

## Effect of thermocycling and khat extract staining on the optical and mechanical properties of ceramic materials

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**ABSTRACT**

**Background:** Color stability of CAD/CAM prosthetic materials is important for long-term clinical and survival success during daily chewing habit. **Aims:** To assess the effects and outcome of thermocycling and khat extract (KE) staining on the optical (i.e., TP and OP) and physical properties of multilayer zirconia and feldspathic CAD/CAM ceramic. **Methods:** Forty specimens with dimensions of 16 mm × 12 mm × 1.8 mm were produced from multilayer zirconia and feldspathic CAD/CAM ceramic materials. Specimen's surfaces were prepared in glazed and polished forms. Before thermocycling, the color parameters of specimens were measured using a spectrophotometer, again and after the thermal aging process, the color measurement was recorded and analyzing the collected data using descriptive statistics, correlation, one-way ANOVA, and Tukey's tests. **Results:** After thermocycling and KE staining, the color parameters L, a, and b decreased, and  $\Delta L^*$ ,  $\Delta a^*$ ,  $\Delta b^*$  had significant differences between groups (p values  $\geq 0.050$ ). Polished surfaces had higher TPs compared with glazed surfaces. OP values ranged from 22.1 to 16.2 and were higher in glazed than in polished surfaces. Glazed and polished zirconia (777.42±72.67N and 533±50.71 N) had significantly higher compressive fracture forces compared with glazed and polished Vita Triluxe (396.25±42.24N and 338.66±40.17N). **Conclusion:** KE staining and thermocycling caused slight color changes in TP and OP values, which were high in polished surfaces. TP and OP values were reduced after KE immersion and thermocycling. Compressive forces were under clinically accepted values for zirconia and less than half values for Vita Triluxe specimens. The three types of failures were presented.

**Keywords:** color measurements, feldspathic ceramic, khat, opalescence parameter, thermocycling, translucency parameter, zirconia



## 1. INTRODUCTION

All ceramics have become tremendously important materials in dental prostheses fabrication because of their many advantages, such as biocompatibility, durability, survival for extended period, and excellent aesthetic capabilities with long-term follow up. These materials show translucency and color brightness and intensity similar to those of natural non-stained teeth (Powers et al., 2012). As a result of continuing improvements, several materials have been utilized to fabricate ceramic prosthesis, including feldspathic porcelain, glass-based ceramics, and zirconia-based ceramics (Singh et al., 2014). Currently, in modern dental practice, zircon-based ceramics are considered as one of the most popular materials to fabricate different types of all ceramic restoration (Fehmer et al., 2014) due to its superior mechanical properties (Arena et al., 2019; Haralur et al., 2019) and excellent biocompatibility compared with other dental ceramics (Abu-Obaid et al., 2020).

Khat plants are regularly chewed by people in Jazan region because of their psychostimulant effect, which is similar to that produced by amphetamine-like elements (Wabe, 2011). These leaves are regularly chewed in the left side of the mouth. Fresh leaves are masticated in the form of bolus and held in the lower buccal area unilaterally or bilaterally for 4–6 hours or longer (Al-Alimi et al., 2018; Al-Anesi et al., 2019). Exploring the effect of khat on materials used for dental restorations and prosthesis is essential. Nevertheless, studies on the effect of khat on oral health remain scarce. Few studies investigated the association among khat mastication and dental ceramic materials. Moreover, the role of khat as a staining material contributing to dental ceramic discoloration and surface effect in relation to maintaining the lustering surfaces of ceramic materials should be studied (Al Moaleem et al., 2020a; Al Moaleem et al., 2020b; Al Moaleem et al., 2020c; Al Moaleem et al., 2020d).

The CIE  $L^*$   $a^*$   $b^*$  color system is used for the determination of color values (Al Moaleem et al., 2020c; Al Moaleem et al., 2020d). CIE  $L^*$ ,  $a^*$ , and  $b^*$  values are known as “chromaticity coordinates”. The  $L^*$  value refers to “lightness”, and a high  $L$  value indicates high lightness. The  $a^*$  value reveals red and green colors, whereas the  $b^*$  value indicates yellow and blue colors (Alghazali et al., 2012; Alghazali et al., 2019). The translucency parameter (TP) of ceramic materials is defined as the percentage of light transmitted through a ceramic (Tuncel et al., 2013). TP is measured by placing the examined specimens under white (W) and black (B) backgrounds (Tabatabaian, 2019). Opalescence is an optical property in which visible light disperses short wavelengths, thereby giving the object a bluish appearance in the reflected color and an orange/brown appearance in the transmitted color. The opalescence parameter (OP) is used to regulate the opalescence of materials used in oral cavity, which is the difference in yellow–blue (CIE  $\Delta b^*$ ) and red–green (CIE  $\Delta a^*$ ) color coordinates between the reflected and transmitted colors (Oh and KIM, 2015; Kim and Kim, 2014).

