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FRACTURE TOUGHNESS OF DENTAL CAD/CAM RESIN-MATRIX CERAMICS EXPOSED TO AN ACIDIC ENVIRONMENT

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This study aimed to assess the effects of an acidic environment on the fracture toughness of dental CAD/CAM resin-matrix ceramics. One hundred rectangular specimens (18 × 4 × 3 mm^3) were obtained from four CAD/CAM resin-matrix ceramic blocks — Crystal Ultra (CU), Vita Enamic (VE), Lava Ultimate (LU), Cerasmart (CS) — and a Vitablocs Mark II (VMII) glass-matrix ceramic. Specimens from each material group were aged either in artificial saliva or cola for one week (n = 10). The fracture toughness (KIc) was evaluated using the single-edge v-notch beam (SEVNB) method in a three-point bending set-up. The fractured specimen surfaces were analysed using a scanning electron microscope (SEM). The data were analysed using a two-way ANOVA with the post hoc Bonferroni and paired t-test ($p < 0.05$). The two-way ANOVA suggested that the materials significantly affected the KIc ($p < 0.001$), while the ageing environment had no significant effect on the KIc ($p = 0.285$). The highest KIc was observed in the CU group aged in cola (1.53 ± 0.12 MPa·m$^{1/2}$), while the lowest mean KIc was observed in the CS group aged in saliva (1.17 ± 0.08 MPa·m$^{1/2}$). All the tested CAD/CAM resin-matrix ceramics showed improved fracture toughness in an acidic environment compared to the artificial saliva. However, the fracture toughness of the CS and CU groups significantly improved in an acidic environment compared to the artificial saliva, demonstrating the ability of these materials to resist an erosive acidic environment.

INTRODUCTION

Recent developments involving computer-aided design/computer-aided manufacturing (CAD/CAM) systems have significantly improved the fabrication and restoration processes in dentistry [1]. Hence, CAD/CAM systems based on the latest dental biomaterials, advanced intraoral camera scans, and modern design software have considerably contributed to prosthodontics and restorative dentistry [2]. Increased accuracy, less waiting time and better quality are a few of the benefits of CAD/CAM technologies in modern-day clinical practice. The conventional procedures of dental fabrication and restoration are plagued by shrinkage, risk of microleakage, technique sensitivity, strict oral hygiene, time consumption, and weak fracture toughness [2]. Using CAD/CAM technologies can overcome such drawbacks; thus, high-quality restorations can be fabricated using CAD/CAM processing [3, 4].

Previous studies have demonstrated that ceramic materials are the choice for fabricating dental restoration via conventional processes [4, 5]. Dental ceramic restorations provide natural aesthetics, insoluble at different temperatures and pH, and are biocompatible with periodontal tissue [6]. However, dental ceramics are less durable and prone to chipping, and their repair is less practical [7]. Therefore, to overcome these deficits, resin-matrix ceramic materials were introduced for dental restorations [8]. Resin-ceramic materials are easy and fast to fabricate with less wear and tear from milling tools, less chipping is also witnessed due to the polymer-based composition, and the fabricated restorations have superior aesthetics [9]. Nevertheless, despite the debate surrounding the definition, the manufacturers' intention to develop resin-matrix ceramic materials was to establish a material that more accurately mimics the elastic modulus of dentin as compared to conventional ceramics, to develop a material that is simpler to mill and conform than polycrystalline or glass-matrix ceramics, and to expedite the repair process with composite resin [10].

Dental restorations used for the in situ restoration in oral cavities are continuously exposed to temperature changes caused by the consumption of food

and or beverages. Consequently, the material properties are weakened with time, and the restoration's ability to withstand fracture in an oral environment with frequent temperature and pH changes could be impeded [11, 12]. Furthermore, dental restorations are intensely affected by the wet environment, mainly due to the saliva in the oral cavity [12]. Although resin-matrix ceramics are milled more quickly and are less prone to chipping fracture, they are still considered to have a lower hardness than conventional dental ceramics. Due to the high polymer content, resin-matrix ceramics can strongly influence the pH or wet oral environment [13].

