Variance
Aromatic Aliphatic-UDMA	:	Tetramethyl-Xylylendiurethane Dimethacrylate
AS	:	Artificial Saliva
b*	:	Colour Along The Yellow And Blue Dimension
BI	:	Beautifil Ii Ls
Bis-GMA	:	Bisphenol-A Glycidyl Methacrylate
Bis-MPEPP	:	2,2-Bis (4-Methacryloxypolyethoxyphenyl) Propane
CIE	:	Commission Internationale Del'eclairege
CN	:	Cention-N
CR	:	Composite Resin
DC	:	Tricyclodecan-Dimethanol Dimethacrylate
F-PRG	:	Full Pre-Reacted Glass Ionomer
GIC	:	Glass Ionomer Cement
HEMA	:	2-Hydroxyethyl Methacrylate
HVGIC	:	High Viscosity Glass Ionomer Cement
IRM	:	Ion-Releasing Restorative Material
ISO	:	International Standarisation Organisation
L*	:	Lightness From Black To White
LC	:	Light Cure
LED	:	Light-Emitting Diode
LN	:	Luna

mgL^{-1}	Milligram per Litre
MPA	Mega Pascal
PEG-400 DMA	Polyethylene Glycol 400 Dimethacrylate
PN	Pola Night
PR	Pola Rapid
RMGIC	Resin-Modified Glass Ionomer Cement
RV	Riva Light Cure Hv
S-PRG	Surface Pre-Reacted Glass Ionomer
Subscript 0	The Measurement Value At Baseline (T0)
Subscript i	The Measurement Value At 24 Hours (T1), Two Weeks (T2), And One Month (T3)
T0	Before Bleaching At Baseline
T1	24 Hours After Bleaching
T2	Two Weeks After Bleaching
T3	One Month After Bleaching
TEGDMA	Triethylene Glycol Dimethacrylate
UDMA	Urethane Dimethacrylate
Δa*	Change In The Colour Along The Red And Green Dimension
Δb*	Change In The Colour Along The Yellow And Blue Dimension
ΔE*	Colour Change
ΔE_{00}	CIEDE2000 Colour-Difference Formula
ΔL*	Change In The Lightness

CHAPTER 1: INTRODUCTION

1.1 Introduction

Direct tooth-coloured restorations are frequently utilised to replace damaged tooth structures and enhance dental aesthetics. Composite resin (CR) remains the preferred restorative material in modern dentistry owing to its excellent mechanical and aesthetic properties. Nevertheless, polymerisation shrinkage in CR restorations can contribute to bonding failures and microleakage (Bilgrami et al., 2022). The existence of a gap at the interface between the tooth-restoration margin facilitates plaque accumulation and bacterial infiltration, leading to the potential development of recurrent caries (Fabianelli et al., 2007). Indeed, systematic reviews have indicated that recurrent caries significantly contribute to the failures of CR restorations (Demarco et al., 2015; Opdam et al., 2014). Hence, ion-releasing restorative materials (IRMs) were developed to prevent secondary caries. The first IRM introduced in 1972, glass ionomer cement (GIC) was capable of creating an ion-interchange layer which facilitates chemical bonding at the tooth-restoration interface and the release of fluoride ions from the material (Pires et al., 2020). Fluoride ions promote the formation of fluorapatite crystals, which are highly resistant to acid attacks, thus effectively inhibiting demineralisation (Dionysopoulos, 2014). GIC has been documented to offer enhanced clinical performance in terms of secondary caries prevention in comparison to CR, due to its superior biocompatibility, chemical adhesion, similar coefficient of thermal expansion to dentine, and its capacity for fluoride release and recharge (Dias et al., 2018).

Conventional GIC has poor mechanical properties and translucency which limit its usage in stress-bearing and aesthetic zones of the dentition. To overcome these drawbacks, contemporary IRMs were introduced as hybrid materials that contain resin monomers and fluoroaluminosilicate fillers (Francois et al., 2020). New classes of IRMs were also introduced through the integration of patented fillers such as Isofiller in alkasite (Ivoclar-Vivadent, AG, Schaan, Liechtenstein) (Alla et al., 2023) and surface pre-reacted glass ionomer (S-PRG) in giomer (Inside Dentistry, 2022). The handling properties of IRMs were also improved in high viscosity GIC and Activa Bioactive Restoratives (Pulpdent Corporation, Watertown, MA, USA) (SDI, 2023; Pulpdent, 2023).

As the awareness of dental aesthetics increases in the general population, the tooth bleaching market is rapidly progressing globally, with its market in the United States expected to reach over USD 2 billion in 2024 (Kwon et al., 2020). Vital or external tooth bleaching, is further classified into in-office, take-home and over-the-counter products (Alqahtani, 2014). These bleaching products comprise hydrogen peroxide or its precursor, carbamide peroxide. In-office bleaching agents contain the highest concentration of hydrogen peroxide at 25-40%, followed by take-home bleaching agents containing 3.5-6.5% (Alqahtani, 2014). The bleaching mechanism of hydrogen peroxide is its dissociation into free radicals that break the long-chained staining chromophore molecules in the tooth structure (Alqahtani, 2014). Complications may arise when oxidising agents affect the restorations of bleached teeth. The discolouration of restorative materials is often attributed to the oxidation of surface pigments and amine compounds (Alqahtani, 2014). The impact of these agents on the surface and optical properties of CR varies depending on material composition (Alqahtani, 2014; El-Murr et al., 2011; Gonulol et al., 2015). Moreover, the dissolution of functional filler particles in IRMs creates internal voids that enhance water sorption, thereby increasing their susceptibility to discolouration (Marovic et al., 2022).

The clinical significance of colour change is the poor colour match between the restoration and adjacent tooth structure (Lempel et al., 2017). Although IRMs share some similarities in their compositions, varying physical properties and clinical success have been reported (Francois et al., 2020; Gracia et al., 2021; Karakaş et al., 2021; Molina et al., 2019; Eissa et al., 2021; Inthihas et al., 2019). Literature comparing the effect of bleaching products on the colour stability of contemporary IRMs is limited.

1.2 Aim

To evaluate the effects of bleaching products on the colour stability of IRMs.

1.3 Objective

1. To evaluate the effect of in-office and take-home bleaching products on the colour stability of IRMs over time.

1.4 Null Hypotheses

1. In-office and take-home bleaching products do not affect the colour stability of IRMs.
2. The colour of IRMs remains stable over time following their exposure to bleaching products.

CHAPTER 2: LITERATURE REVIEW

2.1 Ion-releasing restorative material (IRM)

2.1.1 Background and historical perspectives

The potential of ion-releasing silicate cement in preventing secondary caries was explored in the 1940s. Fluoride-containing amalgam and luting cement became available commercially in the mid-1970s (Eichmiller & Marjenhoff, 1998). Forsten (1976) compared the quantity of fluoride release between fluoride-containing amalgam and two other fluoride-releasing luting cements over five weeks. The findings revealed that while the initial fluoride release from the amalgam was significant, its continuous release decreased after the first week. Conversely, silicophosphate and polycarboxylate cements exhibited a consistent and prolonged fluoride release which is diffusion-controlled, and dictated by the concentration gradient between the set matrix and surrounding environment (Forsten, 1976).

Norman et al. (1960) reported that increasing fluoride concentration in luting cements to reduce enamel solubility had limitations. This supported the notion that although the initial "burst" of high fluoride release (4.37 – 28.28 ppm) is desirable in dental materials, a constant release of relatively low concentration (0.45 – 2.34 ppm) has a higher efficiency in caries prevention (Surabhilakshan et al., 2021). Additionally, fluoride availability in luting cement was limited clinically due to the thin application of the cement layer (Forsten et al., 1976).

Hydrophilic materials, known for their ability to wet and react with hydroxyapatite and collagen, can form a durable bond to the tooth structure. This property was demonstrated by polyacrylic acid, which showed significant adhesive capability. This was attributed to its unique capacity for chelating calcium ions and forming hydrogen bonds with organic polymers that resemble collagen, a key component of tooth structure (Lohbauer, 2009).

In the early 1970s, the concept of merging the strength and fluoride-release properties of silicate cement with the biocompatibility and bonding quality of polyacrylic cement led to the development of GIC (Wilson & Kent, 1972; McLean & Wilson, 1976). GIC releases more fluoride than silicate cement (Forsten, 1977). Its versatility allows it to be used as a filling material, luting agent, and cavity liner. However, it has limited usage in aesthetic and stress-bearing areas of the dentition due to a lack of translucency and mechanical strength. These drawbacks drove the development of enhanced hybrid IRM aiming to overcome the drawbacks of GIC.

2.1.2 Development of hybrid IRM

2.1.2.1 Metal-reinforcement

One approach to reinforce GIC was through metal reinforcement using a silver-tin alloy, which provides superior mechanical strength in amalgam restoratives compared to other commercially available materials. Two methods of metal incorporation were described, i.e. (i) a silver alloy admixed GIC that combines one part of spherical silver alloy powder with eight parts of conventional GIC powder; (ii) a cermet mixture which involves pelletising silver alloy with glass particles at high temperature and pressure (Sikka and Brizuela, 2022). While cermets exhibited superior compressive strength and fatigue limit, their flexural strength and wear resistance were similar to those of conventional (GIC). Moreover, a metallic phase reduces fluoride release and results in a grey appearance (McCabe & Walls, 2013).

2.1.2.2 Fibre reinforcement

Fibre reinforcement of the conventional GIC (Chembond, Dentsply) was reported by Sced and Wilson (1980). The addition of carbon and alumina fibres of length >1000 µm and diameters ranging from 10 to 200 mm at 25 vol% significantly improved the flexural strength to 53 MPa and 44 MPa respectively, as compared to the 10 MPa from the control group. Glass fibres based on the SiO_2-Al_2O_3-CaO-P_2O_5 were also added as a strengthening material, resulting in increased diametrical tensile and flexural strengths (Kobayashi et al., 2000).

2.1.2.3 Resin-modified glass ionomer cement

The more popular strategy for improving GIC is resin incorporation. In the 1980s, resin-modified glass ionomer cement (RMGIC) was developed. It is composed of a fluoroaluminosilicate glass powder and liquid, which has resin monomers, a polyacid, a hydrophilic resin monomer, 2-hydroxyethylmethacrylate (HEMA), water and a photoinitiator. This formulation allows an acid-base reaction and photopolymerisation to co-occur during its setting, subsequently improving early strength and aesthetic appearance, and allowing longer working time than the conventional GIC (Lohbauer, 2009).

2.1.2.4 Polyacid-modified composite

Manufacturers have also developed other variants of IRM with a resin component. Polyacid-modified composite or compomer, for instance, has a resin matrix, ion-leachable aluminosilicate glass filler, and a small proportion of dehydrated functional monomers with carboxylic acid. The primary setting reaction occurs through the photopolymerisation of the methacrylate monomers. The acid-base reaction and subsequent fluoride release occur after its placement upon water uptake from the saliva (Francois et al., 2020).

2.1.2.5 Giomer

Giomer and compomer differ primarily in their aluminosilicate glass fillers, which are pre-activated with polyacid, resulting in pre-reacted glass ionomer (PRG). This pre-activation ensures that remineralising ions are readily available for release upon contact with saliva. The products can be categorised into two types based on the depth of chemical activation: one entails complete filler activation (F-PRG), while the other involves only the surface of the fillers (S-PRG). The latter type is utilised in giomer restoratives (Francois et al., 2020).

2.1.2.6 Bioactive composite

Activa Bioactive Restorative (Pulpdent, Watertown, MA, USA) has been labelled by its manufacturer as a bioactive composite, constituting a new class of IRM. However, some authors classify it under RMGIC. Its composition, akin to RMGIC, includes fluoroaluminosilicate glass and polyacid components. Its Embrace bioactive ionic dual-cure resin sets it apart from other IRMs, allowing chemical and light polymerisation upon activation (Garoushi et al., 2018). The manufacturer claims this technology reduces post-operative sensitivity by minimising exothermic heat and polymerisation stress compared to typical photopolymerised resin materials. With a 4mm depth of cure and a flowable form, it offers clinical ease of use and is less technique-sensitive as bigger increments and shorter time are needed to complete the restoration (Pulpdent, 2023; Lardani et al., 2022).

2.1.2.7 Alkasite

Alkasite restorative, marketed as Cention-N (Ivoclar-Vivadent, AG, Schaan, Liechtenstein), distinguishes itself through its utilisation of an alkaline filler, calcium fluorosilicate, which purportedly releases more remineralising ions in an acidic oral environment. Additionally, it is claimed to generate lower polymerisation stress due to its patented silane functionalised filler, Isofiller, acting as a shrinkage stress reliever (Alla et al., 2023). Compositionally, it resembles giomer, featuring reactive fillers that do not necessitate polyacid for an acid-base reaction during clinical application (Francois et al., 2020). The properties of Cention-N were reportedly comparable to conventional CR and better than GICs (Justen et al., 2024). Figure 2.1 shows Francois et al.'s (2020) proposal for categorising IRMs based on their setting reaction, bioactivity, and bulk-fill properties.

