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RESEARCH ARTICLE

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Stainability and translucency of potassium aluminum sulfate applied computer-aided design and computer-aided manufacturing materials after coffee thermocycling

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Abstract

Objective: The purpose of this in vitro study was to evaluate the effect of potassium aluminum sulfate (alum) application on the stainability and translucency of computer-aided design and computer-aided manufacturing (CAD-CAM) materials after coffee thermocycling (CTC).

Materials and Methods: Disk-shaped specimens (Ø10 × 1 mm; $N = 200$) were fabricated by using additively (Crowntec [CT] and Varseo Smile Crown Plus [VS]) and subtractively manufactured (Brilliant Crios [RCR], CEREC Block [FC], and Vita Enamic [VE]) CAD-CAM materials and polished. All specimens were randomly divided into two groups as alum applied and control ($n = 10$). All specimens were then subjected to CTC (10,000 cycles at 5–55°C) and color coordinates were measured at each time interval. Color differences (ΔE_{00}) and relative translucency parameters (RTPs) were calculated and the data were statistically analyzed ($a = 0.05$).

Results: Among tested time intervals, alum applied specimens had their lowest ΔE_{00} after alum application ($p \leq 0.006$), except for FC ($p = 0.177$). In addition, alum applied RCR had lower ΔE_{00} values than its control specimens ($p = 0.029$). Alum applied specimens had their lowest RTP after CTC ($p < 0.001$) and alum application decreased the RTP of CT ($p = 0.010$). CTC reduced the RTP of all materials in control groups ($p < 0.001$). Alum applied CT had higher RTP than its control specimens ($p = 0.049$).

Conclusions: Alum application's effect on color change varied depending on the material and alum mostly resulted in clinically acceptable changes in translucency. CTC led to unacceptable color and translucency changes based on previously reported threshold values.

Clinical Significance: Optical properties of CAD-CAM materials and the sustainability of these properties over time is critical for longevity. Alum may improve the color stability of reinforced composite resin when subjected to long-term coffee consumption.

KEYWORDS
CAD-CAM, coffee thermocycling, potassium aluminum sulfate, stainability, translucency

1 | INTRODUCTION

Hydrated double sulfate salt of ammonium ($M^{+}{}_2SO_4 \cdot M^{3+}{}_2(SO_4)_3 \cdot 24H_2O$), also known as alum, has been used to increase the potency of drugs as an adjuvant since the 20th century[1] and is the only licensed adjuvant in the United States.[2] This material was shown to induce a stronger response to produce antibodies than sole toxoid.[3] Among the various types of alum available, potassium alum has been the most commonly used derivative.[4] Alum has also been a part of dentistry, as previous studies have reported that mouthwashes containing alum inhibited plaque growth.[5–12] While most of those previous studies have used 0.02 M (500 ppm) alum,[5,7,8,10–12] one study showed that 5%, 10%, and 15% alum affected the plaque growth and increased concentration has also increased the effect of alum.[6] However, alum has been preferred as a mordant in the textile industry for the fixation of natural dyes without acting as a colorant.[13] This color stabilizing effect of alum, along with its plaque inhibition,[5–12] may be beneficial for clinicians, particularly for esthetic restorations.

Computer-aided design and computer-aided manufacturing (CAD-CAM) technologies introduced different types of materials such as feldspathic ceramics, composite resins, and polymer-infiltrated ceramic network.[14] Feldspathic ceramics are indicated for minimally invasive restorative treatments,[15] while composite resins are easier to fabricate, finish, and repair, and cause less wear on opposing dental tissues.[16] Polymer-infiltrated ceramic network combines the advantages of ceramics and composite resins as it is composed of 86% of ceramic and 14% acrylate polymer network (urethane dimethacrylate and triethylene glycol dimethacrylate) in weight.[16] Until recently, the main fabrication method for CAD-CAM materials was milling by using prefabricated blocks or disks. However, additive manufacturing has started to become an essential part of dentistry as it is used for the fabrication of various dental products.[17] Composite resins are among the materials that can be additively manufactured.[18–22] Recently, additively manufactured composite resins that contain ceramic particles have been marketed as definitive restorative materials.[23–25] In addition, previous studies have reported promising results while evaluating the mechanical and optical properties of these materials when compared with subtractively manufactured materials of similar chemical composition.[18,26–28]

