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EFFECT OF VARIOUS BEVERAGES ON COLOR STABILITY OF RESIN CEMENTS

MASTER'S THESIS

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APPROVAL

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DECLARATION

I hereby declare that this thesis is my own work and that, to the best of my knowledge and belief, it contains no material previously published or written by another person nor material which has been accepted for the award of any other degree except where due acknowledgment has been made in the text.

20.10.2020

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LIST OF SYMBOLS AND ABBREVIATIONS

3M.DC	3M Rely-X Ultimate Clicker Dual-Cure
3M.LC	3M Rely-X Veneer Light-Cure
ANOVA test	Analysis of Variance
Bis-GMA	Bisphenol A-glycidyl Di Methyl Acrylate
ΔE	Change in Color
CAD / CAM	Computer Aided Design / Computer Aided Manufacture
EBPADMA	Ethoxylated Bisphenol A Dimethacrylate
EDMAB	Ethyl 4-Dimethylaminobenzoate
HEMA	Hydroxyethyl Methacrylate
Lux	Illuminance
CIE	International Commission on Illumination
K	Kelvin
K.DC	Kerr NX-3 Dual-Cure
K.LC	Kerr NX-3 Light-Cure
LED	Light-Emitting Diode Light
$V(\lambda)$	Luminous Efficiency Function
MPa	Megapascal
PTU	Pyridyl Thiourea
RMGI	Resin-Modified Glass-Ionomer Cement
SD	Standard Deviation
TEGDMA	Triethylene Glycol Di Methyl Acrylate
UDMA	Urethane Di Methyl Acrylate
W/cm ²	Watt/square centimeter
mm	millimeter
nm	nanometer

ABSTRACT

Mostawe, L. (2021). Effect of Various Beverages on Color Stability of Resin Cements. Yeditepe University, Institute of Health Sciences, Department of Prosthodontics, MSc thesis, Istanbul.

The purpose of this *in vitro* study was to investigate the effects of various staining solutions on the color of resin cements. 4 commercially available resin cements (Rely-X Ultimate Clicker Dual-Cured, Rely-X Veneer Light-Cured, NX-3 Dual-Cured, NX-3 Light-Cured) were tested. The specimens were immersed in 5 different solutions: cola, tea, coffee, red wine as test groups and distilled water as the control group. Before immersion, the initial color value of each specimen was recorded by using a spectrophotometer. Color changes (ΔE) were determined after 24 hours, 72 hours, 144 hours, 216 hours, and 288 hours of immersion. The CIE L*a*b* color space was used to identify color differences. At the end of 288 hours of immersion, the greatest color change (ΔE) was observed for the NX-3 dual-cured resin cement group immersed in red wine ($\Delta E=21.66\pm2.73$); and the lowest ΔE value was seen when the NX-3 light-cured resin cement group was immersed in cola ($\Delta E=1.86\pm0.33$). In all resin cement groups, red wine and coffee caused the highest the amount of staining and caused clinically marked color change starting from 24 hours immersion, whereas tea caused marked color change following 72 hours of immersion ($NBS>3$). All the specimens immersed in distilled water and cola did not show clinically marked discoloration.-The effect of time, material, and solution factors on color stability levels of resin cements were significant. As the immersion time increased, the degree of discoloration of all resin cements also increased. The light-cured resin cements were generally more color stable than the dual-cured resin cements.

Keywords: Resin cements, color stability, staining, spectrophotometer.

ÖZET

Mostawe, L. (2021). Rezin Simanların Renk Stabilitesi Üzerine Çeşitli İçeceklerin Etkisi. Yeditepe Üniversitesi, Sağlık Bilimleri Enstitüsü, Protetik Diş Tedavisi ABD., Master Tezi, İstanbul.

Bu *in vitro* çalışmanın amacı, rezin simanların rengi üzerinde çeşitli boyayıcı solüsyonların etkilerini araştırmaktır. Piyasada bulunan 4 rezin siman (Rely-X Veneer Light-Cured, Rely-X Ultimate Clicker Dual-Cured, NX-3 Light-Cured, NX-3 Dual-Cured,) test edildi. Örnekler, kontrol grubu olarak distile suya, test grupları olarak da kola, çay, kahve ve kırmızı şarap olmak üzere 5 farklı solüsyona maruz bırakıldı. Solüsyonlarda bekletilmeden önce, her örneğin başlangıç renk değeri bir spektrofotometre kullanılarak kaydedildi. Rezin siman örneklerin renk değişiklikleri (ΔE), solüsyonlara 24 saat, 72 saat, 144 saat, 216 saat ve 288 saat maruz bırakıldıktan sonra belirlendi. Renk farklılıklarını belirlemek için CIE $L^*a^*b^*$ renk alanı kullanıldı. 288 saat sonunda en büyük renk değişimi (ΔE) kırmızı şarap içinde bekletilmiş NX-3 dual-sertleşen rezin siman grubunda ($\Delta E=21.66\pm 2.73$); en düşük ΔE değeri ise kola içinde bekletilmiş NX-3 ışıkla-sertleşen rezin siman grubunda ($\Delta E=1.86\pm 0.33$) görülmüştür. Tüm rezin siman gruplarında, en fazla boyama etkisi gösteren solüsyonlar olan kırmızı şarap ve kahve, 24 saatlik bekletmeden başlayarak, çay ise 72 saatlik bekletmeden başlayarak klinik olarak belirgin bir renk değişikliğine ($NBS>3$) neden olmuştur. Distile su ve kola içinde bekletilen tüm örnekler klinik olarak belirgin renk değişikliği göstermemiştir. Zaman, materyal ve solüsyon faktörlerinin rezin simanların renk stabilitesi üzerine etkisi anlamlı bulunmuştur. Solüsyonlar içinde bekletme süresi arttıkça, tüm rezin simanlarda görülen renk değişim miktarı da artmıştır. Işıkla sertleşen rezin simanların renk stabilitesi, dual-sertleşen rezin simanların renk stabilitesinden daha fazla olmuştur.

Anahtar kelimeler: Rezin simanlar, renk stabilitesi, renklenme, spektrofotometre.

1. INTRODUCTION AND PURPOSE

There has been a growing increase in the esthetic demands of patients in recent years; therefore, all-ceramic crowns/bridges, porcelain laminate veneers and porcelain inlays/onlays have started to be widely used for the restoration of both anterior and posterior teeth owing to their natural and esthetic appearance. These types of restorations are built for the correction of function, discolorations, angulation problems, shape, closure of diastema and other esthetic issues (1,2,3).

Resin cements are used for cementing all-ceramic restorations as they contribute to favorable esthetic and superior retention as well as fracture resistance of the restorations. Additionally, they show low solubility in oral environment and favorable mechanical properties including flexural strength shear strength, compressive and tensile bond strength, and good adhesion (3,4,5,6).

Resin cements are not only expected to match with adjacent teeth in terms of shade, but they also should maintain color stability all through out their service in the oral environment (7,8,9). Restoration margins are areas where resin cements are exposed to the oral environment, therefore, any kind of discoloration of resin cement at this region may compromise the esthetic properties of all ceramic restorations (4,10). The color change is much more significant when thin-translucent ceramic veneers are used (4,11).

Resin cements can be classified according to the polymerization system: self-cured, light-cured, and dual-cured cements. Self-cured resin cements are mostly utilized for cementation of metallic and metal-ceramic restorations or cast posts. The use of light cured cements is more limited and these materials are indicated only for luting laminate veneers because the light intensity during the transmission through the restoration is decreased (4,12). When areas of difficult access are concerned such as restorations with high thickness, dual cured cement can be preferred because they have the ability to be polymerized in sites where the curing light cannot penetrate. Furthermore, their degree of conversion is high both in the presence and absence of light (4,13).

Discoloration of resin cements can be caused by either intrinsic or extrinsic factors. Intrinsic factors are related with the material including the composition of resin matrix, filler content, type of photo initiator and polymerization systems, and the conversion ratio of the carbon-carbon double bonds. Intrinsic factors are expedited by ultraviolet irradiation or alterations in temperature (6,13). A solution for the prevention of discoloration is production of the resin cement without a benzoyl peroxide/tertiary amine photo initiator system. These types of resin cements have exhibited higher degree of color

stability (13). It has also been shown that triethylene glycol dimethacrylate has a higher degree of susceptibility to discoloration, because they can be detached from the organic matrix along with larger filler particles. This causes a rougher surface which can be easily stained (14). On the other hand, the influence of intrinsic factors on color stability is lower in completely polymerized composite resins. The most important factor that affects the color stability of composite resins is thought to be external discoloration (9). Therefore, resin cements under the restoration may undergo discoloration, especially at the margin of the restoration that is exposed to the oral environment. Discoloration of the margins would compromise the esthetics quality of the restoration and require its replacement, which can be considered as one type of treatment failure (2,9). The adsorption and absorption of media including food and beverage dyes are the main factors which cause external discoloration as well as smoking. the discoloration is also influenced by the conversion ration of the carbon-carbon double bonds as well as surface roughness of resin cements (6,13,14). Some *in-vitro* studies have suggested that the color stability of dual-cured resin cements is lower compared to light-cured resin cements due to the oxidation of aromatic tertiary amine which are included in the composition of dual cured cements. These amines serve as accelerators for the auto polymerizing reaction (2). In light-cured cements, inadequately polymerized camphorquinone gradually turns into a yellowish color. On the other hand, the chemical stability of light cured resin cements is higher and they cause less color change over time.

However, light-cured resin cements are more chemically stable and tend to result in less color change over time (13,15,16).

A common methodology that is used to investigate the staining properties of dental materials is the immersion of specimens in staining solutions for specific periods. It has been indicated that significant discoloration may occur in composite resins when they are immersed in commonly consumed drinks such as coffee, red wine, tea, cola, and fruit juices for prolonged periods (9).

Although various studies have examined the color stability of restorative composite resins (17-26), only few studies have evaluated the use of composite resins as adhesive cements (3,6,9). Therefore, the purpose of this study was to evaluate the effects of various beverages on the color of light-cured and dual-cured resin cements. The tested null hypothesis was that: the immersion solution and the type of adhesive resin cements have no influence on the color change of resin cements.

2. GENERAL INFORMATION

2.1. Luting Cements

The word “luting” is used to describe an adaptable material to attach two components together. ‘Luting cements’ are used in dental practice primarily to hold or seal restorations and prosthetic appliances (crowns, bridges, posts, pin restorations, inlays & onlays) in a fixed position in the mouth as well as direct restorations, cavity lining, orthodontic, surgical, and periodontal procedures (27).

Conventionally, cements constitute an acid–base reaction where a liquid containing the acidic element is mixed with the powder containing the basic element. An ideal luting cement should have physico-mechanical properties which show resemblance to those of dentine. Practically, these cements are expected to maintain their integrity in the oral environment and tolerate masticatory stresses by having an overall good combination of viscoelastic properties while transmitting the stresses from the fixed-prostheses to the dental structure (27).

There are multiple factors determining the retentivity of fixed prosthesis, starting from fit of the crown and geometry of the abutment preparation to luting cement-related properties which include film-thickness of the luting cement, minimal solubility and erosion, and adhesiveness. These materials used for dental treatments must be biocompatible and need to be applied in a way that provides sufficient marginal seal to reduce plaque accumulation, periodontal diseases, and recurrent caries (27).

Cements containing high levels of sustained fluoride are of a good benefit in reducing the risk of recurrent caries due to their ability to release fluoride over a prolonged period. Together with the physico-chemical properties, the handling and manipulative characteristics of the luting cements are the factors influencing the clinician in deciding the most appropriate cement for a particular application. For instance, delivery system, mixing properties, working time, setting time and ease of clean-up are some of the handling and manipulative characteristics (27).

The two- paste systems that usually compose the luting cements are to be mixed to achieve the chemical reaction which results in the setting of the luting cement. Nonetheless, luting cements were produced as a powder–liquid formulation previously. Even though many of the modern materials depend on the acid-base reaction, the resin-based luting cements significantly differ in terms of cement-forming reaction (27).

2.1.1. Classification of Luting Cements

2.1.1.1. Zinc Phosphate Cements

The oldest luting cement, zinc phosphate cement, has been used for crown and bridge cementations since 1879, and has encountered many alterations in formulation and compounding (27). It is a mixture of zinc oxide powder; consisting of amorphous zinc oxide as the major component with minor amounts of magnesium and bismuth oxides added to facilitate the calcining process, and phosphoric acid liquid; consisting of an aqueous mixture of orthophosphoric acid with minor percentages of aluminum and zinc phosphates which serve as a buffer to reduce the reactivity of the free acid (28).

These cements are indicated in all-metal and metal-supported restorations, where according to literature, they achieved impressive mechanical retention and success for a long period of time. Yet, due to their poor compressive and flexural strength; they cannot be used in cementing ceramic and composite crowns (28).

The solubility of zinc phosphate cement in oral fluids, low adhesion to dentine, and post-operative sensitivity due to low pH while setting are major disadvantages of the material which surpass its advantages (28,29). On the other hand, the pH value of the material rises to 5.9 in 24 hours and attains neutral degree (pH 7.0) in 48 hours. (30). Despite their limited usage in dentistry nowadays, they can still be found in the dental market (27).

2.1.1.2. Zinc Polycarboxylate Cements

Zinc polycarboxylate cements have been introduced in 1968 and can be referred to as 'polycarboxylate' or 'polyacrylate' cements. These cements are produced by the reaction of zinc oxide with polycarboxylic (polyalkenoic) acid in water to prevent its dissolvment in oral fluids (27).

Polycarboxylates are also supplied as powder–liquid compositions, similar to zinc phosphate cements. The powder consists of sintered zinc oxide with fine particle sizes, minor amounts of magnesium oxide (1–5%), fumed silica and fluoride salts for the improvement of the mechanical strength and release of fluoride (23). The liquid consists of an aqueous solution of a polycarboxylic acid, usually a homopolymer of polyacrylic acid or a copolymer of acrylic acid with itaconic or maleic acids. Polycarboxylate cements have the ability to bond to enamel and dentine by ionic bond formation through the calcium ions present in the hydroxyapatite mineral (28).

The tensile strength for cement is one third higher than that of zinc phosphate cement. In addition, the compressive strength of polycarboxylate is one half or two thirds higher than that of zinc phosphate cement. The modulus of elasticity is about one third of zinc phosphate, and this provides the material with the ability to display significant amount plastic deformation while loading. These properties result in a material with significant plastic deformation upon loading, therefore, zinc polycarboxylate is not recommended for long-span bridges, or in areas which have to bear high degree of functional stresses (31).

Although its acidity is higher compared to zinc phosphate, the pH degree of polycarboxylate rapidly increases after mixing and this causes very small amount of penetration of organic acid molecules into dentinal tubules. Consequently, the response of the pulp to the material is mild and it has superior biocompatibility. (31).

Despite its greater acidic conditions, compared to zinc phosphate, when mixed, polycarboxylate pH rises rapidly with minimal penetration of the large organic acid molecules into dentin tubules. Therefore, the pulp response is mild, and its biocompatibility is excellent. (31).

Nowadays, polycarboxylate cements are primarily used for cementation of long-term provisional restorations, cast alloy and metal-supported inlays, onlays and crowns/ short-span bridges (27).

2.1.1.3. Conventional Glass-Ionomer Cements (GICs)

Glass-ionomer cement has been introduced to the dental market in 1969. Today, the major constituents of glass ionomer cements are polycarboxylic acid, fluoroaluminosilicate glass, water, and tartaric acid. The conventional glass-ionomer cements combine the favorable properties of both zinc phosphate and zinc polycarboxylate cements. As a result, the fluoride-release property is preserved, and the phosphoric acid and its salts are replaced with polymeric carboxylic acids of the zinc polycarboxylate cements to prevent their susceptibility to dissolution (27).

Glass-ionomer cement provides slightly better antibacterial properties, higher compressive strength, and crown retention compared to zinc phosphate. It also allows for working time that is shorter when compared with zinc phosphate or polycarboxylate (~2–3 1/2 min.) (31). These cements provide chemical adhesion to some degree owing to their polycarboxylic acid component (the liquid). They also have the ability to release fluoride

from the fluoroaluminosilicate glass component (the powder). The utilization of this material is especially advantageous for patients with high degree of caries risk (28, 31).

Glass-ionomer cement demonstrates lower modulus of elasticity compared to zinc phosphate and is therefore not recommended for luting multiple abutments or long-span bridges or in areas subjected to excessive masticatory stress (31). Due to the hygroscopic expansion property of the glass ionomer cements, they are not indicated for luting ceramic crowns as they cause cracks during expansion, however, they can be used to lute metal crowns and metal posts (28). Shelf life may be another concern as it has been reported that viscosity increase after 24 months. (31).

2.1.1.4. Resin-Modified Glass-Ionomer Cements (RMGIs)

RMGIs were developed in the late 1980s and were first introduced in the market as luting cement in 1994 (27). This type of cements is less technique-sensitive and maintain favorable physico-mechanical properties in comparison with conventional glass ionomer cements, yet they have similar levels of fluoride release (32,33,34). They provide the ability to remove excess cements easily, demonstrate minimal postoperative sensitivity (35) and exhibit good clinical performance and durability. The reason for these favorable properties is because part of the poly acrylic acid liquid is replaced with hydrophilic monomers. This results in higher degree of compressive and tensile strength and lower solubility in oral fluids (28) Compared with GIC, RMGI shows improved mechanical strength but decreased biocompatibility (36).

The resin-modified glass ionomer is a hybrid material consisting of conventional glass ionomer cement and a small amount of light-curing resin. Therefore, it possess properties between the two materials and they have superior characteristics compared to conventional glass ionomer materials. Good adhesion to the dental structure, esthetic advantageous, release of fluoride and hardening speedily by visible light are some of the advantages of resin modified glass ionomer cement (37).

RMGIs are indicated for cementing crown and bridge applications, orthodontic brackets, zirconia, and alumina-based ceramics as well as lithium disilicate pressed and milled (CAD/CAM) inlays and onlays (35). However, due to the hydrophilic nature, they exhibit increased water sorption which, therefore, leads to hygroscopic expansion. For this reason, they are contraindicated for cementing low-strength all-ceramic crowns and veneers because clinical fractures may occur (28).

2.1.1.5. Polyacid-Modified Composite Resins (Compomers)

In the late 1990s, compomers were produced which are also known as polyacid-modified composite resin. They are a combination of composite resin (comp) and glass-ionomer (omer) and offer the advantages of both materials.

The compomers are more similar to composite resins in their physical behavior than glass-ionomer with very limited fluoride recharge compared to that of conventional glass-ionomer. On the other hand, they show higher compressive and flexural strengths compared to RMGI, but lower than unmodified composite and they provide very small amount of tooth adhesion without using a resin bonding agent (31).

2.1.1.6. Resin Cements

Resin cements have been in the market for a long time. Their early materials which were developed in the 1950s were primarily based on acrylic resin chemistry. Since the 1970s the dimethacrylate resin chemistry of resin composites and adhesives based on acid-etching technique and dentine conditioning have been adopted for their production (27, 38). Resin cements are methyl methacrylate, Bis-GMA dimethacrylate, or urethane dimethacrylate based, with fillers of colloidal silica or barium glass ranging between 20% to 80% by weight. They are produced as powder/liquid, encapsulated, or paste/paste systems (31).

Resin bonds to the tooth micromechanically by interlocking into an acid etched enamel surface, and bond to dentin after performing multiple steps which include removal of the smear layer and surface demineralization. Later an unfilled resin bonding agent or primer is applied for the resin to form a chemical bond. The retentive properties of resin cements are higher compared to conventional luting cements, so they are utilized for the provision of bonding to substrates such as dentine and enamel, porcelain and other ceramics, gold and other metal alloys, and indirect resin composites (28,31,38,39,40). Since esthetics and strength are important properties for all-ceramic restorations, resin cements serve as good options for luting all-ceramic restorations due to their high esthetic properties and fracture resistance (9,16,31,39).

The clinical performance of the resin cements is directly affected by multiple physical and mechanical properties as follows: Compressive strength, flexural strength, film thickness, solubility, and water sorption. High compressive strength is an essential property of the resin cement to compensate masticatory forces in the mouth. This property

also enhances the fracture resistance of the restoration when the resin cement bonds to both the tooth structure and the restoration (28).

In addition, adequate flexural strength helps the resin cement in transferring the stresses between the tooth and restoration without causing any fracture, thus shows a protective effect on the fragile restorative material. Flexural strength can be described as very important property of the material to endure bending forces without fracture (28).

Luting cements are expected to possess adequate film thickness because it causes an improved seating of the restoration and decreases marginal gap formation. As a result, plaque accumulation, periodontal disease, cement dissolution, and secondary caries formation are significantly reduced. It has been reported that high film thickness may hamper a suitable seating of the restoration and lowers the tensile strength of cast restorations (28)

Moreover, the flexural strength of resin cements is decreased by the absorption of water, there is higher amount of decrease in flexural strength as the thickness of the cement increases. This prevents the cement to distribute masticatory forces between the tooth and the restoration. Therefore, resin cements should be used in thin layers to reduce the plasticizing phenomenon (28).

When compared to the other luting agents, resin cements have equal or higher compressive and tensile strength, toughness, and resilience. On the other hand, resin cements do not offer fluoride release or uptake and their solubility is exceptionally low. Pulpal compatibility may be a concern to due its high film thickness, specifically in deep restorations. Furthermore, there may be complete destruction of the tooth during removal of the restoration. The support of brittle multi-unit all-ceramic prosthesis may also be compromised due to the low elastic modulus of the material(stiffness). Depending on the bulk, polymerization shrinkage of the luting resins may produce stresses enough to open small voids at the cement/tooth interface. In addition to good esthetic qualities, resin cements offer the dental practitioner different color choices. It is recommended to use non-eugenol provisional cement when resin is to be used for the definitive restoration, because the residual eugenol released from the provisional cement can adversely affect the setting reaction of the bonding agent. The technical sensitivity of resin is higher compared to conventional cements and they are more costly (up to 175 times the cost of zinc phosphate). The practitioner should meticulously follow all steps of cementation in correct order and with the recommended duration of each step (31,39).

Resin cements are classified according to their polymerization mechanisms as; self-cured, light-cured, and dual-cured (28).

2.1.1.6.A. Self-Cured Resin Cements

Self-cured resin cements have a setting reaction based on chemical reaction initiated by tertiary amine benzoyl peroxide. They are used in areas where it is difficult for the light to reach. Therefore, they are used to lute metal restorations, porcelain-fused-metals, and thick ceramic restorations (28).

2.1.1.6.B. Light-Cured Resin Cements

The setting reaction in the light-cured resin cements takes place due to the presence of camphorquinone. Camphorquinone photo initiator is more color stable than the tertiary amine benzoyl peroxide that is present in both self-cured and dual-cured resins and has the tendency to darken over time. Light-cured resin cements provide advantages such as extended working time, initiation of setting reaction on demand, and better color stability. Their indication is constricted to situations where a shallow preparation space is to be restored by veneers or inlays, keeping in mind that the thickness of the restoration will not prevent the curing light from passing through the restoration to polymerize the cement (38,41,42).

It is essential to make sure that the applied resin cement is polymerized sufficiently, as it may lead to hygroscopic expansion, marginal gaps, secondary caries, pulpal reactions, decreased hardness and fracture toughness, wear resistance, lower bond strengths and color alterations due to the unreacted camphorquinone photo initiator. Nowadays some resin cements utilize a different type of photo initiator rather than tertiary amines for the prevention of color shift (28).

2.1.1.6.C. Dual-Cured Resin Cements

Dual-cured resin cements are generally produced as two-paste systems. They usually have a setting reaction that is based on redox reaction of benzoyl peroxide with aromatic tertiary amines. Also, at least one of the pastes contains the light-activated portion (camphorquinone) which initiates the light-cure setting mechanism (photoinitiation). However, it has been observed that tertiary amines and benzoyl peroxide have the tendency to darken over time, therefore, some types of resin cements have been

manufactured which do not contain a tertiary amine photo initiator. Consequently, discoloration is prevented, and color stability is improved (13,38,41,42).

Dual-cured resin cements are indicated when delivering restorations including crowns, porcelain inlays and onlays, all-ceramic or pre-cured composite restorations as well as cementing metal or porcelain-fused-metal restorations where material thickness and components may obstruct the transmission of sufficient light energy to the cement. Under these circumstances, light intensity reaching the cement may be adequate to initiate the light-activated polymerization process. However, a self-polymerized catalyst is required to ensure maximal cure and this property helps to control working time by the ratio between inhibitors of the self-curing reaction and the quantity of peroxide and aromatic tertiary amines (27,38).