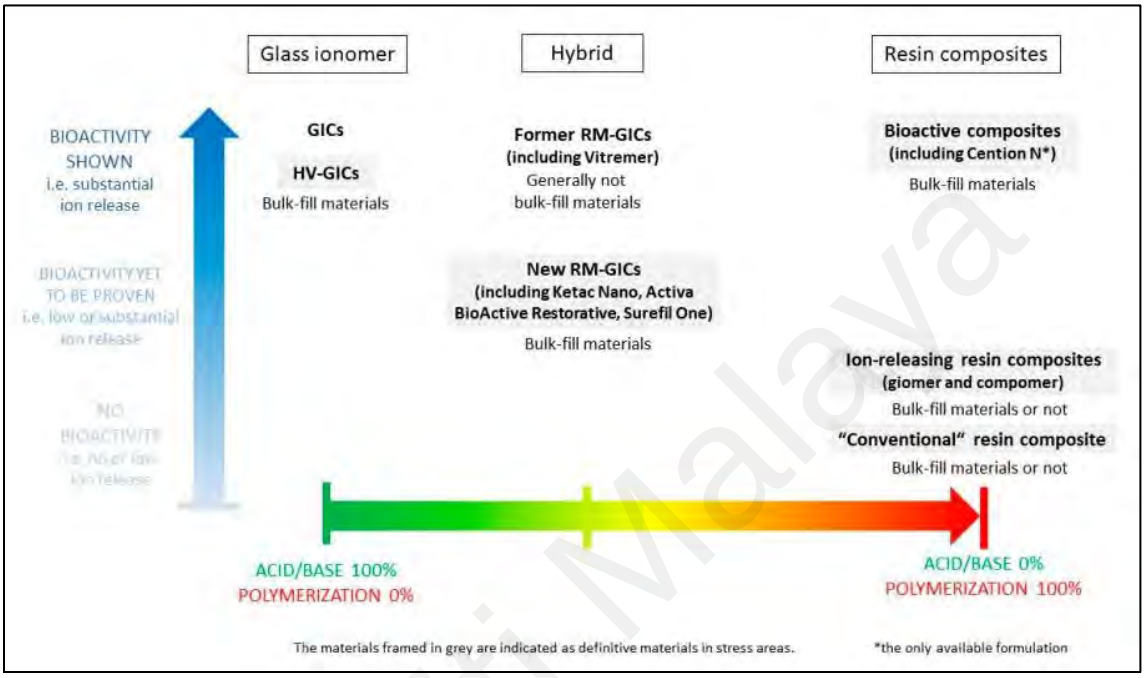


Figure 2.1: A classification of IRMs (Francois et al., 2020)

2.1.3 The properties of IRM

The oral environment presents many challenges to dental biomaterials, such as wear and fatigue from cyclical chewing forces, temperature fluctuations, biofilm that may lead to secondary caries, and long-term exposure to water and colour staining from foodstuffs. To date, no single material has the ideal properties that can ensure its longevity in function. Hence, the choice of the type of restorative materials depends on case selection. For instance, GICs are often used to restore non-carious cervical lesions due to their less sensitivity to moisture in cavities near the gingival margin. Conventional GIC is not indicated for posterior stress-bearing areas due to its weaker mechanical strength (McCabe, 2013). On the other hand, contemporary IRMs have a more comprehensive range of uses owing to the enhancements led by resin reinforcement. The type of monomer determines the differences in the physicochemical and mechanical properties of the formulation and its degree of conversion upon polymerisation (Alrahlah, 2018).

2.1.3.1 Remineralising properties

The significant advantage of IRM is its anti-cariogenic potential, which is achieved through the release of fluoride ions. The release of fluoride from IRM occurs in two phases. The initial burst phase releases a high amount of fluoride ions induced by the superficial rinsing effect, followed by a sustained release of lower concentrations over the subsequent days. The diffusion of water into the material triggers the release of fluoride from the fluoride-containing fillers, which subsequently moves into the saliva driven by a diffusion gradient (Harhash et al., 2017). The materials can also form a fluoride reservoir by recharging fluoride ions absorbed from the surrounding media, such as toothpaste and fluoride varnish applications (McCabe, 2013). The remineralising potential of GICs is also contributed by the release of calcium and phosphate ions which are key components of tooth hydroxyapatite (Tuguynov et al., 2024).

The amount of fluoride released depends on intrinsic factors such as the composition, solubility and porosity present in the material. In contrast to GICs, compomer, giomer, and Activa Bioactive showed no initial burst phase of fluoride release (McCabe, 2013; Wiegand et al., 2007; Garoushi et al., 2018). This is due to the absence of hydrophilic HEMA monomer in these products, which forms a polymeric hydrogel that facilitates water diffusion. Another reason is that the fluoride in giomer and compomer is bound within the filler particles, which are in turn encapsulated within the polymerised matrix (Wiegand et al., 2007). An acid-base reaction which occurs in GICs or RMGICs does not take place in compomer and giomer, and the release of fluoride is dependent on the water absorption of the material facilitating ion diffusion (McCabe, 2013).

Conventional and resin-modified GICs have been reported to form a better fluoride reservoir than resin-based materials owing to their higher permeability. The higher number of porosities in the polymerised GICs allows more water and solutes to be readily available for ion exchange and diffusion to the environment (Wiegand et al., 2007).

2.1.3.2 Water sorption and solubility

Prolonged exposure of restorations to water is inevitable, especially concerning IRMs, where water absorption plays a pivotal role in their acid-base setting reaction and ion-releasing capabilities. This process of water sorption, predominantly occurring within the resin matrix, operates under diffusion-controlled mechanisms. As water permeates the matrix, it induces plasticisation and causes debonding between the filler and matrix components (Toledano et al., 2003). Consequently, this phenomenon triggers the release of uncured monomers and filler ions, resulting in the material's weight loss, quantified as solubility (Gonulol et al., 2015). These interconnected processes of water sorption and solubility exert adverse effects on the restoration's strength, colour stability, and overall longevity (Gonulol et al., 2015). The ISO 4049 standardised the maximum acceptable solubility value for restorative materials is capped at 7.5 μg/mm^3, with the sorption value set at 40 μg/mm^3.(ISO, 2019)

The water sorption and solubility depend on the compositions of the material. The RMGIC appeared to have the highest water sorption and solubility compared to compomer and CR (Toledano et al., 2003), due to the presence of the hydrophilic 2-hydroxyethylmethacrylate monomer. Alkasite Cention-N has lower sorption and solubility than a conventional GIC, Fuji IX due to the presence of a rigid network formed by urethane dimethacrylate (UDMA) (Nayak & Shenoy, 2015).

2.1.3.3 Flexural strength

The flexural strength of restorative materials is widely tested and a fundamental mechanical property of the International Organization for Standardization (ISO) (Alrahlah, 2018). The minimum required flexural strength for restorative materials on occlusal surfaces is 80 MPa (ISO, 2019). Kasraei et al. (2022) compared the flexural strengths between three resin-based IRMs and a conventional CR at 24 hours and six months post-polymerisation, and reported that Cention-N had the highest flexural strength at both time points, followed by Activa Bioactive, Filtek Z350 and Fuji II LC. However, there was a reduction in the flexural strength of all materials except Fuji II LC after six months of storage in artificial saliva. The authors attributed these findings to the UDMA monomer content in the materials, and a higher resin matrix composition led to increased water sorption and degradation over time (Kasraei et al., 2022). Similar results have been reported by Alrahlah (2018), who reported a reduction in the flexural and diametrical tensile strengths after thermocycling. Ong et al. (2023) compared the flexural strength and modulus between IRMs when subjected to different environmental pH. Activa Bioactive and RMGIC exhibited lower values in both parameters due to their low filler weight, water absorption characteristics, and matrix dissolution tendencies.

2.1.3.4 Fracture toughness

The fracture toughness of restorative materials is also crucial in determining their survival. Restoration failure had been largely attributed to its fracture (Demarco et al., 2015; Opdam et al., 2014). Activa Bioactive's fracture toughness was reportedly the highest compared to Fuji II LC, Cention-N and Filtek Z350 CR. This can be due to its rubberised resin matrix acting as a shock absorber (Daabash et al., 2022). After 30 days of water storage, fracture toughness values decreased significantly for all materials except Fuji II LC, with comparable values exhibited by Activa Bioactive, Cention-N and Filtek

Z350 (Daabash et al., 2022). Despite having the lowest flexural strength (Kasraei et al., 2022) and fracture toughness (Daabash et al., 2022) compared to other materials, RMGIC demonstrated the best stability upon water ageing, potentially attributable to its capacity for forming a more resilient polysalt matrix in aqueous environments.

2.1.3.5 Surface roughness

The surface roughness of restorations affects its affinity to biofilm accumulation (Bollen et al., 1997) and staining (Alqahtani, 2014). The critical surface roughness value for bacterial colonisation was reportedly 0.2 μm below which no further reduction in bacterial accumulation could be expected (Bollen et al., 1997). The surface roughness of IRMs can be attributed to the incorporation of air bubbles during hand mixing and mechanical vibrations in the amalgamator (Pacifici et al., 2013). Larger particle sizes also led to a rougher restoration surface (Pacifici et al., 2013). The change in the surface properties of restorations is inevitable in clinical situations, as they are subjected to cyclic wear during mastication, gradually removing material from the surface upon contact with the opposing surface (Garcia et al., 2021).

When comparing the surface roughness of IRMs after being subjected to chewing simulations, Garcia et al. (2021) reported that giomer was superior to Activa possibly due to its better filler distribution and smaller filler size. For similar reasons, RMGIC and Activa were found to have a smoother surface than conventional GIC (Ismail et al., 2020). When exposed to cariogenic environments, Activa was also found to have the highest surface roughness among all other materials (Ibrahim et al., 2024). Self-cured IRMs were found to have a smoother surface than their light-cured counterparts, due to the incomplete light polymerisation that affects the surface finish (Maktabi et al., 2019; Kaptan et al., 2023).

2.1.3.6 Colour stability

Lempel et al. (2017) reported that colour mismatch can cause failures of restorations. This highlights the importance of restorative materials having an acceptable aesthetic appearance and colour stability over an extended duration. Restorations are prone to discolour through the absorption of fluids with various pigments in the oral environment, with frequent exposure to staining agents from the diet. It is also influenced by intrinsic factors such as the matrix-filler content. The higher the material's water sorption and hydrophilicity, the greater its susceptibility to staining (Gonulol et al., 2015).

Gonulol et al. (2015) reported that giomer exhibited the highest water sorption and discolouration compared to CR. This difference is caused by the presence of Bis-GMA and TEGDMA in giomer's resin matrix, while the other materials utilised UDMA monomer, which has less water sorption and is more stain resistant. Similarly, Iazzetti et al. (2000) reported that CR, which is more hydrophobic, has excellent stain resistance and colour stability compared to conventional and resin-modified GICs. Another study comparing the colour stability of giomer and RMGIC exposed to orange juice, milk, and coke found giomer to be more stain-resistant than RMGIC. The possible explanation for this finding is that the smaller filler size in giomer gave rise to a smoother surface finish that is more stain-resistant (Hotwani et al., 2014).

Santos et al. (2023) reported a significantly better clinical colour match for class V cavities restored with RMGICs compared to conventional GIC after 2.5-3.5 years. The findings are attributed to GIC having a longer acid-base polymerisation time that is more sensitive to water sorption and desiccation, while RMGIC has a rapid photopolymerisation mechanism. Other clinical studies reported comparable colour stability for Cention-N and CR (Oz et al., 2023), while compomer was found to have a higher prevalence of discolouration than RMGIC and flowable CR (Hussainy et al., 2018).

2.2 Tooth and restoration discolouration

Aesthetic concern is one of the main reasons patients seek dental treatment today (Kwon et al., 2020). Patients may request enhancement of their dental aesthetics caused by the discolouration of teeth and fillings, malalignment of the dentition and loss of tooth structure secondary to caries or tooth wear. Maintaining a viable dentition is critical to maintaining an individual's physical and psychosocial well-being in this ageing population. Discolouration of the teeth can arise due to extrinsic and intrinsic factors. Depending on the aetiology, its clinical presentation, severity, and adherence to tooth structure vary (Dahl & Pallesen, 2003).

2.2.1 Extrinsic tooth discolouration

Extrinsic factors are further categorised into non-metallic and metallic stains. Non-metallic stains are present in the diet and tobacco. In smokers, it often presents as a yellowish-brown to black discolouration on the palatal or lingual surfaces. Coffee, tea and red wine contain tannin, a dark pigmentation that can enter the porosities of the enamel. The beverage's acidity can also cause erosion on the tooth surface, leaving it more susceptible to staining (Watts & Addy, 2001; Sarembe et al., 2022). Metallic stains can occur through occupational and medicinal exposure to metallic salts. For instance, teeth can be stained black when taking iron supplements or working in an iron foundry (Watts & Addy, 2001).

