Original Article

The Evaluation of the Colour Changes of Traditional Composites, Ceramic Blocks and CAD/CAM Composites in Different Solutions

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ABSTRACT

Aims: The aim of this study is to investigate the color changes of three different traditional composites, one ceramic and two resin-based composites CAD/CAM blocks in different solutions. **Methods:** The materials used in the study were CAD/CAM block containing lithium disilicate glass ceramic (Ivoclar), Vita Enamic containing resin (VITA), Lava Ultimate Block containing resin (3M ESPE), G-aenial anterior composite (GC,), Filtek™ Ultimate Universal composite (3M ESPE) and Clearfil Majesty Esthetic composite (Kuaray). As colouring solutions, red wine (Buzbağ), black tea (Lipton), coffee (Nescafe) and distilled water (EAU distillee) were used. For the preparation of the traditional composite samples to be used in the study, 7 × 7 mm square-shaped plexiglass moulds, 1.2 mm in thickness, were used. The CAD/CAM blocks with ceramic and resin content were cut at the same thickness using a Struers sensitive cutting device. The samples were then randomly separated into grups of 10 and of the 240 samples, groups were separated into 6 different materials and 4 different solutions. The colour measurements of the 240 samples were taken at baseline, 30 days and 120 days with a Lovibond spectrophotometer (Tintometer). **Results:** A statistically significant difference was determined between the materials in respect of the ΔE values in the 30-day solution groups ($P < 0.05$). No statistically significant difference was determined in the ΔE values of the different materials in the 30-day and 120-day distilled water groups ($P > 0.05$). A statistically significant difference was determined between the materials in respect of the ΔE values in the 120-day solution groups ($P < 0.05$). **Conclusion:** In respect of discolouration, ceramic blocks are more successful. Resin-based blocks and traditional aesthetic composites showed more discolouration. The dietary habits of the patient should be taken into consideration in the selection of the restorative material.

Keywords: *CAD/CAM blocks, coloration, composite resins*

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Introduction

Aesthetic restorations are currently one of the primary requests of patients. There are many aesthetic materials which have been produced and used successfully in recent years, of which the leading material is composite resins. One of the most important criteria affecting the success of restoration is the long-term durability of aesthetics in dental treatments.[1]

The ability of the material to resist staining of internal and external origin is associated with several factors such as the oral hygiene of the patient and the frequency of consuming discolourants such as tea, coffee, and cigarettes.[2] It has been shown in previous studies that composite resins cannot maintain their existing aesthetic structure over time because of discolouration.[3]

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Since the entry of composite resins into the dentistry market, the formulation has undergone many physical and mechanical changes. Insufficient and incomplete polymerisation has been reported to decrease mechanical properties and increase colour sensitivity.[4] Improvements in the polymerisation rate can be guaranteed with prefabricated blocks with computer-assisted design/computer-assisted production (CAD/CAM). To provide optimal physical and mechanical quality, these blocks are industrially polymerised under standard parameters of high temperature and pressure.[5]

With developments in CAD/CAM technology, manufacturers have produced PMMA-based blocks in recent years.[6] Examples of these are Vita Enamic in a ceramic network structure infiltrated by felspathic polymer containing 86% ceramic by weight, and Lava Ultimate, which is a resin nano-ceramic material with 80% by weight silica and zirconia nanoparticles and nano-clusters.[7] In comparison with other restorative materials, these blocks have been reported to show values close to those of natural teeth in respect of hardness, flexible resistance and elasticity modulus.[8]

IPS E-max CAD blocks have been produced for CAD/CAM systems, and have the same chemical structure as IPS E-max Press. The difference is partial crystallisation with heat processing, the aim of which is to provide rapid and easy shaping of the blocks and to obtain sufficient resistance to ceramic. The basic crystallisation phase is lithium metasilicate (Li_2SiO_3). With a length of 0.2 to 1 µm, lithium metasilicates are present at the rate of 40% by volume.[9] Thus the ceramic structure acquires resistance up to 130 MPa and after the filing process, the lithium metasilicate crystals are transformed to lithium silicate crystals by oven processing at 850°C. With this transformation, a lithium disilicate crystal ceramic structure is produced at mean 1.5 µm dimension in the CAM matrix and which regresses at the rate of 70% by volume.[10]