Dual-cured resin cements can also be used in cementing restorations on abutments with minimal coronal tooth structure due to their high bond strength and in cementing endodontic posts, amalgam restorations and orthodontic appliances. Considering the absence of inherent adhesion between conventional resin cements and dental tissue, it is essential to provide sufficient bond strength of the luting cement to the tooth structure by using dental bonding agents. In addition, several commercial products recommend the use of an etchant, such as 37% phosphoric acid gel. It is also important to prepare the surface of the prosthetic restoration by sand-blasting or chemical treatments, e.g., with a silane primer. Dual-cured resin cements are sensitive to moisture, have inherent polymerization shrinkage and require multiple steps to perform the right cementation procedure and obtain the desired results (27).

Laboratory studies have been conducted to measure the bond strength of different restorations cemented to dentine with resin luting cements, and it has been reported that the highest values were observed when resin cements were used. These cements do not have an anticariogenic effect since they do not contain intrinsic fluoride release (27).

2.2. Color

To make successful esthetic restorations, imitating the color and appearance of adjacent tooth perfectly is needed, and to manage this, the knowledge of color and light is essential (43).

Color is a sense which is created by visual perception of light reflected, transmitted, or emitted from various objects. In other words, color is an impression left

in our visual sense by light which either comes directly from a light source or comes to the objects first and is reflected or transmitted to our eye then (43) (Figure 1).

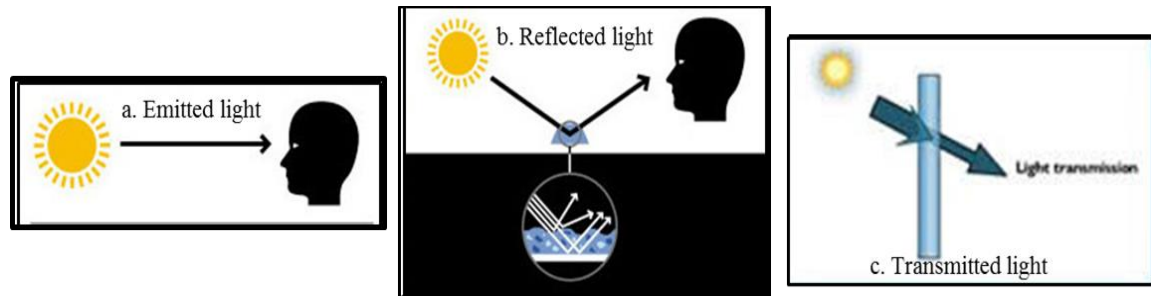


Figure 1. a. Emitted light, b. Reflected light, c. Transmitted light

Light is only a part of diverse electromagnetic waves available in the space (44). The electromagnetic spectrum covers an extremely broad field, ranging between radio waves having wavelengths of several thousand kilometers to gamma rays having wavelengths of 10^{-13} m and shorter. A very small part of this range is considered the visible light region; from approximately 380 to 780 nm (1 nm is one billionth of 1 m). Only the energy which is transmitted by waves which have wavelengths changing between 380 and 780 nm can stimulate the receptors in retina and generate color sense. The reflected light that is recognized by the eye as color is (except man-made monochromatic light) a mixture of light at various wavelengths within the visible region (38,44,45,46,47).

In 1666 Isaac Newton found that a beam of white light could be separated into component colors, or wavelengths, by passing it through a prism. After passing through a prism, the white light is refracted and divided into seven colors of rainbow. This color distribution is called a spectrum. The reason that the human eye is able to see the spectrum because of the stimulation of the retina in the human eye by the specific wave lengths. The spectrum is arranged in the order red, orange, yellow, green, blue, indigo, and violet according to the different wavelengths of light. The light in the region with the longest wavelengths is perceived as red by the human eye, and the light in the region with the shortest wavelengths is perceived as violet. The light region which the human eye can see is called the visible light region. Movement beyond the visible light region toward longer

wavelengths causes us to enter the infrared region whereas movement toward shorter wavelengths results in entering the ultraviolet region. Both of these regions are invisible to the human eye (47,48) (Figure 2).

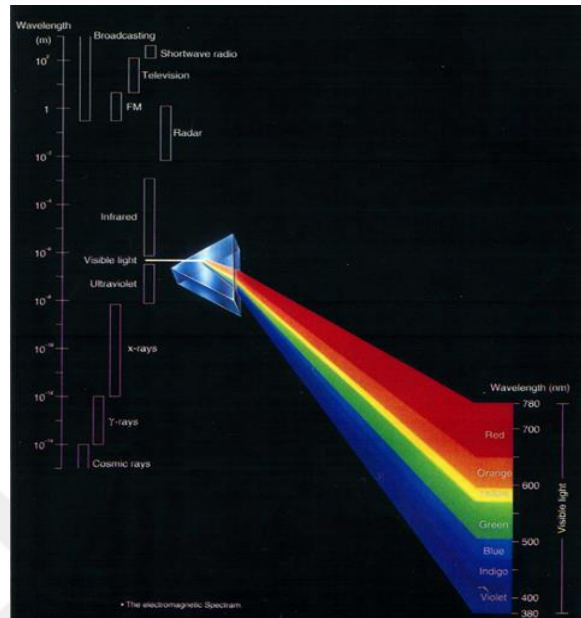


Figure 2. The electromagnetic spectrum.

The observation of Newton was simple: White light contains all colors. If an object appears to be of a specific color to the human eye it means that light that reaches our eye when viewing the object has been changed by the object itself. In other words, the interaction of the light with the object is what allows the perception of color (47,48).

2.2.1. Color Perception

The basic process of color perception can be explained as follows:

After light is emitted from a source it may directly reach our eye or it may strike or pass through an object. Some of the light is absorbed by the object after interaction with an object. Receptors as in retina perceive wavelengths that are not absorbed (i.e., those that are reflected or transmitted to the eye). These signals are transmitted to the brain through optical nerve (44,45,48,49).

The rods provide our vision when illumination levels are low. They are helpful for night vision, peripheral vision, and motion changes, while the cones help central vision and color. At higher levels of illumination, color vision is mediated by the responses of the cones (45). There are three types of cones, each of which contains a

distinctive a specific type of pigment; one cone is able to absorb longer wavelengths (red light), another middle wavelength (green light), and the third type shorter wavelengths (blue light). A given color is able to stimulate all three types of receptors with different effectiveness, and the pattern of the responses is responsible of color perceived. In 1986 researchers identified the genes that correspond to the red, green, and blue pigments (48).

2.2.1.1. Emission

Emission of light from a source occurs through a chemical or physical process. Every process releases higher amount of light at certain wavelengths compared to others. For the creation of perfectly white light, the light source would have to emit the identical amount of each wavelength. Since no light source can emit perfectly white light because of the difficulty of providing exactly the same amount of each wavelength, color perception is affected. Color perception is affected, because there are only certain wavelengths (colors) to interact with an object which explains why the same object will appear to be different colors when viewed using different light sources (48).

2.2.1.2. Reflection and Absorption

Light reflection occurs when light rays hit a solid object, such as an apple and then bounce off of it. The molecular structure or density of the object or medium determines whether certain wavelengths (colors) will be absorbed rather than reflected. The wavelengths that are reflected compose the color perceived. For example, an object that reflects red wavelengths and absorbs the rest of wavelengths is perceived as red (Figure 3a). Theoretically, an object that reflects all light would be perceived as white, and an object that absorbs all light would be perceived as black (48,49) (Figure 3b and Figure 3c).

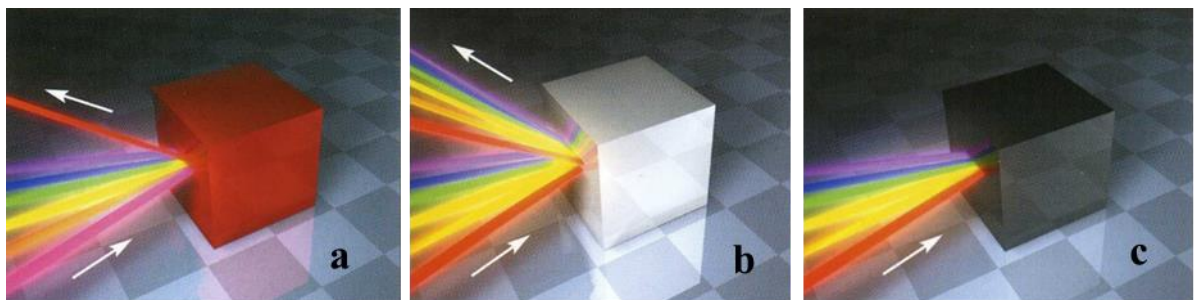


Figure 3. a. Red object reflects only red wavelengths, b. White object reflects all light, c. Black object absorbs all light

Light is absorbed and reflected by each object from different parts of the spectrum with different amounts. These differences in absorptance and reflectance are responsible of the varying colors displayed by different objects. A spectrophotometer is a device which can measure the light reflected from an object at each wavelength (46). This data can be displayed on a graph which is called spectral reflectance graph (45). When we look at the spectral reflectance graph of an apple, it is observed that in the red wavelength region the reflectance (the amount of reflected light) is high, whereas in other wavelength regions the reflectance is low. The apple reflects light mostly in the red wavelength region, and a small amount in the orange and yellow wavelength regions and absorbs light in the green, blue, indigo, and violet wavelength regions (Figure 4). On the other hand, when we look at the spectral reflectance graph of a lemon, we observe that in the red, orange, yellow and green wavelength regions reflectance is high, however, in the indigo and violet wavelength regions reflectance is low. The lemon reflects light in the red, orange, yellow and green wavelength regions. On the other hand, it absorbs light in the and violet wavelength regions (47,50) (Figure 5).

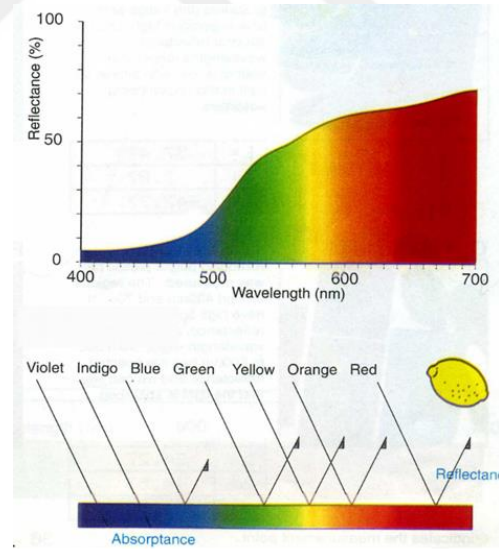
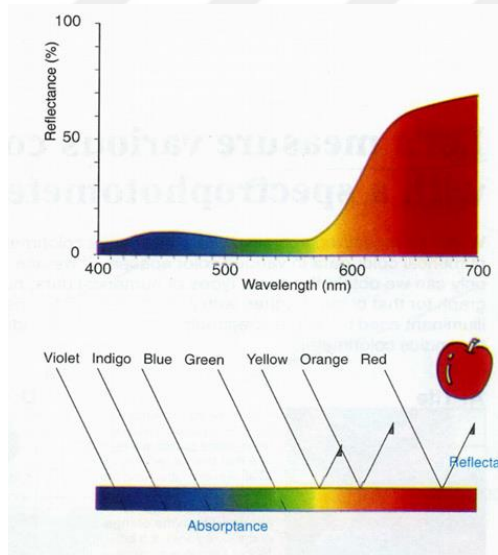


Figure 4. Spectral reflectance graph for an apple Figure 5. Spectral reflectance graph for a lemon

2.2.1.3. Transmission and Absorption

Transmission occurs when light passes through a transparent or translucent material. If the light meets molecules or larger particles within the material, some wavelengths will be absorbed.

The density and the structure of the material through which light passes determine the number of absorptions of light wave and specific wavelengths. The transmitted wavelengths form the color which is perceived by the human eye. For example, if there is an apple picture in the slide, all the wavelengths except red will be absorbed and only the red wavelength (color) will be transmitted, and the color of apple will be perceived red. Such a material which allows part of the light to pass through is regarded as translucent. If all the light is allowed to pass through by the material, then it is called transparent, when all light is transmitted, white color is perceived. The opposite side of transparency is opacity. If the material is completely opaque, all light is absorbed, and the color black is perceived. Clinically, incisal edges of natural teeth are translucent and to achieve success esthetically, it is very important to determine the amount of translucency correctly (48).

2.2.1.4. The Factors Affecting Perception of Color

The appearance of color is dependent on different factors:

Observer Differences: The sensitivity of eye differs from person to person. A person's eyesight generally changes with age. Aging has harmful effects on color perception because the cornea and lens change into a yellowish color with aging. This makes it very difficult for the individual to distinguish between white and yellow (48).

Eyestrain is another factor affecting color perception. It is not possible for weary eyes to have the same perception of colors as alert eyes (48).

Nutrition of individuals differs from person to person. Food supplement intake such as vitamins C and E, nutrients such as zinc and lutein have been shown to have positive effects on eyes while there is an association between macular degeneration and a high consumption of food which is rich in saturated fat (48).

Disorders such as color blindness and binocular difference, some medications such as alcohol, morphine, caffeine, Viagra, and oral contraceptives also affect perceiving color (48).

The emotional status of the individual may influence pupillary diameter and may cause dilation or constriction; this directly affect the discrimination of the color (48).

Size Differences: Colors which are dispersed in a larger area appear brighter and more vivid compared to those which are confined to a smaller area, this is called area effect (47).

Background Differences: If an object is situated in front of a bright background, it will appear duller to a dark background. This is called contrast effect and adversely affects the correct judgment of color (47).

Directional Differences: Specific coloring materials such as metallic paints have highly directional properties. An accurate color communication can be achieved if the angle of viewing the object and the angle of illumination are constant (47).

Light Intensity: At low levels of light, the rods of the eye are more dominant compared to cones; therefore, there is a loss of color perception. To perform the most accurate color reading the light intensity must be 150-200 foot-candles, as verified by a light meter. A color change is observed as the intensity of brightness increases (48).

Light Source Differences: Emission of light occurs through a chemical or physical process. Every light source releases more light at certain wavelengths compared to others. To create perfectly white light, emission of the identical amount of each wavelength is essential. Since no light source can emit perfectly white light because of the difficulty of providing exactly the same amount of each wavelength, color perception is affected. Color perception is affected, because there are only certain wavelengths (colors) to interact with an object which explains why the same object will appear to be different colors when viewed using different light sources (47).

2.2.2. Color spaces¹

One of the major problems that are experienced occurs when colors are attempted to be communicated to other individuals. Color scales have been produced to solve this problem (50).

2.2.2.1 Munsell Color System:

In 1905 the American artist A.H. Munsell developed a method for the expression of colors. This system was used in the past for the quantification of color and still remains

¹Color space: Method of expressing the color of an object or light source using some kind of notation, such as numbers

to be a popular method for the visual description of color. The three parameters of color in this system are **hue**, **chroma** and **value** (49, 50, 51) (Figure 6).

Hue describes the dominant color of an object, such as red, green, or blue. This description of color refers to the dominant wavelengths which are present (44).

Chroma is defined as the intensity of the hue. It represents the degree of saturation of a particular hue (51). The more wavelengths of a particular color that are reflected (relative to all other color wavelengths), the higher the chroma of that hue will be (i.e., the color is deeper and purer). When saturation of the color increases it becomes more vivid. When saturation of the color decreases, the color comes closer to gray and if the saturation is zero, the color will be gray. White, black, and gray tones between black and white are called achromatic colors. They do not have color characteristics and saturation (chroma) (47).

Value can be described as the relative lightness or darkness of a color or the **brightness** of an object. The value increases with total amount of reflected light. Teeth and other objects can be divided into lighter shades (higher value) and darker shades (lower value) (47, 48, 51).

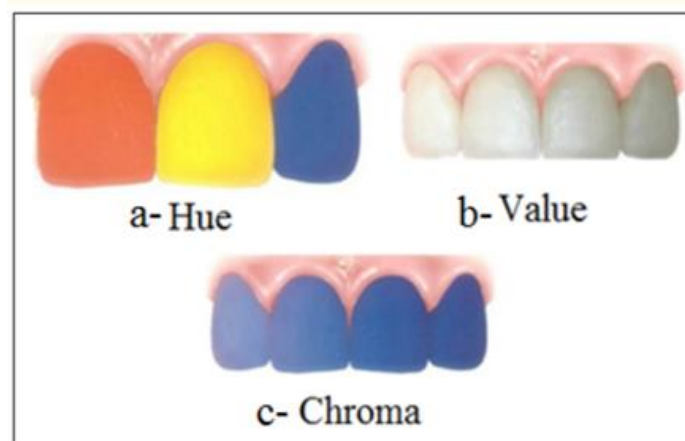


Figure 6. The three parameters of color in Munsell color system.

When the two ends of rainbow colors are connected, a color wheel is obtained. In the cross section of the color wheel, the chroma of red and green colors increases as you move away from the center horizontally. Colors appear dull near the center and become gray at the center. The value of colors increases toward the top where they become white.

In reverse, they tend to decrease toward the bottom where they have a black color at the end (Figure 7).

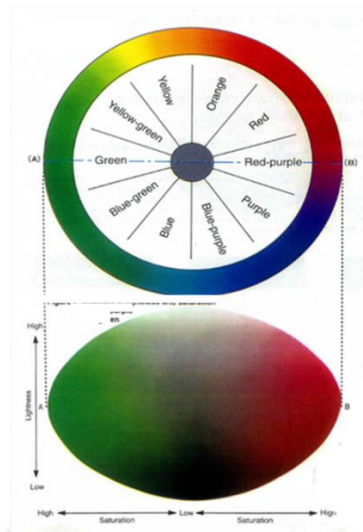


Figure 7. Changes in lightness and saturation for red-purple and green

Figure 8 shows the parameters of color on the three-dimensional color diagram. In the Munsell color system Hues are arranged around the wheel and the intensity of Chroma of a particular Hue gets more intense on the outer rim compared to the hub of the wheel (Figure 9 and 10).

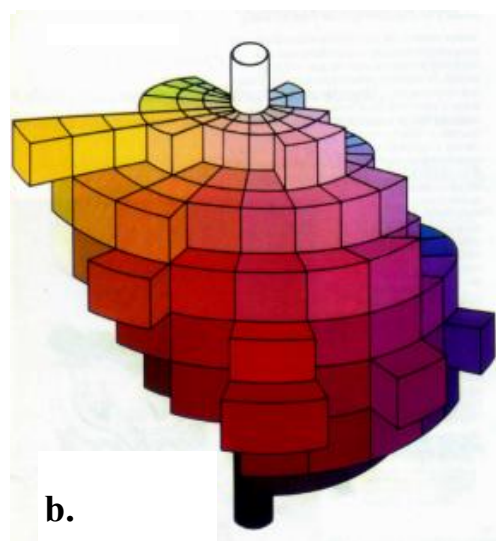
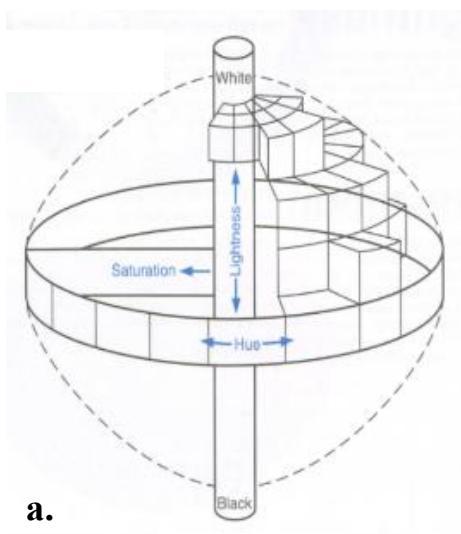


Figure 8. a. and b. The three dimensions of color space.

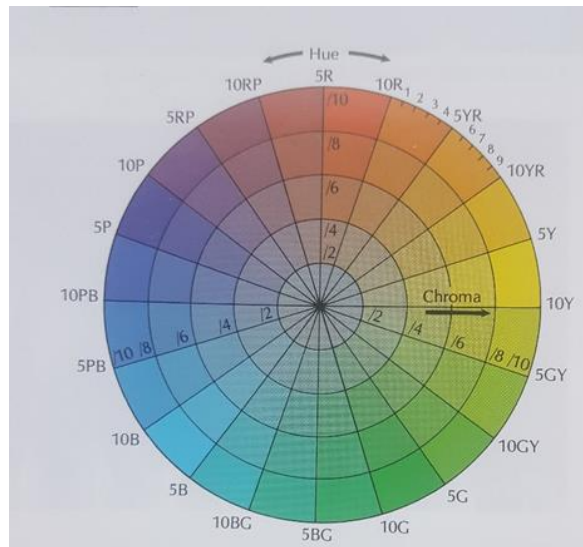


Figure 9. Arrangement of Hue and Chroma in the Munsell system. R, red; YR, yellow-red; Y, yellow; GY, green-yellow; G, green; BG, blue-green; B, blue; PB purple-blue; P, purple; RP, red-purple.

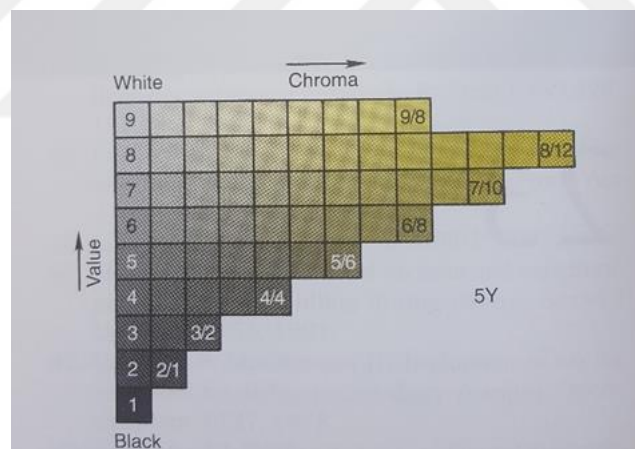


Figure 10. Arrangement of Value and Chroma in the Munsell system.

2.2.2.2. CIE Color Spaces

The CIE (Commission Internationale de l'Éclairage = International Commission on Illumination, usually abbreviated **CIE** for its French name) is an organization which provides standardization in areas such as color and appearance. In 1931, the commission defined a Standard Light Source, developed a Standard Observer and defined the Yxy

color space based on the tristimulus values XYZ, which is representative of the way the human visual system reacts to a given color. In 1976, the CIE also defined a color space, CIE Lab, which is supportive of the color perception theory based on three separate color receptors (red, green, and blue) in the eye. This is currently considered as one of the most popular color spaces and is used as a means of color communication in the world (45,50).

2.2.2.2.A. CIE XYZ Tristimulus Values and CIE Yxy Color Space

As indicated previously there are four types of cone cells in the human eye which are stimulated by different wavelengths of light, this results in the perception of color by the individual. The quantification of colors can be made by the amount of stimulation they make on the cones. These cone cells affect human color perception during medium or high brightness. On the other hand, there is a decrease in color vision in very dim light, which causes the low brightness, monochromatic "night vision" receptors, "rod cells" to become effective. Three parameters which correspond to levels of stimulus of the three kinds of cone cells are called the LMS tristimulus values. LMS (long, medium, short), is a color space representing the response of the three types of cones of the human eye, named for their responsivity (sensitivity) peaks at long (560 nm – 580 nm), medium (530 nm – 540 nm), and short (420 nm – 440 nm) wavelengths. However, the wavelength ranges overlap, which complicates calculations of LMS tristimulus values. The International Commission on Illumination (CIE) developed a new set of values, called XYZ tristimulus values, which are equivalent to the LMS tristimulus values but do not overlap. In mathematical terms, the LMS values were transformed into XYZ values that are linearly independent from each other. Y represents the radiance/luminance, Z represents mostly blue wavelengths, and X represents the remaining wavelengths. The numerical range is not indicated, except that the lower end is generally bounded by zero (Figure 11). The LMS color space is commonly used when conducting chromatic adaptation (estimating the appearance of a sample under a different illuminant). It is also helpful in the studying color blindness, when there is a defect in one or more cone types (45, 47).

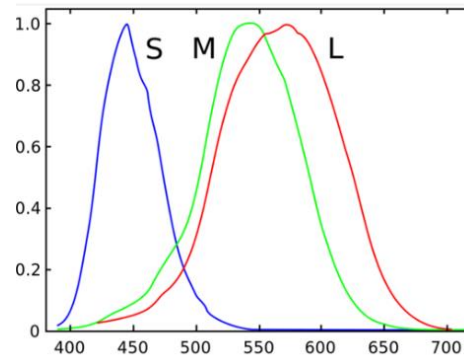


Figure 11. The normalized spectral sensitivity of human cone cells of short, middle, and long wavelength types.

