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Stress intensity factor threshold in dental porcelains

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Abstract The stress intensity factor threshold (K_{I0}) is related to the stress level at which cracks start to grow stably, causing the weakening of porcelain prostheses during their use. The values of K_{I0} of seven dental porcelains (with and without reinforcing leucite crystal, $KAlSi_2O_6$) stored in air (22 °C, 60% relative humidity) and artificial saliva (37 °C) were determined by measuring the crack growth velocity of radial cracks generated at the corner of Vickers indentations. The results of K_{I0} were correlated with the leucite content, fracture toughness (K_{Ic}), and chemical composition of the porcelains. It was observed that K_{I0} increased with the increase of leucite content (only for the leucite-based porcelains) and with the increase of K_{Ic}. The increase in Al_2O_3 content or the decrease in the alkali oxide (K_2O and Na_2O) content of the material's glassy matrix tended to increase the K_{I0} values. Storage media (air and saliva) did not significantly affect the K_{I0} of porcelains tested, indicating that the control parameter of K_{I0} value was not the water content of the storage media.

1 Introduction

Porcelains are aesthetic materials largely used in dentistry to construct varied types of works such as inlays, onlays, overlays, crowns, and fixed partial dentures (FPDs). However, these materials have very low fracture toughness (around 1.0 MPa.m$^{1/2}$) [1], which leads to the unwanted fracture rates reported in clinical trials [2–4]. The fracture of porcelain structures occurs when a pre-existing flaw propagates in an unstable manner under applied tensile stresses, leading to catastrophic failure [5], but before fast fracture occurs, the superficial defects may present a slow and stable growth, mainly in humid environment, such as the oral cavity. This phenomenon is referred as slow or subcritical crack growth (SCG) and leads to strength degradation over time [6].

The phenomenon of SCG is usually described by the relation between crack velocity (v) and stress intensity factor (K_I) [7, 8]. Some aspects of this relationship are well documented, such as the three distinct regions, designated I, II and III, each with its own characteristic. Region I has attracted the most attention in literature because it is highly dependent on K_I and environmental conditions [9]. Dental prostheses are usually subjected to stress levels corresponding to this region during most part of their lifetimes. In region I, the environmental species react with the ceramic interatomic bonds in the crack front, leading to crack propagation [10]. Region II is associated to intermediate crack velocities and also depends on environment, but is much less sensitive to K_I [11]. This region depends on the diffusion of the corrosive species from the environment to the crack tip. In Region III, ultrasonic velocities are reached, and crack growth is strongly dependent on K_I, but is environment-insensitive, since it is usually associated with the kinetic of intrinsic bond rupture [12].

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One of the most important aspects of the v-K_I curve is the presence of a stress intensity factor threshold (K_{I0}) under which no crack propagation occurs. For ceramic prostheses, this threshold is temperature-insensitive and determines a safety range of use, since higher thresholds will lead to higher reliability and longer service life [11]. Determining K_{I0} in ceramic materials is difficult because crack velocities involved are low (below 1 nm/s) and there is a lack of experimental data in the literature [10]. Nevertheless, Wan et al. [11] have determined K_{I0} values for soda lime glass using crack velocity measurements obtained from Vickers indentation flaws. The procedure consists of observing post-indentation virgin radial crack growth under the exclusive action of the residual contact stress field. Since the residual stress field is stable, the crack driving force decreases with crack growth, so the system progresses naturally down the v-K_I curve. Therefore, this test configuration is especially suited to extending data into the threshold region.

There is a lack of data regarding stress intensity factor threshold for dental porcelains, and the relationship between the porcelain's microstructure and K_{I0} still needs to be determined. One work [13] compared the K_{I0} of varied dental ceramics stored in air and found values ranging from ~0.6 to 0.8 MPa.m$^{0.5}$ for dental porcelains (CergoGold, Cerec Mark II and VMK 95), ~0.6 to 1.0 MPa.m$^{0.5}$ for glass-ceramics (Dicor, Empress 1 and Empress 2), and ~2.3 MPa.m$^{0.5}$ for Y-TZP ceramics (Lava and Cercon). Moreover, the influence of the relative humidity on K_{I0} of dental porcelains has not been determined yet. For soda-lime glass at room temperature, it has been observed that an increase in relative humidity (RH) leads to a significant decrease in K_{I0} value [11, 14]. Therefore, the objective of the present study was to determine the K_{I0} of seven dental porcelains and correlate results with their leucite ($KAlSi_2O_6$) content, fracture toughness values, and chemical composition. In addition, the effect of the test ambient (air or saliva) on K_{I0} was investigated.

