Research article

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Discoloration of various CAD/CAM blocks after immersion in coffee

Sasipin Lauvahutanon[1], Maho Shiozawa[2], Hidekazu Takahashi[3]*, Naohiko Iwasaki[3], Meiko Oki[4], Werner J. Finger[5], Mansuang Arksornnukit[1]

[1]Department of Prosthodontics, Faculty of Dentistry, Chulalongkorn University, Bangkok, Thailand
[2]Department of Removable Partial Prosthodontics, Division of Oral Health Sciences, [3]Department of Oral Biomaterials Engineering, [4]Department of Basic Oral Health Engineering, Graduate School of Medical and Dental Sciences, Tokyo Medical and Dental University (TMDU), Tokyo, Japan
[5]Liaison Center for Innovative Dentistry, Tohoku University Graduate School of Dentistry, Miyagi, Japan

Objectives: This study evaluated color differences (ΔEs) and translucency parameter changes (ΔTPs) of various computer-aided design/computer-aided manufacturing (CAD/CAM) blocks after immersion in coffee. ***Materials and Methods:*** Eight CAD/CAM blocks and four restorative composite resins were evaluated. The CIE $L^*a^*b^*$ values of 2.0 mm thick disk-shaped specimens were measured using the spectrophotometer on white and black backgrounds (n = 6). The ΔEs and ΔTPs of one day, one week, and one month immersion in coffee or water were calculated. The values of each material were analyzed by two-way ANOVA and Tukey's multiple comparisons (α = 0.05). The ΔEs after prophylaxis paste polishing of 1 month coffee immersion specimens, water sorption and solubility were also evaluated. ***Results:*** After one month in coffee, ΔEs of CAD/CAM composite resin blocks and restorative composites ranged from 1.6 to 3.7 and from 2.1 to 7.9, respectively, and ΔTPs decreased. The ANOVA of ΔEs and ΔTPs revealed significant differences in two main factors, immersion periods and media, and their interaction except for ΔEs of TEL (Telio CAD, Ivoclar Vivadent). The ΔEs significantly decreased after prophylaxis polishing except GRA (Gradia Block, GC). There was no significant correlation between ΔEs and water sorption or solubility in water. ***Conclusions:*** The ΔEs of CAD/CAM blocks after immersion in coffee varied among products and were comparable to those of restorative composite resins. The discoloration of CAD/CAM composite resin blocks could be effectively removed with prophylaxis paste polishing, while that of some restorative composites could not be removed. (*Restor Dent Endod* 2017;42(1):9-18)

Key words: CAD/CAM block; Discoloration; Translucency parameter; Water solubility; Water sorption

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Lauvahutanon S, Shiozawa M, Takahashi H, Iwasaki N, Oki M, Finger WJ, Arksornnukit M

***Correspondence to**
Hidekazu Takahashi, DDS, PhD.
Professor, Department of Oral Biomaterials Engineering, Graduate School of Medical and Dental Sciences, Tokyo Medical and Dental University (TMDU), Yushima 1-5-45, Bunkyo-ku, Tokyo, Japan 113-8549
TEL, +81-3-5803-5379; FAX, +81-3-5803-5379; E-mail, takahashi.bmoe@tmd.ac.jp

Introduction

During the last decade, computer-aided design/computer-aided manufacturing (CAD/CAM) technologies and materials have rapidly gained importance for indirect prosthetic restorations.[1,2] Composite resin block materials have been developed and manufactured for CAD/CAM systems since 2000.[3,4] Currently, there are several new products of CAD/CAM composite resin blocks available. The novel CAD/CAM composite resin blocks are industrially polymerized under standardized parameters at high temperature and pressure to achieve optimum properties at microstructure level and high degree of conversion. As a result, material characteristics were improved compared to direct restorative composite resin.[4] A CAD/CAM hybrid ceramic block is also introduced which is a polymer-infiltrated feldspar ceramic network enriched with aluminum oxide.[5] Mechanical properties of CAD/CAM composite resin blocks have been evaluated and suggested that they are suitable for single restorations according to the applicable ISO standard.[6,7]

However, the success of restorations depends not only on mechanical and physical properties, but also on the esthetic appearance.[8-10] Tooth-colored restorative materials should feature excellent color match and high color stability during clinical service.[11,12] Visual color difference thresholds can be used as a quality control tool and guideline for selecting esthetic materials.[13]

Restorative materials are exposed to multiple and frequent changes in oral conditions such as temperature, humidity, food, beverage, and smoking. These changes may be associated with discoloration of dental restorative materials. It is known that composite resin restorations show a tendency to discolor in oral environment.[14,15] Previous studies have demonstrated that composite resins were susceptible to discoloration due to extrinsic and instrinsic factors.[16] Extrinsic factors include the influence of staining solutions such as coffee, tea, cola, and red wine, whilst intrinsic factors are related to the resin matrix composition, initiator system, mode and duration of polymerization, conversion of the matrix monomers, particle size, and oxidation of the unreacted carbon double bonds.[17-22] Due to its high potential for staining, the effect of immersion in coffee is considered a reasonable test procedure to estimate the materials' tendency to discolor in resin-based materials.[17] CAD/CAM composite resin blocks were expected to show high resistance to discoloration due to the industrially optimized polymerization process. Discoloration of resin-based materials might be attributed to water sorption. Moreover, correlation between color stability and water sorption has also been demonstrated.[23]