Because of the distribution of the cones, the observer field of view determine the tristimulus values. To exclude this variable, the CIE defined a color-mapping function called the **2° Standard Observer**, which represents the chromatic response of an average human being within a 2° arc inside the fovea. This angle was specifically chosen because it was believed that the color-sensitive cones were present within a 2° arc of the fovea (Figure 12). In 1964, the CIE defined an additional standard observer, based upon 10° field of view, which is referred to as the **10° Supplementary Standard Observer** (Figure 12). The following explanation provides an idea of what a 2° field of view is like compared to a 10° field of view: at a viewing distance of 50 cm, a 2° field of view would be a 1.7 cm circle while a 10° field of view at the same distance would be a 8.8 cm circle. The 2° Standard Observer should be used for viewing angles of 1° to 4°; the 10° Supplementary Standard Observer should be used for viewing angles of more than 4° (45, 47).

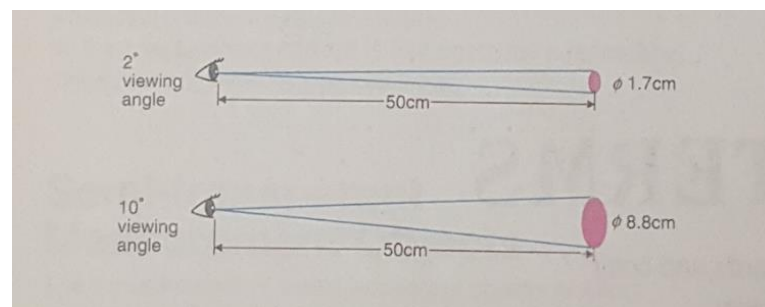


Figure 12. Comparison of 2° and 10° field of view

The standard observer is characterized by **color matching functions** $\bar{x}(\lambda)$, $\bar{y}(\lambda)$ and $\bar{z}(\lambda)$ as shown in Figure 13. The XYZ tristimulus values are obtained by using these Standard Observer color matching functions (45, 47).

The color matching functions can be described as the tristimulus values of spectrum as a function of wavelength. These functions are thought to correspond to the sensitivity of the human eye. Separate sets of three color-matching functions are specified for the 2° Standard Observer and 10° Supplementary Standard Observer (45, 47).

The tristimulus values XYZ are helpful for defining a specific color, however the results are not readily visualized. Therefore, another definition of color space has been made by the CIE in 1931, for graphing color in two dimensions independent of lightness; this is the **Yxy color space**; in which Y corresponds to the lightness (and is identical to tristimulus value Y) and x and y are the chromaticity coordinates which are calculated by using the tristimulus values XYZ. The CIE x, y chromaticity diagram for the color space is shown in Figure 14. In this diagram, achromatic colors are positioned toward the center of the diagram, whereas there is an increase in the chromaticity toward the edges.

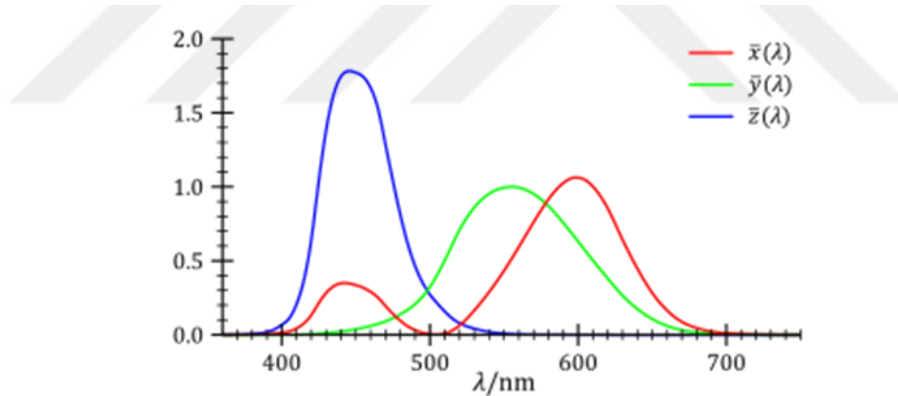


Figure 13. Spectral sensitivity corresponding to the human eye (Color- matching functions of the CIE 1931 Standard Observer)

If the color of apple in Figure 15 is measured using the Yxy color space, the values of $x=0.4832$, $y=0.3045$ are obtained as the chromaticity coordinates, and this corresponds to point A on the diagram in Figure 14. The Y value of 13.37 indicates the reflectance of the apple which is 13.37% (compared to an ideal reflecting diffuser with a reflectance of 100%) (45, 47).

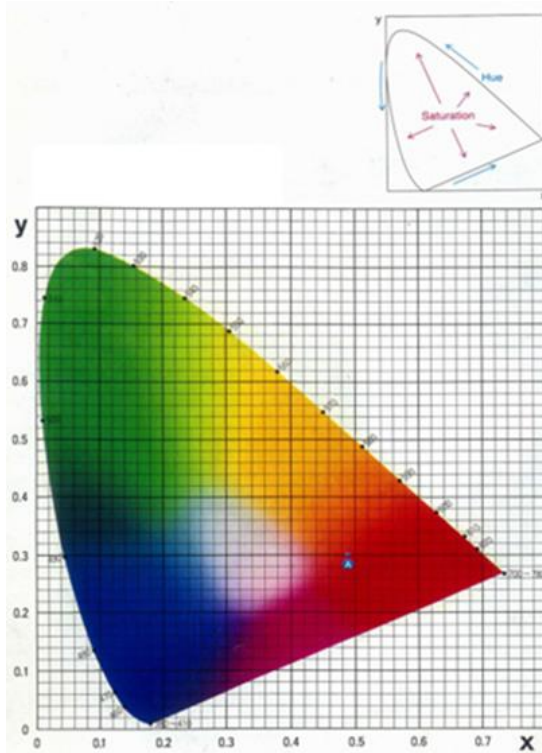


Figure 14. Chromaticity diagram of Yxy color space.

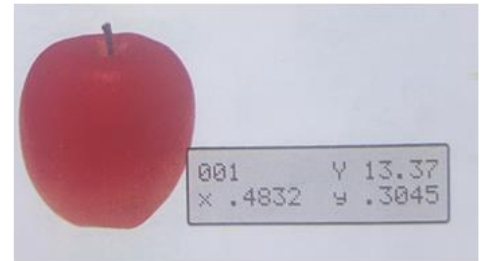


Figure 15. Chromaticity coordinates of the apple using the Yxz color space

2.2.2.2.B. CIE $L^*a^*b^*$ Color Space

The $L^*a^*b^*$ color space (also referred to as CIELAB) is presently one of the most commonly used color spaces for the measurement of object color and is frequently used in all fields (50). It is one of the uniform color spaces defined by CIE in 1976 to reduce one of the major problems associated with the original Yxy color space in which equal distances on the x, y chromaticity diagram does not correspond to equally perceived color differences. CIELAB provides a three-dimensional color space where the a^* - b^* axes form one plane to which the L^* axis orthogonal (45, 47, 51). In this color space, L^* stands for lightness and a^* and b^* refer to chromaticity coordinates (47,50,51) (Figure 16). In this diagram, the a^* and b^* indicate the direction of colors: $+a^*$ is the red direction, $-a^*$ is the green direction, $+b^*$ is the yellow direction and $-b^*$ is the blue direction (47,50).

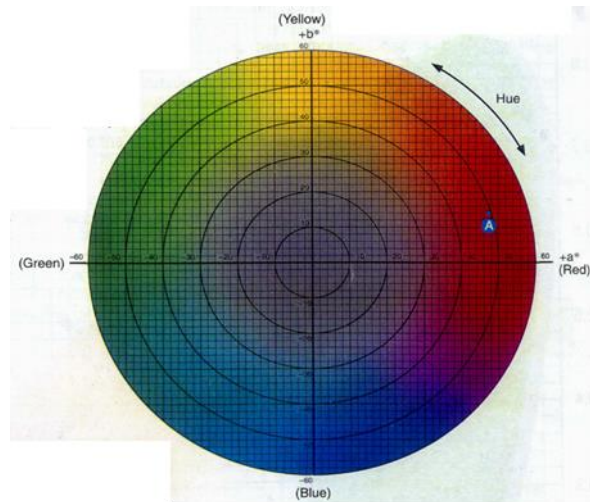


Figure 16. Chromaticity diagram of Yxy color space.

Figure 17 represents the color solid for the $L^*a^*b^*$ color space. Figure 16 is a view of this solid cut horizontally at a constant L^* value. If the color of apple in Figure 18 is measured using the $L^*a^*b^*$ color space, the values of $a^*=+47.63$, $b^*=0.30+14.12$ are obtained as the chromaticity coordinates, which are shown by point A on the diagram in Figure 16. If the color solid of Figure 17 is cut vertically through point A and centered a view of chromaticity versus lightness, part of which is shown in Figure 19 (47).

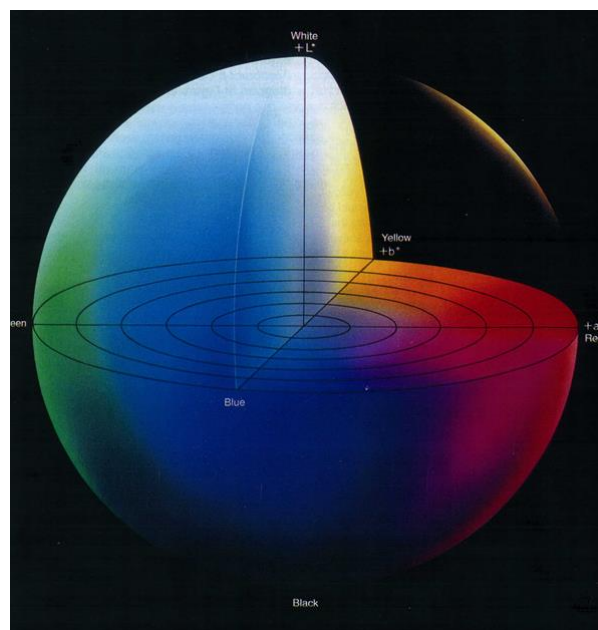


Figure 17. Representation of color solid for $L^*a^*b^*$ color space

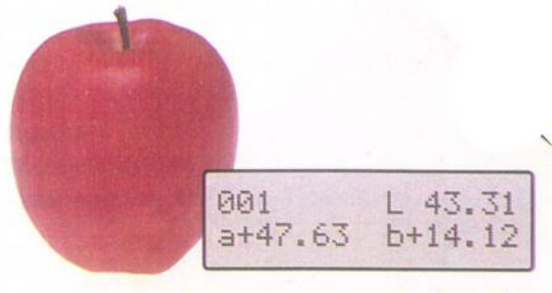


Figure 18. Chromaticity coordinates of the apple using the $L^*a^*b^*$ color space

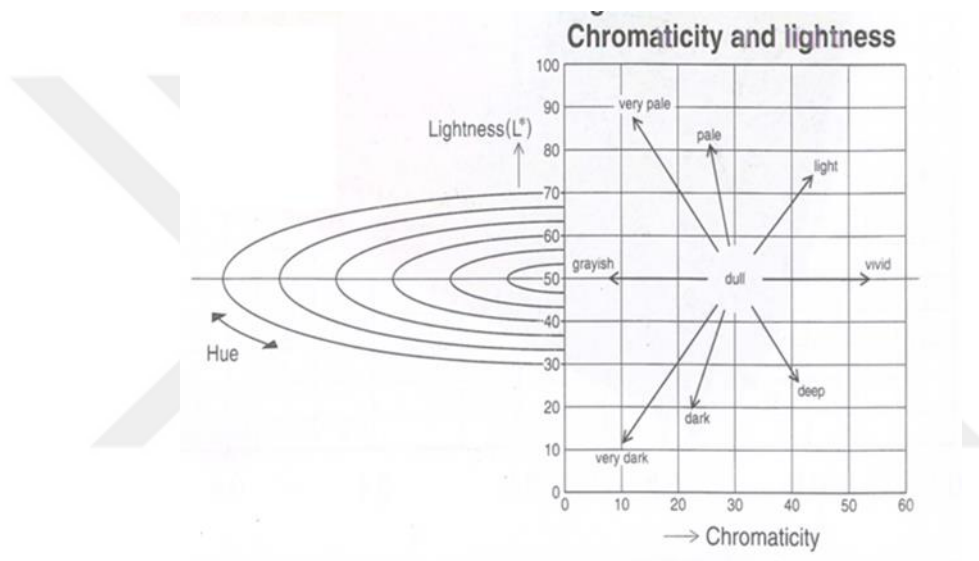


Figure 19. Chromaticity and lightness.

2.2.3 Measurement of Color

Evaluation of color can be made either visually or by using instrumental techniques. Instrumental measurements have the advantage of eliminating subjective interpretation of color comparison. Some devices have been developed for this purpose, such as spectrophotometers or colorimeters.

The human eye is not able to make a precise quantification of color; on the other hand, measurement devices perform a numerical expression of color in accordance with international standards. This way of expressing colors makes it possible to understand the specific color that is being expressed. Color measuring instruments can be categorized as colorimeters, Spectrocolorimeters and spectrophotometers (50,52). Additionally,

spectroradiometers and digital cameras spectroradiometers and digital cameras, can also be used for color evaluation (53).

2.2.3.1 Colorimeters

Colorimeters were the first standardized color measuring devices, and they played a very significant role in the in the development of the science of color which is also referred to as colorimetry. A colorimeter (Figure 20) is a simple device which is developed based upon the visual concepts of color. The sensitivity of colorimeters corresponds to the human eye; however, the measurement conditions will be the same because they always take measurements using the same light source and illumination method (47, 52).

In the most common design, the sample is illuminated at a 45° angle relative to the perpendicular line to the plane of the mounted sample. The reflected light is measured directly perpendicular to the sample through a series of colored filters in order to identify the amounts of red, green, and blue light reflected from the sample. These filters are produced to simulate the three functions x , y , z for the Standard Observer so that the instrument directly measures the three tristimulus values X , Y , Z for a Standard Illuminant. Colorimeters are designed to measure light by using tristimulus method which is identical to the way light is perceived by the human eye (52).



Figure 20. Colorimeter

Colorimeters are easier to produce compared to spectrophotometers, thus they are generally cheaper. They are commonly used as quality control tools for detecting color

difference, shade sorting etc. despite their financial advantages and ease of usage they also possess some disadvantages. Firstly, the colorimeter measures the tristimulus values for one illuminant and one observer. Consequently, it is not possible to identify and quantify illuminant metamerism. Thus, the colorimeter is preferred where the standard and the measured batch are non-metameric such as checking production batches against standard made with the same dyes. Colorimeters are preferred because they are handheld, measurements are quick and easy enough for anyone to use (52).

2.2.3.2 Spectrocolorimeters

A spectrocolorimeter is a hybrid instrument which has the ability to provide colorimetric data such as X, Y, Z or CIE $L^*a^*b^*$ values for various CIE Standard Illuminants. Therefore, they have the ability to provide much better quality control compared to colorimeters. Their price is slightly higher compared to tristimulus colorimeters, but lower than the majority of most spectrophotometers. Spectrocolorimeter can only be used for quality control applications (52).

2.2.3.3. Spectrophotometers

Spectrophotometers (Figure 21) are devices which are different from colorimeters because they measure reflectance, transmittance, or absorbance at various wavelengths in the spectrum. They also measure the amount of light energy which is reflected from an object at 1–25 nm intervals along the visible spectrum.



Figure 21. Spectrophotometer

These instruments perform the measurement of the spectral characteristics of light and then make a calculation of the tristimulus values based on equations for the CIE Standard Observer functions. In addition to showing numerical data in various color spaces, instruments working according to the spectrophotometric method have the ability to display the spectral data directly, revealing more detailed information about the investigated object (47,52).

Using a tristimulus colorimeter, only numerical color data in various color spaces is provided. On the other hand, by using spectrophotometer it is possible to obtain both the numerical data and the spectral reflectance graph. Additionally, using its high precision sensor and the inclusion of data for various illuminant conditions, the spectrophotometer can yield highly accurate results which are much more precise compare to a tristimulus colorimeter (47,52).

Spectrophotometers consists of three parts (54):

1. A lighting system, generally a xenon-flash lamp, or a lamp with a tungsten filament suitably filtered to correct the power imbalance between long and short wavelengths.
2. An optical apparatus for illuminating the surface under examination and collecting the light to the spectrometer-entrance slit, according to the CIE geometrics.
3. One or more spectrometers for spectral measurements. A computer, usually external to the instrument, manages the measurement and processes the raw data, providing the results of the measurement. Two basic optical light geometries (Figure 7) are utilized in reflectance spectrophotometer instruments: (1) illumination at 0 degrees and observation at 45 degrees (0/45), and (2) illumination at 45 degrees and observation at 0 degrees (45/0). Because of the limited access allowed by the oral cavity, only the 45/0 option is suitable clinically (54).

The following comparison can be made between colorimeters and spectrophotometers; colorimeters have comparatively low price, compact size, superior mobility, and simple operation. They are able to identify the tristimulus values easily. However, a colorimeter is not a suitable for complex color analysis such as metamerism and colorant strength. A spectrophotometer, on the other hand, is highly precise and has increased versatility. It is a more suitable instrument for the analysis of complex colors because it has the ability to determine the spectral reflectance at each wavelength. The colorimeter is essentially used in application of production and inspection for color

difference measurements and color chart measurement. On the other hand, the spectrophotometer can be used for high-precision analysis and color management specifically in laboratories and research and development applications (47).

2.2.3.4 Spectroradiometers

A spectroradiometer is a device designed to measure the relative/absolute spectral radiance of a source through optical devices and has inside a spectrometer calibrated on the wavelength scale and the radiometric scale (54).

2.2.3.5. Digital Cameras

Digital photography is one of the newer technologies in dentistry industry as it offers important advantages to the practice. The digital camera (Figure 22) is extremely productive and easy to use. However, basic knowledge of computer technology and photography is needed to use the digital camera efficiently and utilize their benefits to the maximum. Although digital photography and digital imaging are getting popular around the world, their use in dentistry is still limited. Their limitations are usually due to the necessity of providing proper training for the staff members to implement a more cost-effective experience for both the practice and the patient (48).



Figure 22. A digital camera used in dental photography.

Another limitation of digital cameras is the necessity of a gray card and Adobe Photoshop literature-based protocols in order to learn the shade-matching protocol.

However, these are not available for everyday practice. Furthermore, digital camera by itself is not an effective instrument for shade analysis (48).

2.2.4. Illumination

The quality of lighting is a very important factor for correct color perception. Color cannot be accurately perceived or correctly evaluated without adequate illumination (48).

2.2.4.1. Light Sources

The light from a source can be described by the relative power emitted at each wavelength in the visible spectrum. Visible light is commonly described by its **color temperature**. Color temperature is a terminology that is originally in the science of physics. In physics it is known that "black body" will radiate light when heated. The spectrum of the radiated light as well as its color depend on the temperature of the body (45).

It gives dark red color first, when heating is continued it turns to yellow like a bulb filament and if it heated more it becomes bluish white (Figure 7). The colors in this spectrum show illuminations of several light sources. Because of each color generated from a different degree of temperature, it is called **correlated color temperature** (CCT), and its unit is Kelvin. 0°K equals -273°C and known as absolute zero. CCT is an industrial standard which categorizes the color of several illuminants' lightening (45).

Some common examples are as follows:

- 1700 K: Light of matches
- 1850 K: A candle
- 2800 K: Tungsten lamp
- 3350 K: Studio "CP" light
- 3400 K: Studio lamps, photofloods,
- 5000 K: Daylight
- 5500 K: Electronic flash
- 5770 K: Effective sun temperature
- 6420 K: Xenon arc lamp
- 6500 K: Daylight
- 9300 K: TV screen (analog)
- 28000 - 30000 K: A lightning bolt

The CIE has defined various **standard illuminants** with different types of white light. Each of these is known by a letter or by a letter-number combination. These can be used as standard light sources both in calculations of colorimetry and in experimental measurements, using real light sources which will approximate those standard illuminants (48).

2.2.5. Metamerism

The colors of two objects can appear the under daylight whereas they may be different under indoor lighting. This phenomenon is called metamerism and describes the condition where colors may appear different under varying light sources. In metameric objects, the spectral reflectance characteristics of colors of the two objects are different but resulting tristimulus values are the same under one light source and different from each other under another. This problem often occurs due to the utilization of different pigments or materials (48, 51).

2.2.6. Shade Matching

Shade matching is done to record the color and translucency information of the tooth adjacent to the tooth to be restored. This can be performed through either visual or instrumental shade matching techniques (48, 49, 51).

2.2.6.1. Visual Shade Matching Technique

Visual color assessment is generally the most frequently performed method in dentistry (46). It is a subjective method, which is performed by comparing a patient's tooth with a color standard that is manufactured commercially as shade guides (Figure 23), in order to decide the standard color that matches the color of the tooth. However, since visual color assessment may depend on the observer's psychological condition, factors such as fatigue, aging and emotions may influence the results (55, 56).

Physical conditions of the environment such as lighting conditions, and illuminants' position are also factors affecting proper shade matching (55, 56). Besides, shade guide has some important limitations. For example, the range of available color shades is not adequate and does not include the entire color space of natural tooth color; the shades are not systematic in their color space; there are discrepancies in the color of similar nominal shades of different producers; there is no proper control of various batches of one shade guide produced by the same manufacturer; different

reflection curves and surface textures are present due to the gloss with regard to natural teeth and may cause confusions. There is a lack of agreement among and within dentists in color match; the results cannot be transformed into the CIE Lab color scale; and none of the commercially available shade guides are identical (50). In spite of these factors, the shade guides are quick, practical, and economic method for measuring tooth color.



Figure 23. Shade guide

To allow for precise communication of color differences in terms of magnitude and the nature of the difference, more scientific and consistent aids of shade matching are needed in dentistry. Hence, spectrophotometers and colorimeters have been introduced, however, they have been primarily used in research and not in clinical practice. Nevertheless, mostly with modification there are attempts to use these instruments to overcome problems with visual matching clinically (52,53).

2.2.6.2. Instrumental Shade Matching Technique

Various clinical color measuring devices are available. They range from simple to complicated in terms of capabilities and prices to match. There are generally three types of devices: colorimeters, spectrophotometers or digital color analyzers (RGB devices) with various measuring geometries (48, 50, 51).

In vitro testing of some of these devices with various shade tabs have shown them to have reliability of approximately 90%, whereas their accuracy is approximately 70% to 80%. Initial clinical testing of some of these instruments shows similar clinical outcomes for visual matching (51). Digital cameras can also be utilized specially to communicate with the laboratory (48).

3. MATERIALS AND METHODS

This study was carried out in the Hard Tissue Laboratory at the Faculty of Dentistry, Yeditepe University, Turkey.

3.1. Preparation of the Resin Cement Specimens

Four commercially available adhesive resin cements, including 2 light-cured adhesive resin cements (Rely-X veneer and NX-3 Light-Cure) and 2 dual-cured adhesive resin cements (Rely-X Ultimate Clicker, Kerr NX-3 Dual-Cure) were tested (Figure 24). The tested resin cements are listed in Table 1.

For each resin cement, 50 disc-shaped specimens having 10 mm diameter and 0.5 mm height were prepared by using customized Plexiglas molds. The samples were first made a little thicker and then reduced to 0.5 mm standard thickness by grinding (section 3.2). The resin cements were injected into the molds that have been placed on a glass slab and covered with a microscope slide glass with Mylar strips in between. Finger pressure was applied to the slide glass to remove excess material and prevent air-bubble formation. The specimens were then light-cured with a Light-Emitting Diode light unit (LED.B, Woodpecker, Germany) with an intensity of 1000mW/cm² -1700mW/cm² for 40 seconds. The instruments used to prepare specimens and the LED light unit are illustrated in (Figure 25-a and 25-b).



Figure 24. Tested resin cement materials.

Table 1: Resin cement materials tested in the study.