2.2.2 Intrinsic tooth discolouration

Intrinsic discolouration is caused by the incorporation of chromogenic material into the tooth structure during odontogenesis and after eruption (Dahl & Pallesen, 2003). Pre-eruptive discolourations are attributed to the ingestion of high levels of fluoride which results in fluorosis, tetracycline stain, and inherited dental development disorders such as amelogenesis and dentinogenesis imperfecta. Dental trauma and pulpal necrosis can lead to grey discolouration due to the deposition of haemoglobin breakdown byproducts. The deposition of bilirubin in jaundice patients leads to a green discolouration (Watts & Addy, 2001).

2.2.3 Restoration discolouration

There are three types of restoration discolouration: (1) external discolouration caused by plaque and surface stain accumulation, (2) changes in subsurface colour consisting of degradation and the reaction of colourants within the superficial layer of resins, and (3) body or intrinsic discolouration due to physicochemical reactions in the deepest layer of the material. Similar to teeth discolouration, restorative materials are affected by the colourants and acidity of the diet. Ceci et al. (2017) reported that coffee caused higher colour changes than red wine. Immersion in acidic carbonated drinks further increased the staining susceptibility of all materials by softening the surface of CR, resulting in chemical wear. As mentioned before, the colour stability of restorations is material dependent and is associated with the water sorption rate, the type and amount of resin monomer, filler load and size. A low filler load with a large resin volume and the presence of hydrophilic monomers can increase the material’s water sorption, resulting in a higher hydrolysis rate and permeability to pigments (Ceci et al., 2017).

2.3 Bleaching technique in dentistry

The tooth bleaching procedures are indicated for persistent extrinsic and intrinsic stains that cannot be removed by scaling and polishing. It can be categorised into vital and non-vital tooth bleaching. It involves the application of 15-40% hydrogen peroxide either directly onto the tooth structure or indirectly through a chemical reaction of sodium perborate or carbamide peroxide (Alqahtani, 2014).

2.3.1 Historical background

Truman (1864) first described the bleaching of non-vital teeth using chlorine from a solution of calcium hydrochlorite and acetic acid. Various non-vital bleaching agents, such as potassium cyanide, oxalic acid, sulfurous acid, hydrogen dioxide and sodium peroxide, were introduced in the late nineteenth century. In 1961, Spasser developed the "walking bleach" technique, which remains popular today. It involves the insertion of a sodium perborate and water paste mixture in the empty pulp chamber and sealing it with a temporary restoration. The technique is further modified by using 30-35% hydrogen peroxide as the bleaching paste and providing heat to increase the effectiveness and accelerate the bleaching process. The bleaching paste is then renewed weekly until the tooth colour is satisfactory (Coelho et al., 2020).

The vital bleaching technique using oxalic acid and pyrozone was done in the mid-late 1800s (Alqahtani, 2014). Fischer (1911) described using a combination of hydrogen peroxide and a heating instrument or light source, similar to the in-office bleaching used today (Alqahtani, 2014). Later, Dr Klusmier in the 1960s incidentally discovered the lightening effect of 10% carbamide peroxide used in a custom-fitting tray when prescribing it for the treatment of gingivitis. This formed the basis of the modern nightguard vital bleaching technique introduced in 1989 (Haywood & Heymann, 1991). Moreover, over-the-counter bleaching products containing low hydrogen peroxide or carbamide peroxide became commercially available for home use in the 1990s.

2.3.2 The chemistry and mechanism

Bleaching products contain active and inactive ingredients. The common inactive ingredients are carbapol, propylene glycol, surfactants, sodium benzoate and flavourings, which control bleaching products' consistency, moisture, shelf-life, and surface-wetting properties (Alqahtani, 2014).

The bleaching chemical process is mediated by its active ingredients, hydrogen peroxide and its precursor, carbamide peroxide. Carbamide peroxide, or urea peroxide, reacts with water to form hydrogen peroxide and urea. A 10% carbamide peroxide degrades into approximately 3% hydrogen peroxide and 7% urea (Haywood, 2000). The hydrogen peroxide then undergoes further breakdown to release reactive oxygen species, or perhydroxyl ions under higher pH levels >7, of which the latter has better whitening capacity. Both radical oxygen species and perhydroxyl ions attack the double-bonded organic chromogens between the inorganic tooth structure, destabilising and converting them to smaller and lighter-pigmented molecules (Alkahtani, 2020).

2.3.3 The types of vital bleaching techniques and products

In-office or chairside tooth bleaching can only be performed by dental professionals. It contains a higher concentration of hydrogen peroxide (30-38%) and is typically applied for 45 minutes to 1 hour. Pola Rapid (SDI, Bayswater, Australia) contains 38% hydrogen peroxide, and the manufacturer advised three cycles of 8-minute-application (SDI, 2024). It releases a high level of reactive oxygen species upon activation resulting in immediate post-bleaching shade improvement (Alkahtani, 2020). Some products may require chemical activation with manganese gluconate or blue light exposure with a 480 and 520 nm wavelength to accelerate the reaction rate. The efficacy of these activation steps remains controversial (Carey et al., 2014; Epple et al., 2019).

Nightguard or take-home bleaching is considered the gold standard technique due to its fewer side effects and cost-effectiveness. The procedure involves the application of bleaching products containing 10-20% carbamide peroxide onto a custom-fabricated nightguard and wearing it for 8 hours nightly over one or two weeks. The outcome of this treatment relies on the patient’s compliance (Alkahtani et al., 2020)

Over-the-counter bleaching products are available in many delivery systems, such as gels, toothpastes, whitening strips, and mouth rinses. The concentration of hydrogen peroxide is low, generally at 3-6%. Strips and gels are applied over the buccal surfaces of teeth over two weeks and can usually improve the colour by one or two shades (Carey et al., 2014). Apart from the bleaching action from peroxides, whitening toothpaste utilises a coarser and higher quantity of abrasives in its formulation compared to conventional toothpaste. This provides a higher abrasivity in removing extrinsic stains present in the superficial layer of the tooth structure (Epple et al., 2019).

2.3.4 The adverse effects of bleaching procedures on the teeth

Due to its oxidising capability, hydrogen peroxide is widely applied as a bleaching or deodorising agent for the textile industry, and as a disinfectant for water and sewage treatment. The radical oxygen species liberated during the decomposition of hydrogen peroxide can potentially lead to cellular damage through double-strand DNA breaks. This effect is counteracted by antioxidants in our body, which provide electrons that reduce these radicals to water. During bleaching procedures, adverse effects on the oral tissues may occur from the high level of hydrogen peroxide, which overwhelms our protective mechanisms (Tredwin et al., 2006). To prevent caustic burns to the soft tissue around the teeth, such as the gingiva and buccal mucosa, a barrier is employed by placing a rubber dam or close-fitting tray that exclusively contacts the teeth.

The incidence of cervical root resorption has been reported in non-vital bleaching procedures. The prolonged placement of hydrogen peroxide in the pulp chamber may reach the periodontal tissue through the dentinal tubules to initiate inflammation. AlOtaibi (2019) reported that this adverse effect did not occur when sodium perborate was used, while a high concentration of hydrogen peroxide in the presence of heat increased the risk for cervical root resorption. Rotstein et al. (1991) showed that elevating the temperature and duration of bleaching resulted in increased radicular infiltration of hydrogen peroxide. Crown fractures have also been associated with bleaching procedures. The high hydrogen peroxide concentration decreases the dentine’s microhardness and weakens it mechanically (Chng et al., 2002). This weakening effect depends on the bleaching agent’s pH, dentine's buffering capacity and dentinal tubules' properties around the bleaching agent placement (Chng et al., 2002).

Tooth hypersensitivity is commonly reported in external tooth bleaching procedures. The occurrence rate ranged from 15% to 65% for individuals subjected to 10% carbamide peroxide, and 67% to 78% for in-office bleaching combining hydrogen peroxide and heat activation (Dahl & Pallesen, 2003). It has been demonstrated that the peroxide molecules can penetrate the enamel and dentine, causing transient reversible pulpitis (Epple et al., 2019). It typically lasts four days, though its persistence for up to 39 days has been observed (Dahl & Pallesen, 2003).

Hydrogen peroxide affects the composition of tooth structure by causing the dissolution of inorganic components, protein oxidation of the organic matrix and reducing the calcium-phosphorus ratio (Chng et al., 2022). It can also lead to the loss of interprismatic substances, including sodium and magnesium ions. These chemical and morphological structure alterations increases the surface roughness and decreases the microhardness (Goyal et al., 2021). These, in turn, increase the staining susceptibility of the tooth. However, negligible changes to the bleached tooth structure have been reported in other literature (Farawati et al., 2019; Cobankara et al., 2004). Vilhena et al. (2019) compared morphological changes in the bovine dental enamel exposed to 10% carbamide peroxide for different periods. The surface roughness was not afflicted by bleaching, while the severity of ultrastructural changes and microhardness reduction increased with prolonged bleaching.

2.3.5 The adverse effects of bleaching procedures on restorations

The damaging effects of bleaching agents on dental restorations are widely investigated. This occurs due to the unspecific action of the reactive oxygen species that may be exerted within the tooth structure and in the resin-filler interphase of restorations (Attin et al., 2004). A systematic review by Attin et al. (2004) reported that the quantity of porosities and surface roughness of CRs increased after exposure to 10-16% carbamide peroxide. Popescu et al. (2023) observed an increase in the surface roughness of bleached microhybrid and nanohybrid CRs, and the effect was more prominent in the specimens bleached with 40% hydrogen peroxide. This was attributed to the absorption of water and the attack by the free radicals, which resulted in the surface degradation of CR materials. In contrast, Dogan et al. (2008) reported that the bleaching agents reduced the surface roughness of polished CRs. The author explained that this observation might be due to the extension of fillers onto the restoration surface after polishing. The application of hydrogen peroxide dissolves the filler extensions, resulting in a smoother surface.

The surface microhardness of restorations can be affected by bleaching. Turker & Biskin (2002) reported that the effect of 10-16% carbamide peroxide on the microhardness of restorations is material dependent. While feldspathic porcelain showed a reduction in microhardness after bleaching, the opposite was observed on RMGIC. Carbamide peroxide was found to decrease the silicon dioxide content of porcelain and thus affect the microhardness. On the other hand, the increase in the microhardness of RMGIC was attributed to the silica core exposed at the superficial layer after the erosion of the GIC. Karatas et al. (2021) reported a reduction in the microhardness of microhybrid CR after bleaching. This observation was associated with the deterioration of the resin matrix of CR.

A decrease in the bond strength between CR restorations and the tooth structure was related to the bleaching agent's interference with resin bonding, polymerisation and its alteration of protein and mineral content of the bonding tooth surface (Alqahtani, 2014). The resin tags formed in bleached bovine enamel were poorly defined and demonstrated less penetrating depth than the negative control group (Titley et al., 1991). Halabi et al. (2019) corroborated that in-office bleaching decreased the immediate bond strength between the enamel and a self-etch adhesive. The residual oxygen radicals remaining in the tooth structure negatively affect the polymerisation of the bonding resin.

2.3.6 The effect of bleaching on the colour stability of restorations

Restorations' discolouration after placement negatively affects the colour match with the surrounding tooth structure and its aesthetic appearance. The affinity of dental restorations to extrinsic stains is affected by its surface roughness, the susceptibility of its intrinsic pigments and amides to discolouration, the type and amount of resin and the material's degree of conversion (Alqahtani, 2014).

The effect of bleaching on the colour of restorations is material dependent (Inokoshi et al., 1996; Yu et al., 2009; Li et al., 2009; Kara et al., 2013; Hubbezoglu et al., 2008). Inokoshi et al. (1996) reported that the colour and opacity of light-cured CR were more stable than chemically cured CR, owing to the materials' different composition and activator system. The same study also demonstrated macrofilled CR to exhibit less discolouration than microfilled CR due to the lower resin component as the source of discolouration. Yu et al. (2009) compared the colour change of compomer, conventional GIC, and two light-cured CRs after bleaching using 15% carbamide peroxide for two weeks. The colour change, ΔE* of all except one CR exceeded the acceptability threshold. Compomer and conventional GIC exhibited higher ΔE* than the CRs, owing to their hydrophilic nature. Using the same materials, Li et al. (2009) measured their ΔE* over five consecutive weeks following the end of bleaching. They observed that the initial significant discolouration reduced over time for all materials except the compomer.

Interestingly, GIC exhibited the least colour change after five weeks, which can be attributed to the ionic exchange between saliva and the cement, resulting in bleaching resistance. Another study found an insignificant difference in ΔE* between a microhybrid (1.94 ± 0.67) and a nanohybrid (2.34 ± 0.65) CR bleached with 16% carbamide peroxide (Korac et al., 2021). The change in the lightness of materials contributed the most to the colour change of all materials (Yu et al., 2009; Li et al., 2009; Korac et al., 2021).

Torres et al. (2012) compared the effect of different concentrations of hydrogen peroxide on the colour of CRs. 35% hydrogen peroxide was found to cause more colour change in CRs than in the 20% group. This is due to the greater content of active oxidising agents in bleaching products with a higher hydrogen peroxide concentration. Kara et al. (2013) reported similar results where 40% hydrogen peroxide exposure led to a ΔE* of 5.08, compared to 2.37 from the 10% hydrogen peroxide group. Nevertheless, the study revealed that varying concentrations of hydrogen peroxide had a negligible impact on feldspathic porcelain and leucite-based core, as ceramics are more chemically stable than CRs (Hubbezoglu et al., 2008).