Under current conditions, many methods are used to determine tooth colour. These may be subjective comparisons using scales of colour tones of porcelain or acrylic resins, or objective techniques which use spectrophotometry, colorimetry or image analysis techniques.[11] A spectrophotometer is a device that measures the permeablity, reflection and absorption of colour.[12] When taking the measurement, the result is reached by totalling all the light energy reflected in all the wave lengths perceptible to the human eye, in other words, in the 380-720 nm range, and therefore net results are given.[13]

The CIE Lab Delta E (ΔE) value in the spectrophotometry system is the numerical value showing the degree of colour difference perceived of the two parts.[14] When the rules are taken into consideration, if no colour change is determined in a material test environment, the colour integrity is stable and $\Delta E = 0$. In many studies, more than one cutoff value has been reported of the sensitivity of the human eye to colour change.[15] Esmaeili *et al.* showed a ΔE value of ≤ 3.3.[16]

The aim of this study was to examine the colour changes that can occur in daily life with the consumption of red wine, tea and coffee and a control group of distilled water in 3 different traditional composites, 2 resin-based and 1 ceramic CAD/CAM blocks, which have different physical and chemical properties in their content and are routinely used in clinics.

Method

The materials used in the study were CAD/CAM block containing lithium disilicate glass ceramic (Ivoclar, Vivadent AG, Switzerland), Vita Enamic containing resin (VITA Zahnfabrik, Bad Säckingen, Germany), Lava Ultimate Block containing resin (3M ESPE St Paul, MN, USA), G-aenial anterior composite (GC, Tokyo, Japan), Filtek™ Ultimate Universal composite (3M ESPE St Paul, MN, USA) and Clearfil Majesty Esthetic composite (Okayama, Japan). The materials used are shown in Table 1.

As colouring solutions, red wine (Buzbağ klasik Elazığ-Diyarbakır), black tea (Lipton yellow label, UK), coffee (Nescafe classic) and distilled water (EAU distillee, Istanbul) were used.

For the preparation of the traditional composite samples to be used in the study, 7 × 7 mm square-shaped plexiglass moulds, 1.2 mm in thickness, were used. A2-coloured samples of the composite groups were placed into the moulds, covered with a clear band, then a glass slide was placed and with finger pressure, any overflowing restorative material was removed. Light was applied with an LED Light Filling Device for a total of 40 seconds as 20 secs on each side.

The CAD/CAM blocks with ceramic and resin content were cut at the same thickness using a Struers sensitive cutting device (Struers Aps Pederstrupvej, Denmark). A polishing procedure was applied to all the groups for 10 seconds with an Optidisk device (KerrHave SA, Bioggio, Switzerland).

The samples were then randomly separated into groups of 10 and of the 240 samples, groups were separated into 6 different materials and 4 different solutions. All the samples were then left for 24 hours in an MST series incubator at 37°C for water absorption to occur. After taking the first measurements, the samples of each group were placed so that they were covered in 5mlt of distilled water, tea, coffee and red wine. The colour measurements of the 240 samples were taken at baseline, 30 days and 120 days with a Lovibond spectrophotometer (Tintometer, India). The solutions were changed every 10 days.

The distilled water and red wine used in the study were added directly to the samples without any other procedure. For the coffee solution, the instant coffee was added to 200mlt of 100°C water according to the instructions for use and was stirred immediately and after 5 mins, then added to the samples. For the tea solution, a teabag was placed in 200mlt of 100°C water according to the instructions for use, was lightly shaken immediately and after 2 and 5 minutes. The teabag was then removed and the solution was added to the samples.