Substances, such as alkaloids, sterols, terpenoids, tannins, flavonoids, glycosides, amino acids, minerals, and vitamins are found in khat (Wabe, 2011). The continuous contact of khat with oral soft and hard tissues may affect oral tissues and salivary glands, leading to xerostomia and increased rate of caries (Nyanchoka et al., 2008). Furthermore, the habit of khat chewing minimizes salivary flow, increases viscosity, and decreases pH (Al-Alimi et al., 2018). Huge amounts of soft drinks or beverages and sugar tablets are used to minimize the unpleasant taste of khat, which may cause cervical discoloration in enamel and dentine for a long period and lead to staining of teeth, attrition, and cervical caries at the chewing side (El-Wajeh and Thornhill 2009; Al-Meshal et al., 1991).

Intraoral occlusal adjustments for CAD/CAM prostheses are usually performed by a dental practitioner. Such alterations include the removal of a part of the glazed layer from the outer ceramic surfaces, leaving some pores in the exposed material and creating a rough surface. These corrections may cause the staining of the prostheses that needs either reglazing or polishing to maintain luster and an unchanging restoration color (Alghazali et al., 2019; Motro et al., 2012). Thus, this study was conducted to assess the effects of khat extract (KE) on the optical (i.e., TP and OP) and physical (i.e., compressive strength and fracture mode) properties of a multilayer zirconia (Ceramill Zolid PS) and feldspathic (Vita Triluxe Forte) CAD/CAM ceramic materials. The null hypothesis is no difference in TP or OP values after thermocycling and khat immersion based on 50%:50% perceptibility (PT) and acceptability (AT) thresholds and no difference in the values and type of compressive strength or fracture modes before and after thermocycling among the glazed or polished CAD/CAM restorative materials and immersion in KE.

## 2. METHODOLOGY

### Study design

This study was conducted in Jazan University, College of Dentistry, Saudi Arabia in October 2021. The effect of KE on the optical properties, such as TP and OP, of the specimens were assessed, and the color changes in the above specimens in relation to VITA

classical shade guides were compared. The physical properties, such as CS and modulus of elasticity (ME) tests, were performed in all specimens.

### Specimens' fabrications and surface treatments

Forty specimens were fabricated using the CAD/CAM system from a multilayer zirconia (Ceramill Zolid PS) and feldspathic (Vita Triluxe Forte) CAD/CAM ceramic materials (Vita Zahnfabrik). The dimensions of the disc-shaped specimens were  $12 \times 2.0 \pm 0.2$  mm in diameter and thickness. Twenty specimens were placed in each group. Specimens were sintered and glazed in a furnace for 2 hours at  $1550^{\circ}\text{C}$ . After cooling the specimens at room temperature, half of the specimens were glazed. The other half was subjected to a surface finishing process under water with sand paper and polished with a porcelain polishing kit to represent the clinical simulation after polishing. The thicknesses of specimens were measured using a digital caliper.

### Optical properties and color measurements

Following the manufacturer's instructions, a spectrophotometer with 6mm diameter tip (VITA Easyshade 3 Advance, Vita Zahnfabrik, Bad Säckingen, Germany) was calibrated before each measurement. After numbering of 40 specimens, CIE  $L^*$ ,  $a^*$ , and  $b^*$  values were recorded for each specimen by using a spectrophotometer. For each specimen, measurements were accomplished from different points on B and W backgrounds, and the average values were obtained. Color parameters, such as  $L1$ ,  $a1$ , and  $b1$  values, of the specimens were carried out by spectrophotometry on W and B backgrounds. A square window opening with  $2 \times 2$  cm was used and fixed to B and W backgrounds to ensure that the specimen was in the same area during the recording of the optical reading. TP measurements were obtained by calculating the color difference in the specimen over B and W backgrounds by using this formula:

$$TP = [(L_B - L_W)^2 + (a_B - a_W)^2 + (b_B - b_W)^2]^{1/2}$$

Where subscripts B and W refer to the color coordinates over B and W backgrounds, respectively (Koseoglu et al., 2019; Vasiliu et al., 2020). A TP of 0 corresponds to a completely opaque material. A high TP indicates high actual translucency of the material. OP was calculated to determine the opalescence of specimen, which is the modification in yellow–blue (CIE  $\Delta b^*$ ) and red–green (CIE  $\Delta a^*$ ) color coordinates amongst reflected and transmitted colors (Vasiliu et al., 2020; Ardu et al., 2008) by using this equation:

$$OP = [(a_B^* - a_W^*)^2 + (b_B^* - b_W^*)^2]^{1/2}$$

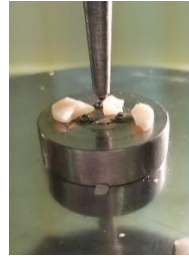
### KE immersion and thermocycling

After the baseline or first measurements of color parameters  $L$ ,  $a$ , and  $b$ , KE was prepared and offered by the Substance Abuse and Toxicology Research Center, Jazan University. The homogenate of khat greenery was prepared by finely mincing fresh khat leaves in 100% distilled water (v/w). KE was then kept in a  $-80^{\circ}\text{C}$  ultra low-temperature freezer until use. Afterward, KE was mixed with NaOH until its pH was comparable with the pH of saliva and oral cavity. All specimens were immersed in KE for 15 days as mentioned in previous in vitro studies (Al Moaleem et al., 2020c; Al Moaleem et al., 2020d). The same procedures were repeated daily with fresh KE. Thermocycling was applied to CAD/CAM restorative materials specimens in an especially designed device containing four chambers with deionized water at standard temperatures (Koseoglu et al., 2019; Vasiliu et al., 2020; Aldosari et al., 2021). All specimen groups were subjected to 5000 thermocycles. Specimens were immersed for 15 seconds in each tank in accordance with the following sequence and ISO 11405 standards:  $5^{\circ}\text{C}$  to  $37^{\circ}\text{C}$  to  $55^{\circ}\text{C}$  to  $37^{\circ}\text{C}$  (Papageorgiou-Kyrana et al., 2018).

The color parameters of specimens were measured again after KE staining and thermocycling and these values were considered as "after".  $L2$ ,  $a2$ , and  $b2$  were assessed again under the same position, operator, and background, and the differences between second and first values were calculated as the average values for  $\Delta L$ ,  $\Delta a$ , and  $\Delta b$ . These values were used in the equation for color parameter measurements.

### Comprehensive force tests

The compressive force test is a shared in vitro assessment to calculate the strength of dental restorative ceramics. The piston-on-three-ball test was used to measure the biaxial flexural force. Disc specimens were centered and supported on 3 evenly spaced steel balls (3.4mm diameter). Diameters of the piston tip and the support circle were 1.2 and 12.0 mm, respectively (Figure 1). Forces were focused to the center of the specimen through a flat tip of the piston of 1.4mm radius, and at a cross head of 1.0 mm/min as suggested in ISO 6872 and in air at room temperature by using a universal mechanical testing apparatus. A thin plastic film (50  $\mu\text{m}$  in thickness) was stuck to the external surfaces of specimens. Thus, the piston distributed the load uniformly (Stawarczyk et al., 2015; Alakkad et al., 2021). The forces of fracture load were recorded in Newtons (N), and the mean compressive force of each group was calculated and compared.



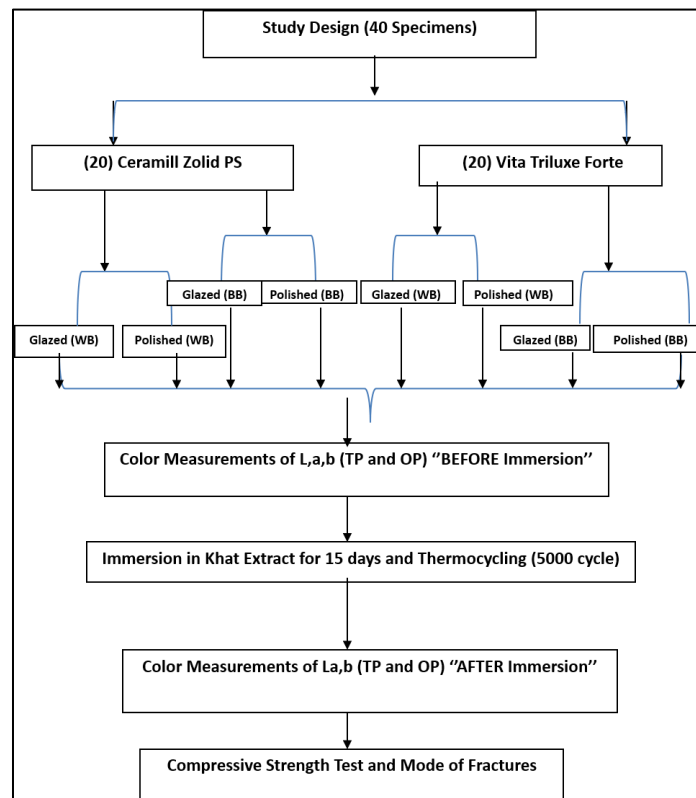
**Figure 1** Specimens during compressive strength application

**Recording of failure modes**

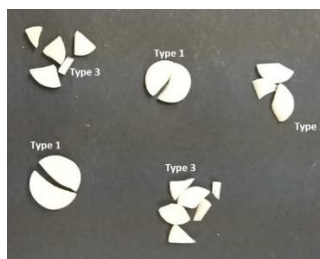
The mode of failure was classified in accordance with the criteria presented in Table 1. For each specimen, the number of pieces after fractures and its percentage from the original shape of the specimens are recorded as follows: type 1, broken in two half or 50%–50% of the original size; type 2, 3–4 fractures where each piece measured 20%–35% of the original size of the specimen; and type 3, each piece measured <20% from the original size of the specimen (Table 1). The study design of the specimens is shown in Figure 2. Figure 3 shows the specimens after fracture and failure mode.