2.4 Colour change

2.4.1 Colour science relevant to dentistry

Colour perception is dependent on the combination of a light source, an object and an observer. The light source illuminates the object, the object changes how light is absorbed, reflected and transmitted through it, and the light which enters the eyes of different observers may be interpreted varyingly (Burkinshaw, 2004).

Colour is often described based on the Munsell colour space, which specifies colour into three parameters, hue, chroma and value. Hue is the property that differentiates one family of colour from another (i.e. red, blue, yellow). Chroma denotes the saturation or intensity of the colour. Value indicates the lightness from white to black

(Sikri, 2010). Other properties also influence the visual appearance of restorative materials. Translucency can be defined as the slope between transparent and opaque (Vadher et al., 2014). Fluorescence involves the absorption of light by a substance followed by the spontaneous emission of light at a longer wavelength. Opalescence is the phenomenon where the colour of the same object appears different between light passing through it and light reflecting from it (Sikri, 2010). Metamerism means two differently coloured objects may appear the same under one lighting condition, which the objects are known as metamers (Burkinshaw, 2004).

Colour science is relevant to dentistry due to the interest in dental aesthetics, as well as the nature of human dentition and dental restorations which are translucent and composed of layers with different organic and inorganic compositions. Shade guides have been developed according to the Munsell Colour System. Clinicians are advised to determine the shade of restorations in the order of value, chroma, and hue (Sikri, 2010). The procedure of shade matching should also be conducted under multiple lighting conditions, including natural daylight, incandescent and fluorescent lighting (Sikri, 2010).

2.4.2 The measurement of colour and the calculation of colour change, ΔE*

Colour is often measured using the International Commission on Illumination (CIE-L*a*b*) colour model developed in 1976. The system is a three-dimensional colour space, where the L*, a* and b* values of the specimen are coordinates that represent the lightness from black to white, red to green and yellow to blue dimensions, respectively, as shown in Figure 2.2. Subsequently, the colour change, ΔE*, is calculated using the following formula: $\Delta E *= \sqrt{(\Delta L *)^2 + (\Delta a *)^2 + (\Delta b *)^2}$.

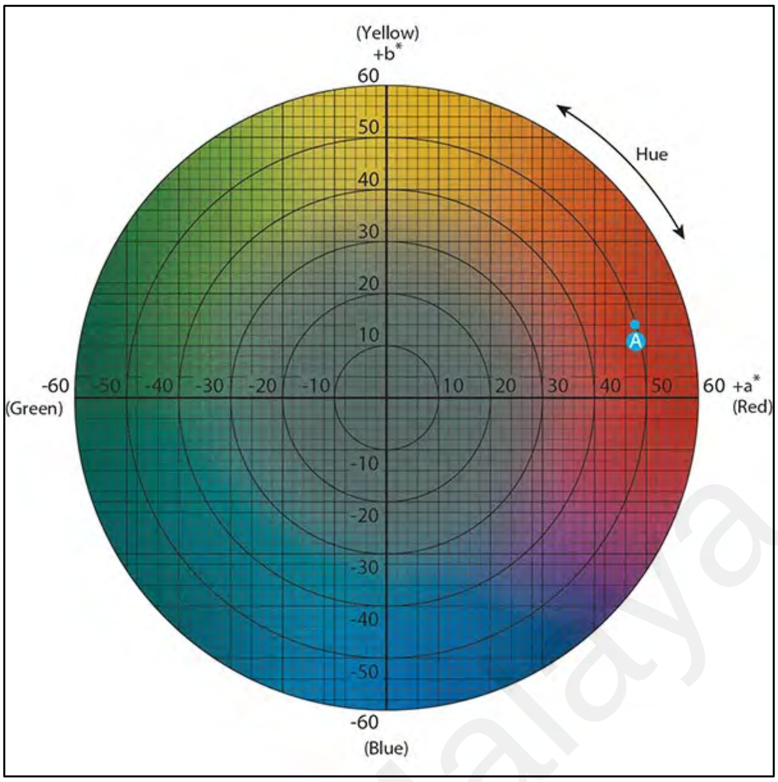


Figure 2.2: The CIE 1976 L*a*b* Colour Space

2.4.3 The thresholds of colour change in dentistry

The ΔE* value is clinically significant if it exceeds specific values: the perceptibility and acceptability threshold. Various threshold values have been proposed in the literature, with the 50:50% perceptibility and acceptability thresholds in dentistry ranging from 0.4 to 2.6 and 1.7 to 5.5, respectively (Paravina et al., 2019). The 50:50% perceptibility threshold defines a clinical scenario where half of the observers noticed a difference in colour while the remaining half noticed no difference. Similarly, a 50:50% acceptability threshold describes a colour variation acceptable by 50% of the observers. According to a review by Khasyahar et al. (2014) regarding colour-related studies, half of the studies adopted a perceptibility threshold of ΔE* = 1 (Kuehni & Marcus, 1979), while one-third of the studies referred to ΔE* = 3.7 (Johnston & Kao, 1989) as the acceptability threshold. A more recent study by Paravina et al. (2015) investigated the threshold values using monochromatic ceramic specimens judged by 175 observers comprising dentists, dental students, dental auxiliaries, dental technicians and lay persons.

They reported 1.2 and 2.7 as the perceptibility and acceptability thresholds, respectively.

2.5 Summary

Novel IRMs have been enhanced in many aspects compared to their traditional formulation (Francois et al., 2020). The flexural strength of RMGIC (65-80 MPa) and giomer (110 MPa) is significantly higher than that of conventional GIC (15-20 MPa) (McCabe and Walls, 2013). The wide range of shades available for these modern materials also broadens their clinical applications and caters to the variation in the aesthetic requirements of patients.

Notwithstanding, an ideal restorative material that can withstand the array of degradations imparted by the oral environment is not yet available. Although modern IRMs can be used in aesthetic and stress-bearing restorations, their fracture rate and longevity were reported to be inferior to CR (Van Dijken and Pallesen, 2010). This implies that CR remains the best material for restoring dentition where high wear and incisal stress are anticipated. Moreover, incorporating a resin component can subject modern materials to hydrolytic degradation, leading to filler dissolution, wear, surface roughening and discolouration of restorations (Ferracane, 2006). This effect is exacerbated in the porous IRMs that rely on water diffusion to release and recharge fluoride ions.

With the increasing demand for bleaching procedures, concerns regarding its detrimental effect on restorations exist. The non-selective oxidation reaction between hydrogen peroxide and the pigmented molecules within the tooth structure may also affect the filler-matrix interphase of restorations (Attin et al., 2004). Consequently, bleaching gels have been found to increase surface roughness and decrease microhardness and bond strength, as well as colour alterations in restorations (Popescu et al., 2023; Turker and Biskin, 2002; Alqahtani, 2014; Kara et al., 2013). A review by El-Murr et al. (2011)

suggested the replacement of GIC, RMGIC, and compomer exposed to bleaching gel, but it did not include details regarding giomer and alkasite restoratives.

The colour of restorations is critical for their success, and their discolouration can lead to an aesthetic failure that ultimately necessitates a replacement (Lempel et al., 2017). This underscores the importance of understanding the interactions between modern restorative materials and bleaching products.

CHAPTER 3: MATERIALS AND METHOD

3.1 Evaluated materials

The technical profiles of restorative materials under evaluation are presented in Figures 3.1-3.5 and Table 3.1. These included a conventional CR SDI Luna (LN) as the control, and four ion-releasing restorative materials (IRMs) [bioactive composite Activa (AB), giomer Beautifil II LS (BI), bulk-fill alkasite Cention-N (CN) and encapsulated Riva light-cured resin-reinforced high viscosity glass ionomer cement (RV)]. The shade A2 was selected for all materials. The in-office and take-home bleaching products, SDI Pola Rapid (PR) and SDI Pola Night (PN), respectively, were used in this study, as depicted in Figures 3.6-3.7 and Table 3.2.

Figure 3.1: Light-cured conventional CR, SDI Luna (LN) [as control]

Figure 3.2: Bioactive composite, Activa Bioactive Restorative (AB)

Figure 3.3: Giomer, Beautifil II LS (BI)

Figure 3.4: Bulk-fill alkasite, Cention-N (CN)

Figure 3.5: Encapsulated Riva light-cured resin-reinforced high viscosity glass ionomer cement (RV)

Figure 3.6: In-office bleaching product, SDI Pola Rapid (PR)

Figure 3.7: Take-home bleaching product, SDI Pola Night (PN)

Table 3.1: Technical profiles and manufacturers of materials to be evaluated.

Material (Abbreviation)	Manufacturer	Type and Curing Method	Resin	Filler	Filler Weight, %
Luna (LN) [As Control]	SDI Limited, Bayswater, Victoria, Australia	Conventional composite (Light-cured)	UDMA, Bis-GMA, TEGDMA	Strontium aluminosilicate, Amorphous silica	76
Activa Bioactive Restorative (AB)	Pulpdent, Watertown, MA, USA	Bioactive composite (Dual-cured/ Light-cured)	A blend of UDMA and other methacrylates with modified polyacrylic acid	Amorphous silica, Sodium fluoride	55.4
Beautifil II LS (BI)	Shofu, Kyoto, Japan	Giomer (Light-cured)	Low-shrinkage urethane diacrylate, Bis-MPEPP, Bis-GMA, TEGDMA	Multi-functional glass and S-PRG filler based on fluoroboroalumino silicate glass, pre-polymerized filler, nanofiller	83.0
Cention-N bulk fill (CN)	Ivoclar-Vivadent, AG, Schaan, Liechtenstein	Alkasite (Self-curing powder/liquid with optional additional light-curing)	UDMA, DCP, Aromatic aliphatic-UDMA, PEG-400 DMA	Br-Al-Si glass filler, Ytterbium trifluoride, Isofiller (copolymer), Calcium barium aluminium fluorosilicate glass, Calcium fluorosilicate glass	78.4
Riva light-cure HVGIC (RV)	SDI Limited, Bayswater, Victoria, Australia	Encapsulated resin-reinforced high viscosity glass ionomer cement (Light-cured)	Compartment 1: Polyacrylic acid, Tartaric acid, HEMA, Dimethacrylate-cross-linker, Acid monomer	Compartment 2: Fluoro-alumino-silicate glass	95.0

Bis-GMA = Bisphenol-A glycidyl methacrylate
Bis-MPEPP = 2,2-Bis (4-methacryloxypolyethoxyphenyl) propane
DCP = Tricyclodecan-dimethanol dimethacrylate
UDMA = Urethane dimethacrylate
TEGDMA = Triethylene glycol dimethacrylate
HEMA = 2-hydroxyethyl methacrylate
HVGIC = high viscosity glass ionomer cement
S-PRG = Surface pre-reacted glass ionomer
Aromatic aliphatic-UDMA = Tetramethyl-xylylendiurethane dimethacrylate
PEG-400 DMA= Polyethylene glycol 400 dimethacrylate

Table 3.2: Bleaching products to be evaluated.

Material (Abbreviation)	Manufacturer	Content
Pola Rapid (PR)	SDI Limited, Bayswater, Victoria, Australia	Liquid: 38% Hydrogen peroxide, 62% Water Powder: 73.26% Thickeners, 26.2% Catalysts, 0.04% Dye, 0.5% Desensitizing agents
Pola Night (PN)		22 wt% Carbamide peroxide (6.6% hydrogen peroxide), < 40 wt% Additives, 30 wt% Glycerol, 20 wt% Water, 0.1 wt% Flavour

3.2 Sample size calculation

Based on an analysis of variance (ANOVA) model with an effect size of 0.63, alpha error of 0.05, and power at 95% for 15 groups, a minimum total sample size of 90 (i.e. n=6) was determined using the G*Power Software version 3.1.9.4 (Yu et al., 2009; Faul et al., 2007).

3.3 Specimen Preparation

Figure 3.10 summarises the flow of the methodology. 45 disc-shaped specimens were prepared for each material according to manufacturers' instructions using a customised cylindrical acrylic mould measuring 8 x 2 mm. A cellulose acetate matrix strip and a glass slide were positioned on the mould's top and bottom surfaces to eliminate excess materials and achieve a smooth surface finish. Subsequently, the specimens were light-cured through the glass slide for 20 seconds using a calibrated LED light curing unit (Radii Cal LED Curing Light, SDI, Bayswater, Victoria, Australia) with an output irradiance of 1200mW/cm^2 and wavelength ranging between 468-470 nm. The light-curing unit was charged and calibrated every 15 specimens to ensure a consistent output.