CIE $L^*a^*b^*$ values are the representative parameters for indication of color, and can be related to human perception of color. Several studies evaluated the perceptibility threshold (PT) and acceptability threshold (AT) for CIE $L^*a^*b^*$ values. The PT is the magnitude of color difference (ΔE) that is visually detectable by the human eye, while the AT is the magnitude of color difference that constitutes the acceptablility between tooth-colored restorative materials.[13,24-26] In an *in vitro* study on ceramics, the ΔE of the 50:50% PTs, which means that 50% of the observers can distinguish the color difference of the 2 objects, was 1.2; while that of 50:50% ATs, which means that 50% of the observers clinically accept the color differences between 2 objects was 2.7.[13]

Translucency of the tooth also plays an important role in esthetic outcome. The translucency parameter (*TP*) is derived from the color difference between the material evaluated on white and black backgrounds, and corresponds directly to the common visual assessments of translucency.[27]

To date, there has been no published study comparing and evaluating the discoloration and changes in translucency of current CAD/CAM blocks after immersion in discoloring media. Therefore, the purpose of this study was to evaluate the discoloration susceptibility and translucency parameters of various CAD/CAM block compared with conventional restorative composite resins after immersion in coffee. The null hypothesis was that there would be no significant difference regarding the discoloration and translucency change among various CAD/CAM blocks after immersion in coffee.

Materials and Methods

Eight CAD/CAM blocks including five composite resin blocks (Block HC [BLO, Shofu, Kyoto, Japan], Cerasmart [CER, GC, Tokyo, Japan], Gradia Block [GRA, GC], KZR-CAD Hybrid Resin Block [KZR, Yamamota Precious Metal, Osaka, Japan], Lava Ultimate [ULT, 3M/ESPE, St. Paul, MN, USA]), one hybrid ceramic block (Vita Enamic [ENA, Vita Zahnfabrik GMbH, Bad Säckingen, Germany]), one PMMA block (Telio CAD [TEL, Ivoclar Vivadent, Schaan, Liechtenstein]), and one feldspar ceramic block (Vitablocs Mark II [VIT, Vita Zahnfabrik GMbH]) and four conventional restorative composite resins (one hybrid composite Clearfil AP-X [APX, Kuraray, Okayama, Japan], one microfilled composite Durafill VS [DUR, Heraeus Kulzer, Hanau, Germany], and two nanohybrid composites Estelite Sigma Quick [ESQ, Tokuyama Dental, Tokyo, Japan] and Filtek Supreme Ultra [FSU, 3M/ESPE]), were examined in the present study. The materials and their compositions, published by the respective manufacturers are shown in Table 1.

Discoloration tendency and translucency parameter

Twelve disk-shaped specimens, 10 mm in diameter and 2.1 mm in thickness, were prepared from each material. CAD/CAM block specimens were shaped using a stone abrasive wheel (Vitrified Dia, Shofu Inc.) attached to a lathe (YS-550V, Towa seiki Co., Ltd., Aichi, Japan) to make cylindrical specimens from which disk-shaped specimens were cut with a low-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA). The conventional composite resin specimens were placed in cylindrical acrylic molds (10 mm in diameter and 2.1 mm in thickness) and light activated for 40 seconds on each side using a light-emitting diode (LED) curing unit (Elipar S10, 3M ESPE, Seefeld, Germany) with a light intensity of 600 mW/cm^2. The specimens were ground sequentially on wet SiC paper

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Table 1. Materials investigated and their compositions