Regardless of manufacturing method, a restorative material's initial optical properties and their sustainability are critical for its esthetic appearance[14] and longevity.[29] However, intraoral medium might deteriorate optical as well as mechanical properties due to temperature changes and staining solutions.[29] Alum may be impactful in this regard with its color stabilizing effect; it is not cytotoxic even after aging and alum's application on dental restorations can be considered.[30] Alum's potential color stabilizing advantage should be further investigated when combined with various restorative materials. However, to the authors' knowledge, no study has ever investigated how alum application on different restorative materials affects their initial optical properties and the sustainability of these properties over time. Therefore, the present study aimed to evaluate the stainability and translucency of different alum applied CAD-CAM materials (one additively manufactured hybrid composite resin, one additively manufactured composite resin, one feldspathic ceramic, one reinforced composite, and one polymer-infiltrated ceramic network) after coffee thermocycling (CTC). The hypotheses were that (i) time interval (after polishing-after alum, after alum-after CTC, and after polishing-after CTC) would affect the stainability within each alum applied material, (ii) condition would affect the overall (after polishing-after CTC) stainability within each material, (iii) time interval would affect the translucency within each alum applied material, (iv) CTC would affect the translucency of the control group within each material, and (v) condition would affect the after CTC translucency within each material.

2 | MATERIALS AND METHODS

Twenty disk-shaped specimens were prepared from each of the tested additively (Crowntec; Saremco Dental AG [CT] and Varseo Smile Crown Plus; Bego GmbH [VS]) and subtractively manufactured (Brilliant Crios; Coltene [RCR], CEREC Block; Dentsply Sirona [FC], and Vita Enamic; Vita Zahnfabrik [VE]) CAD-CAM materials. Detailed information and abbreviations of the test groups are listed in Table 1.

A disk-shaped standard tessellation language (STL) file with dimensions of Ø10 × 1 mm was designed by using a design software (Meshmixer v3.5.474; Autodesk Inc.) for the fabrication of CT and VS specimens. This STL file was transported into a nesting software (Composer v1.3.3; Asiga) and positioned on its flat surface. Supports were automatically generated and this configuration was duplicated 10 times. Specimens were printed with a layer thickness of 50 μm by using a digital light processing (DLP) printer (MAX UV; Asiga). After fabrication, CT specimens were cleaned with an alcohol-soaked (96%) cloth until all resin residues were completely removed, while VS specimens were ultrasonically cleaned in ethanol for 5 min (3 min of pre-cleaning in reusable ethanol and an additional 2 min in fresh ethanol). Specimens were then air-dried and light-cured either with 4000 (CT) or 3000 (VS) lighting exposures by using a Xenon lamp-curing device (Otoflash G171; NK Optik) under nitrogen oxide gas atmosphere. A 10 mm-wide cylinder was designed in STL format by using the same software for the fabrication of RCR, FC, and VE specimens. This STL file was used to mill cylinders from CAD-CAM blocks (Milling unit M1; Zirkonzahn), which were then wet-sliced into 1 mm-thick specimens by using a precision cutter (Vari/cut VC-50; Leco Corporation). All specimens were ultrasonically cleaned in distilled water for 10 min (Eltrosonic Ultracleaner 07-08, Eltrosonic GmbH, Wiesbaden, Germany) and dried with a paper towel prior to the measurements. All

TABLE 1 Materials used in this study.