The specimens were then stored in artificial saliva (SAGF) and placed in an incubator (Memmert Incubator IN750, Schwabach, Germany) for 24 hours at 100% relative humidity and a temperature of 37°C to allow post-irradiation polymerisation. Table 3.3 shows the composition of artificial saliva (SAGF) (Gal et al., 2001). Following this, the specimens were finished and polished using medium, fine and superfine Sof-Lex discs (3M ESPE, St Paul, Minnesota, USA), with 10 strokes made in one direction for each disc. The dimensions of all specimens were confirmed using a digital caliper (Mitutoyo Corporation, Kawasaki, Japan). Finally, all polished specimens were stored in 37°C artificial saliva and placed in the incubator for two weeks before commencing the bleaching procedure. The artificial saliva was renewed weekly to prevent the buildup of deposits.

Table 3.3: Composition of artificial saliva (SAGF)

Components	**Concentration (mg L−1)**
NaCl	125.6
KCl	963.9
KSCN	189.2
KH_2PO_4	654.5
Urea	200.0
$NaSO_4 \cdot 10H_2O$	763.2
NH_4Cl	178.0
$CaCl_2 \cdot 2H_2O$	227.8
$NaHCO_3$	630.8

3.4 Bleaching procedure

As illustrated in Figure 3.8, the specimens of each material were randomly distributed into three groups (n=15) as follows:

(Group A) Control group: Immersion in artificial saliva for seven days.

(Group B) Take-home bleaching group: Exposed to PN for 45 minutes daily for seven days.

(Group C) In-office bleaching group: Exposed to PR for three cycles of 8 minutes on day seven.

For both bleaching groups, the specimens were rinsed with distilled water and dried before each application. The specimens from Groups B and C were stored in artificial saliva at 37°C and placed in the incubator when they were not subjected to any bleaching procedure.

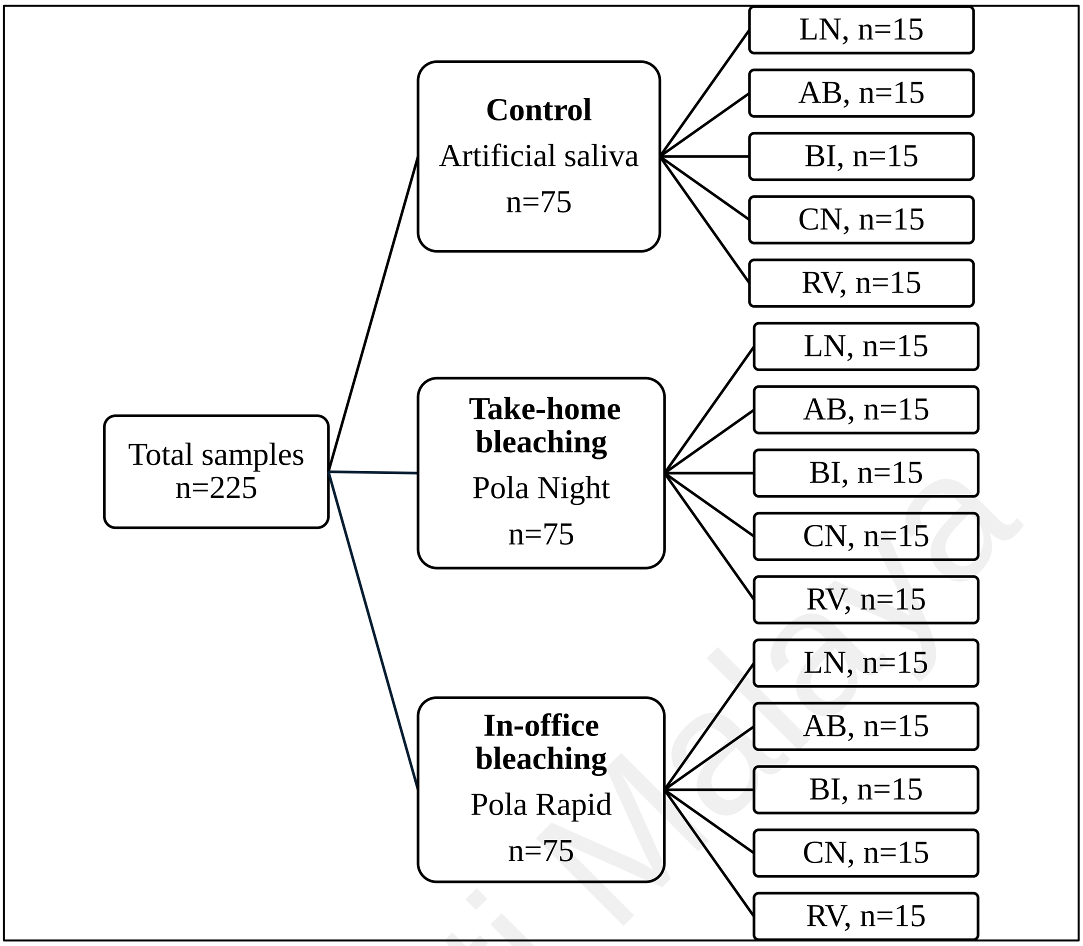


Figure 3.8: Grouping of specimens according to bleaching product for each material.

3.5 Colour measurement

The colour of every specimen was measured using the Commission Internationale del’Eclairege L*a*b* (CIE-L*a*b*) colour space with a spectrophotometer (Konica Minolta Spectrophotometer, CM-5, Tokyo, Japan), as shown in Figure 3.9. The average of three readings per specimen was obtained before bleaching at baseline (T0), 24 hours (T1), two weeks (T2) and one month (T3) after bleaching. Each specimen was placed on the illumination area (a circle of 3 mm diameter) with a standardised black background, and the reflectance values were measured after 1 second of being illuminated by a pulse xenon lamp.

The colour change, ΔE*, was calculated using the following formula:

$$\Delta E *= \sqrt{(L_i - L_0)^2 + (a_i - a_0)^2+(b_i - b_0)^2}$$

where

ΔE* is the colour change;

L is the lightness from black to white

a is the colour along the red and green dimension

b is the colour along the yellow and blue dimension

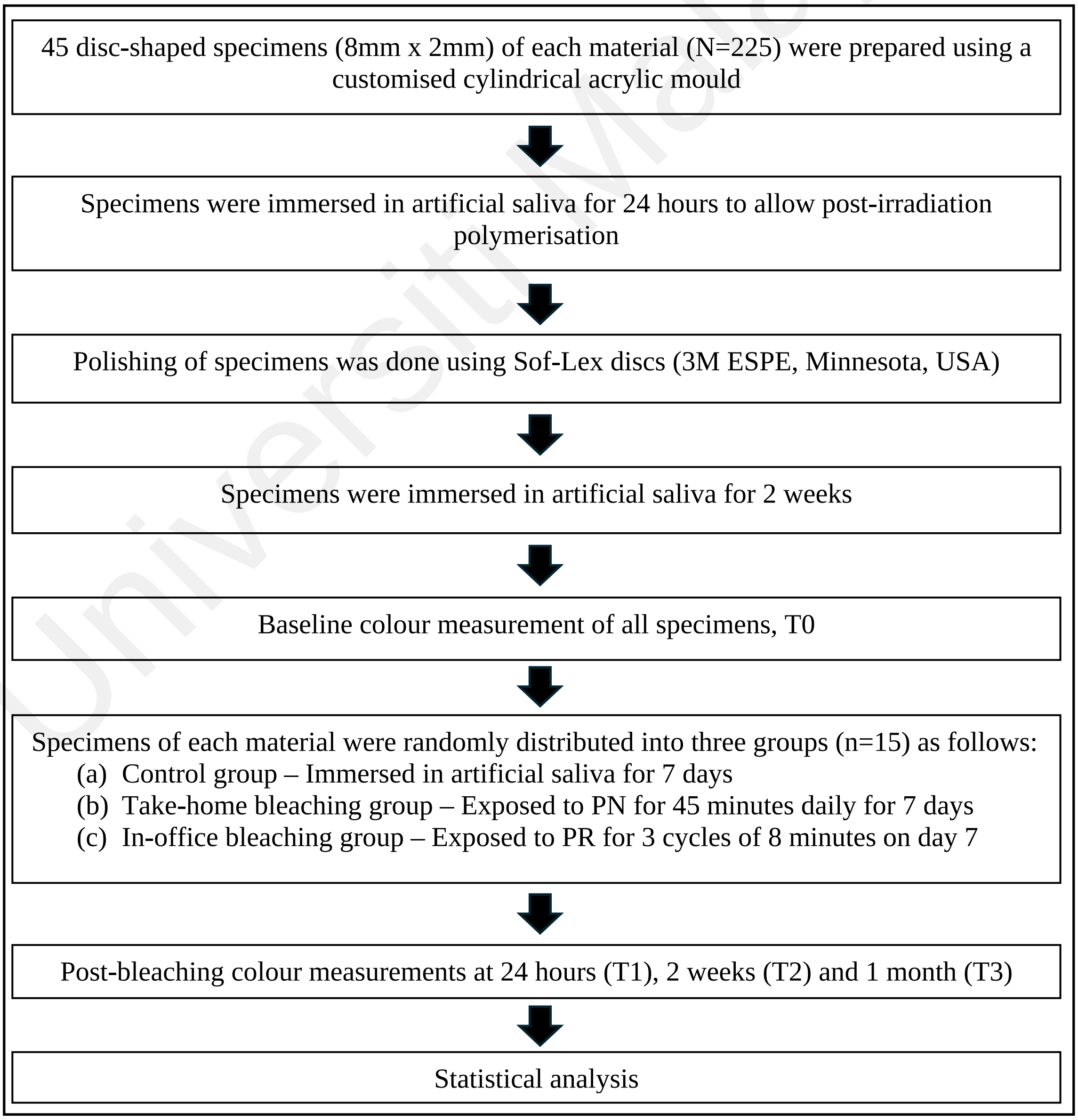
i is the measurement value at 24 hours (T1), two weeks (T2), and one month (T3)

0 is the measurement value at baseline (T0)

Figure 3.9: Konica Minolta Spectrophotometer

3.6 Statistical analysis

Statistical analysis was performed using the statistical software package SPSS 25 for Windows (SPSS Inc., Chicago, Illinois, USA). Shapiro-Wilk test was conducted to assess data normality. As all data were normally distributed, parametric tests were performed at a significance level of $p<0.05$. Differences between IRMs and bleaching products were analysed using one-way analysis of variance (ANOVA) and followed by post-hoc Tukey's test. Two-way ANOVA was done to analyse the effect of the interaction between different IRMs and bleaching products on the ΔE*. Repeated-measures ANOVA was used to analyse the differences in ΔE* between time intervals.



45 disc-shaped specimens (8mm x 2mm) of each material (N=225) were prepared using a customised cylindrical acrylic mould

Specimens were immersed in artificial saliva for 24 hours to allow post-irradiation polymerisation

Polishing of specimens was done using Sof-Lex discs (3M ESPE, Minnesota, USA)

Specimens were immersed in artificial saliva for 2 weeks

Baseline colour measurement of all specimens, T0

Specimens of each material were randomly distributed into three groups (n=15) as follows:
(a) Control group – Immersed in artificial saliva for 7 days
(b) Take-home bleaching group – Exposed to PN for 45 minutes daily for 7 days
(c) In-office bleaching group – Exposed to PR for 3 cycles of 8 minutes on day 7

Post-bleaching colour measurements at 24 hours (T1), 2 weeks (T2) and 1 month (T3)

Statistical analysis

Figure 3.10: Methodological flow chart

CHAPTER 4: RESULTS

Shapiro-Wilk test showed the normal distribution of all data. One-way ANOVA revealed significant differences in the ΔE* between the restorative materials and bleaching products. Two-way ANOVA identified a significant influence of the interaction between the restorative materials and bleaching products on the ΔE*. Moreover, repeated measures ANOVA showed that the colour of similar restorative materials significantly changed over time. Hence, the null hypotheses were rejected.

4.1 L*, a* and b* values

Table 4.1 compares restorative materials' mean L*, a* and b* values at T0, T1, T2 and T3. Most restorative materials increased in L* and a* values from T0 to T3, except for AB bleached with PR and RV immersed in artificial saliva. In contrast, b* values decreased in all restorative materials except for LN bleached with PR and BI immersed in artificial saliva. Change in the L* value, ΔL* ranged from (-0.02 ± 1.23) to (4.94 ± 1.37); change in the a* value, Δa* ranged from (-0.31 ± 0.12) to (0.65 ± 0.13); change in the b* value, Δb* ranged from (-3.08 ± 1.24) to (0.31 ± 0.86).

Table 4.1: Mean (SD) L*, a* and b* values of restorative materials at T0, T1, T2 and T3.