Type	Brand	Code	Manufacturer	Shade/Size	Batch	Composition: Monomer	Composition: Filler	Composition: Mass% (Vol%)
Composite resin block	Block HC	BLO	Shofu Inc., Kyoto, Japan	A3 - HT/M	021401	UDMA, TEGDMA	Silica powder, micro fumed silica, zirconium silicate	61.0
	Cerasmart	CER	GC Corporation, Tokyo, Japan	A3 - LT/14	1308261E	Bis-MEPP, UDMA, DMA	Silica (20 nm), barium glass (300 nm)	71.0
	Gradia Block	GRA	GC Corporation, Tokyo, Japan	A3/14	1308012	UDMA, methacrylate copolymer	Silica, Al-silicate glass, prepolymerized filler	76.0
	KZR-CAD Hybrid Resin Block	KZR	Yamamoto Precious Metal Co., Osaka, Japan	A3	01021410	UDMA,TEGDMA	SiO_2 (20 nm), aggregated SiO_2-Al_2O_3-ZrO_2 (200 – 600 nm) cluster (1 - 6 µm)	74.0
	Lava Ultimate	ULT	3M ESPE, St. Paul, MN, USA	A3 - HT/14L	N494437	Bis-GMA, UDMA, Bis-EMA,TEGDMA	SiO_2 (20 nm), ZrO_2 (4 - 11 nm), aggregated ZrO_2/SiO_2 cluster (SiO_2 = 20 nm, ZrO_2 = 4 - 11 nm)	80.0
Hybrid ceramic block	Vita Enamic	ENA	Vita Zahnfabrik H. Rauter GmbH, Bad Säckingen, Germany	3M2 - HT/EM -14	47630	UDMA, TEGDMA	Feldspar ceramic enriched with aluminum oxide	86.0 (75.0)
PMMA block	Telio CAD	TEL	Ivoclar Vivadent Inc., Schaan, Liechtenstein	A3	T09738	MMA	-	-
Feldspar ceramic block	Vitablocs Mark II	VIT	Vita Zahnfabrik H. Rauter GmbH, Bad Säckingen, Germany	A3C/I14	07BY0803	-	Feldspathic crystalline particles in glassy matrix	-
Conventional restorative composite resin	Clearfil AP-X	APX	Kurarey, Okayama, Japan	A3	01447A	Bis-GMA, TEGDMA	Silanated barium glass, Silanated colloidal silica, Silanated silica (0.1 - 15 µm)	86.0 (70.0)
	Durafill VS	DUR	Heraeus Kulzer, Hanau, Germany	A3	10218	Bis-GMA, UDMA, TEGDMA	SiO_2 (20 - 70 nm), prepolymer < 20 µm, SiO_2 in prepolymer: 32 wt%	75.3 (66.0)
	Estelite Sigma Quick	ESQ	Tokuyama Dental Corporation, Tokyo, Japan	A3	151064P	Bis-GMA, TEGDMA	Silica-zirconia filler, composite filler (0.1 - 0.3 µm)	80.0 (71.0)
	Filtek Supreme Ultra	FSU	3M ESPE, St. Paul, MN, USA	A3	N433373	Bis-GMA, UDMA, Bis-EMA, TEGDMA	SiO_2 (20 nm), ZrO_2 (4 - 11 nm), aggregated ZrO_2/SiO_2 cluster (SiO_2 = 20 nm, ZrO_2 = 4 - 11 nm)	78.5 (63.3)

Bis-GMA, bisphenol A diglycidylether methacrylate; Bis-MEPP, 2,2-Bis(4-methacryloxypolyethoxyphenyl)propane; UDMA, urethane dimethacrylate; TEGDMA, triethylene glycol dimethacrylate; Bis-EMA, ethoxylated bisphenol-A dimethacrylate; DMA, dimethacrylate; MMA, methyl methacrylate.

(600, 1,000, and 1,500 grits). Grinding and polishing was performed on one side of the samples to produce 2.0 ± 0.1 mm thick specimens. The thickness was controlled with a digital micrometer (MCM-25M, Mitsutoyo, Tokyo, Japan), with a reading accuracy of ± 0.001 mm. The specimens were ultrasonically cleaned in deionized water for 10 minutes, shortly dried with compressed air, immersed in deionized water in a brown glass vial and kept in an incubator (TVN480DA, Toyo Seisakusyo Kaisha, Co., Ltd, Chiba, Japan) at 37 ± 0.2°C for 24 hours. Subsequently, the specimens were removed from the vials, washed with deionized water, gently wiped with filter paper, and air-dried by shaking in air for 30 seconds prior to the color measurements.

The CIE $L^*a^*b^*$ values of the polished surfaces of each specimen were measured, backed by a white background (L^* = 92.28, a^* = -1.28, and b^* = -2.05) and by a black background (L^* = 1.50, a^* = -2.37, and b^* = -8.41) using a spectrophotometer (Crystaleye, Olympus Corp., Tokyo, Japan). The light source illumination corresponded to average daylight (D65).

TP was calculated from the color measurements between the specimen obtained with supporting by the white background and the black background using the following equation[27]:

$$TP = [(L^*_B - L^*_W)^2 + (a^*_B - a^*_W)^2 + (b^*_B - b^*_W)^2]^{1/2}$$

where: L^* refers to the brightness, a^* for redness to greenness, and b^* for yellowness to blueness. The subscript B refers to the color coordinates on the black background and W to those on the white background. A high *TP* value indicates high translucency and low opacity. Three measurements were made for each specimen and the average value was recorded.

The *TP* difference (ΔTP) was calculated from the difference of *TP* values before and after immersion:

$$\Delta TP = TP_{after} - TP_{before}$$

Deionized water and coffee were used as the immersion media. The coffee was prepared by dissolving 0.51 g of instant coffee powder (Nescafe Gold Blend, Nestle Japan, Kobe, Japan) in 50 mL of deionized water.[18,23] Six specimens of each CAD/CAM block were immersed in each of the immersion solutions (deionized water and coffee) and placed in a shaking incubator (37 ± 0.2°C, 1 Hz frequency). Both solutions were changed every day. Color measurements were carried out after one day, one week, and one month periods of immersion.