Statistical analysis

Data were analysed statistically using the Kruskal Wallis H-test, and when signifcant differences were determined in that test, the *Post hoc* Multiple Comparison test was used. A value of $P < 0.05$ was considered statistically significant.

Results

The ΔE values of the groups in red wine, tea, coffee and distilled water at 30 days and 120 days are shown in Table 2. In the 30-day tea solution group, the ΔE values of the IPS E-max material group were significantly lower than those of the 3M Filtek Ultimate and the Lava Block material groups, and the ΔE values of the Clearfil Majesty Esthetic and Vita Enamic groups were significantly lower than those of the Lava Block material group ($P < 0.05$).

No statistically significant difference was determined in the ΔE values of the different materials in the 30-day and 120-day distilled water groups ($P > 0.05$).

A statistically significant difference was determined between the materials in respect of the ΔE values in the 30-day coffee solution group ($P < 0.05$). The ΔE values of the IPS E-max and Vita Enamic material groups were significantly lower than those of the 3M Filtek Ultimate, the G-aenial anterior and the Lava Block material groups, and the ΔE values of the Clearfil Majesty Esthetic material group were significantly lower than those of the 3M Filtek Ultimate group.

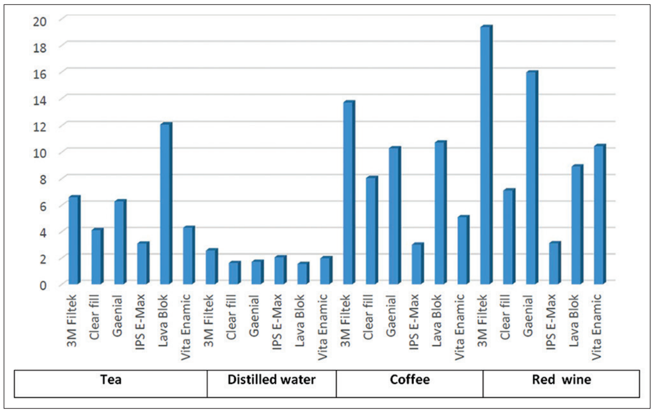
A statistically significant difference was determined between the materials in respect of the ΔE values in the 30-day red wine solution group ($P < 0.05$). The ΔE values of the IPS E-max were significantly lower than those of the 3M Filtek Ultimate, Clearfil Majesty Esthetic and Vita Enamic material groups. The ΔE

Table 1: Restorative materials used in the study

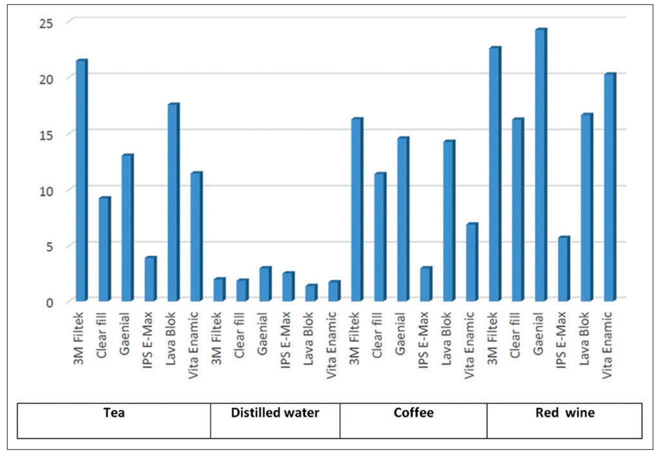
Restorative materials	Material type	Manufacturer	Lot number	Contents
Geanıal Anterıor	Microhybrid	GC, Tokyo, Japan	1610241	Methacrylate monomers, silica, strontium lanthanide fluoride, fumed silica, pigments, catalysts
Clearfil Majesty Esthetıc	Nanohybrid	Kuraray Medical Co. Tokyo, Japan	750178	Bis-GMA, hydrophobic aliphatic dimethacrylate silanized barium glass and nano fillers, Average particle size; 0.7 micron, 78% by weight
3m Fıltek Ultımate	Nanofil	3M ESPE, St. Paul, MN, USA	N750660	Bis-GMA, UDMA, TEGDMA, Bis-EMA Silica, zirconium oxide 78.5% by weight
3m lava Block	Resin nano ceramic	3M ESPE, St. Paul, MN, USA	N652686	Resin Nano-ceramic composite block, BISG Up/ TEGDMA, 80% by weight Nanoceramics
Vıta Enamıc	Hybrid ceramic	VITA Zahnfabrik, Bad Säckingen, Germany	54210	Hybrid Ceramic, UDMA, TEGDMA, 86% by weight feldspar ceramics, 14% by weight polymer, 58-63% SiO_2, 20-23% Al_2O_3, 9-11% Na_2O, 4-6% K_2O, 0.5-2% B_2O_3, <1% Zr_2O and CaO
IPS e max	Lithium Silicate Glass Particles	Ivoclar, Vivadent AG, Switzerland	V33396	SiO_2 %57-80, Li_2O %11-19, K_2O %0-13, P_2O_5, % 0-11, ZrO_2 %0-8, ZnO %0-8, others and coloring oxides %0-12)