2 Material and method

Five leucite-based porcelains indicated for porcelain-fused-to-metal and all-ceramic restorations (A, B, C, D and G), and two porcelains indicated to be used as veneering material for alumina-based cores (E and F, Table 1) were investigated. The different types of porcelains were chosen in order to provide a wide variation of leucite contents. The green specimens (5 × 6 × 30 mm) were prepared by the vibration-condensation method and sintered in a dental porcelain furnace following the firing schedules recommended by the manufacturers. After firing, the specimens were machined to the dimensions of 3 mm × 4 mm × ~25 mm, following the guidelines in ASTM C 1161. Then, the 4-mm side was mirror polished using a polishing machine (Ecomet 3, Buehler, Lake Buff, USA) with diamond suspensions (45, 15, 6 and 1 μm).

Radial cracks were generated on polished surfaces with a Vickers microhardness tester MVK-H-3 (Mitutoyo, São Paulo, Brazil) with load of 19.6 N and dwell time of 20 s. Crack lengths were measured under optical microscopy at various times over a period of 4 months (~0; 1; 3; 10; 30; 100; 300, 1000 and 3000 h) in two storage media (air or artificial saliva). For storage in air (60% RH, 22 °C), four specimens of each material were used, and five indentations were made in each one. Indentations were made next to each other, in the center of the polished surface, and the

Table 1 Porcelains used in the study (shade A3-dentin)

Porcelain	Manufacturer/brand name	Manufacturer's description
A	Dentsply Ceramco R & D, Burlington, USA/Ceramco I	High-fusing, leucite-based porcelain, used for metal-ceramic or all ceramic restorations, containing equiaxied leucite particles. Fusing temperature: 1,000 °C
B	Dentsply Ceramco R & D, Burlington, USA/Ceramco II	High-fusing, leucite-based porcelain, used for metal-ceramic or all ceramic restorations, containing leucite particles. Fusing temperature: 1,000 °C
C	Dentsply Ceramco R & D, Burlington, USA/Ceramco Finesse	Low-fusing, leucite-based porcelain, used for metal-ceramic or all ceramic restorations, containing fine-grained leucite particles. Fusing temperature: 800 °C
D	Ivoclar, Schaan, Liechtenstein/d.Sign	Low-fusing, leucite-based porcelain, used for metal-ceramic or all ceramic restorations, containing leucite particles and crystals of fluorapatite. Fusing temperature: 875°C
E	Noritake, Tokyo, Japan/Cerabien	High-fusing porcelain to be used with alumina frameworks. Fusing temperature: 960 °C
F	Vita/Vitadur-Alpha	High-fusing porcelain to be used with alumina frameworks. Fusing temperature: 970 °C
G	Jendental/Ultropaline	High-fusing, leucite-based porcelain. Fusing temperature: 930 °C

distance between two indentations was at least ten times the size of the radial cracks. Immediately after making each indentation, the crack size was measured and the specimens were stored in a plastic box until next measurement.

For storage in artificial saliva, three specimens of each material were used and five indentations were made in each one, using the same configuration described above. Before making the first indentation, one drop of artificial saliva was placed on the specimen surface. Immediately after making the indention, the surface was dried with a paper cloth to allow visualization of the crack size. Then specimens were stored at constant temperature (37 °C) in a glass box with artificial saliva with the following composition: 100 mL of KH_2PO_4 (25 mM); 100 mL of Na_2HPO_4 (24 mM); 100 mL of $KHCO_3$ (150 mM); 100 mL de NaCl (100 mM); 100 mL of $MgCl_2$ (1.5 mM); 100 mL of $CaCl_2$ (15 mM); and 6 mL of citric acid (25 mM). The pH of artificial saliva was 7.0.