Resin-matrix ceramics are new to routine clinical practice, and several brands and materials with a high polymeric content in their compositions are constantly being introduced to the market. Previous studies have demonstrated that a pre-existing crack is a common problem with dental restorations [14]. The fracture toughness describes a material's resistance to the progression of a pre-existing crack and is considered a significant factor in determining the restorative materials' mechanical characteristics [15, 16]. As a result, for dental restorations to be clinically successful in the oral cavity, the restorative materials must be durable and have a high degree of fracture toughness [17]. There are several methods described for determining the fracture toughness of ceramics, including the single-edge v-notched beam (SEVNB), indentation strength (IS), and indentation fracture (IF). Notch and indentation are two different mechanisms that differentiate these measurement techniques [18]. The SEVNB method is the most simple, direct, and accurate method for determining the fracture toughness [14, 19]. The ISO 6872:2015 standard, which outlines the specifications and test procedures for dental ceramic materials, recommends the SEVNB test for determining the fracture toughness [20, 21].

Consequently, this study aimed to assess the effect of an acidic environment on the fracture toughness of dental CAD/CAM resin-matrix ceramics. The null hypothesis stated was that an acidic environment would have no effect on the fracture toughness of resin-ceramic materials.

EXPERIMENTAL

Specimen preparation

One hundred rectangular specimens ($18 \times 4 \times 3$ mm^3) were obtained from four CAD/CAM resin-matrix ceramic blocks — Crystal Ultra (CU), Vita Enamic (VE), Lava Ultimate (LU), Cerasmart (CS) — and the Vitablocs Mark II (VMII) glass-matrix ceramic. The materials used in the study are detailed in Table 1.

A Ceramill Motion 2 milling device (Amann Girrbach AG, Koblach, Austria) was used to section the blocks to the desired dimensions. The upper and lower surfaces of the prepared specimens were polished using a polishing machine (LaboPol-25, Struers Co., Copenhagen, Denmark) at 300 rpm under water coolant at increasing grit sizes (400, 600, 800, 1000, and 1200) of silicon carbide paper (Struers Co., Copenhagen, Denmark). Then, all the specimens were individually cleaned using distilled water in an ultrasonic bath (Quantrex 90, L&R Ultrasonics, NJ, USA) for 5 min and later air-dried for 20 s. A digital calliper (Mitutoyo, Canada Inc., Ontario, Canada) was used to confirm the dimensions of each specimen. The specimens were later stored in an incubator (JSGI-150T, JS Research Inc., Korea) at 37 °C for 24 h before the ageing process [22].

A notch was made by stabilising the specimen in a customised holder. The holder with horizontal grooves allowed the sliding movement of the specimen against a diamond saw on a mounted rotary handpiece

Table 1. Details of the CAD/CAM materials.

Material/Category	Manufacturer	*Composition
Crystal Ultra / Resin-Matrix ceramic	Digital Dental, USA	Cross-linked polymer (BisGMA, UDMA, BDMA) (30 wt. %) and ceramic-like inorganic silicate glass fillers (70 wt. %).
Vita Enamic / Resin-Matrix ceramic	VIRA Zahnfabrik, Germany	Cross-linked polymer (BisGMA, UDMA) (14 wt. %) and feldspathic ceramic enriched with aluminium oxide (86 wt. %).
Lava Ultimate / Resin-Matrix ceramic	3M ESPE, USA	Matrix: BisGMA, UDMA, BisEMA, TEGDMA Filler: silica zirconia nanoparticles and zirconia/silica nanoclusters (80 wt. %).
Cerasmart / Resin-Matrix ceramic	GC Corp., Japan	Matrix: BisMEPP, UDMA, DMA. Filler: silica and barium glass nanoparticles (71 wt. %).
Vitablocs Mark II / Glass matrix ceramic	VITA Zahnfabrik, Germany	> 20 wt. % feldspathic particles (average particle size 4 μm) and 80 wt. % glass-matrix.