Table 2: 30 and 120 days Delta E (ΔE) values of the groups

Restorative materials	Distilled water		Tea		Coffee		Red wine	
	Δ 30 Day	Δ 120 Day	Δ 30 Day	Δ 120 Day	Δ 30 Day	Δ 120 Day	Δ 30 Day	Δ 120 Day
3M filtek ultımate	0.324	0,8711	6,2868	21,174	13,6049	16,1335	18,8419	22,2447
Clearfil majesty esthetıc	0,8023	1,1551	3,746	9,1403	7,7296	11,0623	6,9624	16,1728
Geanıal anterıor	0,6723	1,0096	6,0191	12,8535	10,2308	14,4238	15,8791	24,1644
IPS e max	0,5345	0,6463	1,8025	3,5574	2,0583	1,9374	2,7457	5,6274
3m lava block	0,8503	0,976	11,8789	17,3967	10,546	14,1634	8,6979	16,546
Vıta enamıc	0,5428	0,8684	4,1537	11,3559	4,3881	6,4609	10,3044	20,2202



Graphic 1: 30. day Delta E (ΔE) values of the groups



Graphic 2: 120. day Delta E (ΔE) values of the groups

values of the Clearfil Majesty Esthetic group were significantly lower than those of the 3M Filtek Ultimate and the G-aenial anterior group, and the ΔE values of the Lava Block material group were significantly lower than those of the G-aenial anterior material group.

The 30-day ΔE values of the groups in red wine, tea, coffee and distilled water are shown in Graphic 1. A statistically significant difference was determined between the materials in respect of the ΔE values in the 120-day tea solution group ($P < 0.05$). The ΔE values of the IPS E-max material group were significantly lower than those of the 3M Filtek Ultimate, G-aenial anterior and the Lava Block material groups, the ΔE values of the Clearfil Majesty Esthetic were significantly lower than those of the 3M Filtek Ultimate and the Lava Block material groups, and the ΔE values of the Vita Enamic group were significantly lower than those of the 3M Filtek Ultimate group.

A statistically significant difference was determined between the materials in respect of the ΔE values in the 120-day coffee solution group ($P < 0.05$). The ΔE values of the IPS E-max material group were significantly lower than those of the 3M Filtek Ultimate, Clearfil Majesty Esthetic, G-aenial anterior and Lava Block material groups, and the ΔE values of the Vita Enamic group were significantly lower than those of the 3M Filtek Ultimate, G-aenial anterior and Lava Block material groups.

A statistically significant difference was determined between the materials in respect of the ΔE values in the 120-day red wine solution group ($P < 0.05$). The ΔE values of the IPS E-max material group were significantly lower than those of the G-aenial anterior, and the Vita Enamic material groups, and the ΔE values of the Clearfil Majesty Esthetic group were significantly lower than those of the G-aenial anterior material group.