The color difference (ΔE) was calculated based on the L^*, a^*, and b^* on black background between the same specimen before and after immersion in one of the immersion media and immersion periods using the following equation:

$$\Delta E = [(L^*_1 - L^*_2)^2 + (a^*_1 - a^*_2)^2 + (b^*_1 - b^*_2)^2]^{1/2}$$

where the subscripts 1 and 2 refer to the color coordinates before and after immersion, respectively. A high ΔE value indicates a large color difference. Three measurements were made on each specimen, and the average value was recorded.

After one month in coffee immersion and following six months storage period in a desiccator, all specimens were polished with a fluoride-free prophylaxis paste (Proxyt RDA 36, Ivoclar Vivadent) applied with a soft rubber cup at 5,000 rpm for each 20 seconds on the planar and the peripheral surfaces, and the color was remeasured.

Water sorption and solubility

Water sorption and solubility of CAD/CAM block specimens were determined by modifying ISO 4049:2009[28] from the suggested diameter of 15.0 mm to 13.5 mm due to the size limitation of CAD/CAM blocks. Five disk-shaped specimens (13.5 mm in diameter and 1.1 mm in thickness) from each material were prepared as described above. Conventional restorative composite resin specimens were placed in a metal mold (13.5 mm in diameter and 1.1 mm in thickness) and light activated on each side with nine overlapping footprints for 40 seconds each using the LED curing unit mentioned above. The specimens were ground sequentially with SiC paper (600, 1,000, and 1,500 grits). Grinding and polishing procedures were performed on both sides to achieve 1.0 mm thick specimens. The final thickness was verified using the digital micrometer. Prior to the initial measurement the specimens were ultrasonically cleaned for 10 minutes with deionized water, gently dried with filter paper and kept in a desiccator at 37 ± 2°C for 22 hours. Then, the specimens were transferred to another desiccator kept at 23 ± 2°C for 2 hours and weighed using a precision digital balance (AUW120D, Shimadzu, Kyoto, Japan, the minimum reading is 0.01 mg). This cycle was repeated until a constant mass at three subsequent measurements was obtained (m_1). The diameters and thicknesses of each specimen were measured to calculate the volume (V). The specimens were immersed in distilled water at 37°C for one week, then removed, gently dried with filter paper, and weighed again (m_2). Finally, the specimens were returned to the desiccators using the above mentioned procedure until a constant mass was obtained (m_3). Water sorption and solubility were calculated using the following equations:

$$\text{Water sorption } (\mu g/mm^3) = (m_2 - m_3) / V$$
$$\text{Water solubility } (\mu g/mm^3) = (m_1 - m_3) / V$$

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Statistical analyses

The *ΔE*s except after prophylaxis polishing and *ΔTP*s were separately analyzed by two-way repeated measures analysis of variance (ANOVA) with immersion media and immersion periods as main factors, followed by Tukey's *post-hoc* multiple comparisons. The differences between *ΔE*s of one month coffee immersion and that after polishing with prophylaxis paste were analyzed by paired *t* test. The *ΔE*s and *ΔTP*s after one month water and coffee immersion, water sorption and solubility were analyzed using nested ANOVA with types of materials as the main factor and brands among each type as the nested effect, followed by Tukey's *post-hoc* multiple comparisons. Statistical analysis software (JMP ver. 11.2.0, SAS Institute Inc., Cary, NC, USA) was used. The significance level was set at $\alpha = 0.05$.

Results

After immersion in coffee, the specimens generally became darker in color as the immersion period increased. Color changes of the specimens after water immersion were not clearly detected.

Table 2 shows the *ΔE* values and their standard deviations after immersion in deionized water and coffee media for one day, one week, and one month and after polishing with prophylaxis paste following one month immersion in coffee and six months storage. Color changes measured on black and white backgrounds demonstrated a similar trend (data on white background is not shown). Moreover, CAD/CAM blocks are usually used for crown restoration. Therefore, only *ΔE* values measured on the black background are presented as to mimic the intraoral lighting. After immersion in coffee,

Table 2. *ΔE* after immersion in deionized water and coffee solution for 1 day, 1 week, and 1 month and after polishing 1 month immersion in coffee