Material	Code	Polymerization System	Shade	Composition	Lot No.	Manufacturer
Rely-X Veneer	3M.LC	Light-Cured	A1	BisGMA and TEGDMA polymer, dimethacrylate polymer, Zirconia/silica and fumed silica fillers, pigments, polymer. Zirconia/silica and fumed silica fillers are used to impart radiopacity, wear resistance and physical strength.	NA19280	3M ESPE, USA
Rely-X Ultimate Clicker	3M.DC	Dual-Cured	A1	Base paste: Meth-acrylate monomers, radiopaque, silanated fillers, Initiator components, Stabilizers, Rheological additives. Catalyst Paste: Methacrylate monomers, Radiopaque alkaline (basic) fillers, Initiator components, Stabilizers, Pigments, Rheological additives, Fluorescence dye, Dual-cure activator for Single Bond Universal Adhesive	5201468	3M ESPE, USA
NX-3 Nexus Light-Cure	K.LC	Light-Cured	White	Bis-GMA, TEGDMA, HEMA, EBPADMA, UDMA EDMAB, Fumed silica, Barium Glass, Ytterbium fluoride, Pigments.	6956899	Kerr, Canada
NX-3 Nexus Dual-Cure	K.DC	Dual-Cured	White	Base; Bis-GMA, TEGDMA, HEMA, EBPADMA, UDMA, EDMAB, PTU, Surfactant, Fumed silica, Barium Glass, ytterbium fluoride, Pigments. Catalyst; Bis-GMA, TEGDMA, HEMA, EBPADMA, UDMA, Fumed silica, Barium Glass, ytterbium fluoride, Pigments, GPDM, BHT, Peroxide.	6923633	Kerr, Canada

BISGMA: Bisphenol-A-diglycidyl ether dimethacrylate; TEGDMA: triethylene glycol dimethacrylate; HEMA: hydroxyethyl methacrylate; PTU: pyridyl thiourea; EDMAB: Ethyl 4-Dimethylaminobenzoate; EBPADMA:Ethoxylated Bisphenol A Dimethacrylate; GPDM: GlyceroPhosphoric acid Dimethacrylate; BHT: 2,6-Di-tert-butyl-4-methyl-phenol.

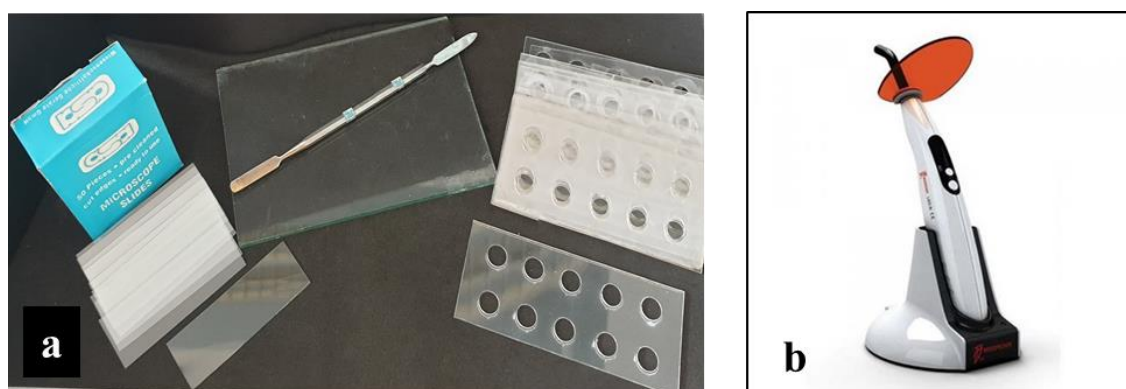


Figure 25-a. Instruments used for preparing the specimens, b. The LED light unit used for polymerizing the specimens.

3.1.1. Application of Rely-X Veneer (3M.LC) Resin Cement: Resin cement was injected into the molds. Afterwards, finger pressure was applied with microscope slide glass on both sides and Mylar strips in between to remove the excess material and minimize air-bubble formation. Later, the resin cement was polymerized for 40 seconds.

3.1.2. Application of Rely-X Ultimate Clicker (3M.DC) Resin Cement: According to the manufacturer instructions, Single Bond Universal Adhesive was applied on the mold and air was applied for 5 seconds without curing the Single Bond Universal Adhesive (Figure 26). Rely-X Ultimate cement catalyst and base pastes were mixed on a mixing pad; the resultant resin cement was injected into the molds. Afterwards, finger pressure was applied with microscope slide glass on both sides and Mylar strips in between to remove the excess material and minimize air-bubble formation (Figure 27-a-d). Later, the resin cement was polymerized for 40 seconds (Figure 27-e).

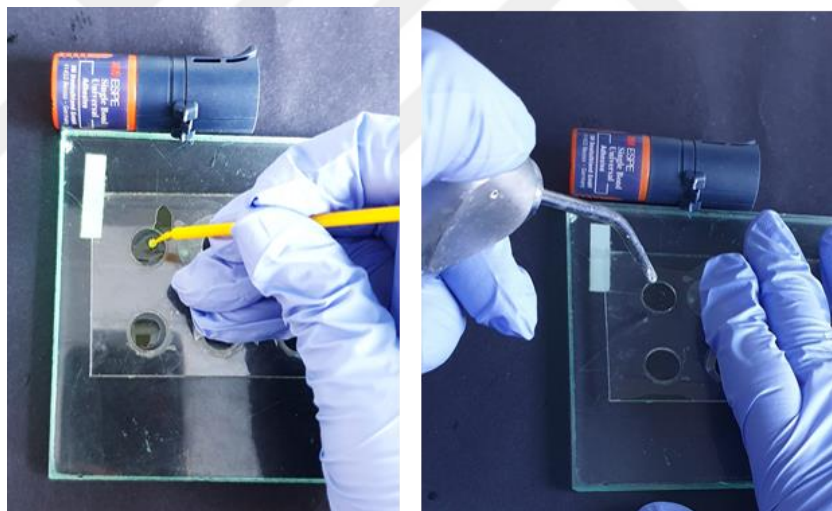


Figure 26. Application of Single Bond Universal Adhesive

3.1.3. Application of NX-3 Light-Cured (K.LC) Resin Cement: Resin cement was injected into the molds. Afterwards, finger pressure was applied with microscope slide glass on both sides and Mylar strips in between to remove the excess material and minimize air-bubble formation. Later, the resin cement was polymerized for 40 seconds.

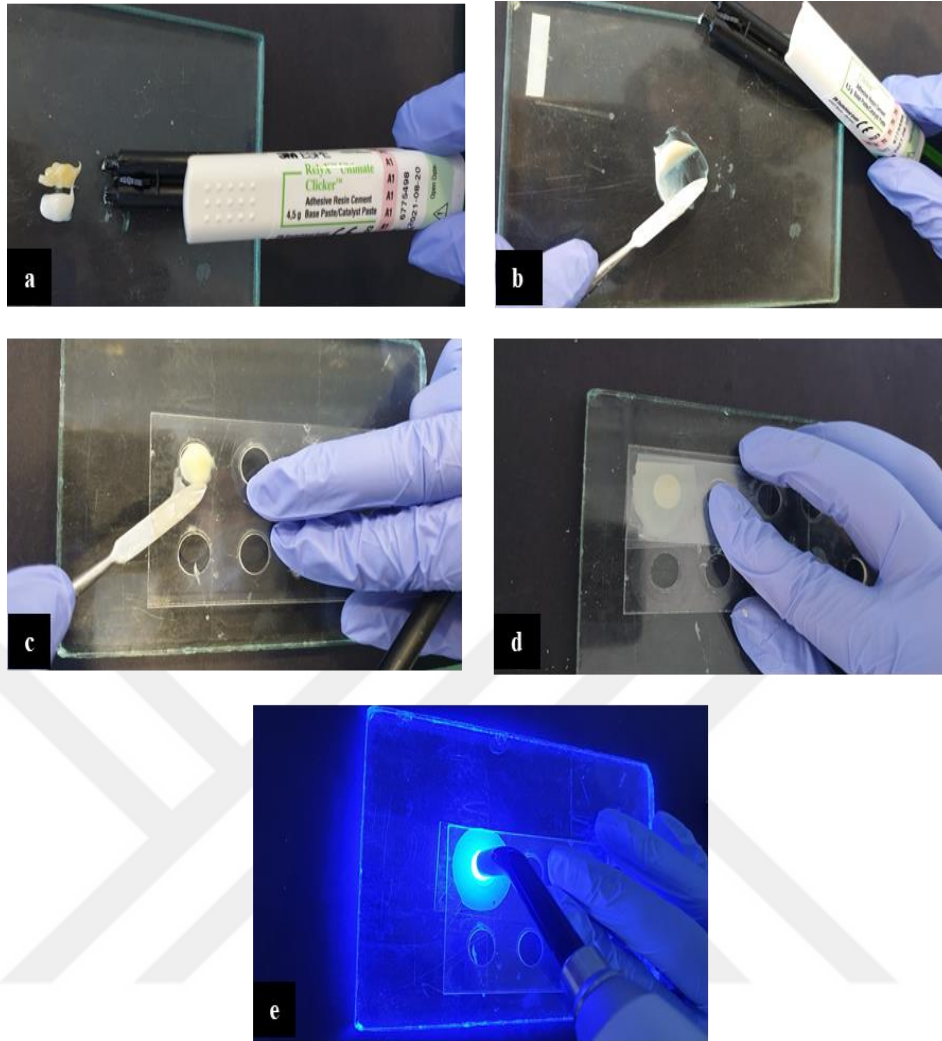


Figure 27- a-e. Preparation of Rely-X Ultimate Clicker

3.1.4 Application of NX-3 Dual-cured (K.DC) Resin Cement: Resin cement was injected into the molds. Afterwards, finger pressure was applied with microscope slide glass on both sides and Mylar strips in between to remove the excess material and minimize air-bubble formation. Later, the resin cement was polymerized for 40 seconds.

3.2. Grinding and Polishing of Specimens

The grinding and polishing machine (Phoenix Beta, Buehler, Illinois, USA) (Figure 28-a) was used to ground the specimens to the final thickness. The specimens were ground and polished in sequence using a series of SiC grinding sheets (Buehler Abrasives, Germany) (#600, #1000, #1500, #2500) (Figure 28-b). These procedures were performed on both sides of the specimens to reduce the thickness to 0.5mm (Figure 28-c). The final

thickness of the specimens was controlled using a digital caliper (Figure 20-d.) having an accuracy of ± 0.01 mm.



Figure 28- a. Grinding and polishing machine, b. SiC grinding sheets, c. Grinding and polishing procedure, d. Digital caliper.

3.3. Pre-staining Procedure

A total of 200 specimens were stored in deionized water at 37°C in the incubator (Mettler GmbH+ Co, Germany) for 24 hours (Figure 29). Before performing the baseline spectrophotometric measurements, the specimens were removed from the vials, washed under running water (Figure 30) and blotted dry by using a paper towel.



Figure 29. Incubator



Figure 30. Washing the specimens under running water.

3.4. Grouping of the Specimens

A total of 200 specimens were randomly assigned into 20 groups, with 10 samples in each solution group. The solutions used in the study were distilled water, cola, tea, coffee, and red wine; and they are listed in Table 2. Grouping of the specimens and their vials used for immersion are shown in Figure 31.

Table 2. Solutions tested in this study.

Solution Type	Trade Name	Lot No.	Manufacturer
Cola	Coca Cola	5449000011114	Coca Cola Co
Teabag	ÇAYKUR Black Tea	8690105000979	Çaykur
Coffee	Nescafe Gold	931402020	Nestle
Red Wine	Doluca	00331-200331	DOLUCA
Distilled water	Pure water	8692534022126	Automix



Figure 31. Grouping of all of the specimens and their vials used for immersion.

3.5. Staining Procedure

The coffee solution (Nescafe Gold, Nestle, Turkey) was prepared by adding 2 g of instant coffee powder in 200 ml of water. The tea solution (Black Teabag, Çaykur, Turkey) was prepared by immersing a tea bag in 200 ml of water. Both solutions were stirred for 10 seconds every 30 minutes until they cooled down to 37°C and they were filtered through a filter paper before being used. The other solutions (distilled water, cola, and red wine) were ready to use and no specific preparation method was applied for them. All the solutions were renewed every 24 hours.

The specimens were stored in the vials containing each solution tested (Figure 32, 33, 34, 35) according to the groups mentioned earlier. All the vials were placed in an incubator at 37°C to imitate the oral environment. The specimens were immersed in the solutions for a total of 288 hours.



Figure 32. Grouping of 3M Rely-X Veneer Light-Cure (3M.LC) specimens according to the solutions (distilled water, cola, tea, coffee, and red wine)

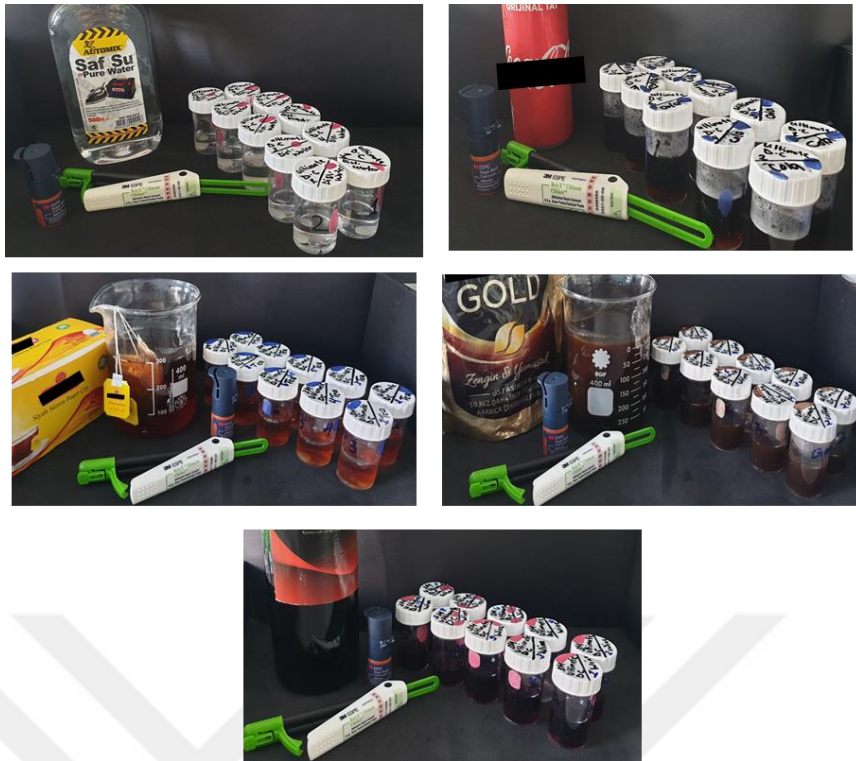


Figure 33. Grouping of 3M Ultimate Dual-Cure (3M.DC) specimens according to the solutions (distilled water, cola, tea, coffee, and red wine)

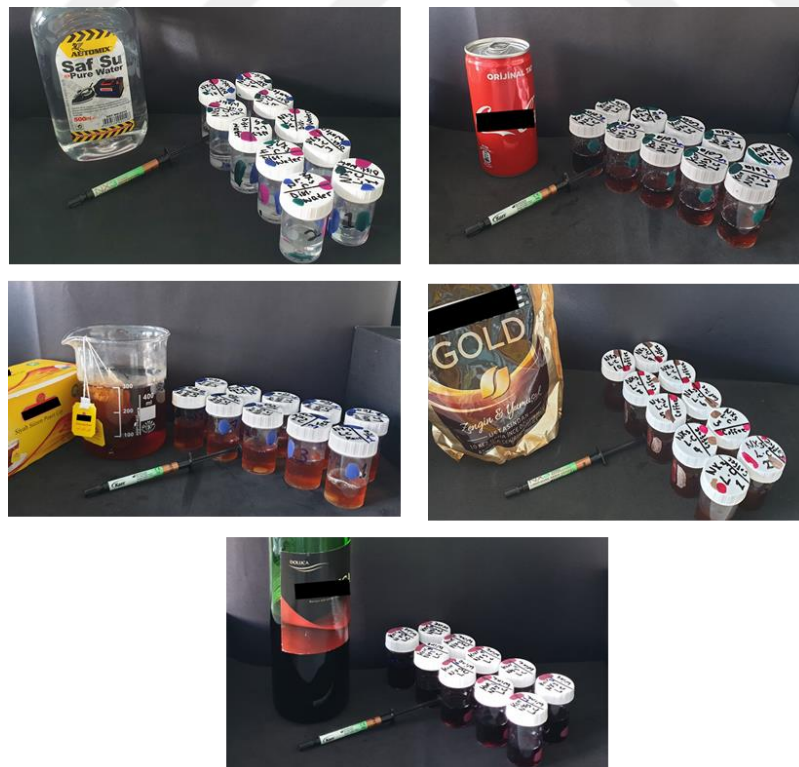


Figure 34. Grouping of Kerr NX-3 Light-Cure (K.LC) specimens according to the solutions (distilled water, cola, tea, coffee, and red wine)

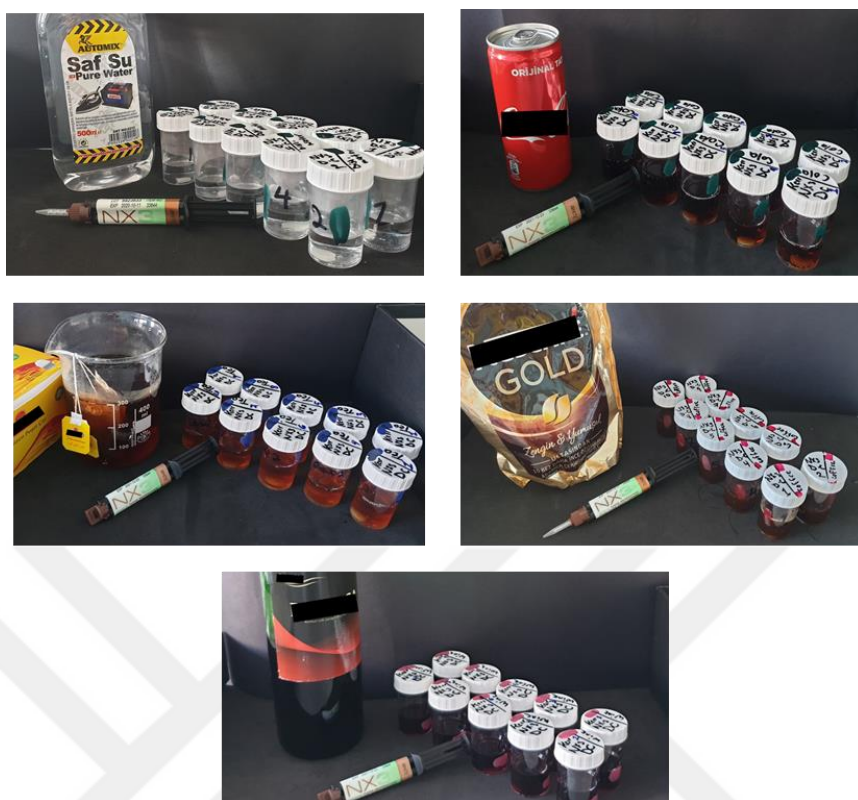


Figure 35. Grouping of the Kerr NX-3 Dual-Cure (K.DC) specimens according to the solutions (distilled water, cola, tea, coffee, and red wine)

3.6. Spectrophotometric Measurements

Before immersing the specimens into the solutions, they were kept in distilled water at $37\pm1^{\circ}\text{C}$ for 24 hours. After 24 hours of immersion, the base-line color measurements of each specimen (T0) were performed using a spectrophotometer (CM-2600d, Konica Minolta Sensing Inc., Japan) (Figure 36). The specimens were then placed in vials containing the tested solutions until further measurements were made.

It was reported that the average consumption time of 1 cup of coffee is 15 minutes and an average of 3.2 cups of coffee is consumed per day (9). Therefore, assuming that 3.2 cups of coffee are consumed per day, the exposure time to coffee is 48 minutes each day. It can thus be calculated that the specimen will be exposed to coffee for 1440 minutes (=24 hours), which is equal to approximately 30 days of consumption. Similarly calculated, color measurements of specimens were made, 24 hours (T1: 30-day consumption of the drink), 72 hours (T2: 90-day consumption of the drink), 144 hours (T3: 180-day consumption of the drink), 216 hours (T4: 270-day consumption of the

drink), and 288 hours (T5- 360 day consumption of the drink) after they were immersed in solutions, respectively.

Prior to each measurement, the specimens were rinsed under running water for 10 seconds, and after each measurement the specimens were kept in artificial saliva for 30 minutes before getting stored in the immersion solution again.

The color of the specimens was assessed with the Commission Internationale de L'Eclairage L*a*b* (CIE L*a*b*) color space using a spectrophotometer (CM-2600d, Konica Minolta Sensing Inc., Japan) (Figure 36) and “Spectra-Magic 3.1” computer Program.

Three measurements were taken for each specimen on white background with daylight and average values were calculated and recorded.

The difference of color (ΔE) before and after staining was calculated using the following formula (9):

$$\Delta E = [(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2]^{1/2}$$

where “L” represents lightness and brightness; greater “L” values indicates lighter samples. The “a” coordinate is a measure of chroma along the red-green axis; a positive “a” stands for redness while a negative “a” represents greenness. The “b” coordinate is a measure of chroma along the yellow-blue axis; a positive “b” represents yellowness while a negative “b” stands for blueness. ΔL , Δa , Δb are differences in L*, a*, b* values before (T0) and after immersion at each time interval (T1, T2, T3, T4, T5).

High ΔE values indicate a large color change.



Figure 36. Spectrophotometer device (CM-2600d, Konica Minolta Sensing Inc., Japan).

In order to make a correlation between the amount of color change (ΔE) recorded by the spectrophotometer and the clinical environment, the data were converted into units of the National Bureau of Standards (NBS) using the following formula:

$$\text{NBS units} = \Delta E \times 0.92.$$

Table 3 explains the National Bureau of Standards (NBS) ratings (57).

Table 3: National Bureau of Standards (NBS) ratings.

NBS UNIT	Critical remarks of color differences
0.0–0.5	Trace: Extremely slight change
0.5–1.5	Slight: Slight change
1.5–3.0	Noticeable: Perceivable change
3.0–6.0	Appreciable: Marked change
6.0–12.0	Much: Extremely marked change
12.0 or more	Very much: Change to other color

3.7. Statistical Analysis:

A power analysis was conducted with 80% power, the common standard deviation 0.45 and 5% Type I error, and a sample size of 10 specimens was selected for each group.

Statistical analysis was performed using the statistical package (IBM SPSS Statistics for Windows v26.0; IBM Corp).

The data set was purged of outliers. The normal distribution status of the data for each group was evaluated by looking at the kurtosis and skewness values. Kalaycı (58) stated that the coefficients of skewness-kurtosis being in the ± 3 range can be evaluated within normality. It was seen that the normal distribution assumption was met. Therefore, parametric tests were used in this research.

Firstly, data obtained from color stability tests were analyzed with One-Way ANOVA Repeated Measures for researching differences between 5 measurements on different times for each brand & solution pair. When sphericity assumption is not met Greenhouse-Geisser results were interpreted in analysis. Pairwise comparisons for time

were completed with the EM Means contrasts with sequential Bonferroni adjustments for multiple comparisons.

Afterwards, 3 Way Repeated Measure ANOVA test was used for researching main and interaction effects of time, brand, and solution on measurements. When sphericity assumption is not met Greenhouse-Geisser results were interpreted in analysis. Pairwise comparisons for brand and solution groups were completed with the EMMeans contrasts with sequential Bonferroni adjustments for multiple comparisons.

The statistical significance was set at the 0.05 level.



4. RESULTS

The means of color difference (ΔE) with standard deviations and corresponding NBS unit values of all groups tested after 24 hours (T1), 72 hours (T2), 144 hours (T3), 216 hours (T4), and 288 hours (T5) of immersion in all solutions are presented in Table 4. Table 4 and Table 5 also show results of statistical analysis for the comparison of mean ΔE values against different variables.

Pairwise comparisons of color change for different solution groups are shown in Table 6. Descriptive statistics of color change (ΔE) for each resin cement and results of comparison between measurements are also shown in Tables 7 to 10. In addition, multiple comparisons for resin cement between all brand groups for dependent variable of ΔE values are shown in the Table 11. In addition, the comparison among the discoloration of all resin cements tested against different staining solutions is shown in Table 12.

Results of multivariate analysis revealed that both resin cements and solution factors had a significant effect on color change over time. Therefore, the null hypothesis that the immersion solution and the type of resin cements has no influence on the color change of resin cements was rejected. Repeated measure analysis showed that the color stability of all resin cements was significantly affected by the immersion period ($p=0.000$) (Table 5). Accordingly, the color change (ΔE) of resin cements increased as the immersion period increased (Table 4).

When comparing color stability of the resin cements for **distilled water** and **cola**, there was no statistically significant difference between any material ($p> 0.05$) (Table 11). Therefore, the material has no effect on color stability while these solutions were used.

When the **tea** solution was used, a high statistical significance ($p= 0.000$) was observed in paired comparisons between all materials except between 3M.LC and K.DC ($p= 0.158$) and between K.LC and K.DC ($p= 0.096$) groups (Table 11). The resin cement showing the most color change in the tea solution was 3M.DC specimens (Table 4 and Table 12).