The 120-day ΔE values of the groups in red wine, tea, coffee, and distilled water are shown in Graphic 2.

A statistically significant difference was determined between the solutions in respect of the 120-day ΔE values ($P < 0.05$). The ΔE values on the 120th day of the distilled water group were statistically significantly lower than those of the tea, coffee, and red wine groups, and the ΔE values of the tea and coffee groups were statistically significantly lower than those of the red wine group.

A statistically significant difference was determined between the solutions in respect of the 30-day ΔE values ($P < 0.05$). The ΔE values on the 30th day of the distilled water group were statistically significantly lower than those of the tea, coffee, and red wine groups, and the ΔE values of the tea group were statistically significantly lower than those of the red wine group.

A statistically significant difference was determined between the materials in respect of the 120-day ΔE values ($P < 0.05$). The ΔE values of the IPS E-max material group were significantly lower than those of the 3M Filtek Ultimate, Clearfil Majesty Esthetic, G-aenial anterior, Lava Block, and Vita Enamic material groups.

A statistically significant difference was determined between the materials in respect of the 30-day ΔE values ($P < 0.05$). The ΔE values of the IPS E-max material group were significantly lower than those of the 3M Filtek Ultimate, G-aenial anterior, Lava Block, and Vita Enamic material groups, and the ΔE values of the Clearfil Majesty and Vita Enamic material groups were statistically significantly lower than those of the 3M Filtek Ultimate material group.

Discussion

Colour incompatibility is one of the leading reasons for changing resin-based composite restorations.[17] Of the complaints in dentistry, 38% are due to discolouration.[18] In traditional composite restorations, discolouration is associated with various factors such as insufficient polymerisation, dietary habits, water absorption, chemical reactions, oral hygiene and surface properties.[19] Aluminium oxide discs have been used in many studies, and this has been proven to be the most successful method of obtaining smooth, shiny surfaces.[20] In the current study, the polishing procedure was applied for 10 secs using the OptiDisc polishing system.

In a study by Hamiyet *et al.*, different polishing procedures were applied to CAD/CAM blocks in a ceramic structure with resin content. According to the colouring results, Lava Block manual polishing was said to be more appropriate than the Vita Enamic glaze process. It has been reported that manual polishing or glaze processes can be recommended for ceramic materials.[21] In the current study, to provide the ideal conditions while preparing the samples with standard moulds, clear bands were used to reduce oxygen inhibition and to be able to compare the resin-content blocks with the traditional composites.

In a study by Ligon *et al.*, it was shown that polymerisation could not be completed in the presence of oxygen inhibition. This was observed to then impair mechanical performance and surface integrity.[22]

Complex events occurring throughout the time in the oral cavity will cause changes in the colour of the material within a certain period of time.[2] When the consumption habits of the patient are taken into consideration, especially in respect of drinks, studies that have examined the discolouration of anterior region restorations have generally used distilled water, cola, coffee and red wine.[23] Tea has been used in fewer studies.[24]

In some studies, artificial saliva has been used instead of distilled water. However, artificial saliva does not contain intra-oral enzymes, which cause softening of dimethacrylate polymers on the composite surface and hydrolysis of methacrylate ester connections.[25] Therefore, in the current study, distilled water was used as the control group. In addition to coffee and red wine, a tea solution was used as that has been less examined previously.