**Table 1** Criteria for failure classifications

Fracture mode	Definitions
Type 1/ Uniform fracture passing at the middle	Specimens were divided into two equal sizes, reparable fracture
Type 2/ Mixed fracture resulting in 3–4 fractures	Specimens were divided into 3–4 pieces, less reparable fracture
Type 3/Complicated fracture resulted in more than four fractures	Specimens were divided into several pieces, irreparable fracture



**Figure 2** Study design and different steps of the study



**Figure 3** Specimens' failure types

**Statistical analysis**

Using the IBM SPSS 20.0 package program descriptive statistics was used to calculate the mean L\*, a\*, and b\* values of the CeramillZolid and Vita Triluxe CAD/CAM ceramic materials at B and W backgrounds and before and after thermocycling and KE immersion. Also, for CS forces and type of failures after thermocycling and KE immersion, the Pearson correlation test was performed to determine the relationship of the type of ceramic specimens with L\*, a\*, b\*, and TP or OP values. For repeated measurements, the one-way ANOVA was performed to compare the specimens subjected to thermocycling in terms of TP or TO, ΔL, Δa, and Δb. The Tukey's test was performed to compare different glazed or polished ceramics. P < 0.05 indicated statistically significance.

**3. RESULTS**

Spectral distributions and color coordinates of the L\*, a\*, and b\* values were measured in the reflectance mode over the W and B backgrounds separately. The changes in L\* (L1 and L2), a\* (a1 and a2), and b\* (b1 and b2) values are summarized in Table 2 for Ceramill Zolid zirconia in glazed forms and polished surfaces over W and B backgrounds and before or after immersion in KE and thermocycling for 15 days. Table 3 shows the same parameters for feldspathic Vita Triluxe specimens. All values for L, a, and b were within normal values in relation to type of ceramic, surface, background, and thermocycling. Before and after thermocycling, the maximum L1 and L2 (81.22 and 80.64) values were observed in polished Vita Triluxe and under B and W backgrounds. The Ceramill Zolid recorded the highest value before and after thermocycling in glazed and polished forms (20.62 and 20.80, respectively) under Band W backgrounds. Overall, color values of Ceramill Zolid were slightly higher than those of Vita Triluxe specimens.

**Table 2** Color values of Ceramill Zolid and Vita Triluxe (glazed and polished) before (L1, a1, b1) and after (L2, a2, b2) khat extract immersion and thermocycling over black and white backgrounds.

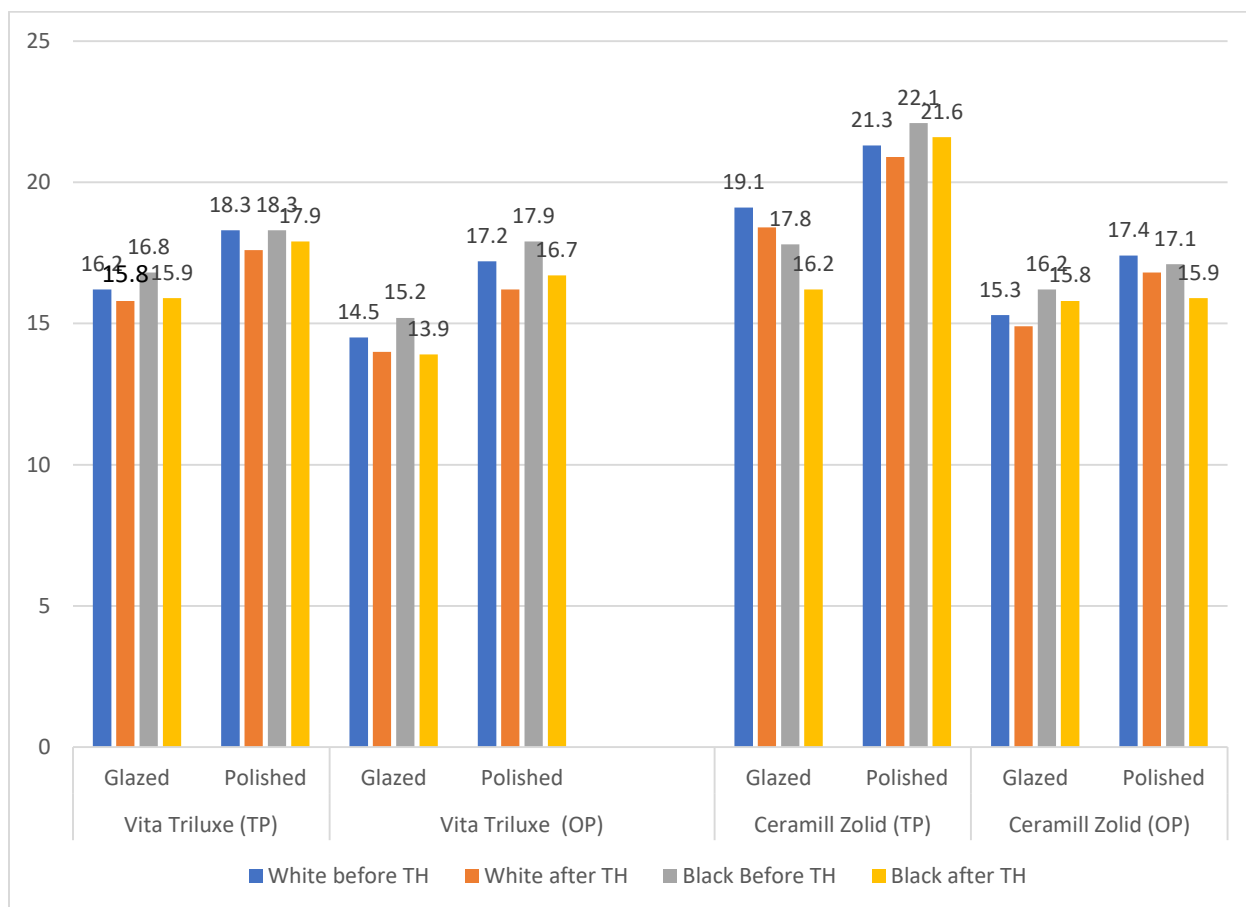
Ceramic type and Surface	Color parameters under black background					
	L1/ baseline Before TH	L2 / 15 days After TH	a1/ baseline Before TH	a2 /15 days After TH	b1/Baseline Before TH	b2/15 days After TH
CeramillZolid, Glazed	75.08	74.62	1.24	1.12	20.62	19.04
CeramillZolid, Polished	77.30	76.48	1.65	1.22	20.04	19.84
	Color parameters under white background					
	L1 /baseline Before TH	L2 / 15 days After TH	a1 /baseline Before TH	a2 /15 days After TH	b1 /Baseline Before TH	b2/15 days After TH
Ceramill Zolid, Glazed	79.12	77.80	2.48	2.28	19.62	18.04
Ceramill Zolid, Polished	80.02	77.88	2.38	2.16	20.08	19.86
	Color parameters under black background					
	L1/ baseline Before TH	L2 / 15 days After TH	a1/ baseline Before TH	a2 /15 days After TH	b1/Baseline Before TH	b2/15 days After TH
Vita Triluxe, Glazed	76.04	75.28	1.46	1.12	20.26	19.40