Material	Type	Composition	Manufacturer
VarseoSmile Crown Plus (VS)	3D-printed composite resin	Esterification products of 4,4′-isopropylidiphenol, ethoxylated and 2-methylprop-2enoic acid, silanized dental glass, methyl benzoylformate, diphenyl (2,4,6-trimethylbenzoyl) phosphine oxide, 30–50 wt%—inorganic fillers (particle size 0.7 μm)	Bego, Bremen, Germany
CEREC Blocs (FC)	Feldspathic ceramic	SiO_2: 56–64 wt% Al_2O_3: 20–23 wt% Na_2O: 6–9 wt% K_2O: 6–8 wt% CaO: 0.3–0.8 wt% TiO_2: 0.0–0.1 wt% Pigments: <0.1 wt%	Dentsply Sirona, Bensheim, Germany
Brilliant Crios (RCR)	Reinforced composite resin	70.7 wt% barium glass (<1 μm) and amorphous silica (SiO_2; <20 nm), cross-linked methacrylates (Bis-GMA, Bis-EMA, TEGDMA)	Coltène AG, Altstätten, Switzerland
Crowntec (CT)	3D-printed composite resin	Esterification products of 4,4′-isopropylidiphenol, ethoxylated and 2-methylprop-2enoic acid, silanized dental glass, pyrogenic silica, initiators. Total content of inorganic fillers (particle size 0.7 μm) is 30–50 wt%	Saremco Dental AG, Rebstein, Switzerland
Enamic (VE)	Polymer-infiltrated ceramic network	14 wt% methacrylate polymer (UDMA, TEGDMA) and 86 wt% fine-structure feldspathic ceramic network	Vita Zahnfabrick, Bad Säckingen, Germany

specimens were controlled with a digital caliper (Model number NB60; Mitutoyo American Corp) to ensure final dimensions of the specimens. After fabrication, each specimen was polished in line with its respective manufacturer's recommendation.

Specimens were randomly into two groups as alum applied and control ($n = 10$). Baseline color coordinate measurement (L^*, a^*, and b^* values) of each specimen was performed on black, gray, and white backgrounds by using a digital spectrophotometer (CM-26d; Konica Minolta).[28,31,32] This spectrophotometer had 2° human observer characteristics, medium area view, and Commission International de l'Eclairage (CIE) D65 illumination. Optical contact was achieved by using saturated sucrose solution between the specimens and the backgrounds, and the spectrophotometer was calibrated before each measurement. Three measurements were recorded on each background for each specimen and these values were averaged. Same clinician performed all measurements in a temperature and humidity-controlled room with day light.

To apply alum, a solution was prepared by adding 5 g of nano potassium alum nano powder (Persian Chemistry Company) into 500 mL of water and stirred by using a magnetic mixer and heater device (MR 3001; Heidolph) for 5 min until homogenous. This homogenous mixture was then heated up to 90°C in a heater and held at this temperature for 5 min. The temperature was then reduced to 70°C and kept stable. The specimens were placed into the solution and kept in it for 15 min. Specimens were then removed and kept at room temperature until dry.[30] Color coordinate measurements were repeated for alum applied specimens.

All specimens were than subjected to thermocycling of 10,000 cycles (SD Mechatronik Thermocycler; SD Mechatronik GmbH) at 5–55°C in a coffee solution with a dwell time of 30 s and a transfer time of 10 s. A tablespoon of coffee (Intenso Roasted and Grounded; Kaffeehof GmbH) was dissolved in 177 mL of water to prepare the filtered coffee solution. The coffee solution was renewed with a freshly made one in every 12 h.[14,28,29,31] Coffee extracts were cleaned by brushing the surface of each specimen 10 times with a toothpaste (Colgate Total Pro Breath Health; Colgate-Palmolive) under running water. Specimens were then ultrasonically cleaned in distilled water for 10 min and dried (Figure 1). Color coordinate measurements were repeated for all specimens. Color difference (ΔE_{00}) of each specimen between different time intervals was calculated by using the coordinates measured on gray background and the CIEDE2000 formula[33] with parametric factors set as 1[1,14,31]:

$$\text{CIEDE2000} = \left[(\Delta L'/k_L S_L)^2 + (\Delta C'/k_C S_C)^2 + (\Delta H'/k_H S_H)^2 + R_T(\Delta C'/k_C S_C)(\Delta H'/k_H S_H) \right]^{1/2}.$$