*BisGMA: Bisphenol-A-glycidyldimethacrylate, UDMA: Urethane Dimethacrylate, BisEMA: Ethoxylated Bisphenol A Dimethacrylate, TEGDMA: Triethyleneglycoldimethacrylate, BisMEPP: Bisphenol A Bis (2-Hydroxyethyl Ether) Dimethacrylate, DMA: Dimethacrylate, BDMA: 1, 4-Butanediol Dimethacrylate.

(Figure 1a). A 2-mm deep V-shaped notch was created at the centre of each specimen under water coolant. The notch was shaped and finished to provide a smooth to-and-fro movement using a razor blade and diamond polishing paste. A digital microscope (KH-7700 Hirox, New Jersey, USA) was used to evaluate and confirm the tip of the V-shaped notch. Then, all the specimens were cleaned in distilled water for 5 min, followed by air drying for 20 s.

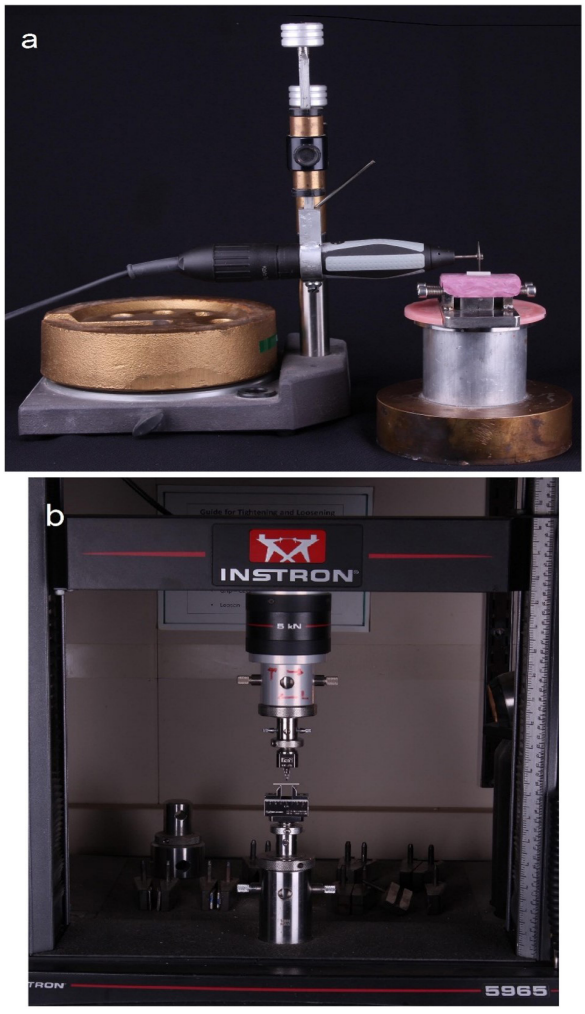


Figure 1. Preparation of the V-notch using the customised holder and diamond saw on a mounted rotary handpiece (a); Three-point bending test in a universal testing machine (b).

Ageing process

The specimens from each material were randomly assigned into two groups (n = 10) according to the ageing solution (artificial saliva (AS) and cola) used. The artificial saliva was prepared in the laboratory at the college of pharmacy, King Saud University, Saudi Arabia, under a previous study [23]. A commercial soda from the Coca-Cola Company, Riyadh, Saudi Arabia, was used as another ageing solution. The pH of the solutions was determined using a pH meter (Jenway, Essex, UK), which was 7.2 and 2.3 for AS and Cola, respectively. The specimens were immersed in large Petri dishes filled with either AS or Cola for 7 days [24]. The solutions were changed every 24 h, and the pH of both solutions was regularly monitored. After ageing, the specimens were individually and gently cleaned with a soft toothbrush under tap water to remove any remnants from the specimen surface. Then, the specimens were air-dried before the fracture toughness test.