Vita Triluxe, Polished	76.36	75.02	1.98	1.82	20.40	19.48
Ceramic type and Surface	Color parameters under white background					
	L1 /baseline Before TH	L2 / 15 days After TH	a1 /baseline Before TH	a2 /15 days After TH	b1 /Baseline Before TH	b2/15 days After TH
Vita Triluxe, Glazed	80.14	79.77	2.66	2.48	19.22	18.48
Vita Triluxe, Polished	81.22	80.64	2.28	2.02	20.80	20.22

Table 3 shows a significant difference (p values  $\geq 0.05$ ) in the three-color parameters  $\Delta L^*$ ,  $\Delta a^*$ , and  $\Delta b^*$  between the zirconia Ceramill Zolid and feldspathic vita Triluxe in both surfaces. A uniform reduction in the TP and OP values for both ceramics with glazed and polished surfaces after KE immersion and thermocycling was documented (Figure 4). For both materials, polished surfaces had higher TPs compared with glazed surfaces. The TP for polished Ceramill Zolid recorded the highest values compared with that for polished Vita Triluxe under both backgrounds and thermocycling followed by glazed Ceramill Zolid. OP values were ranged from 22.1 to 16.2 and were higher than those of glazed surfaces with reductions in all parameters. All specimens showed a slight uniform reduction in TP and OP values after thermocycling and KE immersion (Figure 4).

**Table 3**  $\Delta L^*$ ,  $\Delta a^*$ ,  $\Delta b^*$  values of glazed and polished specimens after KE immersion and thermocycling

Ceramic type	$\Delta L^*$	$\Delta a^*$	$\Delta b^*$	P values
Ceramill Zolid BLACK				0.042*
Glazed	0.80±0.24	0.18±0.41	0.17±1.12	
Polished	0.79±1.24	0.24±0.44	0.22±0.07	
Vita Triluxe WHITE				0.008*
Glazed	0.82±0.02	0.19±0.49	0.19±1.21	
Polished	0.81±1.02	0.27±0.53	0.25±0.14	



**Figure 4** TP and OP values for glazed and polished ceramics before and after thermocycling and KE immersion.



Glazed and polished Ceramill Zolid ( $777.42 \pm 72.67\text{N}$  and  $533 \pm 50.71\text{N}$ ) had higher compressive fracture forces strength compared with glazed and polished Vita Triluxe ( $396.25 \pm 42.24$  and  $338.66 \pm 40.17$  MPa, respectively) (Figure 5). Significant differences were recorded between the two ceramic materials and two surfaces ( $p$  value  $\geq 0.050$ ). The percentage of failure modes after CS application is shown in Figure 6. In CeramillZolid specimens, most fracture modes were “specimens divided into two equal sizes/type 1” for glazed samples, whereas the opposite was recorded in polished surfaces with 40% for type 1 and 60% for type 2” Specimens were divided into 3-4 pieces (between 20%-35%”. In Vita Triluxe, the higher percentage to the glazed surfaces were 60%” Specimens were divided into 3-4 pieces (between 20%-35%), while for polished samples the higher was type 3 mode of fractures” Specimens were divided into several pieces (each piece was lesser than 20%)”.