K_{I0} was determined by plotting log crack velocity versus K_I (the plateau at low K_I values is indicative of threshold behavior). Velocities were determined from measurements of radial crack growth increments over successive time intervals for each indentation crack. K_I at the crack tip was calculated according to the following equation [15]:

$$K_I = \chi P / c^{3/2} \tag{1}$$

where P is indentation load, c is the mean size of the radial cracks in a time interval, and χ is a residual stress coefficient related to the materials Young's modulus, E, and hardness, H, given by:

$$\chi = 0.016 (E/H)^{1/2} \tag{2}$$

The elastic modulus of each material was determined by the pulse-echo method. A soda-lime glass slide (3 mm-thick) was used as reference material in order to verify the reproducibility of the indentation fracture method [11]. Fracture toughness, K_{Ic}, was calculated by Eq. 1 using measurements made in air immediately after indenting specimens [16].

Statistical analysis of K_{Ic} and K_{I0} data was performed by means of one-way analysis of variance (ANOVA). Microstructural analysis of the materials studied was also performed in order to determine the leucite content by means of an image analyzer software. This analysis was made on polished discs by etching their surfaces with 2% hydrofluoric acid (HF) for 15 seconds. After that, they were taken into a scanning electron microscope (SEM) (Jeol–JSM 6300, Peabody, MA, USA) coupled to an energy dispersive spectroscope (EDS) (Noran Instruments, Middletown, WI, USA). In addition, a semi-quantitative chemical analysis of all materials was performed by means of X-ray fluorescence (Shimadzu, XRF 1500, Japan).

3 Results

Figure 1 shows the variation of crack velocity as a function of the stress intensity factor for soda lime glass and porcelains E and G. These graphs were constructed to demonstrate how data obtained from Vickers indentations can be used to define K_{Ic}, K_{I0} and Region I. The value of K_{I0} determined for soda-lime glass in air at 60% RH (0.39 MPa $m^{0.5}$, Fig. 1a) was similar to that obtained by Wan et al. [11] for the same material in air at 50% RH. This result showed that the indentation fracture methodology is reproducible for K_{I0} determination. The calculated values of residual stress coefficient, χ (Eq. 2), of all porcelains were around 0.04 (Table 2), similar to that reported for soda-lime glass [11].

Table 2 shows that materials were grouped in 3 statistically different groups in terms of fracture toughness. The first group, with higher K_{Ic}, comprised materials A, B and G, which had leucite contents (LC) of 22, 22, and 13%, respectively. The intermediary group showed K_{Ic} varying from 0.73 to 0.78 $MPa.m^{1/2}$ and included porcelains D, E and F (LC = 15, 0, and 0%, respectively). Porcelain C (LC = 6%) showed significantly lower fracture toughness compared to the other materials. With respect to fracture toughness measurements, it is important to note that measurements were performed in air, what inevitably underestimates the K_{Ic} values. Moreover, for dental porcelains, it is expected an uncertainty of K_{Ic} values determined by IF method, measured in air, of ±10% of the value determined by a standardized SCF (surface crack in flexure) method [17].

As for K_{I0} values obtained in air, porcelains A, B and G showed the highest mean values, while porcelains D, E and F showed intermediate values ranging from 0.48 to 0.50 $MPa.m^{1/2}$. Porcelain C showed statistically lower K_{I0} compared to other materials in air. In saliva, the highest K_{I0} values were obtained by porcelains A, B and F, while intermediary values were obtained for materials D, E and G (varying from 0.47 to 0.53 Mpa $m^{1/2}$), and the lowest value was obtained for porcelain C.

Storage in saliva did not result in a significant change in K_{I0}, in comparison to air storage, for any of the materials tested. However, different trends were observed depending on the porcelain tested. For instance, the mean values of porcelains B and G tended to be lower in saliva, while for porcelains A and F, a numeric increase in K_{I0} was observed after storage in saliva. No significant correlation between leucite content and K_{I0} was observed in air or saliva Fig. (2). Nevertheless, if porcelains without leucite are disregarded, a strong relationship can be noted between leucite content and K_{I0}. The r^2 values obtained when the regression analysis was performed only for leucite-based porcelains were 0.60 (air) and 0.97 (saliva), as shown in