Fracture toughness (KIc) test

The fracture toughness was determined using the SEVNB method in a three-point bending set-up according to ISO specification 6872:2015 [25]. The specimen was placed on the customised jig of a universal testing machine (Instron Corp, MA, USA) with the beam stabilised at the ends (Figure 1b). The loading rod was directed towards the specimen at a crosshead speed of 0.5 mm·min^{-1} and ambient room temperature. The fracture toughness was determined by calculating the critical stress intensity factor using equations (1-2) [26].

$$K_{IC} = g\left[\frac{P_{max}\, S_0 10^{-6}}{BW^{3/2}}\right]\left[\frac{3[a/w]^{1/2}}{2[1-a/w]^{3/2}}\right] \quad (1)$$

where $g = g\,(a/w) =$

$$\left[\frac{1.99 - [a/w][1-a/w][2.15 - 3.93[a/w] + 2.7[a/w^2]]}{1 + 2[a/w]}\right] \quad (2)$$

Where *KIc* is the fracture toughness, g is the function of the ratio a/W, P_{max} is the maximum load (N) at fracture, S_o is the supporting outer span (in mm), B is the specimen width (in mm), W is the specimen thickness (in mm), and a is the depth of the V-notch (in mm).

Scanning electron microscope (SEM) evaluation

A random fractured specimen from each material group was selected for the SEM analysis. The specimens were gold sputter coated for one minute in a coating machine (Quorum Q150R, Essex, USA). The coated specimen surface was visualised using SEM (JEOL JSM-5900 LV SEM, Tokyo, Japan) at 10 kV and a working distance of 10 mm. The SEM micrographs were obtained at ×1000 magnification.

Statistical analysis

All the data were analysed using statistical analysis software (v.29, IBM SPSS Inc., Chicago, USA). The mean ± standard deviation (SD) was used to describe the quantitative outcome of the fracture toughness test. A two-way analysis of variance (ANOVA) was used to statistically test the factors (material and storage environment) influencing the fracture toughness.

The Bonferroni post-hoc test was applied to detect the significant differences between the groups. The paired t-test determined the differences between two variables for the same material group. A *p*-value of < 0.05 was used to report the statistical significance.

RESULTS

The two-way ANOVA for the interaction of the material and the ageing on the fracture toughness of the CAD/CAM materials is presented in Table 2. The outcome suggests that the material type significantly influenced the fracture toughness ($p < 0.001$). However, the ageing ($p = 0.285$) and the interaction between the factors ($p = 0.846$) had no significant effect on the fracture toughness.

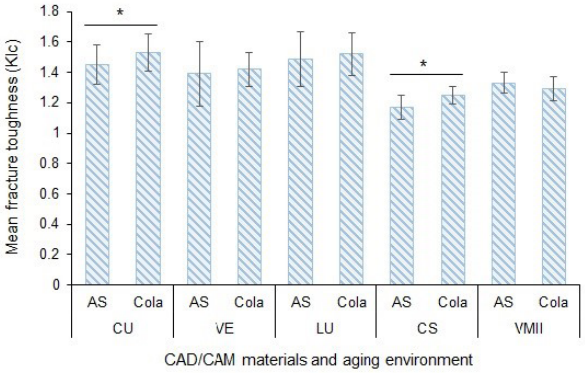


Figure 2. Mean fracture toughness of the materials aged in AS and Cola. The bars indicate the SD.
*indicate a significant difference between the AS and Cola groups within the material ($p < 0.05$; paired t-test)

Table 2. Two-way ANOVA for the interaction between the material type and the ageing on the fracture toughness.