When the **coffee** solution was used, a high statistical significance ($p= 0.000$) was observed in paired comparisons between all materials except between 3M.DC and K.LC ($p=0.234$) and between 3M.DC and K.DC ($p=1000$) and between K.LC and K.DC groups ($p=0.180$) (Table 11). The resin cement showing the most color change in the coffee solution was 3M.DC specimens (Table 4 and Table 12).

When the **red wine** was used, a high statistical significance ($p= 0.000$) was observed in paired comparisons between all materials except between 3M.LC and K.LC ($p=1000$) (Table 11). The resin cement showing the most color change in the red wine was K.DC specimens (Table 4 and Table 12).

At the end of the study (after 288 hours: approximately equal to 360 days of drink consumption), all test group specimens (immersed in cola, tea, coffee, red wine) demonstrated higher ΔE values compared to the control group specimens (immersed in distilled water) (Table 4).

Among all the staining solutions for each time period (T1 to T5), **cola** was the least staining solution for all resin cements. ΔE values of samples immersed in cola were statistically significantly lower than ΔE values of samples immersed in tea, coffee, and red wine for all resin cements ($p= 0.000$). Among the resin cements in the cola solution, the most color change belonged to the K.DC samples ($\Delta E=2.96$, NBS unit=2.72), and this was obtained in the measurements after 288 hours of immersion (T5) (Table 4).

Red wine generally caused the most pronounced color change in all resin cement groups, except in the K.LC group (at T3-T5) and K.DC group (at T1) where at these measurements the specimens showed the highest ΔE value when immersed in coffee (Table 4).

The greatest color change was exhibited in the K.DC specimens immersed in red wine at the end of the 288 hours immersion ($\Delta E=21.66$, NBS unit=19.72, T5).

When the **NBS unit** values obtained in this study were evaluated, NBS values of all resin cement groups immersed in **distilled water** ranged from 0.49 ($\Delta E=0.54$ – K.DC at T1) to 2.16 ($\Delta E=2.35$ – 3M.DC at T5). Also, the NBS values of all resin cement samples immersed in the **cola** solution ranged from 0.72 ($\Delta E=0.79$ – K.LC at T1) to 2.72 ($\Delta E=2.96$ – K.DC at T5) (Table 4). Since these values are less than 3, there was no clinically marked change in all resin cements immersed in distilled water and cola for all immersion intervals according to the NBS rating system.

NBS values of all resin cement groups immersed in **tea** ranged from 2.25 ($\Delta E=2.45$ – 3M.LC at T1) to 13.41 ($\Delta E=14.58$ – 3M.DC at T5). All resin cements in tea solution exhibited “marked” discoloration starting from T2 (72 hours of immersion, namely 90 days of consuming tea). Moreover, the 3M.DC samples immersed in tea had “Extremely marked” discoloration (NBS>6) at the end of 72 hours, while the 3M.LC

samples at the end of 144 hours, and the K.LC and K.DC samples at the end of 216 hours immersion (Table 3 and Table 4).

The specimens immersed in **coffee** and **red wine** showed “marked” color changes starting from the first 24 hours of immersion (T1) in the staining solutions (30 days of consuming). NBS values of all resin cement groups immersed in coffee were ranged from 3.42 ($\Delta E=3.72$ – 3M.LC at T1) to 16.09 ($\Delta E=17.49$ – 3M.DC at T5) (Table 4). For 3M.DC, K.LC, and K.DC group samples, coffee caused “extremely marked” color change after 72 hours of immersion. In the 3M.LC samples, “extremely marked” change (NBS> 6) was observed when immersed in coffee for 144 hours (Table 4).

NBS values of all resin cement groups immersed in red wine ranged from 5.04 ($\Delta E=5.48$ – K.DC at T1) to 19.92 ($\Delta E=21.66$ – K.DC at T5). When immersed in red wine, the “Extremely marked” (NBS>6) color change occurred in the first 24 hours for 3M.DC samples, and after 72 hours for all other resin cement brands (Table 4).

When Tables 4 and 12 are examined, it can be observed that light-cured cements undergo less color change in staining solutions compared to dual-cured cements in general.

Table 4. Means and standard deviations of color difference (ΔE) and corresponding NBS values for resin cements after staining at different testing intervals, and descriptive statistics of color change for resin cements and results of comparison between measurements.

MATERIAL	SOLUTION	T1		T2		T3		T4		T5		ANOVA		Post ^a Hoc
		ΔE ($\pm$ Sd)	NBS	ΔE ($\pm$ Sd)	NBS	ΔE ($\pm$ Sd)	NBS	ΔE ($\pm$ Sd)	NBS	ΔE ($\pm$ Sd)	NBS	F	P	
3M.LC	Distilled water	0.67 ($\pm$ 0.26)	0.61	0.89 ($\pm$ 0.10)	0.81	1.06 ($\pm$ 0.23)	0.97	1.35 ($\pm$ 0.34)	1.24	1.52 ($\pm$ 0.41)	1.39	21.815	0.000	T1-T4, T1-T5, T2-T4, T2-T5
	Cola	0.82 ($\pm$ 0.47)	0.75	1.21 ($\pm$ 0.36)	1.11	1.54 ($\pm$ 0.43)	1.41	1.76 ($\pm$ 0.47)	1.61	1.95 ($\pm$ 0.57)	1.79	32.738	0.000	T1-T3, T1-T4, T1-T5, T2-T3, T2-T4, T2-T5, T3-T5
	Tea	2.45 ($\pm$ 0.62)	2.25	5.03 ($\pm$ 1.11)	4.62	7.29 ($\pm$ 1.19)	6.70	10.33 ($\pm$ 1.23)	9.50	12.43 ($\pm$ 1.64)	11.43	233.150	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T3, T2-T4, T2-T5, T3-T4, T3-T5, T4-T5
	Coffee	3.72 ($\pm$ 0.65)	3.42	5.76 ($\pm$ 0.56)	5.29	9.09 ($\pm$ 1.21)	8.36	11.35 ($\pm$ 0.71)	10.44	12.96 ($\pm$ 0.76)	11.92	463.438	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T3, T2-T4, T2-T5, T3-T4, T3-T5, T4-T5
	Red wine	6.37 ($\pm$ 1.69)	5.86	8.85 ($\pm$ 1.99)	8.14	10.38 ($\pm$ 1.65)	9.54	12.63 ($\pm$ 1.65)	11.61	14.34 ($\pm$ 1.95)	13.19	141.665	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T3, T2-T4, T2-T5, T3-T4, T3-T5 T4-T5
3M.DC	Distilled water	0.72 ($\pm$ 0.22)	0.66	1.39 ($\pm$ 0.44)	1.27	1.66 ($\pm$ 0.56)	1.52	2.03 ($\pm$ 0.65)	1.86	2.35 ($\pm$ 0.73)	2.16	39.443	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T3, T2-T4, T2-T5, T3-T4, T3-T5, T4-T5
	Cola	1.00 ($\pm$ 0.27)	0.92	1.25 ($\pm$ 0.29)	1.15	1.67 ($\pm$ 0.33)	1.53	2.01 ($\pm$ 0.42)	1.84	2.59 ($\pm$ 0.61)	2.38	47.791	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T3, T2-T4, T2-T5, T3-T5, T4-T5
	Tea	2.93 ($\pm$ 0.43)	2.69	6.70 ($\pm$ 1.04)	6.16	10.63 ($\pm$ 1.87)	9.77	13.01 ($\pm$ 1.33)	11.96	14.58 ($\pm$ 1.33)	13.41	390.232	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T3, T2-T4, T2-T5, T3-T4, T3-T5, T4-T5
	Coffee	3.99 ($\pm$ 0.90)	3.67	7.29 ($\pm$ 1.50)	6.70	12.81 ($\pm$ 1.98)	11.78	16.03 ($\pm$ 1.62)	14.74	17.49 ($\pm$ 2.33)	16.09	314.614	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T3, T2-T4, T2-T5, T3-T4, T3-T5, T4-T5
	Red wine	8.10 ($\pm$ 1.12)	7.45	13.46 ($\pm$ 2.03)	12.38	16.69 ($\pm$ 1.91)	15.35	18.74 ($\pm$ 1.99)	17.24	19.75 ($\pm$ 2.02)	18.17	462.786	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T3, T2-T4, T2-T5, T3-T4, T3-T5 T4-T5

	NBS: 0.5—1.5	Slight: Slight change
	NBS: 1.5—3.0	Noticeable: Perceivable change
	NBS: 3.0—6.0	Appreciable: Marked change
	NBS: 6.0—12.0	Much: Extremely marked change
	NBS: 12.0 or more	Very much: Change to other color

Table 4-continued

MATERIAL	SOLUTION	T1		T2		T3		T4		T5		ANOVA		Post ^a Hoc
		ΔE ($\pm$ Sd)	NBS	ΔE ($\pm$ Sd)	NBS	ΔE ($\pm$ Sd)	NBS	ΔE ($\pm$ Sd)	NBS	ΔE ($\pm$ Sd)	NBS	F	P	
K.LC	Distilled water	0.79 (± 0.14)	0.72	1.38 (± 0.28)	1.26	1.52 (± 0.27)	1.39	1.62 (± 0.26)	1.49	1.75 (± 0.33)	1.61	44.515	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T4, T2-T5, T3-T4, T3-T5, T4-T5
	Cola	0.79 (± 0.35)	0.72	1.20 (± 0.30)	1.01	1.46 (± 0.22)	1.34	1.61 (± 0.25)	1.48	1.86 (± 0.33)	1.71	54.609	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T3, T2-T4, T2-T5, T3-T4, T3-T5, T4-T5
	Tea	2.63 (± 0.77)	2.41	3.82 (± 1.23)	3.51	4.54 (± 1.17)	4.17	6.90 (± 1.18)	6.34	9.99 (± 1.81)	9.19	232.258	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T3, T2-T4, T2-T5, T3-T4, T3-T5, T4-T5
	Coffee	4.86 (± 0.71)	4.47	7.11 (± 0.60)	6.54	11.51 (± 0.94)	10.58	14.11 (± 1.45)	12.98	15.73 (± 1.54)	14.47	354.628	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T3, T2-T4, T2-T5, T3-T4, T3-T5, T4-T5
	Red wine	6.16 m(± 0.77)	5.66	8.75 (± 0.88)	8.05	9.90 (± 0.71)	9.10	11.84 (± 0.45)	10.89	13.85 (± 0.69)	12.74	170.264	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T3, T2-T4, T2-T5, T3-T4, T3-T5, T4-T5
K.DC	Distilled water	0.54 (± 0.15)	0.49	0.57 (± 0.19)	0.52	0.71 (± 0.24)	0.65	0.96 (± 0.35)	0.88	1.25 (± 0.46)	1.15	18.464	0.003	T1-T5, T2-T4, T2-T5, T3-T5
	Cola	1.48 (± 0.25)	1.36	1.92 (± 0.22)	1.76	2.43 (± 0.39)	2.23	2.67 (± 0.33)	2.45	2.96 (± 0.55)	2.72	37.182	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T3, T2-T4, T2-T5, T3-T5
	Tea	3.20 (± 0.90)	2.94	5.36 (± 1.32)	4.93	5.90 (± 0.85)	5.42	8.18 (± 1.17)	7.52	10.26 (± 1.15)	9.43	402.355	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T4, T2-T5, T3-T4, T3-T5, T4-T5
	Coffee	5.63 (± 1.04)	5.17	7.86 (± 0.79)	7.23	12.74 (± 1.10)	11.72	14.58 (± 1.14)	13.41	17.03 (± 1.63)	15.66	411.609	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T3, T2-T4, T2-T5, T3-T4, T3-T5, T4-T5
	Red wine	5.48 (± 0.52)	5.04	8.92 (± 0.67)	8.20	13.81 (± 1.09)	12.70	18.32 (± 1.63)	16.85	21.66 (± 2.73)	19.92	277.740	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T3, T2-T4, T2-T5, T3-T4, T3-T5, T4-T5
	NBS: 0.5—1.5	Slight: Slight change												
	NBS: 1.5—3.0	Noticeable: Perceivable change												
	NBS: 3.0—6.0	Appreciable: Marked change												
	NBS: 6.0—12.0	Much: Extremely marked change												
	NBS: 12.0 or more	Very much: Change to other color												

Table 5. Analysis results of the effect of brand of resin cements and solution and time factors on color change.

Source	Type III Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared
<i>Between-Subjects Effect</i>						
Brand	155.515	3	51.838	60.970	.000*	.512
Solution	3933.186	4	983.296	1156.507	.000*	.964
Brand * Solution	181.792	12	15.149	17.818	.000*	.551
Error	147.940	174	.850			
<i>Within-Subjects Effect</i>						
Time	5597.006	2.430	2303.530	3580.280	.000*	.954
Time * Brand	188.267	7.289	25.828	40.144	.000*	.409
Time * Solution	2726.325	9.719	280.515	435.992	.000*	.909
Time * Brand * Solution	444.908	29.157	15.259	23.716	.000*	.621
Error (Time)	272.012	422.777	.643			

3 Way Repeated Measure ANOVA
*significant p-value at 0.05 level

According to results of Table 5, there is a significant difference between ΔE values of 5 repeated measurements ($F=3580.280$, $p=0.000$). This finding can be interpreted as the color stability of the samples change and increase over time without making any brand (resin cement) or solution distinction. Accordingly, the color changes (ΔE) of resin cements intensified with increasing immersion period.

On the other hand, the difference between ΔE values of 5 repeated measurements according to **resin cement groups** was found significant ($F=40.144$, $p=0.000$). Accordingly, it was found that the color stability of the resin cements differed significantly before to after the experiment, that is, the common effects of being in different resin cement groups and repetitive measurements factors on color stability levels were found to be significant (Table 5).

Besides, the difference between ΔE values of 5 repeated measurements by **solution groups** was found significant ($F=435.992$, $p=0.000$). Accordingly, it was found that the color stability of the resin cements applied with 5 different solutions differed significantly from before to after the experiment, that is, the common effects of being in different solution groups and repetitive measurements factors on color stability levels were significant (Table 5).

Also, the difference between ΔE values by **brands of resin cements*solution blocks** was found to be significant ($F=17.818$, $p=0.000$). Accordingly, it was found that the color stability of the 4 different resin cements immersed in 5 different solutions was significant. (Table 5).

Lastly, it was found that the color stability of 4 different resin cement groups immersed in 5 different solutions showed a significant difference ($F=23,716$, $p=0.000$) overtime, that is, the common **effect of time, resin cement and solution** factors on color stability levels were significant (Table 5).



Table 6. Pairwise comparisons of color change for different solution groups

Resin cements	Solution (I)	Solution (J)	Mean Difference (I-J)	Std. Error	p*	95% Confidence Interval for Difference ^a	
						Lower Bound	Upper Bound
3M.LC	Distilled Water	Cola	-.355	.424	1.000	-1.559	.850
		Tea	-6.405*	.424	.000	-7.610	-5.201
		Coffee	-7.475*	.435	.000	-8.711	-6.239
		Red Wine	-9.416*	.424	.000	-10.621	-8.212
	Cola	Distilled Water	.355	.424	1.000	-.850	1.559
		Tea	-6.051*	.412	.000	-7.223	-4.878
		Coffee	-7.120*	.424	.000	-8.324	-5.915
		Red Wine	-9.062*	.412	.000	-10.234	-7.889
	Tea	Distilled Water	6.405*	.424	.000	5.201	7.610
		Cola	6.051*	.412	.000	4.878	7.223
		Coffee	-1.069	.424	.125	-2.274	.135
		Red Wine	-3.011*	.412	.000	-4.183	-1.839
	Coffee	Distilled Water	7.475*	.435	.000	6.239	8.711
		Cola	7.120*	.424	.000	5.915	8.324
		Tea	1.069	.424	.125	-.135	2.274
		Red Wine	-1.942*	.424	.000	-3.146	-.737
	Red Wine	Distilled Water	9.416*	.424	.000	8.212	10.621
		Cola	9.062*	.412	.000	7.889	10.234
		Tea	3.011*	.412	.000	1.839	4.183
		Coffee	1.942*	.424	.000	.737	3.146
3M.DC	Distilled Water	Cola	-.075	.412	1.000	-1.248	1.097
		Tea	-7.942*	.412	.000	-9.114	-6.770
		Coffee	-9.893*	.412	.000	-11.065	-8.720
		Red Wine	-13.720*	.412	.000	-14.892	-12.547
	Cola	Distilled Water	.075	.412	1.000	-1.097	1.248
		Tea	-7.867*	.412	.000	-9.039	-6.694
		Coffee	-9.818*	.412	.000	-10.990	-8.645
		Red Wine	-13.644*	.412	.000	-14.817	-12.472
	Tea	Distilled Water	7.942*	.412	.000	6.770	9.114
		Cola	7.867*	.412	.000	6.694	9.039
		Coffee	-1.951*	.412	.000	-3.123	-.778
		Red Wine	-5.778*	.412	.000	-6.950	-4.605
	Coffee	Distilled Water	9.893*	.412	.000	8.720	11.065
		Cola	9.818*	.412	.000	8.645	10.990
		Tea	1.951*	.412	.000	.778	3.123
		Red Wine	-3.827*	.412	.000	-4.999	-2.654
	Red Wine	Distilled Water	13.720*	.412	.000	12.547	14.892
		Cola	13.644*	.412	.000	12.472	14.817
		Tea	5.778*	.412	.000	4.605	6.950
		Coffee	3.827*	.412	.000	2.654	4.999

Based on estimated marginal means

*significant p-value at 0.05 level

a. Adjustment for multiple comparisons: Bonferroni.

Table 6-continued

Resin cements	Solution (I)	Solution (J)	Mean Difference (I-J)	Std. Error	P*	95% Confidence Interval for Difference ^a	
						Lower Bound	Upper Bound
K.LC	Distilled Water	Cola	.027	.412	1.000	-1.145	1.200
		Tea	-4.166*	.412	.000	-5.338	-2.993
		Coffee	-9.254*	.412	.000	-10.426	-8.081
		Red Wine	-8.691*	.424	.000	-9.895	-7.486
	Cola	Distilled Water	-.027	.412	1.000	-1.200	1.145
		Tea	-4.193*	.412	.000	-5.366	-3.021
		Coffee	-9.281*	.412	.000	-10.453	-8.109
		Red Wine	-8.718*	.424	.000	-9.923	-7.514
	Tea	Distilled Water	4.166*	.412	.000	2.993	5.338
		Cola	4.193*	.412	.000	3.021	5.366
		Coffee	-5.088*	.412	.000	-6.260	-3.915
		Red Wine	-4.525*	.424	.000	-5.730	-3.320
	Coffee	Distilled Water	9.254*	.412	.000	8.081	10.426
		Cola	9.281*	.412	.000	8.109	10.453
		Tea	5.088*	.412	.000	3.915	6.260
		Red Wine	.563	.424	1.000	-.642	1.767
	Red Wine	Distilled Water	8.691*	.424	.000	7.486	9.895
		Cola	8.718*	.424	.000	7.514	9.923
		Tea	4.525*	.424	.000	3.320	5.730
		Coffee	-.563	.424	1.000	-1.767	.642
K.DC	Distilled Water	Cola	-1,485*	,435	,008	-2,721	-,249
		Tea	-5,773*	,424	,000	-6,977	-4,568
		Coffee	-10,759*	,424	,000	-11,964	-9,555
		Red Wine	-12,831*	,435	,000	-14,067	-11,595
	Cola	Distilled Water	1,485*	,435	,008	,249	2,721
		Tea	-4,288*	,424	,000	-5,493	-3,084
		Coffee	-9,275*	,424	,000	-10,479	-8,070
		Red Wine	-11,346*	,435	,000	-12,582	-10,111
	Tea	Distilled Water	5,773*	,424	,000	4,568	6,977
		Cola	4,288*	,424	,000	3,084	5,493
		Coffee	-4,987*	,412	,000	-6,159	-3,814
		Red Wine	-7,058*	,424	,000	-8,263	-5,854
	Coffee	Distilled Water	10,759*	,424	,000	9,555	11,964
		Cola	9,275*	,424	,000	8,070	10,479
		Tea	4,987*	,412	,000	3,814	6,159
		Red Wine	-2,072*	,424	,000	-3,276	-,867
	Red Wine	Distilled Water	12,831*	,435	,000	11,595	14,067
		Cola	11,346*	,435	,000	10,111	12,582
		Tea	7,058*	,424	,000	5,854	8,263
		Coffee	2,072*	,424	,000	,867	3,276

Based on estimated marginal means

*significant p-value at 0.05 level

a. Adjustment for multiple comparisons: Bonferroni.

4.1. Results of the Color Changes of 3M.LC Resin Cement Groups after Immersion Procedure

The 3M.LC specimens exposed to distilled water, cola, tea, coffee, and red wine showed statistically significant increase in terms of ΔE values throughout measurements repeated over-time ($p=0,000$) (Table 7 and Figure 37).

Among all the staining solutions for each time period (T1 to T5), distilled water was the solution that caused the least color change on 3M.LC samples (Table 7). There was no statistically significant difference ($p>0.05$) between ΔE values of 3M.LC specimens in distilled water and cola. However, there were statistically significant differences between the staining effects of distilled water and tea, distilled water and coffee, and distilled water and red wine ($p=0.000$) (Table 6).

Table 7. Descriptive statistics of color change (ΔE) for 3M.LC resin cements and results of comparison between measurements.

MATERIAL	SOLUTION	T1	T2	T3	T4	T5	ANOVA		Post ^a Hoc
		ΔE ($\pm Sd$)	ΔE ($\pm Sd$)	ΔE ($\pm Sd$)	ΔE ($\pm Sd$)	ΔE ($\pm Sd$)	F	P	
3M.LC	Distilled water	0.67 (± 0.26)	0.89 (± 0.10)	1.06 (± 0.23)	1.35 (± 0.34)	1.52 (± 0.41)	21.815	0.000	T1-T4, T1-T5, T2-T4, T2-T5
	Cola	0.82 (± 0.47)	1.21 (± 0.36)	1.54 (± 0.43)	1.76 (± 0.47)	1.95 (± 0.57)	32.738	0.000	T1-T3, T1-T4, T1-T5, T2-T3, T2-T4, T2-T5, T3-T5
	Tea	2.45 (± 0.62)	5.03 (± 1.11)	7.29 (± 1.19)	10.33 (± 1.23)	12.43 (± 1.64)	233.150	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T3, T2-T4, T2-T5, T3-T4, T3-T5, T4-T5
	Coffee	3.72 (± 0.65)	5.76 (± 0.56)	9.09 (± 1.21)	11.35 (± 0.71)	12.96 (± 0.76)	463.438	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T3, T2-T4, T2-T5, T3-T4, T3-T5, T4-T5
	Red wine	6.37 (± 1.69)	8.85 (± 1.99)	10.38 (± 1.65)	12.63 (± 1.65)	14.34 (± 1.95)	141.665	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T3, T2-T4, T2-T5, T3-T4, T3-T5, T4-T5

NBS: 0.5—1.5	Slight: Slight change
NBS: 1.5—3.0	Noticeable: Perceivable change
NBS: 3.0—6.0	Appreciable: Marked change
NBS: 6.0—12.0	Much: Extremely marked change
NBS: 12.0 or more	Very much: Change to other color

The 3M.LC specimens exposed to distilled water showed statistically different ΔE values between repeated measurements ($p=0.000$). While the ΔE of 3M.LC samples

in distilled water was 0.67 in the measurement at T1, this value increased to 1.52 in the measurements at T5 (Table 7 and Figure 37).

ΔE values of samples in the **cola** solution were statistically significantly lower than ΔE values of 3M.LC samples immersed in tea, coffee, and red wine, as was the case with other brands of resins ($p = 0.000$) (Table 4 and 6).

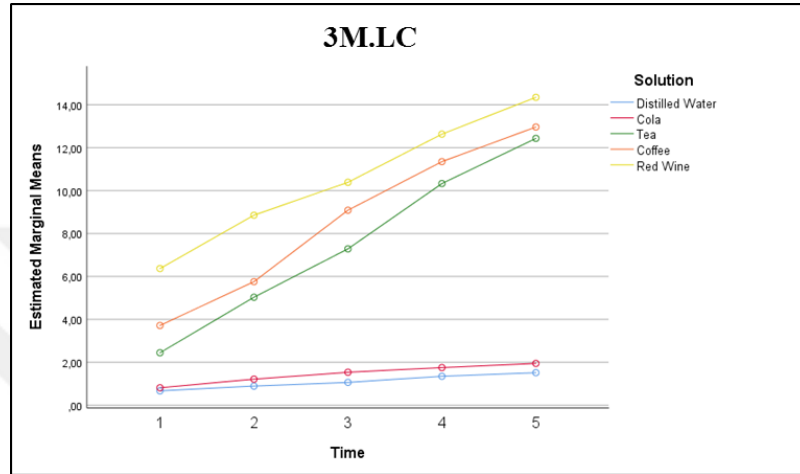


Figure 37. Color changes (ΔE values) of 3M.LC resin cement groups after staining at different testing intervals in different immersion solutions.