Materials	T0			Bleaching Products	T1			T2			T3		
	L*	a*	b*		L*	a*	b*	L*	a*	b*	L*	a*	b*
LN	63.99 (0.96)	-2.49 (0.18)	6.81 (0.50)	**AS**	65.89 (1.47)	-1.92 (0.38)	6.55 (0.96)	66.02 (1.73)	-1.94 (0.46)	6.40 (0.81)	66.34 (1.22)	-1.96 (0.32)	6.03 (0.91)
				PN	64.15 (0.88)	-2.32 (0.17)	6.55 (0.49)	64.01 (1.07)	-2.33 (0.21)	6.60 (0.42)	64.33 (1.32)	-2.26 (0.22)	6.51 (0.71)
				PR	64.57 (1.20)	-2.14 (0.19)	6.93 (0.98)	64.16 (0.94)	-2.06 (0.18)	7.10 (0.72)	64.55 (0.81)	-2.06 (0.14)	7.12 (0.86)
AB	55.00 (1.35)	-1.49 (0.10)	10.36 (1.42)	**AS**	56.78 (0.99)	-1.38 (0.10)	9.20 (1.26)	58.98 (1.80)	-1.06 (0.09)	7.82 (1.46)	59.94 (1.37)	-0.84 (0.13)	7.28 (1.23)
				PN	55.69 (1.41)	-1.34 (0.10)	8.80 (1.23)	57.28 (0.83)	-1.07 (0.07)	7.97 (1.19)	57.69 (1.11)	-0.89 (0.06)	7.89 (1.24)
				PR	54.98 (1.22)	-1.24 (1.26)	9.20 (1.00)	56.26 (1.29)	-1.09 (0.11)	8.49 (1.18)	57.64 (1.05)	-0.90 (0.09)	7.51 (1.18)
BI	60.29 (0.97)	0.13 (0.18)	4.42 (0.56)	**AS**	60.81 (1.37)	0.17 (0.20)	4.66 (0.82)	61.10 (1.58)	0.13 (0.17)	4.35 (0.75)	60.96 (1.20)	0.21 (0.18)	4.50 (0.78)
				PN	60.69 (1.14)	0.13 (0.07)	3.54 (0.45)	60.43 (1.22)	0.17 (0.09)	3.83 (0.48)	60.54 (1.49)	0.24 (0.12)	3.99 (0.55)
				PR	60.32 (1.40)	0.14 (0.10)	4.21 (0.85)	61.21 (1.23)	0.17 (0.11)	3.77 (0.48)	61.39 (1.32)	0.30 (0.15)	3.76 (0.47)
CN	61.81 (1.83)	-0.95 (0.16)	10.28 (0.79)	**AS**	62.15 (2.39)	-0.76 (0.24)	9.81 (0.59)	62.37 (2.56)	-0.49 (0.24)	9.61 (0.93)	62.75 (1.82)	-0.41 (0.18)	8.78 (1.13)
				PN	61.92 (2.00)	-0.70 (0.15)	9.51 (1.21)	62.10 (1.51)	-0.52 (0.23)	9.43 (1.11)	62.22 (1.87)	-0.40 (0.16)	9.29 (1.27)
				PR	62.13 (2.83)	-0.75 (0.22)	10.03 (1.12)	62.21 (2.41)	-0.52 (0.22)	10.16 (1.34)	62.55 (1.93)	-0.41 (0.18)	9.98 (1.13)
RV	57.64 (2.11)	-0.12 (0.22)	9.69 (0.86)	**AS**	57.95 (0.92)	-0.43 (0.12)	8.87 (0.96)	58.88 (1.32)	-0.30 (0.17)	8.79 (0.80)	59.32 (0.81)	-0.24 (0.16)	8.79 (0.71)
				PN	58.59 (1.52)	0.04 (0.11)	6.79 (0.44)	59.77 (1.21)	0.10 (0.10)	6.97 (0.48)	59.78 (1.23)	0.10 (0.11)	6.91 (0.38)
				PR	59.57 (1.18)	0.03 (0.15)	6.88 (0.47)	59.30 (1.26)	0.02 (0.12)	7.03 (0.56)	60.28 (0.75)	0.08 (0.11)	6.97 (0.61)

Abbreviation: LN=Luna, AB=Activa, BI=Beautifil, CN=Cention-N, RV=Riva, AS=Artificial saliva, PR=Pola Rapid, PN=Pola Night, T0=baseline colour measurement, T1=24 hours after bleaching, T2=two weeks after bleaching, T3=one month after bleaching, L*=lightness from black to white, a*=colour along the red and green dimension, b=colour along the yellow and blue dimension.

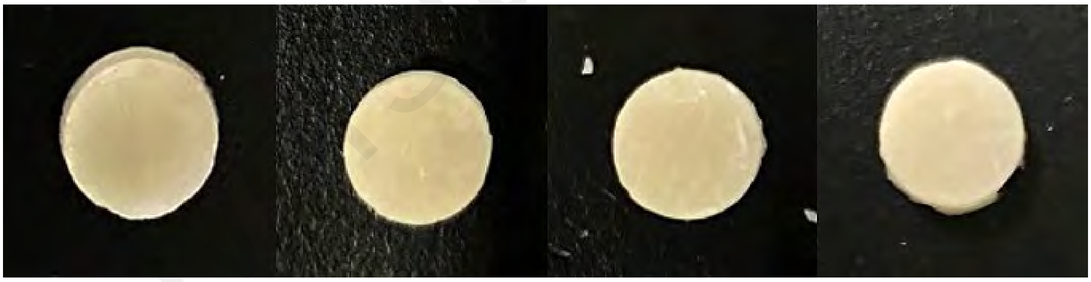
4.2 Colour change, ΔE*

Figure 4.1 illustrates the RV specimens at baseline and T1 when the greatest ΔE* values were observed in the RV bleached with PR (3.55 ± 0.78) and PN (3.34 ± 0.72). Figure 4.2 shows the AB specimens at baseline and T3 when AB exhibited the highest ΔE* for all bleaching groups.

Table 4.2 shows the mean values of ΔE* for all materials at T1, T2 and T3. ΔE* in LN bleached with PN was consistently the lowest throughout the study period, ranging from (0.94 ± 0.48) to (1.22 ± 0.98). LN and AB immersed in artificial saliva showed higher ΔE* compared to those bleached with PN and PR. In contrast, RV bleached with PN and PR resulted in higher ΔE* than those immersed in artificial saliva.

Similar ΔE* between the artificial saliva and bleaching products was noted in BI and CN respectively.

In general, the ΔE* of all groups increased from T1 to T3.



Baseline	Artificial saliva (AS)	Pola Night (PN)	Pola Rapid (PR)

Figure 4.1: RV specimens at baseline and T1

Baseline	Artificial saliva (AS)	Pola Night (PN)	Pola Rapid (PR)

Figure 4.2: AB specimens at baseline and T3

Table 4.2: Mean ΔE* (SD) of restorative materials exposed to bleaching products (n=15), measured at different time intervals

Materials	Bleaching Products	Time Intervals		
		T1	T2	T3
LN	AS	2.33 (1.30)	2.41 (1.58)	2.70 (1.22)
	PN	0.94 (0.48)	1.05 (0.49)	1.22 (0.98)
	PR	1.43 (0.86)	1.21 (0.43)	1.34 (0.40)
AB	AS	2.50 (0.85)	4.95 (1.80)	5.99 (1.33)
	PN	2.39 (0.71)	3.43 (1.14)	3.80 (1.41)
	PR	1.78 (0.78)	2.56 (0.95)	4.02 (1.31)
BI	AS	1.51 (0.71)	1.64 (0.96)	1.47 (0.52)
	PN	1.47 (0.43)	1.28 (0.60)	1.50 (0.63)
	PR	1.35 (0.71)	1.45 (0.94)	1.67 (0.90)
CN	AS	2.15 (1.24)	2.51 (1.32)	2.53 (1.20)
	PN	2.13 (1.15)	1.85 (0.92)	2.20 (1.22)
	PR	2.71 (1.29)	2.52 (1.09)	2.13 (1.09)
RV	AS	1.38 (0.68)	1.88 (1.08)	2.05 (0.77)
	PN	3.34 (0.72)	3.59 (0.88)	3.67 (0.67)
	PR	3.55 (0.78)	3.39 (0.95)	3.86 (0.65)

Abbreviation : LN=Luna, AB=Activa, BI=Beautifil, CN=Cention-N, RV=Riva AS=Artificial saliva, PR=Pola Rapid, PN=Pola Night, T1=24 hours after bleaching, T2=two weeks after bleaching, T3=one month after bleaching

4.2.1 Comparison of ΔL*, Δa*, Δb* and ΔE*

Figure 4.3 shows the mean ΔL*, Δa*, Δb* and ΔE* of restorative materials bleached with PN. The mean ΔE* of all restorative materials increased from T1 to T3, although a slight decrease was noted for BI and CN at T2. At T1 and T2, the highest ΔE* was observed in RV, which was then exceeded by AB at T3, whereas LN consistently remained the lowest.

Figure 4.4 shows the mean ΔL*, Δa*, Δb* and ΔE* of restorative materials after bleaching with PR. From T1 to T3, the mean ΔE* values of AB, BI and RV increased while CN decreased. The highest ΔE* was observed in RV initially but was surpassed by AB at T3.

In both PN and PR, the ΔL* and Δa* values of most restorative materials increased from T1 to T3, while the Δb* values declined. AB and RV exhibited more pronounced changes in the L* and b* values over time compared to other materials.

It can be deduced that the colour change occurred mostly due to the L* and b* value changes. For instance, ΔL*, Δa* and Δb* at T3 for AB bleached with PR were 2.64, 0.59 and -2.84 respectively, resulting in a ΔE* value of 4.02.

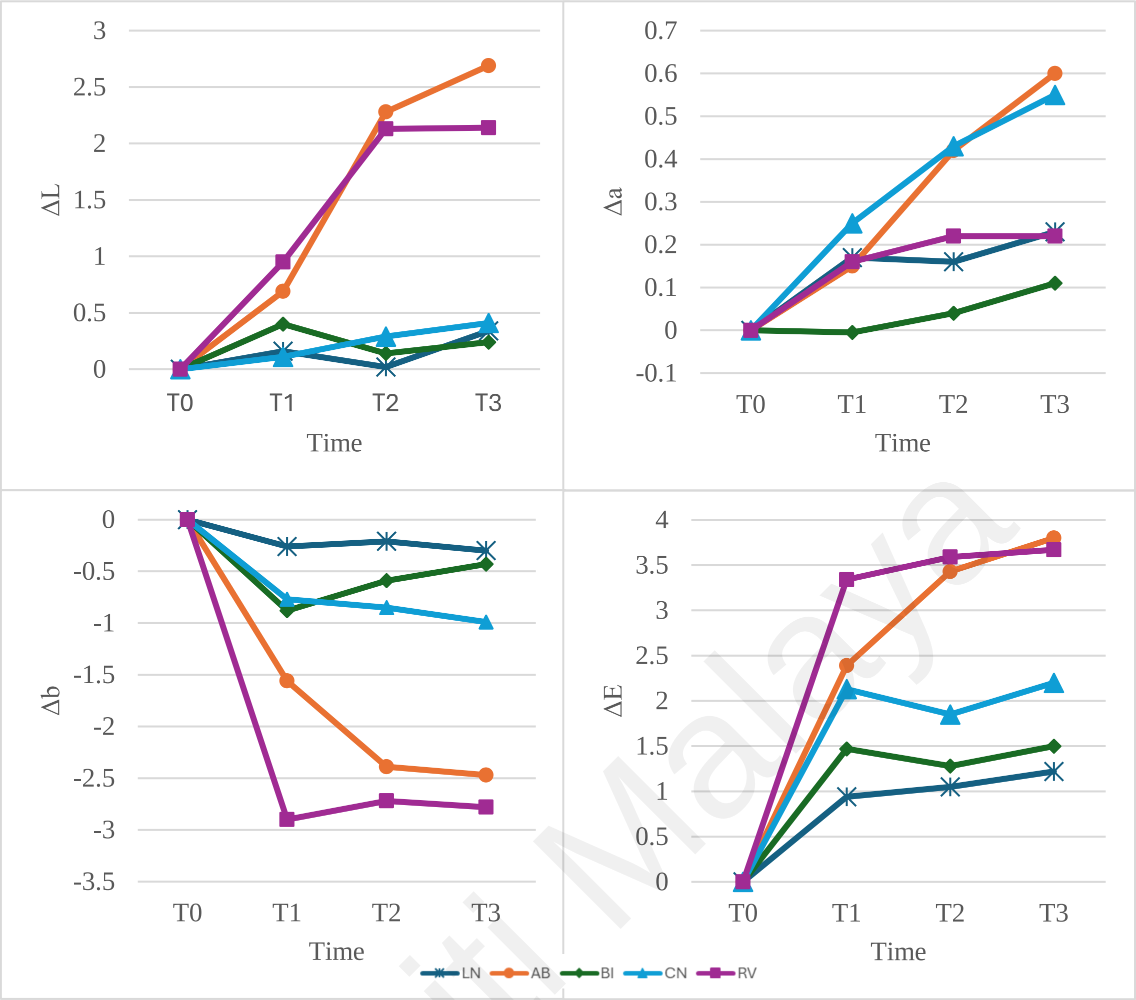


Figure 4.3: Mean ΔL*, Δa*, Δb* and ΔE* of restorative materials bleached with PN.