Source	Type III Sums of Squares	DF	Mean Square	F	Sig.
Corrected Model	1.337	9	0.149	4.153	0.000*
Intercept	192.106	1	192.106	5371.553	0.000*
Material	1.246	4	0.311	8.708	0.000*
Media	0.041	1	0.041	1.155	0.285
Material × Ageing	0.050	4	0.012	0.347	0.846
Error	3.219	90	0.036	-	-
Total	196.661	100	-	-	-
Corrected Total	4.555	99	-	-	-

*statistically significant ($p < 0.05$)

Figure 2 presents the study groups' mean fracture toughness (KIc) values. The highest mean KIc was observed in the CU-group immersed in cola (1.53 ± 0.12 MPa·m$^{1/2}$), while the lowest mean KIc was observed in the CS-group immersed in saliva (1.17 ± 0.08 MPa·m$^{1/2}$). The KIc of the resin-matrix ceramic materials increased whereas the KIc decreased for the glass ceramic aged in cola. The paired sample t-test revealed statistical differences in the KIc among the saliva and cola groups for both the CS and CU materials ($p < 0.05$).

Table 3 presents the Bonferroni post-hoc multiple comparison outcomes to detect the significant differences between the materials. Among the materials aged in AS or Cola, statistical differences were observed only between CS and LU and CS and CU ($p < 0.05$).

Figure 3 presents the SEM micrographs to demonstrate the fracture and surface pattern of the CAD/CAM materials after ageing in AS or Cola. Irrespective of the ageing environment, all the specimens revealed surface flaws and voids, which could have led to the specimen failure. The fractured surfaces of the CS and CU specimens were comparatively less rough than the other tested materials. The VE and VMII specimens showed rougher fracture patterns compared to the other material groups. Interestingly, the LU specimen aged in AS was rougher than those immersed in Cola. The VE and VMII specimens aged in Cola showed more eroded surfaces than those aged in AS.

DISCUSSION

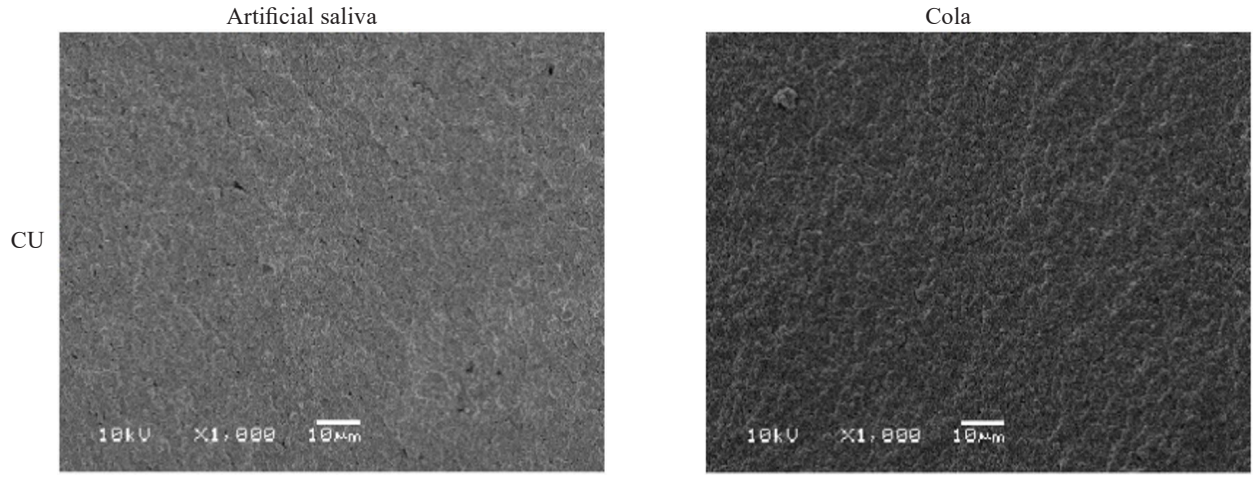
The current study aimed to assess the effect of an acidic environment on the fracture toughness of dental CAD/CAM resin-matrix ceramics. The outcome of the study recommends the rejection of the null hypothesis stating that an acidic environment would have no effect on the fracture toughness of the tested resin-matrix ceramics.