CIE L * a * b *, colour difference ΔE, was the value used to evaluate colour changes, and was calculated with a special formula using differences in the L *, a *, b * values. In 2001, the new, updated ΔE00 formula was introduced and recommended by the CIE.[26] In practice, these formulas can be changed[27] and the values can be linked to a high degree.[28] Therefore, in the current study, it was decided to use the well-known ΔE values in the colour measurements.[1,18,29] When making this evaluation, clinical interpretation is important. It is based on the differentiation of colour difference perceptible to the human eye and a statistically acceptable difference. It has been reported that values >3.3 are not clinically acceptable and ΔE < 1.1 cannot be perceived by the human eye.[29]

The water absorption and solubility of composte resins are affected by the duration of testing. In previous studies using tea, coffee, red wine and cola, they have been applied for various periods. Villata *et al.* left samples in a colouring solution for 3 hours a day and in distilled water for 21 hours a day for 40 days.[17] In another study by Dayan *et al.*, samples were immersed in colouring solution for 15 minutes twice a day, and then left in distilled water.[30] Bagheri *et al.* left samples in distilled water for 1 week then in colouring solutions for 2 weeks.[31] As the expected lifespan of direct and indirect restorations is 8-10 years, the immersion period used in the current study was 120 days. As shown by Ertaş *et al.*, clinical ageing of approximately 10 years should be represented.[32] Taking this period into account, the current study was conducted over 120 days.

Based on the test conditions applied in an *in vitro* study by Alharbi *et al.*, the current study results showed that with the exception of distilled water and artificial saliva, the ΔE value of none of the materials tested was <3.3 in any of the solutions. Therefore, if the same condition is applied clinically, the colour change can be perceived by most people and is not clinically acceptable.[33] Similarly, according to the intensity of staining in this study, red wine has a higher staining potential than coffee and tea solutions in all the materials. These results are consistent with those of previous studies.[5,29,34]

It has been reported in several studies that alcohol facilitates staining by softening the resin matrix.[2,35] However, it is not clear whether the colour change is due to the presence of alcohol or pigments in red wine. In tea and coffee colouring solutions, the solution itself is alkaline and does not have a content that can disrupt the structure of the organic matrix.

Water absorption is related to many things in composites. The fillers within the resin matrix, the distribution, size and volume, depend on the silane (intermediate phase) connecting these two. Other factors affecting absorption are time, temperature, surface properties, stress, and solution intensity.[36]

When the organic structures of composite resins are examined, there is generally seen to be Bis-GMA, UDMA, Bis-EMA and TEGDMA. Of these, TEGDMA is the component with the tightest polymer network but the greatest water absorption. UDMA and Bis-EMA absorb less water, but there are more residual monomers. Although Bis-GMA is the strongest of these structures, it absorbs more water than UDMA and Bis-EMA.[37] In the results of the current study, while the G-aenial anterior composite resin showed less discolouration in the tea and coffee solutions at the end of 120 days, compared to the 3M group composite and Lava Block, there was seen to be more discolouration in the red wine solution.

Although Clearfil Majesty Esthetic is a traditional composite, more resistant 120-day results were seen in the the tea and red wine solutions than the other materials with the exception of IPS E-max. In the coffee solution, in addition to IPS E-max, there was seen to be more discolouration than in Vita Enamic. When this is examined in respect of the material content, in addition to the content of G-aenial anterior organic matrix of urethane dimethacrylate (UDMA) and dimethacrylate co-monomers, that it does not include Bis-GMA has been confirmed in previous studies in respect of discolouration. As the 3M group block (Bis-GMA, UDMA, Bis-EMA, TEGDMA) and traditional composites (Bis-GMA, UDMA, TEGDMA, PEGDMA VE Bis-EMA) contain more water-absorbing structures as the resin matrix, greater sensitivity to discolouration was shown, and this is also supported by the findings of previous studies.

When the Clearfil Majesty Esthetic composte resin is examined, it contains Bis-GMA as the organic matrix. However, the resistance to discolouration was extremely good in all the results with the exception of ceramic material and coffee solution. This result seems to be in conflict with previous studies.[33] It can be thought that this result was due to water absorption having been reduced by the content of hydrophobic aliphatic dimethacrylate. In the comparison with the CAD/CAM blocks with resin content, Vita Enamic showed less discolouration than Lava Block at 120 days in all the solutions except red wine, which could be attributed to Vita Enamic not containing Bis-GMA and having a greater proportion of filler. The Ph value of the intra-oral environment and the solution has a negative effect on the resistance to wear of composite resins. This occurs through the removal of inorganic fillers from composite resin.[38]