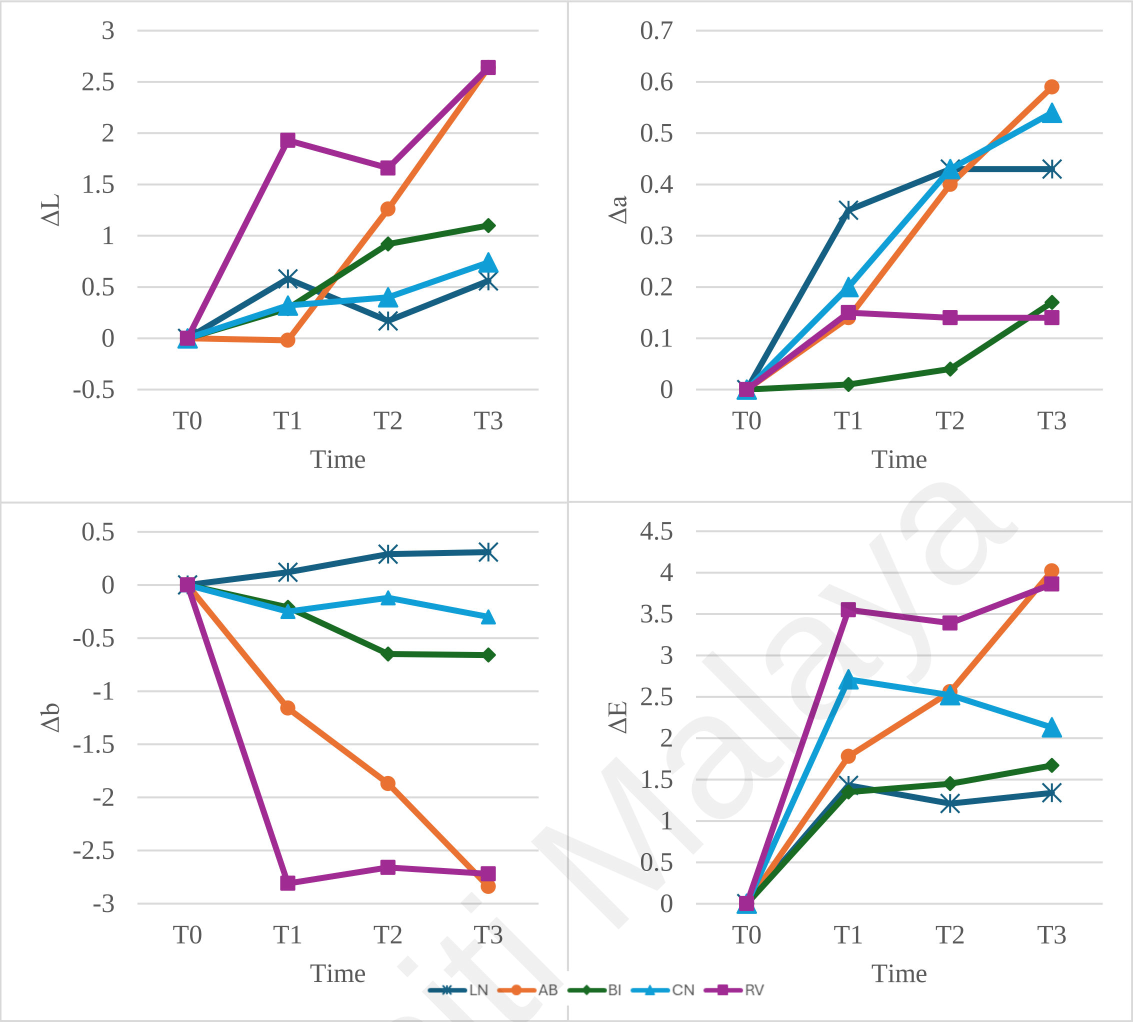


Figure 4.4: Mean ΔL*, Δa*, Δb* and ΔE* of restorative materials bleached with PR.

4.2.2: Comparison of ΔE* between different bleaching products

Table 4.3 compares the restorative materials' ΔE* between different bleaching products**.** The effect of bleaching products on the ΔE* of restorative materials was material dependent. LN demonstrated a significantly higher colour change in artificial saliva than both PR and PN. Similar results were observed in AB at T2 and T3. On the other hand, PR and PN resulted in significantly greater colour changes for RV samples. There were no significant differences in the colour change produced by PR and PN. There were no differences in the colour change produced by artificial saliva and the bleaching products in BI and CN.

Table 4.3: Comparison of restorative materials' ΔE* between different bleaching products.

Materials	**Differences**		
	T1	**T2**	**T3**
LN	AS > PR = PN	AS > PR = PN	AS > PN = PR
AB	=	AS > PR = PN	AS > PN = PR
BI	=	=	=
CN	=	=	=
RV	PR = PN > AS	PR = PN > AS	PR = PN > AS
Abbreviation : LN=Luna, AB=Activa, BI=Beautifil, CN=Cention-N, RV=Riva AS=Artificial saliva, PR=Pola Rapid, PN=Pola Night, T1=24 hours after bleaching, T2=two weeks after bleaching, T3=one month after bleaching			

Results on one-way ANOVA/Tukey's post hoc test with significance level set at ($p<0.05$). = denotes no significant differences between bleaching groups, whereas ">" denotes significantly greater.

4.2.3 Comparison of ΔE^* between different restorative materials

Table 4.4 compares the ΔE^* between different restorative materials subjected to similar bleaching products.

In artificial saliva, AB had the greatest colour change across all time intervals, followed by LN. BI had the least colour change in all time intervals except at T1 which was presented by RV.

When samples were bleached with PN and PR, the highest colour change was seen in RV at T1 and T2. The colour change of RV was then exceeded by AB at T3. LN generally had the lowest colour change across all time intervals except at T1 and T3, which was observed with BI.

Table 4.4: Comparison of ΔE^* between different restorative materials subjected to similar bleaching products.

Time Intervals	**Differences**		
	AS	**PN**	**PR**
T1	AB > LN ≥ CN > BI ≥ RV	RV > AB ≥ CN > BI ≥ LN	RV ≥ CN > AB ≥ LN ≥ BI
T2	AB > LN ≥ CN ≥ RV ≥ BI	RV ≥ AB > CN ≥ BI ≥ LN	RV ≥ AB ≥ CN > BI ≥ LN
T3	AB > LN ≥ CN > RV ≥ BI	AB ≥ RV > CN ≥ LN ≥ BI	AB ≥ RV > CN ≥ BI ≥ LN
Abbreviation : LN=Luna, AB=Activa, BI=Beautifil, CN=Cention-N, RV=Riva AS=Artificial saliva, PR=Pola Rapid, PN=Pola Night, T1=24 hours after bleaching, T2=two weeks after bleaching, T3=one month after bleaching			

Results on one-way ANOVA/Tukey's post hoc test with significance level set at ($p<0.05$). "≥" denotes greater than but not statistically significant, whereas ">" denotes statistical significance.

4.2.4 Comparison of ΔE* between T1, T2 and T3

Table 4.5 compares the restorative materials' ΔE* between T1, T2 and T3. The colour of all restorative materials remained stable over time except for AB. In artificial saliva and PN, the ΔE* of AB significantly increased from T1 to T2 and remained stable up to T3. In PR, the ΔE* significantly increased from T1 to T2, and from T2 to T3.

Table 4.5: Comparison of restorative materials' ΔE* between T1, T2 and T3.

Materials	**Differences**		
	AS	**PN**	**PR**
LN	=	=	=
AB	T3 = T2 > T1	T3 = T2 > T1	T3 > T2 > T1
BI	=	=	=
CN	=	=	=
RV	=	=	=
Abbreviation : LN=Luna, AB=Activa, BI=Beautifil, CN=Cention-N, RV=Riva AS=Artificial saliva, PR=Pola Rapid, PN=Pola Night, T1=24 hours after bleaching, T2=two weeks after bleaching, T3=one monhth after bleaching			

Results on repeated measures ANOVA with significance level set at ($p<0.05$). "=" denotes no statistically significant difference, whereas ">" denotes significantly greater.

CHAPTER 5: DISCUSSION

This study has determined the effects of bleaching products on the colour stability of some IRMs and evaluated their post-bleaching colour stability over time.

5.1 Methodology

5.1.1 The justification for choosing the evaluated materials

The rationale for investigating the properties of IRM is partly due to their ability to promote reminersalisation of adjacent tooth structure. While CR remains popular among clinicians, its failures were frequently caused by secondary caries and fractures (Demarco et al., 2012; Opdam et al., 2014). The prevalence of secondary caries in restorations reported was approximately 60% and often necessitated the replacement of the restorations (Ge et al., 2023). On the other hand, contemporary IRMs offer an added advantage of resistance to recurrent caries. Systematic reviews and meta-analyses have documented the superior efficacy of GIC and RMGIC in preventing secondary caries compared to amalgam and CR restorations (Ge et al., 2023; Pinto et al., 2023).

The occurrence of secondary caries is multifactorial. From the aspects of restorative materials, polymerisation shrinkage exhibited by resin-based dental materials ranges from 1-6% (Soares et al., 2017). The resultant shrinkage stress generated causes debonding at the tooth-restoration interface. Moreover, resin-based restorations and bonding agents are prone to hydrolytic degradation (Vilde et al., 2022). The marginal gaps and microleakage at the tooth-restoration interface facilitate biofilm accumulation and the subsequent development of recurrent caries (Ge et al., 2023).

Considering the high failure rates of CR attributed to recurrent caries, novel IRMs are desirable alternatives due to their ability to combat caries. The main anti-cariogenic action of IRM is associated with its release and recharge of fluoride ions that inhibit demineralisation and promote the remineralisation of dental hard tissues. Its ability to form a chemical bond with dentine provides a good cavity seal. Microleakage at the restoration margin can also be counteracted by the hygroscopic expansion of GIC and RMGIC around 6% at 24 hours (Ge et al., 2023). Hence, the properties of IRM are worth evaluating as they may be the solution to extending the clinical longevity of restorations.

The IRMs were compared against LN, a conventional nanohybrid CR that was reportedly more resistant to stains and discolouration (Al-Shami et al., 2023). The manufacturer claimed that LN has a chameleon effect that can match the natural tooth's translucency, opalescence and fluorescence (SDI, 2024a). AB, BI, CN and RV each represent their respective categories of IRMs. AB is the first bioactive composite as claimed by its manufacturer (Pulpdent Corporation, 2023). BI is a recently introduced giomer with a low volumetric shrinkage of 0.85% (Firla, 2018). CN is an alkasite which delineates itself from others by having an alkaline filler (Alla et al., 2023). RV is a high-viscosity RMGIC which was asserted to have good colour stability (SDI, 2023).

The popular demand for dental aesthetics has sparked the growth of the tooth-bleaching market. Depending on the procedure, restoration surfaces may be subjected to these bleaching agents for an extended period throughout the bleaching process. For instance, take-home bleaching is applied in a customised bleaching tray worn by patients overnight. As bleaching products contain oxidising chemical compounds, their potential adverse effect has been widely discussed (Dahl & Pallesen, 2003; Alqahtani, 2014). To date, their effects on the properties of restorative materials remain controversial, with literature reporting significant negative influences on surface roughness, microhardness and bond strength (Attin et al., 2001; Karatas et al., 2021; Alqahtani 2014), while others reported conflicting results (Dogan et al., 2008; Fernandes et al., 2020). The discrepancies in these findings can be due to the difference in study methodology, and the wide variation in composition of tested materials. The bleaching products examined in this study were

PR and PN, which featured a short application time (i.e. 24 minutes for PR; 45 minutes daily for PN), compared to similar products, which require 40-60 minutes for chairside bleaching, and up to eight hours for take-home bleaching. Both products also contain fluoride which helps to minimise the risk of post-operative hypersensitivity (SDI, 2024b; SDI, 2024c).

5.1.2 Specimen preparation

The 8mm diameter of the specimens corresponded to that of the light-curing tip to ensure uniform light polymerisation (Cardoso et al., 2022). After light-curing, the specimens were incubated for 24 hours in artificial saliva to allow complete post-irradiation polymerisation (Li et al., 2009; Yap et al., 1997). Delayed polishing of the specimens was carried out after the incubation period to prevent plastic deformation of the incompletely cured restoration (Gonder & Fidan, 2022). Fabricating specimens using a Mylar strip produced a smooth and glossy surface. However, as in clinical situations, finishing and polishing were conducted to remove the superficial layer with high resin content that is more susceptible to wear. This improves the restoration’s colour stability and early wear resistance (Yazici et al., 2010). A mylar finish and consistent polishing protocol using SofLex discs were employed to ensure a uniform surface roughness among specimens, thereby minimising its potential influence on colour stability.

The specimens were further immersed in artificial saliva for two weeks before commencing bleaching procedures. This allows for stabilisation of the materials’ water sorption, which typically occurs in 7-14 days after polymerisation (Lima et al., 2018; Li et al., 2009). Resin-based materials are prone to hydrolytic degradation causing the alteration in their optical properties.

5.1.3 Immersion protocol

An artificial saliva, SAGF was used to mimic the oral environment. While many studies have used deionised and distilled water (Yu et al., 2009; Kara et al., 2013; Erturk-Avunduk et al., 2022), it is worth noting that these solutions cannot accurately represent the chemical complexity of the oral environment. Due to the difference in the composition of artificial saliva and distilled water, the leaching of filler was found to be more pronounced in artificial saliva than in distilled water (Soderholm et al., 1996). This is attributed to the discrepancy in ions present in these immersion mediums, of which distilled water only contains hydrogen and oxide ions. In contrast, artificial saliva contains a variety of ions including potassium, chloride, magnesium and calcium ions (Gal et al., 2001).