An important parameter in evaluating a material's durability under mechanical stress is the fracture toughness [27]. The type of dental materials and the testing procedure applied can influence the fracture toughness value [28, 29]. In this study, the SEVNB method in a three-point bending set-up was used to measure the fracture toughness of the CAD/CAM materials. Among the methods used to determine the fracture toughness of ceramics, the SEVNB

Table 3. Bonferroni post-hoc multiple comparisons to detect the significant differences between the materials.

Ageing environment	Group (I)	Group (J)	Mean Difference (I-J)	Sig.	95 % Confidence Interval	
					Lower Bound	Upper Bound
Artificial saliva	CU	VE	0.052	1.000	-0.185	0.291
		CS	0.277	0.013*	0.039	0.515
		VMII	0.118	1.000	-0.120	0.356
		LU	-0.038	1.000	-0.277	0.199
	VE	CS	0.224	0.079	-0.013	0.462
		VMII	0.065	1.000	-0.172	0.303
		LU	-0.091	1.000	-0.330	0.146
		CU	-0.052	1.000	-0.291	0.185
	LU	VE	0.091	1.000	-0.146	0.330
		CS	0.316	0.003*	0.078	0.554
		VMII	0.157	0.579	-0.081	0.395
		CU	0.038	1.000	-0.199	0.277
	CS	VE	-0.224	0.079	-0.462	0.013
		VMII	-0.159	0.548	-0.397	0.079
		LU	-0.316	0.003*	-0.554	-0.078
		CU	-0.277	0.013*	-0.515	-0.039
	VMII	VE	-0.065	1.000	-0.303	0.172
		CS	0.159	0.548	-0.079	0.397
		LU	-0.157	0.579	-0.395	0.081
		CU	-0.118	1.000	-0.356	0.120
Cola	CU	VE	0.109	1.000	-0.150	0.370
		CS	0.283	0.024*	0.0228	0.543
		VMII	0.242	0.087	-0.018	0.5021
		LU	0.009	1.000	-0.250	0.270
	VE	CS	0.173	0.552	-0.086	0.434
		VMII	0.132	1.000	-0.127	0.393
		LU	-0.099	1.000	-0.360	0.160
		CU	-0.109	1.000	-0.370	0.150
	LU	VE	0.099	1.000	-0.160	0.360
		CS	0.273	0.033*	0.012	0.534
		VMII	0.232	0.116	-0.028	0.492
		CU	-0.009	1.000	-0.270	0.250
	CS	VE	-0.173	0.552	-0.434	0.086
		VMII	-0.041	1.000	-0.301	0.219
		LU	-0.273	0.033*	-0.534	-0.012
		CU	-0.283	0.024*	-0.543	-0.022
	VMII	VE	-0.132	1.000	-0.393	0.127
		CS	0.041	1.000	-0.219	0.301
		LU	-0.232	0.116	-0.492	0.028
		CU	-0.242	0.087	-0.502	0.018

*statistically significant difference between the groups ($p < 0.05$)