When the previous results are examined, while colour change with coffee is known to occur with both adsorption and absorption, the colour change from tea is known to be due to adsorption to the surface of the polar colouring materials.[18,29,31] The absorption and penetration of the colorants into the organic phases of the materials is probably due to the compatibility of the polymer phase with the yellow colorants of the coffee.[18] In the current study, according to the 120-day results of G-aenial anterior and Clearfil Majesty Esthetic, coffee solution caused less discolouration than tea solution. In the 30-day results, close values of tea and coffee solutions were seen only in Lava Block. For the other materials, more evident and a greater degree of discolouration was provided by coffee.

With the exception of non-methacrylate-based posterior composite resins, when CAD/CAM blocks were compared directly with resin materials, they had a higher resistance to discolouration in all the solutions.[33] The production of CAD/CAM blocks and the procedure used for polymerisation developed the discolouration resistance behaviours according to appearance.[5] When comparing resin-based CAD/CAM (LU) results and direct composite (F sup), which is basically the same compound, it can be clearly observed that Lava Block has better resistance to discolouration.[33] However, this is not consistent with a recent study which stated that the resistance to discolouration of CAD/CAM blocks containing resin was higher. The results of that study showed a clinically unacceptable colour change when resin nanoceramic (LU) and nano composite resin (F Sup) were discoloured with coffee.[39] With the exception of coffee, the results of Lava Ultimate (composite block with nano ceramic particles) were similar to those of Vita Enamic (hybrid ceramic), demonstrating that composite materials could show similar performance to that of ceramics.[33] Correspondingly, when a colouring solution such as coffee was used, Vita Enamic showed better resistance to discolouration.[39]

This result does not contradict those of the current study to a great extent. When Lava Block and Vita Enamic were compared using tea and coffee, Vita Enamic showed significantly better resistance to discolouration than Lava Block in both tea and coffee. However, for red wine, the results were different as Lava Block showed better resistance to discolouration than Vita Enamic at both 30 and 120 days. This was attributed to the temperature of the solutions, the alcohol content, the polar structure of tea and most importantly the material content of Vita Enamic and Lava Block. As control groups in the current study, distilled water was used and IPS E-max ceramic CAD/CAM blocks.

As in the current study, Özlem *et al.* used IPS E-max, Lava Block and Vita Enamic, and Filtek Supreme as composite. In a study that examined whether or not there was any difference in discolouration between materials of samples of different thicknesses, IPS E-max did not show any greater discolouration than other materials in any solution or at any thickness, and the discolouration was not percetible to the human eye. It has been said that of the resin content blocks, Vita Enamic could be an alternative to IPS E-max.[39]

In a study by Gawriolek *et al.*, 22 different ceramic materials were compared using tea, coffee, red wine, and distilled water, as in the current study, and no colour change was as high as in composte resins.[40] The results of the current study confirm this as IPS E-max showed the least discolouration of all the groups and in all the solutions. After 30 days, the value of 3.3 as perceptible to the human eye was not reached in any solution, whereas at 120 days, perceptible differences were found in tea and red wine solutions. Similarly in the current study, although the results of Vita Enamic were close to those of IPS E-max in coffee solution, in tea and red wine solutions, the results of Clearfil Majesty Esthetic together with Lava Block were close to those of IPS E-max.

Conclusion

In conclusion, the results of this study showed that distilled water does not make any discernible change in restorative materials. IPS E-max did not show any significantly greater discolouration than any other material. In respect of discolouration, ceramic blocks are more successful. Resin-based blocks and traditional aesthetic composites showed more discolouration. This demonstrates the importance of the necessity of polishing and finishing procedures for restorations. The dietary habits of the patient should be taken into consideration in the selection of the restorative material. Nevertheless, there is a need for this *in vitro* study to be supported by clinical studies with long-term follow-up.

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Conflicts of interest

There are no conflicts of interest.

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