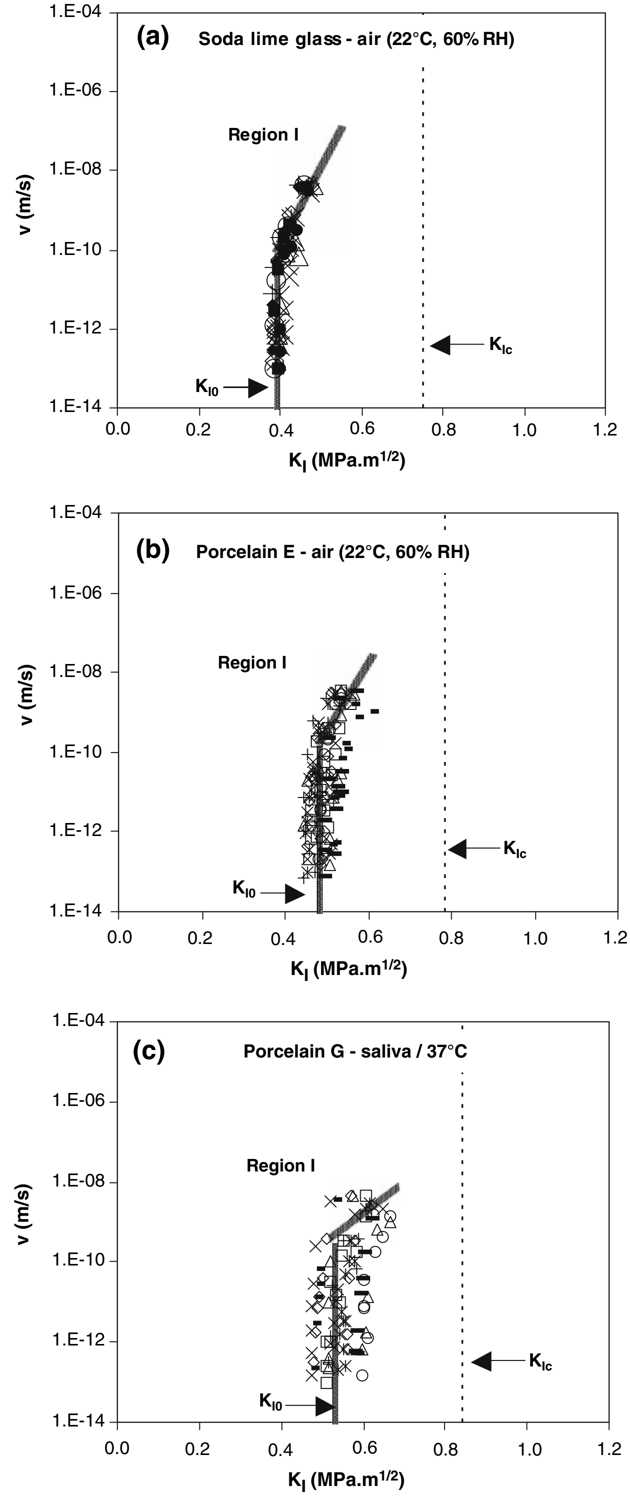

Fig. 1 Crack velocity as a function of stress intensity factor for (**a**) soda-lime glass, (**b**) porcelain E, both in air (60%RH/22 °C), and (**c**) porcelain G in artificial saliva (37 °C). The stress intensity factor threshold (K_{I0}) and the fracture toughness (K_{Ic}) are identified. Different symbols represent results from the different indentations

Fig. 2. The relationship between K_{Ic} and K_{I0} is shown in Fig. 3. It is possible to note that there is a significant correlation between the two variables. Most of the results were in the range of K_{I0}/K_{Ic} ratio from 0.60 to 0.75 (average of 0.67 ± 0.06), which is close to the values reported for oxide ceramics and glasses [18].

Chemical analysis (Table 3) showed that all porcelains were silicate based materials with different contents of Al_2O_3, K_2O, Na_2O, CaO, and other oxides, like MgO, BaO, ZrO_2, and TiO_2. Very low contents (traces) of impurities and colorants, like Fe, Ni, Zn, Rb, and Sr, were also detected. For leucite-based porcelains (A, B, C, D, and G), the composition of the glassy matrix was estimated based on the results of chemical analysis (Table 3) and leucite content (Table 2), and assuming stoichiometric leucite composition ($KAlSi_2O_6$). It was observed that the glassy matrix of leucite-based porcelains (Table 4) contained: ~70 mol% SiO_2; ~7.5 mol% Al_2O_3, except for porcelains C (5.7%) and G (10.1%); ~14 mol% alkali oxides ($K_2O + Na_2O$); and ~5.5 mol% alkaline earth oxides (CaO + MgO + BaO), except for porcelain C (9.3%). For porcelains without leucite (E and F), SiO_2 content was higher than that of the leucite-based porcelains (Table 3). Porcelain E presented, among all porcelains, the lowest Al_2O_3 content (5.6%) and also the lowest contents of alkali (8.2%) and alkaline earth (1.8%) oxides. Porcelain F presented higher Al_2O_3 content (10.0%) and lower contents of alkali (10.7%) and alkaline earth (3.0%) oxides compared to the leucite-based porcelains (Table 3).