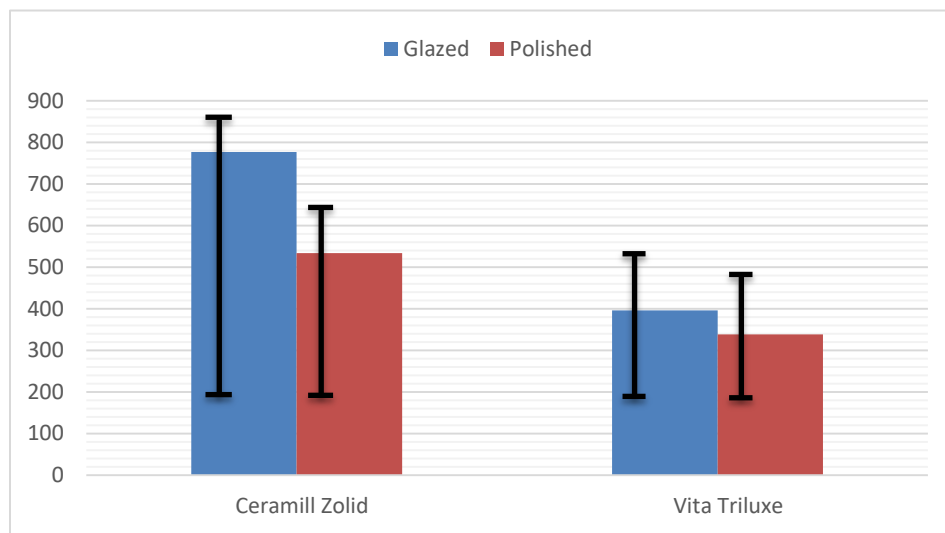


Figure 5 Mean and SD of compressive strength for glazed and polished ceramics

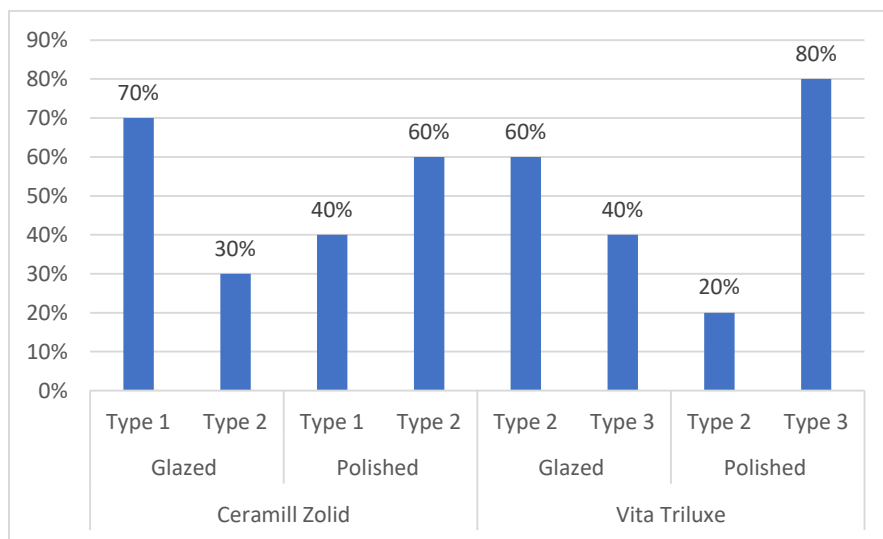


Figure 6 Percentage of the failure mode of different ceramics with their surfaces

#### 4. DISCUSSION

In this invitro study, optical properties, such as TP and OP, were evaluated using two types of ceramics and in forms of glazed and polished surfaces. TP and OP were calculated before and after thermocycling and KE immersion. In addition, CS and fracture type failures were examined and documented. The null hypothesis, i.e., the TP and OP before KE immersion and thermocycling were not different with TP and OP after KE immersion and thermocycling was rejected. In this study, TP, and OP changes after thermocycling were observed in types of ceramics, types of surfaces, and after KE immersion and thermocycling. Also, for the second hypothesis, i.e., no difference in the values of CS or fracture modes before or after thermocycling between glazed or polished CAD/CAM restorative materials after immersion in KE was rejected because we observed different CS values and failure types for

tested materials. Overall, TP and OP values were slightly affected by KE staining followed by thermocycling and were recorded in the majority of the published studies that tested the color changes in CAD/CAM restorative materials strained by coffee, beverage materials, and soft drinks.