Material type	Code	Water			Coffee			After polishing
		1 day	1 wk	1 mon	1 day	1 wk	1 mon	
Composite resin block	BLO	0.2 (0.1)^{a}	0.3 (0.2)^{a}	0.3 (0.1)^{a,3}	1.3 (0.5)^{b}	2.2 (0.5)^{c}	3.7 (0.7)^{d,5}	1.8 (0.5)
	CER	0.3 (0.3)^{e}	0.4 (0.2)^{e}	0.7 (0.2)^{e,g,1,2,3}	1.2 (0.4)^{f,g}	1.9 (0.7)^{f}	2.7 (0.6)^{h,5,6,7}	0.9 (0.4)
	GRA	0.3 (0.2)^{i}	0.3 (0.2)^{i}	0.4 (0.2)^{i,2,3}	0.5 (0.3)^{i}	0.9 (0.2)^{j}	1.6 (0.4)$^{k,\$,6,7}$	1.6 (0.5)$^{\$}$
	KZR	0.2 (0.1)^{l}	0.3 (0.2)^{l}	0.3 (0.1)^{l,3}	1.0 (0.3)^{m}	1.6 (0.3)^{n}	2.9 (0.3)^{o,5,6}	0.9 (0.2)
	ULT	0.7 (0.4)^{p}	0.6 (0.3)^{p}	0.5 (0.3)^{p,2,3}	1.7 (0.3)^{q}	2.7 (0.4)^{r}	3.6 (0.6)^{s,5}	1.2 (0.4)
Hybrid ceramic block	ENA	0.3 (0.2)^{t}	0.4 (0.2)^{t}	0.5 (0.4)^{t,2,3}	0.5 (0.3)^{t}	0.8 (0.4)^{t}	1.4 (0.4)^{u,7}	0.5 (0.2)
PMMA block	TEL$^{\#}$	0.9 (0.6)	0.9 (0.8)	1.0 (0.8)^{1,2}	0.9 (0.7)	1.6 (1.2)	2.0 (1.1)^{6,7}	1.2 (0.9)
Feldspar ceramic block	VIT	0.3 (0.2)^{v}	0.3 (0.1)^{v}	0.3 (0.2)^{v,3}	0.4 (0.2)^{v}	1.1 (0.2)^{w}	1.8 (0.3)^{x,6,7}	0.3 (0.1)
Conventional restorative composite resin	APX	0.9 (0.4)^{y,z}	0.8 (0.4)^{y}	0.8 (0.7)^{y,1,2,3}	1.4 (0.2)^{z}	2.1 (0.3)^{A}	2.1 (0.2)^{A,6,7}	0.5 (0.2)
	DUR	0.5 (0.2)^{B}	0.7 (0.2)^{B,C}	0.9 (0.3)^{B,C,1,2,3}	1.6 (0.6)^{B,C}	4.1 (1.2)^{D}	7.9 (1.5)^{E,4}	5.7 (1.4)
	ESQ	0.3 (0.2)^{F}	0.2 (0.1)^{F}	0.9 (0.1)^{G,1,2,3}	0.8 (0.3)^{G}	1.6 (0.4)^{H}	2.4 (0.5)^{I,5,6,7}	0.7 (0.3)
	FSU	0.3 (0.1)^{J}	0.6 (0.2)^{J}	1.3 (0.1)^{J,K,1}	2.3 (0.7)^{K}	3.7 (1.0)^{L}	6.6 (1.0)^{M,4}	4.4 (0.9)

Standard deviations are in parentheses and the same superscript letter denotes homogenous subsets in each material ($p > 0.05$). The same superscript number denotes homogenous subsets after 1 month immersion ($p > 0.05$).
BLO, Block HC (Shofu, Kyoto, Japan); CER, Cerasmart (GC, Tokyo, Japan); GRA, Gradia Block (GC); KZR, KZR-CAD Hybrid Resin Block (Yamamota Precious Metal, Osaka, Japan); ULT, Lava Ultimate (3M/ESPE, St. Paul, MN, USA); ENA, Vita Enamic (Vita Zahnfabrik GmbH, Bad Säckingen, Germany); TEL, Telio CAD (Ivoclar Vivadent, Schaan, Liechtenstein); VIT, Vitablocs Mark II (Vita Zahnfabrik GmbH); APX, Clearfil AP-X (Kuraray, Okayama, Japan); DUR, Durafill VS (Heraeus Kulzer, Hanau, Germany); ESQ, Estelite Sigma Quick (Tokuyama Dental, Tokyo, Japan); FSU, Filtek Supreme Ultra (3M/ESPE).
[#]Not significantly different in spite of immersion condition.
[$]The difference between *ΔE* of 1 month coffee and after polishing 1 month coffee by paired *t* test was significant in all groups except for GRA denotes.

most changes occurred with a decrease in L^* values and an increase in a^* and b^* values. This demonstrated that the specimens appeared more toward red and yellow and darker in color, but such an increase in a^* and b^* values was minimal. Two-way repeated measures ANOVA of ΔE values revealed that the two main factors, immersion media and immersion period, and their interaction were significant, except TEL. Generally, the ΔE values after immersion in water did not change significantly, whereas those after immersion in coffee showed a significant increase with the increase of immersion time. The ΔE of types of materials after immersion in water were significantly different ($p < 0.001$); feldspathic ceramic block and composite resin blocks were significantly less than conventional restorative composite resins and PMMA block.

The ΔE values in coffee after one month immersion were significantly higher than those after one day and one week immersion except DUR. The ΔE values after one month immersion in coffee of CAD/CAM composite resin blocks, the remaining blocks (ENA, TEL, and VIT), and conventional restorative composite resins ranged from 1.6 to 3.7, from 1.4 to 2.0, and 2.1 to 7.9, respectively. The ΔE of types of materials after immersion in coffee were significantly different ($p < 0.001$) and ranked as follows; hybrid ceramic block = feldspathic ceramic block = PMMA block < composite resin block < conventional restorative composite resin. DUR and FSU were significantly greater than the others; BLO and ULT were significantly greater than APX, TEL, VIT, GRA, and ENA.

After prophylaxis paste polishing for 20 seconds on the planar and the peripheral surfaces, nearly all tested materials showed significant reduction in ΔE values ($p < 0.05$), except GRA. Only DUR and FSU, which is conventional restorative composite resin still demonstrated perceptibly high discoloration (ΔE 4.4 - 5.7) after polishing.