4 Discussion

The results of this work showed that, if porcelains without leucite are disregarded, a strong correlation between LC and K_{I0} is observed in both air and saliva. Therefore, it seems like the addition of leucite to a porcelain does not necessarily result in higher $K_{I0,}$ but once the material already has leucite in its composition, higher K_{I0} values are obtained with the increase in LC. It is likely that the underlying mechanisms that explain the porcelains' behavior in terms of stress intensity threshold are related to both the chemical composition and leucite content. The observed increase in K_{I0} with the increase in LC in leucite-based porcelains may be explained by presence of the tangential compressive stress fields that are created around the leucite-glass interface during cooling of the material, since leucite particles contract more rapidly than the matrix glass [19]. Thus, before a pre-existing defect starts growing slowly, the tensile stresses concentrated at the crack tip

Table 2 Leucite content, residual stress coefficient (χ), fracture toughness (K_{Ic}), and stress intensity factor threshold (K_{I0}) of the seven porcelains tested. Within each column, values followed by the same superscript are statistically similar ($p > 0.05$)

Porcelain	LC (%)	χ	K_{Ic} (MPa.m$^{1/2}$)	K_{I0} (MPa.m$^{1/2}$)	
				Air	Saliva
A	22	0.040	0.93 ± 0.12 ^{a}	0.58 ± 0.09 ^{b,c,d}	0.64 ± 0.11 ^{a,b}
B	22	0.040	0.89 ± 0.11 ^{a}	0.70 ± 0.11 ^{a}	0.63 ± 0.14 ^{a,b,c}
C	6	0.041	0.64 ± 0.06 ^{c}	0.39 ± 0.07 ^{g}	0.41 ± 0.08 ^{f,g}
D	15	0.040	0.77 ± 0.06 ^{b}	0.50 ± 0.05 ^{d,e}	0.52 ± 0.09 ^{d,e}
E	0	0.040	0.78 ± 0.06 ^{b}	0.48 ± 0.03 ^{e,f}	0.47 ± 0.04 ^{e,f}
F	0	0.038	0.73 ± 0.15 ^{b}	0.49 ± 0.07 ^{d,e}	0.55 ± 0.08 ^{b,c,d}
G	13	0.040	0.84 ± 0.11 ^{a}	0.63 ± 0.06 ^{a,b,c}	0.53 ± 0.05 ^{c,d,e}

need to overcome the compressive stresses that hinder crack opening. In accordance to this, a previous study that followed the indentation crack path showed that the higher the porcelain's leucite content, the higher the incidence of deflected cracks, which indicates the importance of the stress fields to the mechanical behavior of the material [1].