KE significantly affects the color of the ceramic materials in forms of glazed or polished feldspathic porcelain, zirconia, and natural teeth. Also, khat chewing significantly affects bacterial biodiversity in oral biofilms and discolors prosthetic dental materials. In the current study, khat affected TP and OP (Figure 4). The highest TP (22.1) observed in polished zirconia, whereas the highest OP (17.9) was observed in polished Vita Triluxe. Similar results were recorded by Koseoglu et al., (2019), who documented that TP values for zirconia reach 18. The TP values of zirconia restorative materials obtained by Wahba MM 2011 those recorded 9.6 TP as a highest value registered among all tested groups.

Vasiliu et al., (2020) examined the TP and OP values of milled zirconia-reinforced lithium silicate glass-based ceramic and a milled feldspathic ceramic and found that the TP values of milled feldspathic and zirconia ceramics are  $15 \pm 1.2$  and  $13 \pm 1$ , respectively. These values are close to the values recorded in the present study. Vasiliu et al., (2020) documented that the values of OP for zirconia and feldspathic ceramics are higher before thermocycling for the heat-pressed feldspathic ceramic ( $8 \pm 0.79$ ) and zirconia ( $9 \pm 0.85$ ) displayed higher OP values. The recorded OP values in the present study ranged between 15 and 17.6. The high differences between the OP values of the present study and Vasiliu et al., (2020) could be attributed to the immersion instaining materials, aging periods, and type of surface. The same study recorded that polished samples of restorative materials are higher than glazed samples of restorative materials. This finding is in parallel with our findings for glazed or polished surfaces. Overall, the TP obtained in the present study was slightly higher before KE immersion compared with that after KE immersion. This finding agreed with studies that investigated the TP of ceramic materials (Eldwakhly et al., 2019; Koseoglu et al., 2019; Vasiliu et al., 2020), which recorded less TP values after immersion than before immersion. Al Moaleem et al., (2020a, b) found that khat is a coloring agent that causes superficial discoloration of the intraoral ceramic materials either feldspathic or all ceramic. Also, he recorded that the mean color changes were more among polished samples (Al Moaleem et al., 2020c, d), this was agreed for polished samples in this study.

Color change, OP, and TP were determined using the VITA Easyshade Compact (Vita, Zahnfabrik H. Rauter Gmb H & Co. KG) spectrophotometer previously used in multiple studies for the same purpose. This method is reliable because of the internal light source illuminant. Thus, environmental lightening conditions do not influence color measurements (Aldosari et al., 2021; Adawi et al., 2021). Relying on large-diameter fiber optics arranged in a stainless-steel probe that can illuminate the surface and receive light that is internally scattered avoids the specular reflected light that contains little or no color information and can negatively affect the color measurement (Steele and Johnson, 1999; Mjor and Gordan, 2002). During measurements, the contact probe tip with 5 mm diameter was placed at the center of each specimen from the veneered side with a circle of rubber base materials to prevent and standardize the application of light during the measurement of color parameters (Haralur et al., 2019).

Thermocycling is a widespread process of artificial accelerated aging of CAD/CAM ceramics that replicates the oral environment as an extrinsic factor (Savaş and Subaşı, 2015; Koseoglu et al., 2019; Vasiliu et al., 2020; Aldosari et al., 2021). The water aging system is an especially designed device consisting of four tanks with deionized water at standard temperatures. All specimen groups were subjected to five days thermocycles, with 1000 per day. Specimens were immersed for approximately 17 seconds in each chamber in accordance with the following sequence and ISO 11405 standards:  $5^{\circ}\text{C}$  to  $37^{\circ}\text{C}$  to  $55^{\circ}\text{C}$  to  $37^{\circ}\text{C}$  (Papageorgiou-Kyranas et al., 2018; Koseoglu et al., 2019). The thermocycling method could affect the longevity of the restoration and simulate the behavior of the ceramic material in the oral environment. As shown in Graph 4, thermocycling showed and recorded lower TP and OP values for all tested CAD/CAM materials in their forms of glazed or polished compared with before aging and thermocycling. This finding totally coincided with the results documented by Koseoglu et al., (2019) who tested zirconia materials after staining in beverages, Vasiliu et al., (2020) who tested ceramic materials and thermocycling, and Aldosari et al., (2021) who tested glazed and polished CAD/CAM materials. Tango1 et al., (2021) tested the TP of different CAD/CAM materials (Cerasmart, IPS e.max, Lava Ultimate, Shofu HC, Vita Enamic, and Vita Suprinity) and concluded that aging and thermocycling (ISO 4892-2 standard) for 15 days resulted in low values of Delta TP after immersion in different beverages. The same was evident among our samples, which showed low TP values after aging and thermocycling for similar CAD/CAM materials.

The values and types of CS or fracture modes before and after thermocycling the tested glazed or polished CAD/CAM restorative materials after immersion in KE are different. Thus, the second null hypothesis was rejected. Alakkad et al., (2021) found a relationship between the surface treatments (surface glazing) in increasing the fracture strength compared with polished surfaces for restorations made CAD/CAM with high significant differences between the two surfaces among the tested samples of such as IPS e.max CAD, Celtra Duo, and IPS Empress CAD. In the present study, glazed surfaces had significantly higher values compared with polished surfaces.