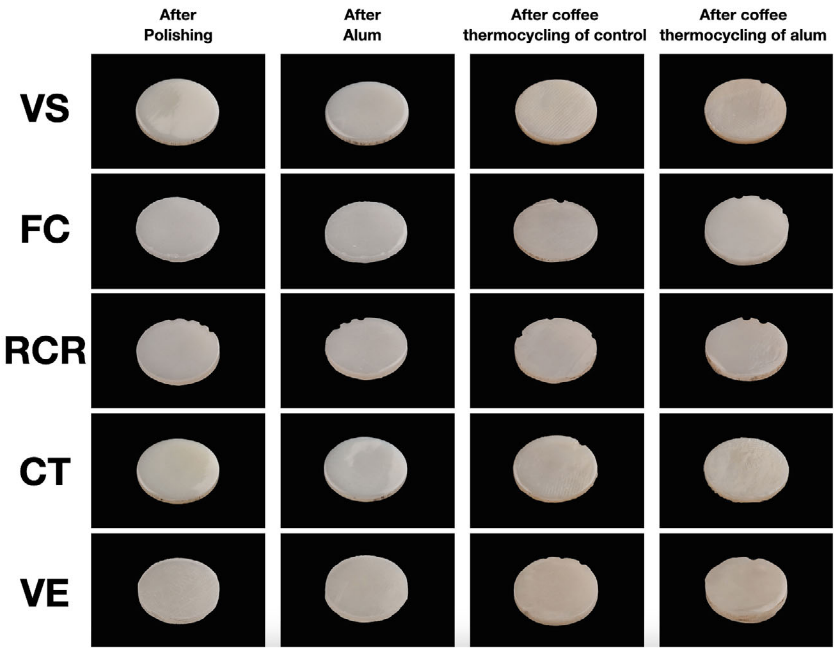

FIGURE 1 Representative image of specimen from each material at different time intervals.

The translucency (RTP) of each specimen at different time intervals was also calculated by using the same formula and the measurements performed on white and black backgrounds.

Distribution of data was analyzed by using Shapiro–Wilks test. Due to normal distribution of data, Bonferroni corrected repeated measures analysis of variance (ANOVA) test was used to evaluate the ΔE_{00} and RTP values among different time intervals within each alum applied group, while paired samples *t*-tests were used to evaluate the RTP values of each control group between different time intervals. Overall ΔE_{00} (after polishing-after CTC) and RTP values within each material depending on condition were analyzed by using either independent samples *t*-tests (ΔE_{00} of VS, FC, and CT, and RTPs, normal distribution) or Mann–Whitney *U* tests (RCR and VE, non-normal distribution). All analyses were performed by using a software (SPSS v23; IBM Corp) at a significance level of $\alpha = 0.05$. Perceptibility and acceptability of ΔE_{00} values (not perceptible: ≤0.8, perceptible but clinically acceptable: ≤1.8, moderately unacceptable: ≤3.6 clearly unacceptable: ≤5.4, and extremely unacceptable: >5.4 units) and differences in RTP values (ΔRTP, not perceptible: ≤0.62 and not acceptable: ≥2.62) were further evaluated by using previously reported threshold values.[34,35]

3 | RESULTS

Significant differences in ΔE_{00} values among different time intervals were observed within each alum applied material ($p \leq 0.004$). All materials had their lowest ΔE_{00} values after alum application ($p \leq 0.006$), except for FC, which had similar ΔE_{00} values after alum application and after CTC ($p = 0.177$) (Table 2). When overall ΔE_{00} values between each condition were evaluated within each material, RCR ($p = 0.029$) had significant differences, in which alum applied specimens had lower ΔE_{00} values (Table 3).

RTP values of all alum applied specimens had significant differences among different time intervals ($p < 0.001$) as all specimens had their lowest RTP after CTC ($p < 0.001$). However, while CT had higher RTP after polishing than after alum application ($p = 0.010$), the difference between these time intervals for other materials was nonsignificant ($p \geq 0.060$). CTC significantly reduced the RTP values of all materials in control groups ($p < 0.001$). Among tested materials, condition affected the RTP of only CT as alum applied group had higher values ($p = 0.049$) (Table 4).