When 3M.LC samples were immersed in **cola**, **tea**, **coffee**, and **red wine**, they showed statistically different ΔE values between repeated measurements (from T1 to T5) ($p=0.000$). While the ΔE of 3M.LC samples in **cola** was 0.82 in the measurement at T1, this value increased to 1.95 in the measurements at T5 (Table 7 and Figure 37). While the ΔE of 3M.LC samples in **tea** was 2.45 in the measurement at T1, this value increased to 12.43 in the measurements at T5. While the ΔE of the samples in **coffee** was 3.72 at T1, this value increased to 12.96 in the measurements at T5. While the ΔE value for **red wine** was 6.37 at T1, this value increased up to T5 and reached 14.34 (Table 7 and Figure 37). In paired comparisons between these 3 solutions, there was no statistically significant difference only between ΔE values of 3M.LC specimens in tea and coffee ($p > 0.05$) (Table 6)

Among all the staining solutions for each time period (T1 to T5), the red wine was the most staining solution for the 3M.LC specimens followed by, coffee, tea, and cola. The difference between the ΔE values of the 3M.LC samples immersed in red wine and

cola, red wine and tea, red wine and coffee were statistically significant ($p= 0.000$) (Table 6).

When comparing solutions for 3M.LC specimens, ranking from high to low of ΔE value was **Red Wine > Coffee, Tea > Cola, Distilled Water**.

When color changes are evaluated **according to NBS rating system**, no clinically “marked” color change was observed in the 3M.LC specimens immersed in distilled water and cola at the 5 different measurements. The highest NBS value was 1.79, which is less than 3, and belonged to cola. Even at the end of the 288-hour immersion, the highest NBS value of the 3M.LC samples immersed in distilled water and cola was below 3. In other words, after 288 hours, distilled water and cola did not cause a clinically “marked” color change in 3M.LC samples (Table 4).

While a clinically “marked” color change (NBS unit > 3) occurred in 3M.LC specimens in tea after 72 hours of immersion (T2, namely 90 days of consuming tea), a “marked” color change occurred in 3M.LC specimens immersed in coffee from the first measurement (T1, namely 30 days consuming). In addition, the 3M.LC samples in red wine had an “extremely marked” color change from the first measurement, because the NBS value of these samples was above 6 even in the first 24 hours (T1: 30 days consuming red wine) (Table 4).

After 288 hours, the highest color changes were seen in 3M.LC samples immersed in red wine ($\Delta E= 14.34$ and NBS unit= 13.19), which is considered to be “very much” color change, as it is more than 12 according to the NBS rating system (Table 4).

At the end of immersing for 288 hours, a “slight” color change occurred in distilled water; “noticeable” color change occurred in cola; “much” color change occurred in tea and coffee; and “very much” change was observed in red wine for 3M.LC specimens (Table 4).

4.2. Results of the Color Changes of 3M.DC Resin Cement Groups after Immersion Procedure

The 3M.DC specimens exposed to distilled water, cola, tea, coffee, and red wine showed statistically significance increase in terms of ΔE values throughout measurements repeated over-time ($p=0,000$) (Table 8 and Figure 38).

Among all the staining solutions for each time period (T1 to T5), **distilled water** and **cola** produced significantly less discoloration in 3M.DC samples than tea, coffee,

and red wine (Table 8). There was no statistically significant difference ($p>0.05$) between ΔE values of 3M.DC specimens in distilled water and cola. However, there were statistically significant differences between distilled water and tea, distilled water and coffee, and distilled water and red wine in their staining effects ($p=0.000$) (Table 6).

Table 8. Descriptive statistics of color change (ΔE) for 3M.DC resin cements and results of comparison between measurements.

MATERIAL	SOLUTION	T1	T2	T3	T4	T5	ANOVA		Post ^a Hoc
		ΔE ($\pm Sd$)	ΔE ($\pm Sd$)	ΔE ($\pm Sd$)	ΔE ($\pm Sd$)	ΔE ($\pm Sd$)	F	P	
3M.DC	Distilled water	0.72 (± 0.22)	1.39 (± 0.44)	1.66 (± 0.56)	2.03 (± 0.65)	2.35 (± 0.73)	39.443	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T3, T2-T4, T2-T5, T3-T4, T3-T5, T4-T5
	Cola	1.00 (± 0.27)	1.25 (± 0.29)	1.67 (± 0.33)	2.01 (± 0.42)	2.59 (± 0.61)	47.791	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T3, T2-T4, T2-T5, T3-T5, T4-T5
	Tea	2.93 (± 0.43)	6.70 (± 1.04)	10.63 (± 1.87)	13.01 (± 1.33)	14.58 (± 1.33)	390.232	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T3, T2-T4, T2-T5, T3-T4, T3-T5, T4-T5
	Coffee	3.99 (± 0.90)	7.29 (± 1.50)	12.81 (± 1.98)	16.03 (± 1.62)	17.49 (± 2.33)	314.614	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T3, T2-T4, T2-T5, T3-T4, T3-T5, T4-T5
	Red wine	8.10 (± 1.12)	13.46 (± 2.03)	16.69 (± 1.91)	18.74 (± 1.99)	19.75 (± 2.02)	462.786	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T3, T2-T4, T2-T5, T3-T4, T3-T5, T4-T5

NBS: 0.5—1.5

Slight: Slight change

NBS: 1.5—3.0

Noticeable: Perceivable change

NBS: 3.0—6.0

Appreciable: Marked change

NBS: 6.0—12.0

Much: Extremely marked change

NBS: 12.0 or more

Very much: Change to other color

The 3M.DC specimens exposed to distilled water showed statistically different ΔE values between repeated measurements ($p=0.000$). While the ΔE of 3M.DC samples in distilled water was 0.72 in the measurement at T1, this value increased to 2.35 in the measurements at T5 (Table 8 and Figure 38).

ΔE values of samples in the **cola** solution were statistically significantly lower than ΔE values of 3M.DC samples immersed in tea, coffee, and red wine, as was the case with other brands of resins ($p = 0.000$) (Table 4 and 6).

When 3M.DC samples were immersed in **cola**, **tea**, **coffee**, and **red wine**, they showed statistically different ΔE values between repeated measurements (from T1 to T5) ($p=0.000$). While the ΔE of 3M.DC samples in cola was 1.00 in the measurement at T1, this value increased to 2.59 in the measurements at T5. While the ΔE of 3M.DC samples

in **tea** was 2.93 in the measurement at T1, this value increased to 14.58 in the measurements at T5. While the ΔE of the samples in **coffee** was 3.99 at T1, this value increased to 17.49 in the measurements at T5. While the ΔE value for **red wine** was 8.10 at T1, this value increased up to T5 and reached 19.75 (Table 8 and Figure 38).

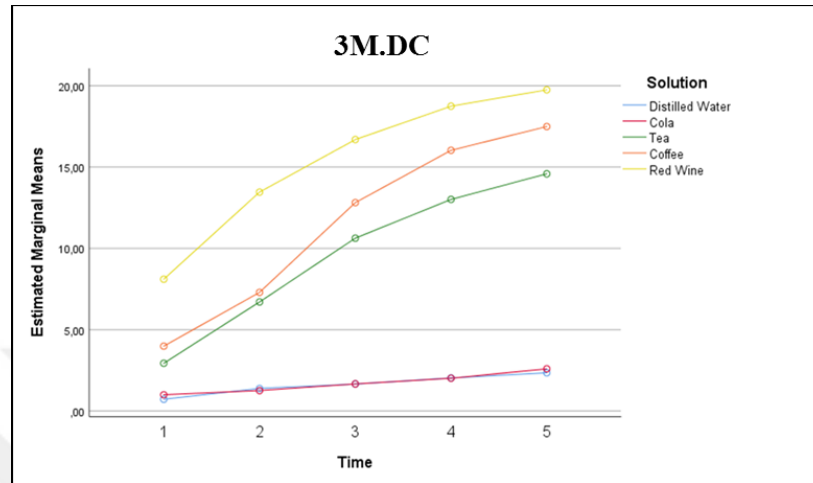


Figure 38. Color changes (ΔE values) of 3M.DC resin cement groups after staining at different testing intervals in different immersion solutions.

Among all the staining solutions for each time period (T1 to T5), the red wine was the most staining solution for the 3M.DC specimens followed by, coffee, tea, and cola. The difference between the ΔE values of the 3M.DC samples exposed to red wine and cola, red wine and tea, red wine and coffee were statistically significant ($p= 0.000$) (Table 6).

When comparing solutions for 3M.DC specimens, ranking from high to low of ΔE value was **Red Wine > Coffee > Tea > Cola, Distilled Water**.

When color changes are evaluated **according to NBS rating system**, no clinically “marked” color change was noted in the 3M.DC specimens exposed to distilled water and cola at the 5 different measurements. The highest NBS value was 2.59, less than 3, and belonged to cola. Even at the end of the 288-hour immersion, the highest NBS value of the 3M.DC samples immersed in distilled water and cola was below 3. In other words, after 288 hours, distilled water and cola did not cause a clinically “marked” color change in 3M.DC samples (Table 4).

For 3M.DC resin cements, a “much” discoloration ($NBS > 6$) occurred in tea and in coffee after 72 hours of immersion (T2, which is equal to 90 days of consuming),

while the samples immersed in red wine started to be seen from the first measurement (T1, which is equal to 30 days consuming). At the end of 288 hours, it has reached a "very much" color change in the coffee (Table 4).

In addition, "very much" color change ($NBS > 12$) occurred in the samples immersed in tea after 288 hours (T5, which is equal to 360 days consuming), in the samples immersed in coffee after 216 hours (T4, which is equal to 144 days consuming) and in the samples immersed in red wine after 72 hours (T2, which is equal to 90 days consuming) (Table 4).

After 288 hours, the highest color changes were seen in 3M.DC samples immersed in red wine ($\Delta E = 19.75$ and $NBS \text{ unit} = 18.17$), which is considered to be "very much" color change, as it is more than 12 according to the NBS rating system (Table 4).

At the end of immersing for 288 hours, a "noticeable" color change occurred in distilled water and cola; and "very much" change was observed in tea, coffee, and red wine for 3M.DC samples (Table 4).

4.3. Results of the Color Changes of K.LC Resin Cement Groups after Immersion Procedure

The K.LC specimens exposed to distilled water, cola, tea, coffee, and red wine exhibited statistically significance increase in the ΔE values throughout measurements repeated over-time ($p = 0.000$) (Table 9 and Figure 39).

Among all the staining solutions for each time period (T1 to T5), **distilled water** and **cola** produced significantly less discoloration in K.LC samples than tea, coffee, and red wine (Table 9). There was no statistically significant difference ($p > 0.05$) between ΔE values of K.LC specimens in distilled water and cola. However, there were statistically significant differences between both solutions (distilled water and cola) and tea, and coffee, and red wine in their staining effects ($p = 0.000$) (Table 6).

The K.LC specimens exposed to **distilled water** showed statistically different ΔE values between repeated measurements ($p = 0.000$). While the ΔE of K.LC samples in distilled water was 0.79 in the measurement at T1, this value increased to 1.75 in the measurements at T5 (Table 9 and Figure 39).

Table 9. Descriptive statistics of color change (ΔE) for K.LC resin cements and results of comparison between measurements.

MATERIAL	SOLUTION	T1	T2	T3	T4	T5	ANOVA		Post ^a Hoc
		ΔE ($\pm Sd$)	ΔE ($\pm Sd$)	ΔE ($\pm Sd$)	ΔE ($\pm Sd$)	ΔE ($\pm Sd$)	F	P	
K.LC	Distilled water	0.79 (± 0.14)	1.38 (± 0.28)	1.52 (± 0.27)	1.62 (± 0.26)	1.75 (± 0.33)	44.515	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T4, T2-T5, T3-T4, T3-T5, T4-T5
	Cola	0.79 (± 0.35)	1.20 (± 0.30)	1.46 (± 0.22)	1.61 (± 0.25)	1.86 (± 0.33)	54.609	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T3, T2-T4, T2-T5, T3-T4, T3-T5, T4-T5
	Tea	2.63 (± 0.77)	3.82 (± 1.23)	4.54 (± 1.17)	6.90 (± 1.18)	9.99 (± 1.81)	232.258	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T3, T2-T4, T2-T5, T3-T4, T3-T5, T4-T5
	Coffee	4.86 (± 0.71)	7.11 (± 0.60)	11.51 (± 0.94)	14.11 (± 1.45)	15.73 (± 1.54)	354.628	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T3, T2-T4, T2-T5, T3-T4, T3-T5, T4-T5
	Red wine	6.16 (± 0.77)	8.75 (± 0.88)	9.90 (± 0.71)	11.84 (± 0.45)	13.85 (± 0.69)	170.264	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T3, T2-T4, T2-T5, T3-T4, T3-T5, T4-T5

NBS: 0.5—1.5	Slight: Slight change
NBS: 1.5—3.0	Noticeable: Perceivable change
NBS: 3.0—6.0	Appreciable: Marked change
NBS: 6.0—12.0	Much: Extremely marked change
NBS: 12.0 or more	Very much: Change to other color

ΔE values of samples in the **cola** solution were statistically significantly lower than ΔE values of K.LC samples exposed to tea, coffee, and red wine, as was the case with other brands of resins ($p = 0.000$) (Table 4 and 6).

When K.LC samples were immersed in **tea**, **coffee**, and **red wine**, they displayed statistically different ΔE values between repeated measurements (from T1 to T5) ($p=0.000$). While the ΔE of K.LC samples in cola was 0.79 in the measurement at T1, this value increased to 1.86 in the measurements at T5. While the ΔE of K.LC samples in **tea** was 2.63 in the measurement at T1, this value increased to 9.99 in the measurements at T5. While the ΔE of the samples in **coffee** was 4.86 at T1, this value increased to 15.73 in the measurements at T5. While the ΔE value for **red wine** was 6.16 at T1, this value increased up to T5 and reached 13.85 (Table 9 and Figure 39).

Among all the staining solutions for T1 and T2 measurement, the red wine was the most staining solution for the K.LC specimens followed by, coffee, tea, and cola. While from T3 to T5 measurement, coffee was the most staining solution for the K.LC specimens followed by red wine, tea, and cola. However, there was no statistical significance ($p>0.05$) between the ΔE values of the samples immersed in red wine and

coffee, while the difference between the ΔE values of the samples immersed in red wine and cola, red wine and tea were statistically significant.

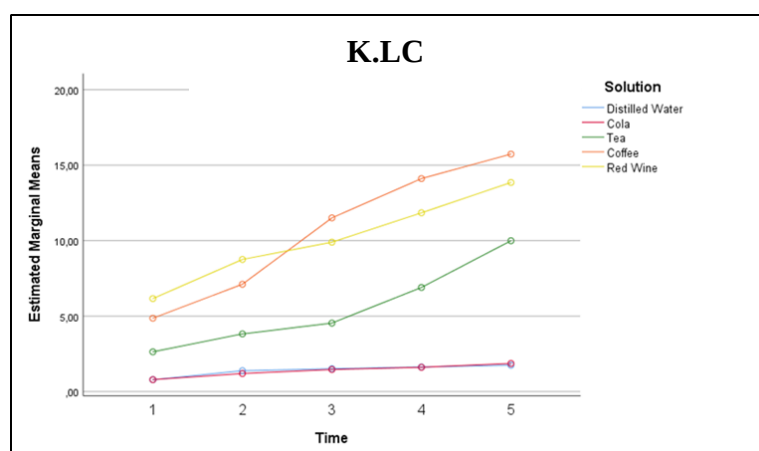


Figure 39. Color changes (ΔE values) of K.LC resin cement groups after staining at different testing intervals in different immersion solutions.

When comparing solutions for 3M.LC specimens, ranking from high to low of ΔE was **Red Wine, Coffee > Tea > Cola, Distilled Water** (Table 6).

When color changes are evaluated **according to NBS rating system**, no clinically “marked” color change was observed in the K.LC specimens immersed in distilled water and cola at the 5 different measurements. The highest NBS unit value was 1.71, which is less than 3, and belonged to cola.

While a clinically “marked” color change ($NBS > 3$) occurred in K.LC specimens in tea after 72 hours of immersion (T2, which is equal to 90 days of consuming tea), a “marked” color change occurred in K.LC specimens immersed in coffee and red wine from the first measurement (T1, which is equal to 30 days consuming). Moreover, K.LC resin specimens immersed in coffee and red wine showed "much" color change ($NBS > 6$) after 72 hours of immersion and "very much" color change ($NBS > 12$) after 216 hours of immersion (T1, which is equal to 270 days consuming) (Table 4).

After 288 hours, the highest color changes were seen in K.LC samples immersed in red wine ($\Delta E = 15.73$ and $NBS \text{ unit} = 14.47$), which is considered to be "very much" color change, as it is more than 12 according to the NBS rating system (Table 4).

At the end of immersing for 288 hours, a “noticeable” color change occurred in distilled water and cola; “much” color change occurred in tea; and "very much" change was observed in coffee, and red wine for K.LC specimens (Table 4).

4.4. Results of the Color Changes of K.DC Resin Cement Groups after Immersion Procedure

The K.DC specimens exposed to distilled water, cola, tea, coffee, and red wine showed statistically significance increase in terms of ΔE values throughout measurements repeated over-time ($p=0,000$) (Table 10 and Figure 40).

Among all the staining solutions for each time period (T1 to T5), **distilled water** was the solution that caused the least color change on K.DC samples (Table 10). There were statistically significant differences between distilled water and cola, distilled water and tea, distilled water and coffee, and distilled water and red wine in their staining effects ($p=0.000$) (Table 6).

Table 10. Descriptive statistics of color change (ΔE) for K.DC resin cements and results of comparison between measurements.

MATERIAL	SOLUTION	T1	T2	T3	T4	T5	ANOVA		Post ^a Hoc
		ΔE ($\pm Sd$)	ΔE ($\pm Sd$)	ΔE ($\pm Sd$)	ΔE ($\pm Sd$)	ΔE ($\pm Sd$)	F	P	
K.DC	Distilled water	0.54 (± 0.15)	0.57 (± 0.19)	0.71 (± 0.24)	0.96 (± 0.35)	1.25 (± 0.46)	18.464	0.003	T1-T5, T2-T4, T2-T5, T3-T5
	Cola	1.48 (± 0.25)	1.92 (± 0.22)	2.43 (± 0.39)	2.67 (± 0.33)	2.96 (± 0.55)	37.182	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T3, T2-T4, T2-T5, T3-T5
	Tea	3.20 (± 0.90)	5.36 (± 1.32)	5.90 (± 0.85)	8.18 (± 1.17)	10.26 (± 1.15)	402.355	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T4, T2-T5, T3-T4, T3-T5, T4-T5
	Coffee	5.63 (± 1.04)	7.86 (± 0.79)	12.74 (± 1.10)	14.58 (± 1.14)	17.03 (± 1.63)	411.609	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T3, T2-T4, T2-T5, T3-T4, T3-T5, T4-T5
	Red wine	5.48 (± 0.52)	8.92 (± 0.67)	13.81 (± 1.09)	18.32 (± 1.63)	21.66 (± 2.73)	277.740	0.000	T1-T2, T1-T3, T1-T4, T1-T5, T2-T3, T2-T4, T2-T5, T3-T4, T3-T5, T4-T5

NBS: 0.5—1.5	Slight: Slight change
NBS: 1.5—3.0	Noticeable: Perceivable change
NBS: 3.0—6.0	Appreciable: Marked change
NBS: 6.0—12.0	Much: Extremely marked change
NBS: 12.0 or more	Very much: Change to other color

The K.DC specimens exposed to distilled water showed statistically different ΔE values between repeated measurements ($p=0.003$). While the ΔE of K.DC samples in distilled water was 0.54 in the measurement at T1, this value increased to 1.25 in the measurements at T5 (Table 10 and Figure 40).

ΔE values of samples in the **cola** solution were statistically significantly lower than ΔE values of K.DC samples immersed in tea, coffee, and red wine, as was the case with other brands of resins ($p = 0.000$) (Table 4 and 6).

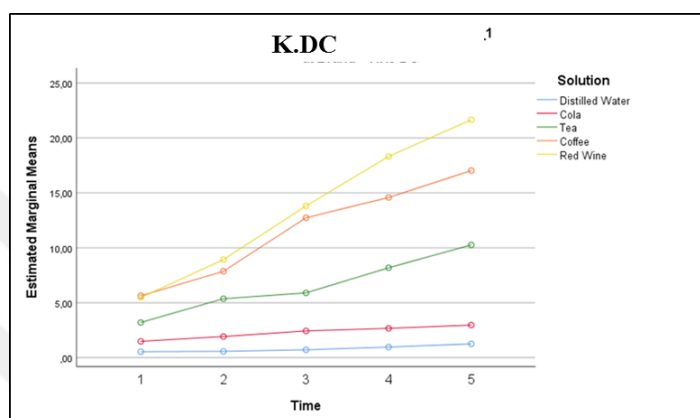


Figure 40. Color changes (ΔE values) of K.DC resin cement groups after staining at different testing intervals in different immersion solutions.

When K.DC samples were immersed in **tea**, **coffee**, and **red wine**, they showed statistically different ΔE values between repeated measurements (from T1 to T5) ($p=0.000$). While the ΔE of K.DC samples in **cola** was 1.48 in the measurement at T1, this value increased to 2.96 in the measurements at T5. While the ΔE of K.DC samples in **tea** was 3.20 in the measurement at T1, this value increased to 10.26 in the measurements at T5. And while the ΔE of K.DC samples in **coffee** was 5.63 in the measurement at T1, this value increased to 17.03 in the measurements at T5. While the ΔE value for **red wine** was 5.48 at T1, this value increased up to 21.66 in T5 (Table 10 and Figure 40).

When comparing all solutions tested for K.DC specimens, there was a statistically significant difference between all solutions ($p= 0.000$). Ranking from high to low of ΔE was **Red Wine > Coffee > Tea > Cola > Distilled Water**.

When color changes are evaluated **according to NBS rating system**, no clinically “marked” color change was observed in the K.DC specimens immersed in distilled water

and cola at the 5 different measurements. The highest NBS value was 2.72, which is less than 3, and belonged to cola. Even at the end of the 288-hour immersion, the highest NBS value of the K.DC samples immersed in distilled water and cola was below 3. In other words, after 288 hours, distilled water and cola did not cause a clinically "marked" color change in K.DC samples (Table 4).

Tea caused clinically "marked" color change in K.DC samples from the first measurement (T1, which is equal to 30 days consuming) and "much" change after 216 hours (T4, which is equal to 270 days consuming) (Table 4).

When K.DC samples were immersed in coffee and red wine, the "marked" color change occurred at the end of the first 24 hours (T1), and "much" color change at 72 hours of immersion (T2) (Table 4).

At the end of the 288 immersion, among all the staining solutions, the red wine was the most staining solution for the K.DC specimens ($\Delta E = 21.66$ and NBS unit = 19.92), which is considered to be "very much" color change, as it is more than 12 according to the NBS rating system.

At the end of immersing for 288 hours, a "slight" color change occurred in distilled water; "noticeable" color change occurred in cola; "much" color change occurred in tea; and "very much" change was observed in coffee and red wine for K.DC specimens (Table 4).

Multiple comparisons for all resin cements between solution groups for dependent variable of ΔE values are shown in Table 11. In addition, Figure 41 demonstrates the color difference (ΔE) of resin cements after immersion in distilled water, tea, cola, coffee, and red wine.

Table 11. Pairwise comparisons of color change for the resin cement groups.