It is known that IRM relies on ionic exchange in its physicochemical properties (Francois et al., 2020). Hence, using SAGF can better replicate the oral environment and provide a better understanding of the materials' properties.

In this study, the colour measurements were taken 24 hours, two weeks and one month after bleaching. This protocol followed other colour stability studies (Torres et al., 2012; Li et al., 2009; Dunn, 2020; Forabosco & Checchi, 2023; Melo et al., 2024). The post-bleaching colour measurement after 24 hours reflects clinical situations where restorations are constantly in an aqueous environment. The interaction between the artificial saliva and the restorative materials may influence their post-bleaching colour. Bleaching procedures also cause dehydration (Alamé & Zogheib, 2023), in which a colour assessment after 24 hours of rehydration may be more reliable (Hatirli et al., 2021). A further two-week and one-month colour remeasurement allows an evaluation of the materials' ageing resistance, which is correlated to their clinical longevity (Yilmaz and Sadeler, 2018). A prolonged immersion time has also been reported to result in significant colour alterations in IRMs (Abuljadayel et al., 2023).

5.1.4 Spectrophotometric analysis of colour stability

Ryge Criteria is frequently employed to evaluate the success of restorations on multiple aspects. The colour of restorations is assessed by observing how well the shade and translucency of restoration match the surrounding tooth tissue (Cvar & Ryge, 1971). However, in-vivo studies evaluating dental restorations can be complex, costly, and cause ethical concerns. Furthermore, a subjective assessment relies on the operators’ experience and can be influenced by environmental factors such as the ambient light conditions, the patient’s sitting position and the application of lipstick (Ugurlu et al., 2022).

Thus, an objective evaluation of colour using the instrumental method is generally considered more practical (Ahamed et al., 2023). This study utilised a bench-top spectrophotometer which measures the light reflectance of the specimens at different wavelengths, which is then calculated into values using the CIE-L*a*b* colour space. Apart from providing a quantitative measurement, its results on the L*, a* and b* colour coordinates also provide information regarding the specific colour dimensions that have led to the colour change of restorations.

The colour is also influenced by the background of which the specimens are measured (Tabatabaian et al., 2021). In this study, the specimens were placed against a standardised black background to stimulate the darkness in the oral cavity (Poggio et al., 2016). A variety of background colours such as white, composite shade A3, grey and pink have been described in other studies (Tabatabaian et al., 2021; Erturk-Avunduk et al., 2022; Li et al., 2009).

5.2 Discussion of results

5.2.1 Clinical perceptibility and acceptability of colour change

The colour difference thresholds are key when discussing the clinical significance of colour change in restorations. Varying values of perceptibility and acceptability thresholds have been utilised in the literature, ranging from 0.4 to 2.6 and 1.7 to 5.5, respectively (Paravina et al., 2019). A recently proposed CIEL*a*b* 50%:50% perceptibility and acceptability thresholds were ΔE*=1.2 and ΔE*=2.7 (Paravina et al., 2015). Most groups exceeded the ΔE*=1.2 threshold at T1, except for LN bleached with PN which exhibited clinically negligible colour change until two weeks after bleaching (T2). Its ΔE then exceeded the perceptibility threshold slightly at one month (ΔE*=1.22). RV bleached with PN and PR, and CN bleached with PR exceeded the acceptability threshold at T1. On the other hand, the colour of AB worsened drastically, causing clinically unacceptable colour alteration for the control and both bleaching groups at T3.

5.2.2 Changes in the colour dimensions, L*, a* and b*

In general, the L* and a* values increased, while the b* value decreased after bleaching. Changes in the L* and b* dimensions were more pronounced than the a* dimension. When relating these changes to the Munsell colour space, the colour of the specimens was altered towards a lighter and bluish hue, causing a lightening of the restorative materials (Attin et al., 2003). Moreover, the a* and b* values were shifted closer to the zero or neutral axis (i.e. negative a* values increased, positive b* values decreased) after bleaching causing desaturation and a duller appearance (McGuire, 1992). Similarly, Karamangar et al. (2014) and Korać et al. (2021) also found that the increase in the L* value was the main factor for colour change in their studies. RV and AB specimens bleached with PN and PR resulted in a larger change in the b* value than the L* value. Yu et al. (2009) reported similar findings where the b* values of RMGIC and compomer declined considerably immediately after bleaching. The oxidising agents present in bleaching products cause surface dissolution. This leads to the formation of tiny pores which can be occupied by saliva and air, changing the material's reflectance and optical properties (Li et al., 2009). Another possible reason for this finding is the elution of monomers changes the surface roughness of materials.

LN appeared more resistant to bleaching-induced colour alterations than the other materials. This finding is in accord with the results reported by Pecho et al. (2019), who observed little changes in all L*, a* and b* dimensions for CR exposed to 35% hydrogen peroxide.

5.2.3 Comparison of the colour changes produced by different bleaching products

There were no statistically significant differences in the ΔE* produced by PR and PN, corresponding to findings from other studies (Silva Costa et al., 2009; Kurtulmus-Yilmaz et al., 2013; Kamangar et al., 2014). This is possibly due to the short application of a high concentration of hydrogen peroxide having an equivalent effect with the prolonged application of a low concentration of hydrogen peroxide. PR and PN have varying concentrations of hydrogen peroxide and bleaching protocol. PR contains 38% hydrogen peroxide while PN contains 22% carbamide peroxide dissociating into 6.6% hydrogen peroxide. Studies have demonstrated a positive correlation between the concentration of hydrogen peroxide and the colour change in restorative materials (Canay & Cehreli, 2003; Yalcin & Gurgan, 2005). This is because a high concentration of hydrogen peroxide releases more reactive oxygen species during the bleaching process. The bleaching protocol in this study followed the manufacturer's instruction, consisting of three cycles of 8 minutes for a single day for PR and 45 minutes daily for seven days for PN. An extended duration of application also increases the exposure of the restorative materials to the detrimental effects of the reactive oxygen species (Yu et al., 2015).

Bleaching significantly increased the colour change of RV samples. The colour change induced by bleaching products is caused by the attack of radical oxygen species and perhydroxyl ions on the resin-filler interphase, causing microporosities and a rougher surface that is less stain-resistant. Bleaching agents also oxidise the surface pigments and amides that are directly related to the shade of the restoration (Alqahtani, 2014).

Colour change can also be induced by immersion in artificial saliva. The discolouration is associated with the water sorption property of the resin matrix. Water absorption facilitates the elution of residual monomers which affects the materials' colour stability (Moldovan et al., 2019). Residual monomers are inevitable in resin-based material due to the gel effect that prevents the total conversion of organic monomers during photo-polymerisation (Huang et al., 2022). It can also lead to chemical debonding between the resin and filler and plasticisation of the restoration which results in increased wear and surface roughness (Attin et al., 2004). Interestingly, LN and AB presented significantly higher ΔE* when immersed in artificial saliva than the bleached samples. The possible reason is the difference in the surface roughness between the control and bleached samples (Dogan et al., 2008). The polished samples in this study may present fillers extending onto the restoration surface at baseline, which were then dissolved by the hydrogen peroxide during bleaching procedures, resulting in a smoother surface. This results in a lower surface roughness in bleached samples than those immersed in artificial saliva, improving the optical appearance (Dogan et al., 2008).

5.2.4 Comparison of colour change between restorative materials

Compared to other materials, the colour change in RV appeared to be most affected by bleaching at T1 and T2. The lack of colour stability may occur due to the degradation of the metal polyacrylate salts in the set material (Rao et al., 2009). This is also shown by Rao et al.'s (2009) study where GIC exhibited the most discolouration compared to giomer and CR. It is expected that IRMs tend to discolour more than CR, attributed to the dissolution of functional filler particles. The release of ions into the external environment of the restoration also requires water diffusion. This could form internal voids that increase water sorption and solubility (Marovic et al., 2022), as shown by the appearance of cracks and pits on the surface of GIC under scanning electron microscopy (Yu et al., 2009). The difference in the colour stability between materials can be explained by their variation in filler content, size, and type of organic monomer. The utilisation of higher filler content and organic monomers such as UDMA increases the hydrophobicity of the material (Gonulol et al., 2015). As AB contains the least filler weight among other materials (55.4%), its ΔE* exceeded RV at T3. In contrast, BI which has 83% filler weight was significantly more resistant to colour change caused by bleaching. Its ΔE* is close to that of LN, which had the best colour stability against bleaching procedures. This result is consistent with other studies (Rao et al., 2009, Li et al., 2009; Yu et al., 2009). The penetration of the bleaching agent into the matrix of LN can be impeded by a tight cross-linkage formed by the high molecular weight polymer molecules (Li et al., 2009). The colour stability of CN can be attributed to its resin matrix's hydrophobicity of UDMA, DCP, an aromatic aliphatic-UDMA, and PEG-400 DMA along with high filler content. CN also utilises a thiocarbamide as a photoinitiator instead of tertiary amines that are susceptible to oxidation over time leading to discolouration (Bhattacharya et al., 2020).

5.2.5 Comparison of colour change over time

In general, the ΔE* of restorative materials increased over time, but no statistically significant differences were detected between T1, T2 and T3 except for AB. This implies that the colour change only occurred immediately after bleaching, and bleaching does not worsen the materials' colour stability in the aqueous environment. This is probably due to the water stabilisation of the IRMs that can occur within 7 to 14 days of artificial saliva immersion (Lima et al., 2018). The samples were subjected to prolonged artificial saliva immersion after the initial setting and between bleaching procedures. Moreover, the samples were aged in artificial saliva for one month after completing the bleaching procedures. Most colour changes occur within the first 7-10 days when most water sorption occurs (Luce & Campbell, 1988).

Water sorption is a diffusion-controlled process which mainly takes place in the resin matrix (Omar, 2015). The higher amount of resin matrix in AB may necessitate a longer time to achieve water sorption equilibrium. This will explain why the ΔE* of AB samples immersed in artificial saliva and bleached with PN only stabilised after T2.

5.3 Clinical recommendations

This study showed that prolonged exposure to an aqueous environment and bleaching products can lead to restoration discolouration. Care must be taken to avoid exposing restorations to bleaching products. RV that presented with unacceptable colour change after bleaching procedures may require replacement. AB which exhibited poor colour stability might not be suitable for use in the aesthetic zone. Alternatively, restoration procedures should be carried out one to three weeks after completion of bleaching procedures (Attin et al., 2004)

5.4 Limitations of study and suggestions for future research

This study cannot accurately simulate the oral environment such as biofilm adhesion and thermal variations. Although SAGF contains ions that mimic natural saliva, it lacks glycoproteins that can affect its viscosity which plays a role in the diffusion and reaction rates between the biomaterials and the immersion medium (Gal et al., 2001). The ageing protocol of specimens can be improved by including a thermocycling process to simulate thermal changes.

The drawback of the CIEL*a*b* colour space is that it only measures the differences in three parameters, the lightness (L*), red to green dimension (a*) and yellow to blue dimension (b*). As previously discussed, human colour perception can be altered by the lighting conditions and the object’s surroundings (Burkinshaw, 2004). Human was also found to be more sensitive to the changes in reddish colours (a*) than yellow (b*) (Gómez-Polo et al., 2020). The most recent colour-difference formula, CIEDE2000 (ΔE_{00}) incorporates weighting functions and parametric factors to account for variation in colour-difference perception dependent on lightness, chroma, hue, chroma-hue interaction and the influence of illumination and vision conditions (del Mar Perez et al., 2011). Hence, the calculation of colour change using the ΔE_{00} formula in future research may be more clinically accurate. However, it is worth noting that the perceptibility and acceptability thresholds of this formula are $\Delta E_{00} = 0.8$ and $\Delta E_{00} = 1.8$ respectively, which are significantly different from the ΔE_{ab} values.

As discussed earlier, the materials’ colour stability is correlated to their surface roughness, microhardness, water sorption and solubility properties. Studies should address these properties of the materials when investigating their colour stability. Other properties that affect restorative materials’ optical appearances such as surface gloss, translucency, fluorescence and opalescence should be assessed in future research (Joiner, 2004).

CHAPTER 6: CONCLUSION

Within the limitations of this study, the following conclusions are drawn:

1. As the bleaching products significantly influenced the colour stability of IRMs and the colour stability of similar IRM over time, the initial null hypotheses were duly rejected.
2. PR and PN produced similar colour changes for all IRMs.
3. The effect of bleaching products on the colour stability of IRMs was material dependent. RV had the highest colour change after bleaching. AB and LN had more colour changes when immersed in artificial saliva than bleaching. For BI and CN, similar colour changes were produced by artificial saliva and bleaching.
4. The colour of IRMs remained stable over one month after bleaching, except AB which significantly increased from T1 to T3. AB had the greatest colour change for all bleaching groups after one month.

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