4 | DISCUSSION

Significant differences were observed among different time intervals within alum applied specimens when ΔE_{00} values were considered. Therefore, the first hypothesis was accepted. However, only RCR had significantly lower ΔE_{00} values after CTC when alum was applied. Therefore, the second hypothesis was rejected. Among alum applied specimens, only CT had an imperceptible mean color change after alum application (0.59 units), while VE and RCR (0.89 and 1.29 units) had clinically acceptable and VS and FC had moderately unacceptable color changes (2.24 and 2.33 units). When after CTC and overall ΔE_{00} values were considered, VS, CT, and VE had extremely unacceptable ($\Delta E_{00} \geq 5.69$ units) and FC and RCR had clearly unacceptable ($\Delta E_{00} \geq 4.65$ units) color change. Increased ΔE_{00} values within each material after CTC, either significantly or nonsignificantly, may be associated with CTC's adverse effect on alum coating. However, it should be noted that the control groups of all materials had extremely unacceptable mean color change after CTC ($\Delta E_{00} \geq 5.46$ units).[34] In addition, for FC and RCR, the difference in overall ΔE_{00} values between alum applied and control specimens was perceptible. Considering these interpretations, it can be speculated that alum application after polishing may result in a color change that

TABLE 2 Descriptive statistics of ΔE_{00} values among different time intervals for each alum applied material.

	After polishing-after alum	After alum-after CTC	After polishing-after CTC
VS	2.24 ± 2.56^{a}	6.30 ± 0.92^{b}	5.69 ± 0.44^{b}
FC	2.33 ± 2.06^{a}	4.65 ± 1.63^{a}	5.27 ± 1.11^{b}
RCR	1.29 ± 0.93^{a}	5.16 ± 1.10^{b}	5.22 ± 0.54^{b}
CT	0.59 ± 0.30^{a}	7.09 ± 0.41^{b}	7.13 ± 0.47^{b}
VE	0.89 ± 1.22^{a}	7.04 ± 2.74^{b}	7.12 ± 2.64^{b}

Note: Different superscript lowercase letters indicate significant differences among time intervals within each material ($p < 0.05$).

TABLE 3 Descriptive statistics of after polishing-after CTC ΔE_{00} values of each material between different conditions.

	Alum applied		Control	
	Mean ± standard deviation	Median (min–max)	Mean ± standard deviation	Median (min–max)
VS	5.69 ± 0.44^{a}	5.64 (5.16–6.31)	5.46 ± 0.86^{a}	5.52 (4.20–7.01)
FC	6.47 ± 1.11^{a}	6.60 (4.70–8.30)	6.77 ± 1.27^{a}	6.85 (5.23–9.61)
RCR	5.22 ± 0.54	5.26^{a} (4.11–5.61)	7.38 ± 3.94	5.99^{b} (5.74–15.07)
CT	7.13 ± 0.47^{a}	7.19 (6.12–7.78)	6.91 ± 0.33^{a}	6.93 (6.51–7.61)
VE	7.12 ± 2.64	6.26^{a} (5.18–13.97)	7.41 ± 0.88	7.31^{a} (6.32–9.27)

Note: Different superscript lowercase letters indicate significant differences within each material ($p < 0.05$).

TABLE 4 Descriptive statistics of RTP values each material-condition pair among different time intervals.

	Alum applied			Control	
Material	After polishing	After alum	After CTC	After polishing	After CTC
VS	20.43 ± 1.35^{a}	20.62 ± 1.80^{a}	9.92 ± 0.81^{bA}	18.78 ± 2.77^{a}	10.07 ± 0.88^{bA}
FC	21.70 ± 2.13^{a}	19.81 ± 1.68^{a}	10.64 ± 0.86^{bA}	24.4 ± 6.94^{a}	11.82 ± 2.20^{bA}
RCR	24.09 ± 2.17^{a}	22.32 ± 3.33^{a}	12.38 ± 0.90^{bA}	24.1 ± 1.96^{a}	12.82 ± 1.08^{bA}
CT	24.87 ± 0.96^{a}	23.89 ± 1.38^{b}	12.29 ± 0.67^{cB}	25.05 ± 0.78^{a}	11.61 ± 0.63^{bA}
VE	26.70 ± 3.49^{a}	24.99 ± 3.21^{a}	13.12 ± 1.57^{bA}	29.49 ± 2.76^{a}	14.11 ± 1.23^{bA}

Note: Different superscript lowercase letters indicate significant differences among time intervals within each material-condition pair, while different superscript uppercase letters indicate significant differences between conditions within each material ($p < 0.05$).

is moderately unacceptable at worst when tested materials were considered and alum application improves the color stability of FC and RCR after long-term coffee exposure. It should also be emphasized that due to the duration of CTC, all specimens were exposed to coffee excessively and for those patients who seldomly consume coffee or do not consume at all, these values may change.