Solution	Brand (I)	Brand (J)	Mean Difference (I-J)	Std. Error	p	95% Confidence Interval for Difference ^a	
						Lower Bound	Upper Bound
Distilled Water	3M.LC	3M.DC	-.530	.424	1.000	-1.661	.601
		K.LC	-.312	.424	1.000	-1.442	.819
		K.DC	.292	.435	1.000	-.868	1.452
	3M.DC	3M.LC	.530	.424	1.000	-.601	1.661
		K.LC	.218	.412	1.000	-.882	1.319
		K.DC	.822	.424	.324	-.309	1.952
	K.LC	3M.LC	.312	.424	1.000	-.819	1.442
		3M.DC	-.218	.412	1.000	-1.319	.882
		K.DC	.603	.424	.938	-.527	1.734
	K.DC	3M.LC	-.292	.435	1.000	-1.452	.868
		3M.DC	-.822	.424	.324	-1.952	.309
		K.LC	-.603	.424	.938	-1.734	.527
Cola	3M.LC	3M.DC	-.250	.412	1.000	-1.351	.850
		K.LC	.071	.412	1.000	-1.030	1.171
		K.DC	-.838	.424	.297	-1.969	.292
	3M.DC	3M.LC	.250	.412	1.000	-.850	1.351
		K.LC	.321	.412	1.000	-.780	1.422
		K.DC	-.588	.424	1.000	-1.719	.543
	K.LC	3M.LC	-.071	.412	1.000	-1.171	1.030
		3M.DC	-.321	.412	1.000	-1.422	.780
		K.DC	-.909	.424	.200	-2.040	.222
	K.DC	3M.LC	.838	.424	.297	-.292	1.969
		3M.DC	.588	.424	1.000	-.543	1.719
		K.LC	.909	.424	.200	-.222	2.040

Based on estimated marginal means

*significant p-value at 0.05 level

a. Adjustment for multiple comparisons: Bonferroni.

Table 11. Continued

Solution	Brand (I)	Brand (J)	Mean Difference (I-J)	Std. Error	p	95% Confidence Interval for Difference ^a	
						Lower Bound	Upper Bound
Tea	3M.LC	3M.DC	-2.067	.412	.000*	-3.167	-.966
		K.LC	1.928	.412	.000*	.827	3.029
		K.DC	.924	.412	.158	-.176	2.025
	3M.DC	3M.LC	2.067	.412	.000*	.966	3.167
		K.LC	3.995	.412	.000*	2.894	5.095
		K.DC	2.991	.412	.000*	1.890	4.091
	K.LC	3M.LC	-1.928	.412	.000*	-3.029	-.827
		3M.DC	-3.995	.412	.000*	-5.095	-2.894
		K.DC	-1.004	.412	.096	-2.104	.097
	K.DC	3M.LC	-.924	.412	.158	-2.025	.176
		3M.DC	-2.991	.412	.000*	-4.091	-1.890
		K.LC	1.004	.412	.096	-.097	2.104
Coffee	3M.LC	3M.DC	-2.948	.424	.000*	-4.079	-1.818
		K.LC	-2.091	.424	.000*	-3.221	-.960
		K.DC	-2.993	.424	.000*	-4.124	-1.863
	3M.DC	3M.LC	2.948	.424	.000*	1.818	4.079
		K.LC	.858	.412	.234	-.243	1.958
		K.DC	-.045	.412	1.000	-1.146	1.056
	K.LC	3M.LC	2.091	.424	.000*	.960	3.221
		3M.DC	-.858	.412	.234	-1.958	.243
		K.DC	-.903	.412	.180	-2.003	.198
	K.DC	3M.LC	2.993	.424	.000*	1.863	4.124
		3M.DC	.045	.412	1.000	-1.056	1.146
		K.LC	.903	.412	.180	-.198	2.003
Red wine	3M.LC	3M.DC	-4,833	,412	,000*	-5,934	-3,733
		K.LC	,414	,424	1,000	-,717	1,545
		K.DC	-3,123	,424	,000*	-4,254	-1,992
	3M.DC	3M.LC	4,833	,412	,000*	3,733	5,934
		K.LC	5,247	,424	,000*	4,117	6,378
		K.DC	1,710	,424	,000*	,579	2,841
	K.LC	3M.LC	-,414	,424	1,000	-1,545	,717
		3M.DC	-5,247	,424	,000*	-6,378	-4,117
		K.DC	-3,537	,435	,000*	-4,697	-2,377
	K.DC	3M.LC	3,123	,424	,000*	1,992	4,254
		3M.DC	-1,710	,424	,000*	-2,841	-,579
		K.LC	3,537	,435	,000*	2,377	4,697

Based on estimated marginal means

*significant p-value at 0.05 level

a. Adjustment for multiple comparisons: Bonferroni.

4.5. Comparison of Color stability between Light-Cured Resin Cements (3M.LC and K.LC)

When comparing the ΔE values of both light-cured resin cements tested, there were no statistically significant differences between 3M.LC and K.LC resin cements immersed in distilled water, cola, and red wine ($p>0.05$). However, when immersed in tea and coffee, there was a statistically significant difference between both light-cured cements ($p=0.000$) (Table 11 and Figure 41).

The ΔE values observed in the 3M.LC and K.LC resin cement specimens immersed in **distilled water** and **cola** varied between 0.67 and 1.95. Hence, both materials in both solutions (distilled water and cola) did not present a clinically “marked” discoloration ($NBS<3$) through the whole immersion period (Table 4).

On the other hand, the specimens immersed in **tea** for both materials (3M.LC and K.LC) demonstrated a clinically “marked” discoloration ($NBS\text{ unit}>3$) from 72 hours of immersion (T2), and their ΔE values were 5.03 and 3.82, respectively (Table 4 and Figure 37, 39 and 41); and the difference between them was statistically significant ($p=0.000$) (Table 11). At the end of 288 hours (360 day consumption of the drink), “much” color change was observed in both light-cured cements immersed in tea.

When immersed in **coffee** and **red wine**, 3M.LC and K.LC cements demonstrated a clinically “marked” discoloration ($NBS>3$) from 24 hours of immersion (T1), having the following ΔE values; 3.72 and 4.86, respectively in the **coffee** group; and 6.37 and 6.16 respectively in the **red wine** group (Table 4 and Figure 37, 39 and 41). At the end of 288 hours (360 day consumption of the drink), “much” or “very much” color change was observed in the samples in both light-cured cements immersed in coffee or red wine.

In summary, at the end of the 288 hours both light-cured resin cements in water and cola did not show any discoloration that could be detected clinically ($NBS<3$). However, both light-cured cements demonstrated clinically marked discoloration that ranged from “much” to “very much” when immersed in the other 3 staining solutions (Table 4).

4.6. Comparison of Color stability between Dual-Cured Resin Cements (3M.DC and K.DC)

When comparing the ΔE values of both the dual-cured resin cements tested, there were no statistically significant differences between 3M.DC and K.DC resin cements

immersed in distilled water, cola, and coffee ($p>0.05$). However, when immersed in tea and red wine, there was a statistically significant difference between both dual-cured cements ($p=0.000$) (Table 11 and Figure 41).

The ΔE values observed in the 3M.DC and K.DC resin cement specimens immersed in **distilled water** and **cola** varied between 0.54 and 2.96. Hence, both materials in both solutions (distilled water and cola) did not present a clinically “marked” discoloration ($NBS<3$) through the whole immersion period (Table 4).

On the other hand, the specimens immersed in **tea** for both materials (3M.DC and K.DC), demonstrated a clinically “marked” discoloration ($NBS>3$) from 72 hours of immersion (T2), and their ΔE values were 6.70 and 5.36, respectively (Table 4 and Figure 38, 40 and 41); and the difference between them was statistically significant ($p=0.000$) (Table 11). At the end of 288 hours (360 day consumption of the drink), “much” or “very much” color change was observed in both dual-cured cements immersed in tea.

When immersed in **coffee** and **red wine**, 3M.DC and K.DC cements demonstrated a clinically “marked” discoloration ($NBS>3$) from 24 hours of immersion (T1), having the following ΔE values; 3.99 and 5.63, respectively in the **coffee** group; and 8.10 and 5.48, respectively in the **red wine** group (Table 4 and Figure 38, 40 and 41). However, it can be noticed that when 3M.DC specimens are immersed in red wine, they demonstrated “much” discoloration from 24 hours of immersion (T1) since its ΔE was equal to 8.10. At the end of 288 hours (360 day consumption of the drink), “very much” color change was observed in both dual-cured cements immersed in coffee or red wine.

In summary, at the end of the 288 hours (360 day consumption of the drink) both dual-cured resin cements in water and cola did not show any discoloration that could be detected clinically ($NBS<3$). However, both dual-cured cements demonstrated clinically marked discoloration that ranged from “much” to “very much” when immersed in the other 3 staining solutions (Table 4).

4.7. Comparison of Color stability between Light-cured and Dual-cured Resin Cements Groups

4.7.1. Comparison of Color Stability between 3M.LC and 3M.DC Resin Cement Groups

When comparing the ΔE values of 3M.LC and the 3M.DC groups, there were no statistically significant differences between both materials immersed in distilled water

and cola ($p>0.05$). However, when immersed in tea, coffee, and red wine, there was a statistically significant difference between both 3M.LC and 3M.DC materials ($p=0.000$) (Table 11 and Figure 41).

The ΔE values observed in 3M.LC and 3M.DC resin cement specimens immersed in **distilled water** and **cola** varied between 0.67 and 2.96. Hence, both materials in both solutions (distilled water and cola) did not present a clinically “marked” discoloration ($NBS<3$) through the whole immersion period (Table 4).

On the other hand, the specimens immersed in **tea** for both materials (3M.LC and 3M.DC) demonstrated a clinically “marked” discoloration ($NBS \text{ unit}>3$) after 72 hours of immersion (T2), and their ΔE values were 5.03 and 6.70, respectively (Table 4 and Figure 37, 38 and 41); and the difference between them was statistically significant ($p=0.000$) (Table 11). At the end of 288 hours (360 day consumption of the drink), “much” or “very much” color change was observed in both resin cements immersed in tea.

When immersed in **coffee** and **red wine**, both resin cements (3M.LC and 3M.DC) demonstrated a clinically “marked” discoloration ($NBS \text{ unit} > 3$) from 24 hours of immersion (T1), having the following ΔE values; 3.72 and 3.99, respectively in the **coffee** group and 6.37 and 8.10, respectively in the **red wine** group. Moreover, it can be noticed that when 3M.DC specimens are immersed in red wine, they demonstrate “much” discoloration from 24 hours of immersion (T1) since its ΔE was equal to 8.10 (Table 4 and Figures 37, 38, 41); and the difference between them were statistically significant ($p=0.000$) (Table 11). At the end of 288 hours (360 day consumption of the drink), “much” or “very much” color change was observed in the samples in both resin cements immersed in coffee or red wine.

At the end of 288 hours (360 day consumption of the drink) 3M.LC specimens showed lower ΔE values than 3M.DC when immersed in tea, coffee, and red wine. According to Table 4, it can be concluded that the dual-cured resin cement (3M.DC) undergoes more discoloration than the light-cured resin cement (3M.LC).

In summary, at the end of the 288 hours (360 day consumption of the drink) 3M.LC and 3M.DC in water and cola did not show any discoloration that could be detected clinically ($NBS<3$). However, these cements demonstrated clinically marked discoloration that ranged from “much” to “very much” when immersed in other 3 staining solutions (Table 4).

4.7.2. Comparison of Color Stability between 3M.LC and K.DC Resin Cement Groups

When comparing the ΔE values of both the 3M.LC and the K.DC groups, there were no statistically significant differences between both materials when immersed in distilled water, cola and tea ($p>0.05$). However, when immersed in coffee, and red wine, there was a statistically significant difference between both 3M.LC and K.DC ($p=0.000$) (Table 11 and Figure 41).

The ΔE values observed in 3M.LC and K.DC resin cement specimens immersed in **distilled water** and **cola** varied between 0.54 and 2.96. Hence, both materials in both solutions (distilled water and cola) did not present a clinically “marked” discoloration ($NBS<3$) through the whole immersion period (Table 4).

On the other hand, the specimens immersed in **tea** for both materials (3M.LC and K.DC) demonstrated a clinically “marked” discoloration ($NBS \text{ unit}>3$) from 72 hours of immersion (T2), and their ΔE values were 5.03 and 5.36, respectively (Table 4 and Figure 37, 40 and 41); and the difference between them was not statistically significant ($p=0.000$) (Table 11). At the end of 288 hours (360 day consumption of the drink), “much” color change was observed in both resin cements immersed in tea.

When immersed in **coffee** and **red wine**, both resin cements (3M.LC and K.DC) demonstrated a clinically “marked” discoloration ($NBS \text{ unit}>3$) from 24 hours of immersion (T1), having the following ΔE values; 3.72 and 5.63, respectively in the **coffee** group; and 6.37 and 5.48, respectively in the **red wine** group (Table 4 and Figures 37, 40, 41); and the difference between them was statistically significant ($p=0.000$) (Table 11). At the end of 288 hours (360 day consumption of the drink), “much” or “very much” color change was observed in the samples in both resin cements immersed in coffee or red wine.

At the end of 288 hours (360 day consumption of the drink) 3M.LC specimens showed lower ΔE values than K.DC when immersed in coffee, and red wine (most colorant solutions) (Table 4).

3M.LC specimens showed lower ΔE values than K.DC when immersed in tea, coffee, and red wine (except at T1 when red wine solution was used, and when tea solution was used from T3 to T5) (Table 4). According to Table 4, it can be concluded that in general the dual-cured resin cement (K.DC) undergoes more discoloration compared to the light-cured resin cement (K.LC).

In summary, at the end of the 288 hours (360 day consumption of the drink) 3M.LC and K.DC in water and cola did not show any discoloration that could be detected

clinically ($NBS < 3$). However, these cements demonstrated clinically marked discoloration that ranged from “much” to “very much” when immersed in other 3 staining solutions (Table 4).

4.7.3. Comparison of Color Stability between K.LC and K.DC Resin Cement Groups

When comparing the ΔE values of both the K.LC and the K.DC groups, there were no statistically significant differences between both the materials when their specimens were immersed in distilled water, cola, tea, and coffee ($p > 0.05$). However, when immersed in red wine, there was a statistically significant difference between both K.LC and K.DC materials ($p = 0.000$) (Table 11 and Figure 41).

The ΔE values observed in K.LC and the K.DC resin cement specimens immersed in **distilled water** and **cola** varied between 0.54 and 2.96. Hence, both materials in both solutions (distilled water and cola) did not present a clinically “marked” discoloration ($NBS < 3$) through the whole immersion period (Table 4).

On the other hand, the specimens immersed in **tea** for both the materials (K.LC and K.DC) demonstrated a clinically “marked” discoloration ($NBS \text{ unit} > 3$) from 72 hours of immersion (T2), and their ΔE values were 3.82 and 5.36, respectively (Table 4 and Figure 39, 40 and 41); and the difference between them was not statistically significant ($p > 0.05$) (Table 11). At the end of 288 hours (360 day consumption of the drink), “much” color change was observed in both resin cements immersed in tea.

When immersed in **coffee** and **red wine**, both resin cements (K.LC and K.DC) demonstrated a clinically “marked” discoloration ($NBS \text{ unit} > 3$) from 24 hours of immersion (T1), having the following ΔE values; 4.86 and 5.63, respectively in the **coffee** group; and 6.16 and 5.48, respectively in the **red wine** group (Table 4 and Figures 39, 40, 41); While the difference between them in coffee was no statistically significant ($p > 0.05$), the difference in red wine was statistically significant ($p = 0.000$) (Table 11). At the end of 288 hours (360 day consumption of the drink), “very much” color change was observed in the samples in both resin cements immersed in coffee or red wine.

K.LC specimens showed lower ΔE values than K.DC when immersed in tea, coffee, and red wine (except at T1 when red wine solution was used) (Table 4). According to Table 4, it can be concluded that the dual-cured resin cement (K.DC) undergoes more discoloration than the light-cured resin cement (K.LC).

In summary, at the end of the 288 hours (360 day consumption of the drink) K.LC and K.DC in water and cola did not show any discoloration that could be detected

clinically (NBS<3). However, these cements demonstrated clinically marked discoloration that ranged from “much” to “very much” when immersed in the other 3 staining solutions (Table 4).

4.7.4. Comparison of Color Stability between K.LC and 3M.DC Resin Cement Groups

When comparing the ΔE values of both the K.LC and the 3M.DC groups, there were no statistically significant differences between both the materials when they were immersed in distilled water, cola, and coffee ($p>0.05$). However, when immersed in tea and red wine, there was a statistically significant difference between both K.LC and K.DC materials ($p=0.000$) (Table 11 and Figure 41).

The ΔE values observed in K.LC and the 3M.DC resin cement specimens immersed in **distilled water** and **cola** varied between 0.72 and 2.59. Hence, both materials in both solutions (distilled water and cola) did not present a clinically “marked” discoloration (NBS<3) through the whole immersion period (Table 4).

On the other hand, the specimens immersed in **tea** for both the materials (K.LC and 3M.DC) demonstrated a clinically “marked” discoloration (NBS unit>3) from 72 hours of immersion (T2), and their ΔE values were 3.82 and 6.70, respectively (Table 4 and Figure 38, 39 and 41); and the difference between them was statistically significant ($p=0.000$) (Table 11). At the end of 288 hours (360 day consumption of the drink), “much” or “very much” color change was observed in both resin cements immersed in tea.

When immersed in **coffee** and **red wine**, both resin cements (K.LC and 3M.DC) demonstrated a clinically “marked” discoloration (NBS unit>3) from 24 hours of immersion (T1), having the following ΔE values; 4.86 and 5.63, respectively in the **coffee** group; and 6.16 and 5.48, respectively in the **red wine** group (Table 4 and Figures 39, 40, 41); While the difference between them in coffee was not statistically significant ($p>0.05$), the difference in red wine was statistically significant ($p=0.000$) (Table 11). At the end of 288 hours (360 day consumption of the drink), “very much” color change was observed in the samples in both resin cements immersed in coffee or red wine.

K.LC specimens showed lower ΔE values than 3M.DC when immersed in tea, coffee, and red wine (Table 4). According to Table 4, it can be concluded that the dual-

cured resin cement (3M.DC) undergoes more discoloration compared to light-cured resin cement (K.LC).

In summary, at the end of the 288 hours (360 day consumption of the drink), K.LC and 3M.DC in water and cola did not show any discoloration that could be detected clinically (NBS<3). However, these cements demonstrated clinically marked discoloration that ranged from “much” to “very much” when immersed in the other 3 staining solutions (Table 4).

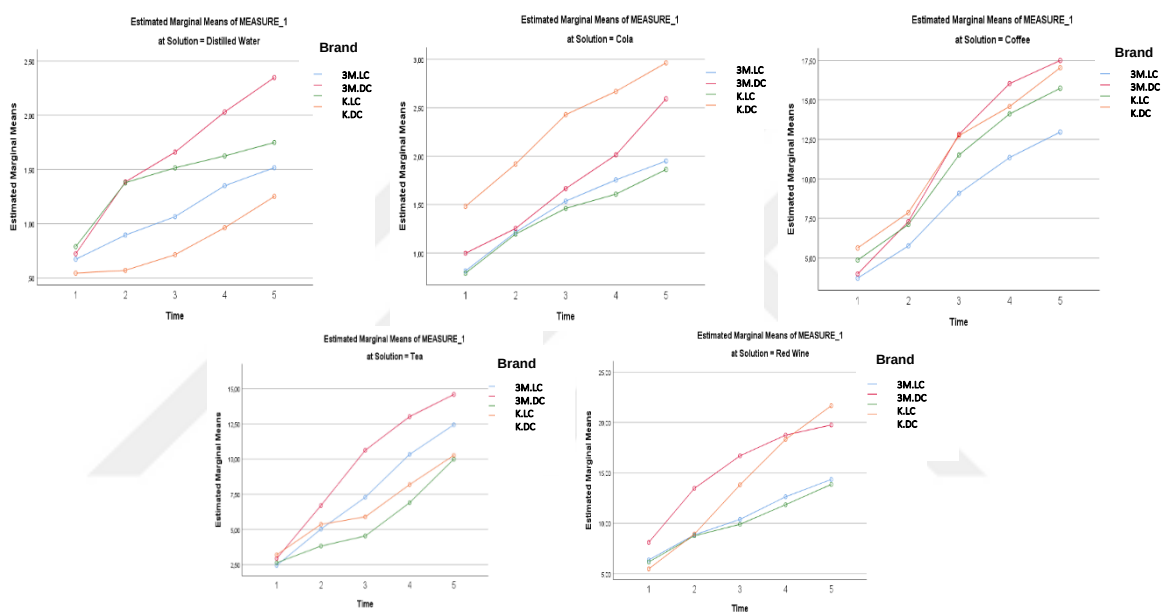


Figure 41. Color difference (ΔE) of resin cements after immersion in distilled water, tea, cola, coffee, and red wine.

4.8. Comparison of Color Stability between the Four Resin Cement Groups 3M.LC, 3M.DC, K.LC, K.DC)

The comparison among the discoloration of all resin cements tested against different staining solutions is shown in Table 12.

Table12. Discoloration of resin cements against different staining solutions

Staining solutions	Discoloration, based on higher ΔE values after staining with respective solution
Red wine	3M.DC > K.DC > 3M.LC, K.LC
Coffee	3M.DC, K.DC, K.LC, > 3M.LC
Tea	3M.DC > 3M.LC > K.LC, additionally 3M.DC > K.DC
Cola	No significant difference observed among groups
Distilled water	No significant difference observed among groups

The > symbol indicates statistical significance ($P < 0.05$).
See Table 2 for definition of material codes.

When 3M.LC, 3M.DC, K.LC and K.DC resin cements were compared, it was observed that the lowest color change (ΔE) was in **distilled water** and **cola** through the whole immersion period (Table 4). There was no statistically significant difference between any brand in distilled water and cola ($p > 0.05$).

When comparing color stability of brands for **tea** solution, there was no statistically significant difference between 3M.LC and K.DC and between K.LC and K.DC groups ($p > 0.05$). There were statistically significant differences in other paired comparisons. Ranking from high to low of ΔE was **3M.DC > 3M.LC > K.LC and additionally 3M.DC > K.DC in tea** (Table 12).

When comparing color stability of brands for **coffee** solution, there was a statistically significant difference between 3M.LC and all other brands ($p < 0.05$). There were also statistically significant differences in other paired comparisons. Ranking from high to low of ΔE was **3M.DC, K.DC, K.LC, > 3M.LC in coffee** (Table 12).

Red wine was the solution that caused the most staining for all resin cements tested. Ranking from high to low of ΔE values was **3M.DC > K.DC > K.LC, 3M.LC in red wine** (Table 12). There was no statistically significant difference between 3M.LC and K.LC specimens ($p > 0.05$). There were statistically significant differences in other paired comparisons (Table 12).

The ΔE values observed in the resin cement specimens immersed in **distilled water** and **cola** varied between 0.54 and 2.96. Hence, all the materials in both solutions (distilled water and cola) did not present a clinically “marked” discoloration ($NBS < 3$) through the whole immersion period (Table 4).

On the other hand, the 3M.LC, K.LC and K.DC specimens immersed in **tea** demonstrated a clinically “marked” discoloration ($NBS > 3$) from 72 hours of immersion (T2), and their ΔE values were 5.03, 3.82 and 5.36, respectively. 3M.DC specimens showed "much" color change from 72 hours immersion in tea. And these ΔE values gradually increased with each measurement during the test period (Table 4).

When immersed in **coffee** and **red wine**, all resin cements demonstrated a clinically “marked” discoloration ($NBS > 3$) after 24 hours of immersion (T1), having the following ΔE values; 3.72, 3.99, 4.86 and 5.63, respectively in the **coffee** group, and 6.37, 8.10, 6.16 and 5.48, respectively in the **red wine** group (Figure 37, 38, 39, 41). And these ΔE values gradually increased with each measurement during the test period (Table 4).

When Table 4 and 12 are examined, it can be concluded that in general, light-cured cements undergo less color change against staining solutions compared to dual-cured cements.

In summary, at the end of the 288 hours (360 day consumption of the drink) all resin cements in water and cola did not show any discoloration that could be detected clinically ($NBS < 3$). However, all cements demonstrated clinically marked discoloration that ranged from “much” to “very much” when immersed in other 3 staining solutions (Table 4).

5. DISCUSSION AND CONCLUSION

As the esthetic demands of patients have increased in the recent years, all-ceramic restorations started to be widely preferred for the restoration of both anterior and posterior teeth owing to their natural and esthetic appearance. All-ceramic restorations such as feldspathic ceramics and glass-ceramics with a high degree of translucency, allow the transmission and dispersion of light, specifically at small thicknesses. Therefore, the dental substrate plays a very important role on light transmission as well as initial color and color stability of luting cements (4).