Table 3 shows the ΔTP values and their standard deviations after immersion in deionized water and coffee media for one day, one week, and one month. The ΔTP values after one month immersion in water were from -0.2 to 0.6, after one month immersion in coffee from

Table 3. ΔTP after immersion in deionized water and coffee solution for 1 day, 1 week, and 1 month

Material type	Code	Water			Coffee		
		1 day	1 wk	1 mon	1 day	1 wk	1 mon
Composite resin block	BLO	0.0 (0.1)^{a}	0.0 (0.2)^{a}	0.0 (0.1)^{a,3}	-0.3 (0.3)^{b}	-0.6 (0.2)^{c}	-1.0 (0.2)^{d,5}
	CER	0.1 (0.2)^{e,f}	0.2 (0.3)^{e}	0.6 (0.3)^{e,1,2}	-0.2 (0.1)^{e}	-0.3 (0.2)^{f}	-0.4 (0.2)^{f,4}
	GRA	0.0 (0.1)^{g}	0.1 (0.2)^{g}	0.6 (0.2)^{h,1}	-0.3 (0.1)^{i}	-0.5 (0.2)^{i}	-0.7 (0.2)^{j,4,5}
	KZR	0.0 (0.1)^{k}	-0.3 (0.2)^{l}	-0.2 (0.2)^{k,l,3}	-0.8 (0.2)^{m}	-1.4 (0.1)^{n}	-1.8 (0.2)^{o,6}
	ULT	0.1 (0.5)^{p}	0.0 (0.1)^{p}	0.1 (0.2)^{p,3}	-0.4 (0.3)^{p,q}	-0.7 (0.3)^{q}	-1.2 (0.4)^{r,2,3}
Hybrid ceramic block	ENA	0.1 (0.2)^{s}	0.1 (0.2)^{s}	0.0 (0.2)^{s,t,3}	-0.1 (0.2)^{s,u}	-0.3 (0.2)^{t,u}	-0.7 (0.1)^{v,4,5}
PMMA block	TEL$^{\#}$	0.0 (0.4)^{w}	-0.1 (0.4)^{w}	0.1 (0.3)^{w,3}	-0.4 (0.2)^{w}	-0.4 (0.3)^{w}	-0.4 (0.2)^{w,4}
Feldspar ceramic block	VIT	0.0 (0.1)^{x}	-0.1 (0.1)^{x,y}	-0.1 (0.1)^{x,3}	-0.4 (0.1)^{y}	-1.2 (0.2)^{z}	-2.1 (0.3)^{A,6}
Conventional restorative composite resin	APX	-0.1 (0.4)^{B}	-0.3 (0.4)^{B,D}	0.0 (0.4)^{B,3}	-0.3 (0.4)^{B,C}	-0.8 (0.2)^{C}	-0.7 (0.3)^{C,D,4,5}
	DUR	-0.3 (0.2)^{E,F}	-0.1 (0.3)^{E}	-0.1 (0.1)^{E,3}	-0.6 (0.2)^{F}	-1.1 (0.2)^{G}	-2.3 (0.4)^{H,6,7}
	ESQ	-0.1 (0.2)^{I,J}	0.2 (0.2)^{I}	0.1 (0.2)^{I,2,3}	-0.3 (0.2)^{J,K}	-0.4 (0.2)^{K}	-0.9 (0.3)^{L,4,5}
	FSU	0.1 (0.2)^{M}	0.1 (0.3)^{M}	0.1 (0.1)^{3}	-0.6 (0.2)^{N}	-1.4 (0.2)^{O}	-2.7 (0.2)^{P,7}

Standard deviations are in parentheses and the same superscript letter denotes homogenous subsets in each material ($p > 0.05$). The same superscript number denotes homogenous subsets after 1 month immersion ($p > 0.05$).

BLO, Block HC (Shofu, Kyoto, Japan); CER, Cerasmart (GC, Tokyo, Japan); GRA, Gradia Block (GC); KZR, KZR-CAD Hybrid Resin Block (Yamamota Precious Metal, Osaka, Japan); ULT, Lava Ultimate (3M/ESPE, St. Paul, MN, USA); ENA, Vita Enamic (Vita Zahnfabrik GMbH, Bad Säckingen, Germany); TEL, Telio CAD (Ivoclar Vivadent, Schaan, Liechtenstein); VIT, Vitablocs Mark II (Vita Zahnfabrik GMbH); APX, Clearfil AP-X (Kuraray, Okayama, Japan); DUR, Durafill VS (Heraeus Kulzer, Hanau, Germany); ESQ, Estelite Sigma Quick (Tokuyama Dental, Tokyo, Japan); FSU, Filtek Supreme Ultra (3M/ESPE).