The lowest RTP values among different time intervals within each alum applied material were observed after CTC, while alum application also reduced the RTP of CT. Therefore, the third hypothesis was accepted. In addition, CTC significantly reduced the RTP values of each control group, which led to the acceptance of the fourth hypothesis. The fifth null hypothesis was also accepted as alum applied CT had higher values than its control group. Even though alum application only reduced the RTP values of CT significantly, it should be noted that all materials had perceptible mean differences in translucency after alum application (ΔRTP ≥ 0.98 units), except for VS (ΔRTP $= 0.19$ units). CTC not only significantly reduced the RTP values of each material regardless of the condition, it also led to unacceptable differences when compared with after alum stage of alum applied and after polishing stage of control group specimens (ΔRTP ≥ 8.71 units). None of the ΔRTP values between the alum applied and the control groups after CTC was unacceptable (ΔRTP ≤ 1.18 units), while the difference between the alum applied and the control groups was also imperceptible for VS and RCR.[35]

Therefore, it can be concluded that alum application after polishing may not lead to translucency changes that result in esthetic complications, but it would not increase the resistance to detrimental effect of coffee consumption on translucency when tested materials were considered.

Previous studies have also investigated the optical properties of tested materials with a similar methodology.[28,32,33] A recent study has evaluated the effect of polishing techniques on the stainability of CT and VS when subjected to CTC, and concluded that polishing technique did not affect the stainability of CT, whereas that of VS was lower when treated with a surface glaze than conventional polishing.[28] In another study, RCR was subjected to 5000 cycles of CTC, and had a mean ΔE_{00} of 0.9 units.[32] Higher mean ΔE_{00} values of RCR in the present study may be related to the higher number of CTC cycles. Arif et al.[33] evaluated the stainability of VE in different thicknesses (0.7 and 1.5 mm) and concluded that 1.5 mm-thick VE had clinically acceptable color change, whereas 0.7 mm-thick VE had unacceptable color change when subjected to 6000 cycles of CTC. However, to the authors' knowledge, the present study was the first on the effect of alum application on the optical properties of additively and subtractively manufactured materials. Therefore, the results of the present study regarding the effect of alum could not be compared with previous studies.

Even though significant differences were reported for each parameter investigated, the absence of a priori power analysis to determine the number of specimens in each group was a limitation. Nevertheless, post hoc power analyses were performed for each parameter investigated. Statistically significant differences were found between conditions, and the sample size was deemed adequate for a minimum of 99% power with a minimum effect size of 2.44 and $\alpha = 0.05$ for comparisons among time intervals. Another limitation of the present study was that CTC arrangement stains both sides of the specimens, which might have aggravated staining and led to a greater color change than the actual clinical situation in which only the polished surface of the restorations is exposed to discolorants. In addition, CTC arrangement did not involve saliva and different staining solutions may affect tested parameters. All specimens were polished in line with their respective manufacturer's recommendations, but alum application after glazing or addition of alum powder to glaze powders may lead to different results. Future studies should focus on alternative application methods of alum on different CAD-CAM materials and how other clinically relevant properties are affected by this process to broaden the knowledge on the applicability of alum.

5 | CONCLUSIONS

Within the limitations of the present study, it can be concluded that:

1. Alum application resulted in imperceptible, perceptible, or moderately unacceptable initial color change of tested materials, based on previously reported threshold values.
2. CTC led to unacceptable color changes within each material, regardless of alum application. However, alum application enabled higher overall color stability for reinforced composite resin than its control.
3. Alum application mostly resulted in clinically acceptable changes in translucency, while CTC reduced the translucency of each material, which was clinically unacceptable, regardless of the condition.

CONFLICT OF INTEREST STATEMENT

The authors declare that they do not have any financial interest in the companies whose materials are included in this article.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available on request from the corresponding author. The data are not publicly available due to privacy or ethical restrictions.

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