Resin cements are used for cementing all-ceramic restorations as they contribute to favorable esthetics and superior retention as well as fracture resistance of the restorations (3,6). However, resin cements may undergo intrinsic or extrinsic discoloration. Intrinsic factors are dependent on the material. External discoloration is considered an essential factor influencing the color stability of composite resins. Luting resin cements at the cervical margin of the restoration are in contact with the oral environment and may undergo discoloration due to the adsorption and absorption of media, including food and beverage dyes, and smoking (9,6). If the resin cement changes color, it can become visible; thus, the shade match of the restoration with the adjacent teeth deteriorates leading to the necessity of restoration replacement. (4). Few studies (3,6,11) have investigated the color stability associated with resin cements in daily consumed beverages. Therefore, color stability of four adhesive resin cements against various beverages were evaluated and the obtained values were then evaluated in the present study. Based on the results, storage in some drinks influenced the color of resin cement materials, therefore, the null hypothesis that the immersion solution and the type of resin cements have no influence on the color change of resin cements was rejected.

Color changes can be evaluated either visually or by using instrumental techniques. Instrumental measurements are advantageous because they exclude the subjective interpretation of visual color comparison; spectrophotometers or colorimeters are used in lieu of visual evaluation (59,60). Instrumental measurement is also more accurate compared to the naked eye in measuring slight differences in colored objects situated on flat surfaces (49,61).

Colorimeters are instruments that resemble the spectral function of the standard observer's eye by filtering light in three or four areas of the visible spectrum to detect the color of an object and are manufactured to measure color in the same way the human eye perceives. One possible drawback associated with these instruments is that they take

measurement using the same light source and illumination methodology, therefore, measurement circumstances will be the same whether it is day or night and indoors or outdoors. Also, it is not possible to identify and make a quantification of illuminant metamerism (48, 52).

Spectrophotometers can be considered as practical, and flexible instruments that allow overall color matching in dentistry. These instruments have the ability to measure spectral features of the light and calculate the tristimulus values based on equations for the CIE Standard Observer functions. Instruments using the spectrophotometric method not only display numerical data in various color spaces, but they can also show spectrophotometric method directly. This provides a very detailed information about the object to be examined (48,52). Spectrophotometer is best used in research as it allows an object to be placed inside the spectrophotometer and exposed to light from many different angles and directions, producing the most accurate and precise spectral analysis of the properties reflected by the object.

Therefore, a spectrophotometer was utilized for the measurement of the color differences in the present study to eliminate potential subjective errors in color assessment (62,63).

Color changes were described using the Commission Internationale d'Eclairage $L^*a^*b^*$ color space (CIE $L^*a^*b^*$). The CIE $L^*a^*b^*$ color space is currently one of the most preferred and extensively used color spaces and it is quite suitable for the determination of small differences in color (64). The advantage of the CIE $L^*a^*b^*$ system is that differences in color can be expressed in units which can be correlated with visual perception and clinical significance (50).

Color difference (ΔE) was calculated using a previously indicated formula (Sec. 3.6). In dental literature, many authors (3,4,9,65-68) used ΔE values for the evaluation of color difference perceptibility. On the other hand, it should be mentioned that the perceptibility criteria preferred by each author may differ. Johnston and Kao (69) and Ertaş *et al.* (70) suggested that ΔE value of 3.7 is unacceptable due to the fact that it is clinically perceptible. Turgut and Bagis (11), on the other hand, reported that ΔE values lower than 3.5 were clinically acceptable, while Ruyter *et al.* (71) reported that color changes with ΔE values lower than 3.3 were acceptable. To eliminate such differences and disagreements in the criteria used, the NBS rating system is frequently used to identify the degree of color difference. This system provides definite criteria by which ΔE values can be converted to remarks having clinical significance (72). Consequently,

in the present study, NBS units were calculated to identify the color differences caused by exposure time and/or solution type. According to NBS rating system, when the NBS value is more than 3, a clinically "marked" color change is observed. Based on the NBS rating system, NBS = 3 was accepted as the threshold value and values above 3 were considered clinically unacceptable in the study. Thus, the $\Delta E = 3.3$ value accepted by Ruyter *et al.* (71) was met ($\text{NBS units} = \Delta E \times 0.92 = 3.3 \times 0.92 = 3.036$).

While preparing resin cement samples in this study, similar to previous studies (9,65,66,73) the resin cements were injected into the molds that have been placed on a glass slab and covered with a microscope slide glass with Mylar strips in between. The use of a Mylar strip not only provides a smooth surface finish (26), but also eliminates the possibility of an uncured layer of resin on the surface (74).

The specimens were light-cured using a Light-Emitting Diode light unit having an intensity of 1000mW/cm^2 - 1700mW/cm^2 for 40 seconds. It is already known that all acrylate-based resin materials used in dental profession form an oxygen inhibited surface layer when cured in the air. Therefore, they were ground and polished sequentially using a series of SiC grinding sheets (#600, #1000, #1500, #2500) to a high gloss finish in order to eliminate the oxygen-inhibited layer similar to the grinding and polishing procedure performed by Yu *et al.* (9) and Bagheri *et al.* (73). This layer might be present in the clinical situation because it is difficult to perform the polishing procedure on the cervical margins of the restoration. Therefore, it would stain much more easily than expressed by the findings of the present study and this result is consistent with Shiozawa *et al.* (6).

Some of the previous studies (3,6,26,66,70) used a thickness of 2 mm, which is considerably higher compare to clinical circumstances. Similar to Schneider *et.al.* (65) and Yu *et al.* (9), the final thickness of the resin cement specimens in this study was found to be 0.5 mm, which is closer to clinical thickness. This amount of thickness, which is slightly higher than in normal clinical conditions, was necessary to facilitate the specimen preparation.

Prior to performing the baseline spectrophotometric measurements, the specimens were stored in distilled water at 37°C in the incubator for 24 hours, similar to previous studies (3,6,9,13,26,65,70). Before each measurement was conducted, the specimens were washed under running water without brushing for 10 seconds and then blotted dry with paper towel similar to the study performed by Bagheri *et al.* (73).

Following 24 hours of immersion, the baseline color measurements of each specimen were performed by using a spectrophotometer.

Many studies examined the color stability of resin based composite materials in various beverages such as coffee (26,59,66,70,73,75-81), black tea (20,26,59,66,70,73,75,77,78,81), red wine (20,26,70,73,75-77,81) and cola (26,70,73,75,78,79,81). These beverages are frequently consumed in people's daily diet and have the potential to cause staining in restorative materials used in dentistry. Therefore, the solutions used in the study were distilled water, cola, tea, coffee, and red wine.

The specimens were immersed in the vials containing each solution tested for a total of 288 hours similar to the study performed by Yu *et al.* (9). All the vials were placed in an incubator at 37°C to imitate the oral environment. After each measurement, similar to Shiozawa *et al.* (6), all the solutions were renewed every 24 hours.

It is reported that the average consumption time of 1 cup of coffee is 15 minutes and 3.2 cups of coffee is consumed per day on average (9,26). Therefore, assuming that 3.2 cups of coffee are consumed per day, the exposure time to coffee is 48 minutes each day. It can thus be calculated that the specimen will be exposed to coffee for 1440 minutes (24 hours), which is equal to 30 days of consumption. In the present study, similarly calculated, color measurements of specimens were made 24 hours (T1: 1 day: 30-day consumption of the drink), 72 hours (T2: 3 days: 90-day consumption of the drink), 144 hours (T3: 6 days: 180-day consumption of the drink), 216 hours (T4: 9 days: 270-day consumption of the drink), and 288 hours (T5: 12 days: 360 day consumption of the drink) after they were immersed in solutions. Namely, the immersion time was set to 288 hours of immersion (nearly 1 year consumption) in this study, as was used in a previous study conducted by Yu *et al.* (9).

During the whole experiment, while the ΔE values of dual-cured resin cement specimens exposed to red wine and coffee, which are the highest staining solutions, ranged from 17.03 to 21.66, those of light-cured cements ranged from 12.96 to 15.70. Therefore, it can be concluded that the light-cured resin cements possessed less susceptibility to staining and color change compared to dual-cured cements tested in the present study. However, the staining values of both cements remained above clinically acceptable levels. This finding was consistent with the results of previous studies (4,9,13,82) which investigated the color stability of resin cements.

In order to evaluate the effects of staining solutions on the color, translucency, substance loss, and surface roughness of contemporary adhesive resin cements. Yu *et al.* (9) utilized six adhesive resin cements (Clearfil SA Cement, Panavia F2.0, Rely-X U200,

Rely-X Veneer, NX-3 Light Polymerize, NX-3 Dual Polymerize) in their study. They subjected the specimens to a cyclic staining model having 4 different solutions (deionized water, cola, coffee, and red wine). All specimens were immersed in their solutions 12 times for 15 minutes daily over a period of 96 days. The specimens were preserved in artificial saliva between staining cycles. The color parameters, surface profile, and surface roughness of the specimens were calculated before and after cyclic staining for a period of 16, 32, 48, 64, 80, and 96 days. They found out that all the tested materials displayed similar behaviors with longer periods of immersion within the staining solutions and the light-polymerized adhesive resin cements were generally more color stable compared to dual-polymerized resin cements. Their findings are consistent with the present study.

Pissaia *et al.* (82) evaluated the influence of different shades and time of aging on the color stability of 2 light-cured and 2 dual-cured resin cements. The color measurements were performed right after sample preparation and at 7th, 30th and 90th days of aging in distilled water. They stated that as aging time progressed, there was an increase in ΔE values and the mean values of ΔE were found to be lower for both light-cured resin cements than the dual-cured cements. Similarly, in our study, the ΔE value of light cured cement samples immersed in distilled water ($\Delta E=1.75$) was lower than that of dual-cured cement samples ($\Delta E= 2.35$) at the end of 288 hours.

Pissaia *et al.* (4) studied the effect of curing mode and resin cements shade on the color stability ceramic veneers of minimum thickness following a three-year preservation time in distilled water. They luted ninety six 0.5-mm-thick feldspathic ceramic veneers (Mark II) onto resin composite substrates (Filtek Z350 XT, shade A2E) using 2 light-cured (NX3 Light cure and AllCem Veneer) and 2 dual-cured resin cements (NX-3 dual-cure and AllCem) of various shades. Color measurements were performed with a spectrophotometer at the following times: 1h and 24h; 7, 30, and 180 days; and 1, 2, and 3 years. According to the authors, light-cured resin cements were less affected by color change compared to dual-cured cements. Therefore, this result is consistent with our study. On the other hand, at the end of the 2 years, Pissaia found out that all cements showed ΔE values which are above the acceptable threshold. This result is conflicting with the present study and can be explained by the prolonged immersion procedure performed by Pissaia *et al.* (4).

Ural *et al.* (13) investigated the short-term (4 weeks) color stability of light-cure and dual-cure resin cements. Sixty disc-shaped test specimens of adhesive resin cement (10×1 mm) were prepared. One feldspathic porcelain specimen was prepared using a

prefabricated ceramic block. The feldspathic sample was placed on the resin cement disc and all the measurements were conducted without cementation. Specific differences in coordinates and total differences in color (ΔE) were calculated following immersion in distilled water for various periods. Results revealed that the light-cured resin cements had lower ΔE values compared to dual-cure cements with no statistical significance, which is consistent with the result of the present study when the distilled water groups are evaluated.

To evaluate the influence of food colorants on the color stability and water absorption of frequently used resin cements, Akay and Tanis (3) stored 3 commercially available resin cements (2 self-adhesive resin cements and 1 dual/light curing resin cement) in 0.15% erythrosine, dark brown and sunset yellow for 30 days. They concluded that the food colorant solutions used for immersion procedure influenced the color stability as well as water absorption of different resin cement materials. Nevertheless, this result coincides with the present study because the used staining beverages affected the color stability of the tested resin cement too and resulted in increased discoloration of the specimens.

Liebermann *et al* (68) analysed the long-term color stability of eight self-adhesive composite resin cements (BeautyCem, Bifix SE, Clearfil SA Cement Automix, RelyX Unicem 2 Automix, SeT, SmartCem 2, SoloCem, SpeedCEM) after storage in red wine, curry-solution, cress-solution, and distilled water for up to one year. Spectrophotometric measurements were performed after 7, 28, 90, 180, and 365 days of immersion. Significant ΔE differences were found between all storage media ($p < 0.001$), with highest values obtained for red wine, followed by curry-solution, cress-solution, and distilled water. In the present study, there was a statistical significance between all the storage solutions except between cola and distilled water. Red wine caused the highest ΔE values similar to Liebermann *et al*. (68).

Shiozawa *et al*. (6) evaluated the color stability of contemporary adhesive resin cements following exposure to coffee. They examined 4 dual-cured resin cements (Clearfil SA cement Automix Universal, Maxcem Elite Clear, Maxcem Elite Yellow, and RelyX Unicem2 Automix A2) and 2 chemical-cured resin cements (Super-Bond C&B Clear and Super-Bond C&B Esthetic). The spectrophotometric measurements for color difference (ΔE) were performed on a white background after 1-day and 1-week immersion in coffee and water. They found that the ΔE values after coffee immersion were significantly higher compared to those after water immersion, except for Super-

Bond C&B Esthetic. The ΔE values after water immersion were not significantly different among the products tested. These results match the results of the present study.

Nathanson and Banasr (83) investigated the color stability of several commercially available resin cements after accelerating aging for 14 weeks. The specimens were divided into 2 groups: 1) cement samples bonded to ceramic 2) noncovered cement samples. Color measurements were conducted at the baseline and after 1 week, 2 weeks, 4 weeks, 6 weeks, and 14 weeks of accelerated aging. They found out that all cements aged up to 4 weeks had ΔE below 3.3, and this is consistent with the present study when all the resin cements were immersed in distilled water; they produced low ΔE below 3.3 after 288 hours of immersion.

In the present study, both resin cements and solution factors had a significant effect on color change over time. The color stability of all resin cements was highly affected by the period of immersion. Accordingly, the color changes (ΔE) of resin cements intensified with increasing immersion period. These findings were consistent with the results of some previous studies (6,9,66,68,82,84-86).

At the end of the study (after 288 hours: approximately equal to 360 days of drink consumption), all test group specimens (immersed in cola, tea, coffee, red wine) demonstrated higher ΔE values compared to control group specimens (immersed in distilled water) and this is in line with the results obtained by Yu *et al.* (9). Yu *et al.* (9) reported that the tested materials displayed varied degrees of resistance to coloring solutions, which is consistent with the results of the present study.

At the end of 288 hours, the ΔE values after exposure to the tea, coffee and red wine ranged from 9.99 to 21.66. According to the NBS rating system, all tested resin cements demonstrated clinically marked discoloration that ranged from “much” to “very much”. However, at the end of the same test period, Yu *et al.* (9) obtained lower ΔE values (3.46-12.25) compared to the results of our study when the samples were immersed in coffee and red wine. This can be attributed to some differences in the methodology applied. The specimens were exposed to cyclic staining procedure. Following 12 daily cycles, the specimens were brushed with toothpaste for 1 minute and then preserved in artificial saliva overnight. While in the present study, the specimens were stored in artificial saliva for 30 minutes only after each measurement and a prolonged staining method was used instead of the cyclic staining and no brushing or polishing procedures were performed after staining the specimens.

In this study, distilled water showed color changes that were imperceptible and clinically acceptable. Similar results were reported by some previous studies (6,70,82,87,88). On the contrary, Alkhadim *et al.* (66) reported perceivable color changes in the distilled water groups and attributed the reason to the chemistry of the resin material that led to some intrinsic color changes. Ergucu *et al.* (74) and Burrow and Makinson (89) stated that water sorption by itself was not a factor that cause extensive discoloration of composites. In the present study, control specimens exposed to distilled water produced similar and clinically acceptable color changes in all materials tested for 288 days and similar results were reported by Shiowaza *et al.* (6) and Yu *et al.* (9).

In this study, cola groups did not show clinically marked discoloration. Throughout the whole experiment, the color change caused by cola ranged from 1.86 to 2.96. No statistical significance was noted between the influence of distilled water and cola. This result was consistent with a study done by Arocha *et al.* (75), where they attributed the reason to the low staining potential of the constituents. Similarly, Mundim *et al.* (90) indicated that although that cola contains phosphoric acid, it did not cause any color change in the composite resins. In previous studies by Ertas *et al.* (70), Bagheri *et al.* (73) and Mundim *et al.* (90), it was concluded that the staining effect of coffee was more intense compared to cola, and these results are consistent with our study.

In the present study, immersion of the resin specimens in tea showed discoloration and the ΔE values of specimens immersed in tea ranged from 9.99 to 14.58 for all resin cement groups. Samples immersed in tea showed greater discoloration than samples immersed in cola. This finding was consistent with studies done by Ertas *et al.* (70), Bagheri *et al.* (73) and Afzali *et al.* (88).

Chan *et al.* (62) evaluated the staining potential of coffee, tea, cola, and soy sauce on two resin composites and indicated that following 6 weeks of immersion, coffee and soy sauce caused discoloration of composite resin restorations to a significantly higher degree compared to tea or cola, which is consistent with our study's findings for tea and coffee solutions.

In some previous studies (66,70) it has been reported that there was no a statistically significant difference between the ΔE values of various composite resins immersed in tea and coffee. However, in the present study, there was a high statistical significance between the ΔE values of resin cement groups immersed in tea and coffee.

Both tea and coffee contain yellow colorants which possess different polarities. Higher polarity components (like those in tea) elute first, while lower polarity

components (like those in coffee) elute later. Discoloration by tea because of the adsorption of polar colorants onto the surface of resin composite materials can be removed by tooth brushing. On the other hand, discoloration caused by coffee is because of both absorption and adsorption of polar colorants. This adsorption and penetration of colorants into the organic phase of the materials were explained by Um and Ruyter (59) and Ertaş *et al.* (70) as possibly due to compatibility of the polymer phase with the yellow colorants of coffee. Previous studies (26,91,92,93) also supported the view that static immersion of composite resin discs in coffee may cause color change which is associated mainly with adsorption on composite resin surfaces.

Moreover, Um and Ruyter (59) defended that lack of yellow colorants in cola solution is the reason for exhibiting lower levels of discoloration compared to tea and coffee which contained yellow colorants with different polarities.

The highest level of discoloration in the study was observed in red wine with ΔE values ranging between 13.85 and 21.66 followed by coffee, tea, cola, and distilled water. Similar results were found in the previous studies (17,68,75,84,85). Studies supporting the highest rate of discoloration associated with red wine are available (9,20,26,70,77,85).

In the present study, at the end of 288 hours immersion, color change (ΔE) values for all resin composite restorative materials immersed in tea ($\Delta E=9.99-14.58$), coffee ($\Delta E= 12.96-17.49$) and red wine ($\Delta E= 13.85- 21.66$), were greater than 3.3. These color change values can be determined by the human eye; therefore, they are unacceptable clinically. This finding confirms the study done by Ertaş *et al.* (70).

In this study, after staining for 288 hours, all materials (except those immersed in distilled water and cola) displayed clinically marked color changes ($\Delta E>3.3$, $NBS>3$). Similarly, Kumar *et al.* (86) reported that the ΔE values of resin composite restorative materials were less than 3.3 after 24 hours of immersion in cola.

In the present study, resin cements immersed in tea exhibited clinically perceptible color changes ($\Delta E>3.3$ and $NBS>3$) from 72 hours of immersion, while clinically perceptible color changes ($\Delta E>3.3$) were observed in the first 24 hours when exposed to coffee and red wine.

On the other hand, Alkhadim *et al.* (66) reported that all materials tested had ΔE values greater than 3.7 after 2 weeks of immersion in coffee and tea. This can be attributed to the fact that Alkhadim *et al.* (66) used resin composites (Z250XT, IPS Empress-Direct, G-aenial, Vit-l-escence, and Ceram.X) in their study. Liebermann *et al.* (70) reported that

all the specimens exposed to the immersion solution (red wine, curry solution, cress solution, distilled water) exhibited clinically unacceptable discoloration ($\Delta E > 3.3$). Although Liebermann *et al.* (68) obtained clinically unacceptable discoloration after 365 days, in the present study, we achieved the same conclusion within 12 days of immersion.

According to Ertas *et al.* (70), the value of color change (ΔE) for all resin composite restorative materials in tea, coffee, and red wine were higher than 3.7.

In the present study, resin cements immersed in tea displayed clinically perceptible color changes ($\Delta E > 3.3$ and $NBS > 3$) from 72 hours of immersion, while clinically perceptible color changes ($\Delta E > 3.3$) were observed in the first 24 hours when exposed to coffee and red wine. These ΔE values obtained after 288 hours of immersion in tea, coffee and red wine for all resin cement samples are compatible with many *in vitro* studies. These ΔE values (9.99-21.66) are quite high color change values. Possible reasons for this finding can be explained as follows: The samples were rinsed only with distilled water and preserved in artificial saliva for 30 minutes following every measurement in the present study. The effects of tooth brushing or thermal cycling were not simulated throughout the experiment. However, oral hygiene procedures are usually done twice a day, creating a kind of polishing that almost completely removes the color particles and food ingredients attached externally and creates resistance to an inherent discoloration (92). On the other hand, the prolonged static immersion of the resin in the colorant beverages does not reveal the natural state of human drinking and eating, since nutrition is a dynamic process in which fluid is not held statically in the oral cavity. Furthermore, according to Bagheri *et al.* (73), the presence of saliva and other fluids in the oral cavity reduces the effect of stains by diluting them, hence, the actual staining in the oral cavity occurs after longer periods of intermittent exposure to staining food and beverages. Additionally, covering resin cements with a ceramic layer would represent the discoloration in the oral cavity in a more specific way (2). Therefore, the limitation of the study is that this methodology mimics oral conditions to some extent. Future studies are warranted to evaluate the effects of these factors on the color stability of adhesive resin cements.

Considering the limitations of this *in-vitro* study, the following conclusions can be drawn:

1. The daily beverages (water, cola, tea, coffee, red wine), type of the resin cements tested (Rely-X Veneer, Rely-X Ultimate Clicker, NX-3 Nexus Light-Cure and

NX-3 Nexus Dual-Cure), and the immersion period (up to 288 hours) had a significant effect on the color stability of the resin cements.

2. ΔE values indicating the color change of resin cements showed an increasing trend as the immersion time increased.
3. Among all the staining solutions for each time period, distilled water and cola caused significantly less discoloration in all resin cement specimens than tea, coffee, and red wine.
4. The color changes of the resin cements immersed in distilled water and cola were within clinically acceptable limits ($\Delta E < 3.3$ and $NBS < 3$).
5. Highest discoloration rates were observed for red wine, followed by coffee, tea, cola and distilled water, respectively.
6. Rely-X Ultimate Clicker dual-cured (3M.DC) resin cement specimens displayed the lowest color stability among the 3 other tested resin cement materials.
7. The resin cements immersed in tea produced clinically marked discoloration from 72 hours of immersion ($\Delta E > 3.3$, and $NBS > 3$), while the resin cements immersed in coffee and red wine produced a clinically marked discoloration from the first 24 hours of immersion.
8. At the end of immersing for 288 hours, all resin cements reached clinically marked discoloration that ranged from “much” ($NBS > 6$) to “very much” ($NBS > 12$) when immersed in tea, coffee, and red wine.
9. While the ΔE values of dual-cured resin cement specimens immersed in wine and coffee, which are the highest colorant solutions, ranged from 17.03 to 21.66, those of light-cured cements ranged from 12.96 to 15.70. Therefore, it can be concluded that the light-cured resin cements had less susceptibility to staining and color change compared to dual-cured cements tested in this study. However, the staining values of both cements remained above clinically acceptable levels.
10. Since the margin discolorations of all-ceramic restorations are diet-related, patients should be warned about the use of colorant beverages and foods, and about the importance of oral hygiene habits to minimize discoloration.
11. Dentists should pay attention to careful management of the perfect fit of the restoration, applying the correct cementation procedure, and re-polishing the restoration margins with recall sessions.

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7. CURRICULUM VITAE

Personal Informations

Name	Leila	Surname	Mostawe
Place of Birth		Date of Birth	
Nationality		TR ID Number	
E-mail		Phone number	

Education

Degree	Department	The name of the Institution Graduated From	Graduation year
Master	Prosthodontics	Yeditepe University	2021
University	Bachelor of Dental Surgery (BDS)	R.A.K. Medical and Health Sciences University	2018
High school	Scientific Section	Al-Massara Private High School	2013

Languages	Grades
Arabic	Native Proficiency
English	Toeﬂ 76
Turkish	Tomer C1

Computer Skills

Program	Level
Microsoft Office; Word, Power Point and Excel	Good

Scientific works

Articles published in other journals

Being a left-handed dentist: boon/ﬂaw? A survey in dental colleges around the U.A.E. Leila Mostawe, Amneh Darwish, Sabrin Ali Azim Journal of Medical Case Reports and Reviews 2:6 (2019).
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Proceedings presented in international scientific meetings and published in proceedings book.

Rehabilitation of fully edentulous patient with two different Prosthetic treatment plans: case report (3 rd place award) Poster presentation: Erdis International Congress, 2020, Kayseri, Turkey