-2.7 to -0.4. Two-way repeated measures ANOVA of *ΔTP* values revealed that the two main factors, immersion media and immersion periods, and their interaction were significant. The *ΔTP* values after immersion in water did not significantly change, except GRA. The *ΔTPs* of types of materials after immersion in one month water were significantly different ($p < 0.021$), but the differences among types of materials were less than 0.28. The *ΔTP* values of GRA, after one month immersion in water, was significantly greater than those after one day and one week immersion in water. All *ΔTP* values apart from CER, TEL, and APX decreased significantly after one month immersion in coffee. The *ΔTPs* of types of materials after immersion in one month coffee were significantly different ($p < 0.001$); feldspar ceramic block and conventional restorative composite resin were significantly less than hybrid ceramic block and PMMA block, the difference between composite resin block and hybrid ceramic block and that between hybrid ceramic block and PMMA block were not significant.

The means and standard deviations of water sorption and solubility are shown in Table 4. The water sorptions of types of materials were significantly different ($p < 0.001$), and ranked as follows: feldspar ceramic block < hybrid ceramic block < conventional restorative composite resin < PMMA block < composite resin block. Water sorption of BLO was the largest (39.7 μg/mm^3). However, this high value was still within the acceptable range specified in ISO 4049 (< 40 μg/mm^3). The water solubility of types of materials were significantly different ($p < 0.001$) and ranked as follows: hybrid ceramic block < composite resin block < PMMA block < feldspar ceramic block < conventional restorative composite resin. VIT and TEL were almost 0. DUR showed the largest water solubility (2.7 μg/mm^3) of all tested materials, however it was still below the maximum permissible value specified in ISO 4049 (< 7.5 μg/mm^3). CER, GRA, KZR, ULT, ENA, VIT, and TEL showed negative values for water solubility.

Discussion

The *ΔE* and *ΔTP* values obtained from the CAD/CAM block materials after immersion in coffee were significantly different according to time and immersion media for each material. Therefore, the null hypothesis that there would be no significant difference regarding discoloration and translucency changes among CAD/CAM blocks after immersion in coffee was rejected.

The *ΔE* values after immersion in water were not significantly different, in contrast to those after immersion in coffee that showed a significant increase, apart from TEL, a PMMA-based block material. This finding is in agreement with a previous study on luting agent which showed greater *ΔE* values of composite resin cements compared to PMMA-based resin cements.[23] ENA, VIT, and TEL demonstrated small discoloration whose *ΔEs*

Table 4. Water sorption and solubility after water immersion for 1 week (μg/mm^3)

Material type	Code	Water sorption	Water solubility
Composite resin block	BLO	39.7 (1.3)^{a}	0.6 (0.5)^{C,D}
	CER	22.0 (0.7)^{e}	-0.2 (0.2)^{F}
	GRA	16.9 (1.3)^{g,h}	-2.7 (0.5)^{G}
	KZR	23.5 (0.7)^{d}	-0.2 (0.1)^{F}
	ULT	30.7 (0.3)^{b}	-0.4 (0.3)^{F}
Hybrid ceramic block	ENA	7.0 (0.2)^{i}	-2.8 (0.0)^{G}
PMMA block	TEL	23.2 (0.1)^{d,e}	-0.1 (0.1)^{E,F}
Feldspar ceramic block	VIT	0.1 (0.2)^{j}	-0.1(0.0)^{E,F}
Conventional restorative composite resin	APX	15.8 (0.2)^{h}	1.1 (0.1)^{C}
	DUR	17.3 (0.2)^{g}	2.7 (0.2)^{A}
	ESQ	19.5 (0.1)^{f}	0.4 (0.1)^{D,E}
	FSU	25.7 (0.6)^{c}	1.7 (0.1)^{B}

Standard deviations are in parentheses and the same superscript letters are not significantly different ($p > 0.05$).
BLO, Block HC (Shofu, Kyoto, Japan); CER, Cerasmart (GC, Tokyo, Japan); GRA, Gradia Block (GC); KZR, KZR-CAD Hybrid Resin Block (Yamamota Precious Metal, Osaka, Japan); ULT, Lava Ultimate (3M/ESPE, St. Paul, MN, USA); ENA, Vita Enamic (Vita Zahnfabrik GMbH, Bad Säckingen, Germany); TEL, Telio CAD (Ivoclar Vivadent, Schaan, Liechtenstein); VIT, Vitablocs Mark II (Vita Zahnfabrik GMbH); APX, Clearfil AP-X (Kuraray, Okayama, Japan); DUR, Durafill VS (Heraeus Kulzer, Hanau, Germany); ESQ, Estelite Sigma Quick (Tokuyama Dental, Tokyo, Japan); FSU, Filtek Supreme Ultra (3M/ESPE).

were less than 2.0. This small value in ΔE unit from the staining media might relate to their different structure and composition when compared with the other materials investigated. This might be attributed to the hydrophobic nature of ceramic in VIT and ceramic network in ENA which demonstrated low water sorption and solubility.[29] The stain resistance of TEL may be due to lower polarity of PMMA in TEL which reflected less affinity to polar liquid and water.[30] Comparing the composite resin blocks to the conventional restorative composite resin, the conventional restorative composite resins showed severe discoloration. The reason for discoloration of these products was not obvious. One of the reasons for discoloration of composite resins might be insufficient silanization.[18] Two composite resins consist of nano-size fillers which have large relative surface areas. Silanization of these nano-size fillers might be difficult. Although the same filler composition was used for ULT and FSU, ULT did not show pronounced discoloration, which might be related to the polymerization process under ULT block fabrication.