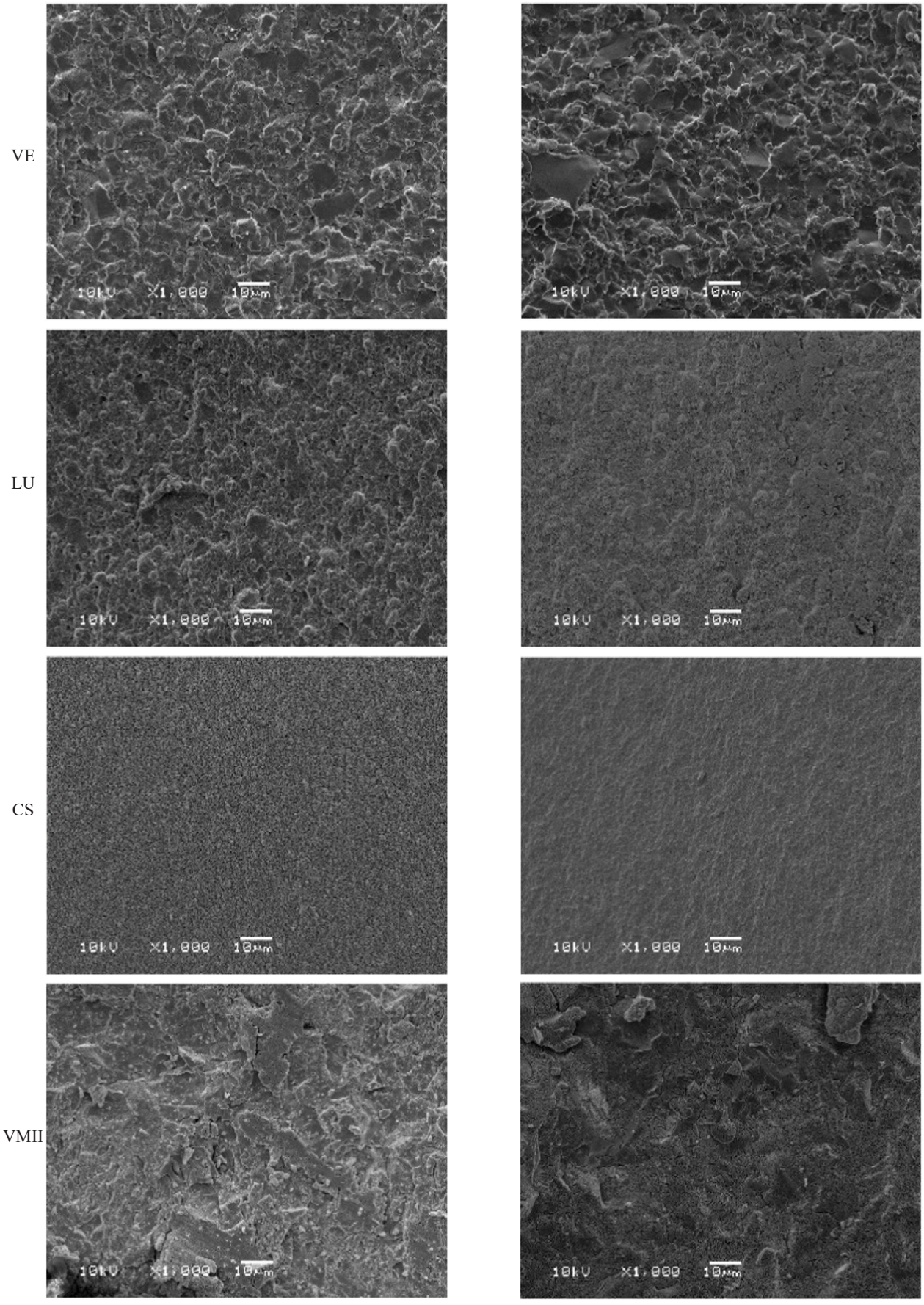


Figure 3. Scanning electron microscopy micrographs (×1000) of the specimens.

method is considered the "gold standard" as it is more reliable, accurate and reproducible [18, 19, 21, 30, 31]. Due to the precise and controlled fracture brought on by the V-notch in the specimens, the SEVNB technique may accurately reflect the material properties. The SEVNB approach, on the other hand, involves a thorough notch preparation and is, hence, highly sensitive [29]. The toughness values will be less than the actual fracture toughness if the crucial notch length is underestimated [29, 30], while the findings could be overestimated if the notch root radius is too high [18].

A comparatively lower fracture toughness value of the VMII specimens immersed in Cola (1.29 ± 0.08 $MPa \cdot m^{1/2}$) was observed than those in artificial saliva (1.33 ± 0.07 $MPa \cdot m^{1/2}$). A possible reason is that the exposure of the VMII specimens to low-pH acidic drinks can directly affect and dissolute the glass matrix of glass-ceramic materials and contributes to the degeneration of the materials' properties [11, 12]. In contrast, the higher fracture toughness values in the salivary medium might suggest that a higher pH has no deleterious effect on this type of ceramic.