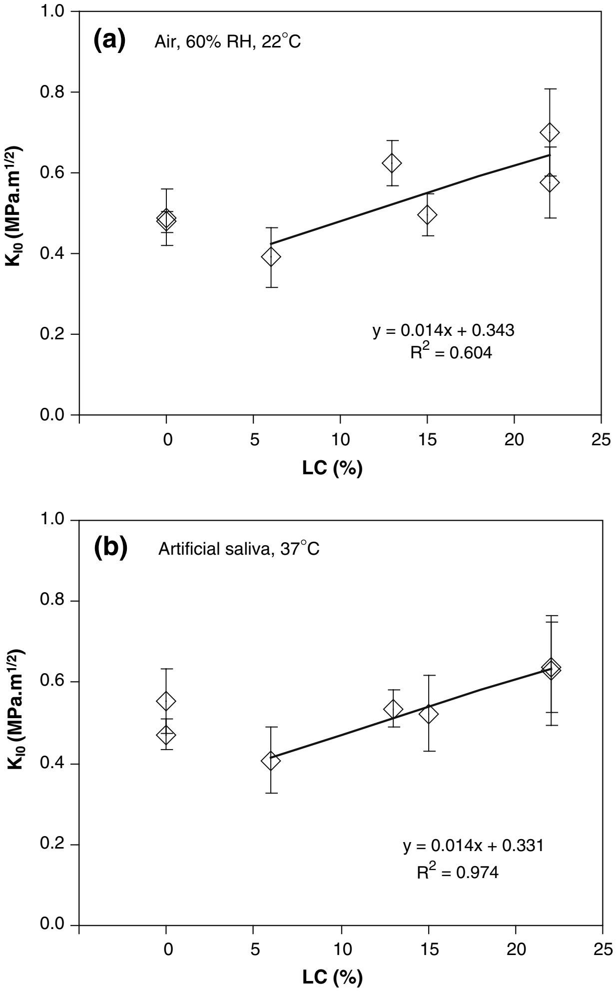

Fig. 2 Stress intensity factor threshold (K_{I0}) versus leucite content (LC) in air (**a**) and in artificial saliva (**b**). Regression analysis was performed disregarding porcelains without leucite

With regards to the influence of the chemical composition on K_{I0}, it is hypothesized that the distributions of both Al_2O_3 and alkali oxides (K_2O and Na_2O) in the glassy matrix of the different materials are responsible for the fact that porcelains without leucite (E and F) had similar or higher K_{I0} compared to some leucite-based materials, like porcelains C and D. For the leucite based materials, a fraction of the Al_2O_3 (and also the K_2O) content detected in their composition (Table 3) is used to form leucite ($KAlSi_2O_6$), whereas the Al_2O_3 content found in vitreous porcelains E and F (5.6 and 10.0 mol%, respectively) is distributed throughout their glassy matrix and avoids crack propagation, since Al_2O_3 enhances inter-atomic bonding and increases surface energy [20]. Nevertheless, the higher K_{I0} of porcelain E compared to the porcelain C can not be explained solely by differences in the Al_2O_3 content, since the estimated Al_2O_3 content in the glassy matrix of porcelain C (Table 4) is similar to that found in porcelain E (Table 3). A possible explanation for this difference in K_{I0} is the fact that, compared to porcelain E, the glassy matrix of porcelain C showed higher amounts of alkali (K_2O and Na_2O) and alkaline earth (CaO, MgO, and BaO) oxides

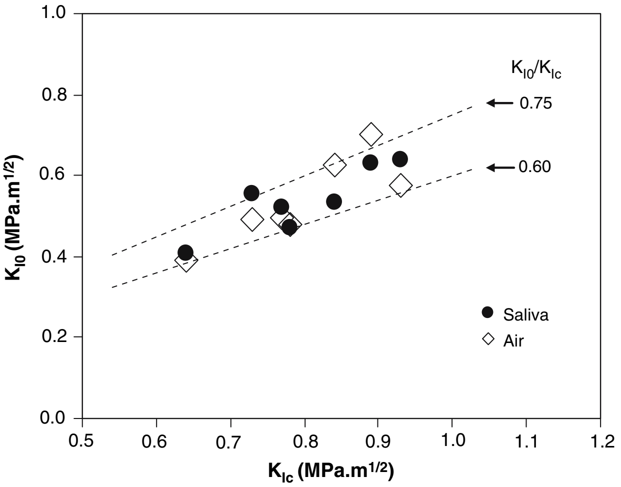

Fig. 3 Stress intensity factor threshold (K_{I0}) versus fracture toughness (K_{Ic}) in air and saliva

Table 3 Chemical analysis by X-ray fluorescence (in mol %) for all porcelains studied

Component	Porcelain						
	A	B	C	D	E	F	G
SiO_2	72.7	72.0	70.1	67.6	82.9	75.9	68.8
Al_2O_3	9.5	9.5	6.4	9.0	5.6	10.0	10.9
K_2O	8.2	9.2	8.7	8.1	4.6	7.1	9.3
Na_2O	4.9	3.8	5.5	4.9	3.6	3.6	5.6
CaO	2.6	3.9	3.8	3.7	1.1	3.0	3.6
Others	1.7 MgO	0.7 BaO 0.6 CeO	5.0 MgO 0.4 Tb_4O_7	3.0 ZnO 1.3 ZrO_2 1.2 BaO 0.6 TiO_2 0.4 P_2O_5	0.8 ZrO_2 0.7 MgO 0.3 CeO	0.3 ZrO_2	0.8 MgO 0.5 TiO_2
Traces (<0.2%)	Fe, Ni, Sn, Zn, Rb, Sr, Ce, Tb, Cl	Fe, Ni, Rb, Sr, Cs, Tb, Cl	Fe, Ni, Zr, Rb, Sr, Re, Cl	Fe, Ni, Cr, Hf	Fe, Ni, Zn, Ti, Cr, Hf, V, I	Fe, Ni, Ti, Rb, Sr, Pb	Fe, Ni, Zr, Rb, Zn, Cr, P, Cl, S