Savaş and Subaşı, (2015) compared the fracture resistance of restoration fabricated by CAD/CAM by using Lava Ultimate (1525±394 N), IPS Empress CAD (1364.3±545.6 N), and CEREC (1231.9±412.2 N) after accelerated artificial aging. These forces were slightly higher than forces recorded in this study for glazed (777.42± 72.67N) and polished (533±50.71N) zirconia. This finding could be explained by the different compositions of CAD/CAM tested materials, which had similar fabrication methods and aging process. In addition, Savaş and Subaşı (2015) showed that the tested materials had higher compressive fracture than the feldspathic CAD/CAM Vita Triluxe ceramic material used in the current study (glazed surface, 396.25±42.24N; polished surface, 338.66±40.17N). This finding might be because fracture forces that resulted from feldspathic ceramic are less than those from other ceramics, such as leucite-based glass-ceramics (IPS Empress CAD), resin nano-ceramic (Lava Ultimate), and fine particle feldspathic glass-based ceramic (CEREC, Table 4). Also, Savaş and Subaşı, (2015) showed no significant difference among the groups in accordance with the one-way ANOVA (p value= 0.304), and this finding was not parallel with the findings of the present study, which showed a significant difference (p values ≥ 0.050, Table 4). The IPS e.max CAD recorded higher fracture forces than LAVA Ultimate and VITA Enamic (Stawarczyk et al., 2015). A ceramic with fracture forces value near or equal to 1000 N is clinically acceptable. In the current study, compressive fracture forces were slightly under the clinically accepted values for zirconia Ceramill Zolid and less than half the clinically accepted values for Vita Triluxe specimens.

*In-vivo* studies on prosthetic materials are critical but are not always possible due to complications related to patient follow-up, ethical considerations, and cost. Thus, *in-vitro* studies are useful for gaining information about behavior of the restorative materials over a short period. Using a spherical headpiece on the middle of an occlusal surface with a punctual compression force is the most suitable method for producing fracture patterns similar to those encountered in clinical practice (Steele and Johnson 1999). This method was used to gain fracture strength values in the current study. Interestingly, duplicating the oral environments of the patients entirely prevents the assessment of the survival rate and compatibility of prosthetic CAD/CAM materials (Major and Gordan, 2002). Savaş and Subaşı (2015) stated that 50% of tested samples made from CAD/CAM by using Lava Ultimate, IPS Empress CAD, and CEREC show repairable fracture types and that half of the teeth show irreparable (catastrophic) fracture types after accelerating artificial aging. This finding marginally agreed with the fracture mode of the tested materials in the current study. Most zirconia failures were type 1, in which the specimens were divided into two equal sizes (repairable fracture). In contrast to Yoon et al., (2018) those described that the tested CAD/CAM materials showed predominantly catastrophic failures. Ceramic prosthesis might collect the stresses to the tooth due to higher elastic modulus compared with dentin until a catastrophic failure occurs. Koseoglu et al., (2019) concluded that 50% of CEREC and IPS Empress CAD groups and 60% of the Lava Ultimate group showed repairable fractures. This result was in parallel with our finding because type 1 failures are high among the tested zirconia and feldspathic CAD/CAM ceramic materials.

A major drawback of the current *in-vitro* study was that it did not mimic actual clinical and oral cavity conditions precisely, which might result in a different outcome due to the existence of restorative or prosthetic materials, individual saliva, continuously changing in pH, absence of the periodontal ligament chewing cycles, saliva, stress in the oral environment, and one-directional axial loading. Therefore, long-term clinical investigations are needed to realize the patterns of fracture and fatigue in recent materials used in prosthetic and restorative dentistry.

## 5. CONCLUSIONS

Within the limitations of this laboratory study, the following conclusions are drawn. KE staining and thermocycling cause slight color changes in TP and OP for glazed or polished CeramillZolid zirconia and feldspathic Vita Triluxe. TP and OP after KE immersion and thermocycling decreased compared with those before thermocycling. Polished surfaces were more affected compared with glazed surfaces. TP and OP values of the tested specimens differed in accordance with surface type. Fracture forces were slightly under the clinically accepted values for zirconia Ceramill Zolid and less than half the clinically accepted values for Vita Triluxe specimens. Fracture types were a mixed of three types. Type 1 failures recorded the maximum percentage (70%) for glazed zirconia, whereas type 3 failures were highest (80%) among polished specimens of vita Triluxe. Type 2 failures were highest in polished zirconia and glazed vita triluxe with equal percentages (60%).

### Author Contributions

All authors contributed evenly with regards to development of study design, data collection and analysis, interpretation of data, drafting the manuscript, and critical revision.

**Ethical approval**

This study was approved by the Research Ethics Committee in Faculty of Dentistry, Jazan University, Jazan, SA, with the ethical approval number (CODJU/21281 at Nov/10/2021).

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**Conflict of Interest**

The authors declare that there are no conflicts of interests.

**Data and materials availability**

All data associated with this study are presented in the paper.

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