In clinical situation, the prospective multi-center study has performed to establish the clinical relevant on color perception. The 50:50% PTs and 50:50% ATs of dental ceramics tested under simulated clinical setting were 1.2 and 2.7, respectively.[13] After one month immersion in water, the 50:50% PTs of the materials tested in this trial were less except FSU. The 50:50% ATs after one week immersion in coffee proved that the investigated materials, except DUR and FSU, fell within the acceptability range. In contrast, the ATs of BLO, KZR, ULT, DUR, and FSU after one month immersion in coffee exceeded the threshold.

Previous literature suggested that immersion in coffee for one week was equivalent to seven months of coffee drinking with the assumption that the coffee remained in the mouth during drinking.[23] Thus, one month immersion of specimens in coffee for evaluation of the resulting staining effect might be an exaggeration of the reality. However, in spite of this severe condition, the ΔE values of all CAD/CAM block specimens after one week immersion in coffee were under the 50:50% AT. The conventional restorative composite resins of DUR and FSU showed pronounced discoloration in this test that might indicate a risk for perceptible staining during clinical service.[31,32] Even after the one month challenge with coffee, the block materials tested except BLO and ULT were within the 50:50% AT. This result indicated that discoloration of CAD/CAM composite resin blocks in coffee varied among products which was similar to conventional restorative composite resins.

After maintenance polishing with prophylaxis paste, the discoloration of the coffee-stained specimens except GRA significantly reduced, indicating that discolorations noticed on most of the products investigated were extrinsic. It is assumed that regular oral hygiene measures can eliminate or reduce surface stains effectively. The ΔE values of DUR and FSU after maintenance polishing were greater than the 50:50% AT. The composition of FSU and ULT was almost identical. These results suggested that the industrially optimized polymerization process was effective to improve discoloration resistance.

Some composite resins showed slight increase of ΔTPs after immersion in water. This might be attributed to the absorption of water into the composite resin. All materials investigated showed negative values of ΔTPs after immersion in coffee indicating a reduction in translucency. Coffee stains, absorbed on the surfaces of materials, might cause changes in translucency.

CAD/CAM composite resin blocks were expected to show less water sorption and solubility compared to direct composite resins. Interestingly, two of the five CAD/CAM composite resin blocks tested, BLO and ULT, showed water sorption values higher than 30 µg/mm^3. However, this high value was still within the acceptable range specified in ISO 4049 (< 40 µg/mm^3). This finding might be attributed to the zirconium oxide content of the filler, eventually resulting in reduced stability of the silane coupling agent.[33] CAD/CAM composite resin blocks, with the exception of BLO showed negative solubility values, whereas all conventional restorative composite resins investigated showed positive solubility values. Negative values might be the result of water penetrated into the surface of the materials. This phenomenon might be ascribed to the hydrophilicity of the resin matrix and/or hydrolytic instability of the interfacial coupling between filler and resin matrix.[34,35]

A significant correlation between ΔEs and water sorption was previously proven,[23] however, no significant correlations among ΔEs, ΔTPs, water sorption, and water solubility could be detected in the present study. Water absorption into the composite resin results in expansion and plasticization of the resin matrix eventually leading to the microcracks in the surface. As a result, colorants may penetrate into the cracks and cause color changes.[36] When CAD/CAM composite resin blocks and conventional restorative composite resins were considered separately, slightly positive yet statistically non-significant correlations between ΔEs and water sorption were noticed for both groups. Considering the various compositions and industrial production methods of the products investigated, obvious correlations between ΔEs and water sorption were unlikely to exist.

In this *in vitro* study, the effects of one month immersion in coffee and water of CAD/CAM blocks on discoloration were evaluated. In the oral environment, restorative materials are also subjected to numerous other liquids, to temperature and load stress, and to tooth brushing. The limitation of this *in vitro* study is that the clinical environment and its effect on the performance of CAD/CAM blocks on the discoloration were not fully mimicked. Therefore, further investigations are recommended to evaluate the effect of additional contributing factors on the long-term discoloration resistance of CAD/CAM block materials.

Conclusions

Within the limitations of this study, the ΔE values after immersion in water did not significantly change, whereas those after immersion in coffee significantly increased with increasing immersion time. The ΔE values from 1.6 to 3.7 of CAD/CAM composite block after one month coffee immersion were significantly less than those of conventional restorative composite resin, from 2.1 to 7.9, and significantly greater than those of hybrid ceramic block, 1.4, and feldspar ceramic block, 1.8. Maintenance polishing with prophylaxis paste could effectively reduce discolorations of all CAD/CAM block materials except GRA as could be done on most of the light-cured composite resins after immersion in coffee. Discoloration upon immersion in coffee was predominantly extrinsic. The ΔTP values after immersion in water did not significantly change except GRA. However, upon immersion in coffee, ΔTP decreased significantly after one month, except CER, TEL, and APX.

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