(Table 3). Among these oxides, the alkali ions (K^+ and Na^+) are more likely to be leached from the glass by means of an ion exchange process with ions present in water [21].

The positive relationship between K_{I0} and K_{Ic} (Fig. 3) indicates that the higher the fracture toughness, the higher the stress intensity factor threshold. Therefore, it seems like the previously mentioned microstructural and chemical characteristics tend to avoid not only the start of slow crack growth, but also fast crack propagation. This finding is relevant for the development of new materials, since leucite, Al_2O_3, and alkali oxide contents can be manipulated in order to obtain restorations with longer lifetime. It is important to keep in mind that for a given material, higher in-service reliability will be achieved when K_{Ic} is the highest possible, and K_{I0} is as close as possible to K_{Ic} [13]. Moreover, restorations and prostheses designed to tolerate stresses that are below their K_{I0} are more likely to have infinite lifetime because of the absence of slow crack growth [10] . Based on this rationale, it is expected that porcelains A and B will have longer lifetimes compared to the other porcelains since they showed both the highest K_{Ic} and K_{I0} (in saliva) compared to the other materials. On the other hand, restorations made with porcelain C will possibly show higher strength degradation over time since this material showed the lowest K_{Ic} and K_{I0} values.

Table 4 Calculated average glass matrix composition (in mol %) of leucite-based porcelains

Component	Porcelain				
	A	B	C	D	G
SiO_2	72.7	71.9	69.9	66.7	68.2
Al_2O_3	7.6	7.5	5.7	7.6	10.1
K_2O	7.6	8.8	8.6	7.7	9.2
Na_2O	6.2	4.9	5.8	5.8	6.4
CaO	3.3	5.0	4.0	4.4	4.1
Others	2.2 MgO	0.9 BaO 0.7 CeO	5.3 MgO 0.5 Tb_4O_7	3.5 ZnO 1.6 ZrO_2 1.4 BaO 0.7 TiO_2 0.5 P_2O_5	1.0 MgO 0.6 TiO_2

The storage media did not significantly affect the K_{I0} results, as it can be observed in Table 2. This finding indicates that for the dental porcelains investigated, the slow crack growth initiation does not depend upon environmental conditions tested (air/60% RH/22 °C and saliva/37 °C). It was expected that immersion in artificial saliva would result in a decrease in the stress intensity factor threshold, since the presence of stresses and water molecules break the chemical bonds of SiO_2-based materials at the crack tip, resulting in crack extension [9]. From the Griffith criteria of crack growth stability [8], it can be inferred that the environment-dependent surface energy was similar in both environments tested. Therefore, it is likely that in this experimental design the control parameter is not water content, but the pH at the crack tip. In this respect, Guin & Wiederhorn [22] have suggested that crack growth behavior in soda-lime glass in water is dependent on the ion exchange process (Na^+ and K^+ ions from glass exchanged by H^+ ion from liquid) at the crack tip, which can increase the local pH up to ~ 12, resulting in the formation of a corrosive fluid. At this moment, the parameters that control the pH at the crack tip in dental porcelains are unknown. Therefore, further investigations are necessary to identify the control parameters that govern the effects of the storage medium in the K_{I0} of dental porcelains.

5 Conclusion

Based on the results of this study it was possible to conclude that:

- If only leucite-based porcelains are considered, the stress intensity factor threshold (K_{I0}) increased with the increase in leucite content.
- Storage media (air/60% RH/22 °C and artificial saliva/37 °C) did not significantly affect the K_{I0} of porcelains tested.
- Porcelains with higher fracture toughness (K_{Ic}) tended to show higher $K_{I0.}$
- The increase in Al_2O_3 content or the decrease in alkali oxide (K_2O and Na_2O) content in the porcelain's glassy matrix tended to increase K_{I0}.

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