Interestingly, the resin-matrix ceramics, except for the CS group, demonstrated a higher fracture toughness than the glass-matrix ceramics in both environments. The increased fracture toughness values among the resin-matrix ceramic groups might suggest that the presence of cross-linked polymers in these materials shows a plasticising effect or stress relaxation mechanism in the immersion environment [13]. However, significantly higher fracture toughness values of the VE, LU and CU groups compared to the CS group are anticipated due to the variations in matrix composition, volume fraction filler, and production techniques [1]. The higher fracture toughness values of the LU and CU groups than the VE group could be attributed to differences in the inorganic content of these three materials: LU has 80 wt. % silica, zirconia nanoparticles and zirconia/silica nanoclusters, and CU has 70 wt. % silicate glass fillers. In contrast, VE has 86 wt. % Al_2O_3 [2]. The composition, size, and dispersion of the filler particles in the resin matrix could be another factor contributing to the differing fracture toughness of these materials [32, 33]. Overall, there was no discernible difference in the fractured specimens immersed in AS and Cola as observed in the SEM micrographs. This outcome was also complemented by the two-way ANOVA, which confirmed that the immersion environment had an insignificant effect on the fracture toughness ($p = 0.285$). The possible reason for such an outcome could be that the damaging effect might not be noticeable for up to 7 days of continuous immersion or is negligible.

Flaws in restorative materials, such as closed pores or surface cracks, negatively affect the mechanical properties, such as fracture toughness and flexural strength [34]. However, the mechanical properties can be optimised by introducing new fabrication process [13]. The void size and number, and internal stress in the microstructure might all be decreased due to the high-pressure, high-temperature polymerisation technique in these materials [13, 34]. The higher fracture toughness values in VE, LU and CU groups might be attributed to the polymer-infiltrated ceramic network technology and resin nanoceramics in their respective formulations. Although, VE and LU have been largely studied, CU is a recently introduced resin matrix-ceramic with a higher polymer/ceramic ratio. According to the manufacturer, the CU material is reported to be the most elastic resin matrix-ceramic available on the market. The improved fracture toughness of CU might be the attributed to the flexible nature of the material and presumably because of the chemical stability of the polymeric content [32]. Elasticity allows the material to flex while chewing or under stress thereby minimising chipping and fractures [35].

The outcome of the present study are in disagreement with those published studies reporting deleterious effect of acidic drinks on the material properties of resin-matrix ceramic materials [2, 7]. Furthermore, few studies have demonstrated superior fracture toughness of CAD/CAM glass-ceramic materials compared to resin-matrx ceramic materials [2, 7, 13]. In contrast, our findings suggest the vice versa. However, for clarity and elimination of any ambiguity, future studies are necessary using other types of glass-ceramic materials. The available literature demonstrates inconsistent KIc values for the same materials, and it is challenging to compare them. In a recent study by Goujat et al., [36], the reported fracture toughness values of VE, CS and LU were similar to those reported in the present study. However, a comparison with previous findings should be made carefully due to the differences in the materials and methodologies applied [32].

Like other laboratory studies, this study, too had some limitations. Since this study only evaluated the fracture toughness property of resin ceramic materials, the other mechanical properties may provide a wider view of the applicability and usefulness. Additionally, more research can examine how these restorative materials respond to different ageing processes and clinical conditions. Moreover, the in vitro outcome may not necessarily replicate the in vivo environment.

CONCLUSIONS

The fracture toughness and fractographic analysis revealed that CAD/CAM resin-matrix ceramic materials are more acid-resistant than glass-ceramic materials (i.e., Vita Bloc Mark II). Hence, resin-matrix ceramic materials may be a viable option as a dental restorative material.

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