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EVALUATION OF MARGINAL ACCURACY AND MICROSTRUCTURE OF HEAT PRESSED LITHIUM DISILICATE AND ZIRCONIA REINFORCED LITHIUM SILICATE CROWNS AFTER THERMAL TEMPERING

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ABSTRACT

Objectives: To evaluate the effect of two thermal tempering cycles on marginal accuracy of crowns fabricated from two heat-pressed glass ceramics.

Materials and methods: Twenty-eight ceramic crowns were pressed from glass ceramic materials in two groups (n=14). Group (E): Lithium disilicate IPS e.max Press, Group (C): Zirconia reinforced lithium silicate (ZLS) Celtra Press. Each group was subdivided according to tempering temperature into (T1) 9% and (T2) 5% below pressing temperature of each ceramic. A stereomicroscope was used to measure the marginal adaptation before and after tempering and microstructure was analyzed. Three-way ANOVA test was used to study the effect of ceramic type, tempering, tempering procedure and their interactions on marginal gap distance. Bonferroni's post-hoc test was used for pair-wise comparisons when ANOVA test is significant. The significance level was set at $P \leq 0.05$.

Results: The interaction between the three variables had statistically significant effect on mean marginal gap distance (P-value <0.001). Before tempering and after (T1) tempering, there was no statistically significant difference between ceramic types (P-value = 0.577). After (T2) tempering, there was a statistically significant difference between ceramic types (P-value <0.001). Celtra showed statistically significantly higher mean marginal gap distance compared to E.max.

Conclusions: Mean marginal gap distance for lithium disilicate was not affected after tempering. ZLS after -5% tempering showed the highest mean marginal gap distance. All tested groups showed clinically acceptable marginal adaptation except ZLS with -5% tempering which showed clinically unacceptable marginal adaptation >120 μm.

KEYWORDS: Lithium disilicate, zirconia lithium silicate, thermal tempering, marginal adaptation, microstructural analysis.

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INTRODUCTION

The goal of dental clinicians, prosthodontists and manufacturers is to achieve esthetically and functionally perfect restorations [1, 2]. One of the main factors required for longevity and clinical success in indirect restorations is marginal accuracy.[3] Inadequate margins contribute to the accumulation of plaque, which increases the risk of periodontal diseases, microleakage, carious lesions, and pulp inflammation especially with subgingival margins [2]. Heat pressed lithium disilicates (LDS) are nowadays frequently used because of their excellent esthetics and mechanical properties, but they have some restrictions regarding clinical indications in posterior area due it their low fracture resistance compared to other polycrystalline materials.[2] To overcome the previous weakness of heat pressed lithium disilicate, Zirconia reinforced lithium silicate (ZLS) material were introduced to the market by Dentsply (Celtra Press, Dentsply, Hanau, Germany). A tampering cycle is suggested by the manufacturer to raise flexural strength up to 500 MPa by pressing then expose the material to a tempering cycle.

Due to increased zirconia (ZrO_2) content in zirconia reinforced materials (ZLS), the crystals size is smaller which can be overcome by exposing the material to additional tempering cycle to allow crystals growth, [4] [5] A thermal tempering (power glaze) protocol has been proposed. [6] The tempering processes, which were proven by the staircase experimental approach, were found to increase the fatigue failure load. [7] Unlike traditional glaze firings, which can produce tensile stresses, this extended glaze firing method, after rigorous machining of ZLS, corrects faults by creating advantageous compressive residual stresses. [6] According to Apel et al 2007 [8] the high ZrO_2 level in glass ceramics causes higher viscosity and a decrease in crystal formation leaving the flexural strength to be affected by crystals orientation to the load beam.[8] A study by Stawarczyk et al 2020 [9] revealed that power firing increased the fracture strength of ZLS (Celtra Press, Dentsply, Hanau, Germany) when compared to unfired samples and attributed it to further crystallization and growth of lithium silicate crystals eventually reinforcing the material. Moreover, the manufacturer prescribes this process as mandatory to bring the mechanical values of this ceramic to the optimum.

Additionally, investigations revealed that firing of ZLS led to changes in dimensions, color, and Weibull modulus. Firing increases flexural strength, but minimal temperature extension may increases materials inhomogeneity and risk of failure. As the ceramic microstructure compacts and refines during the firing process, ZrO_2 particles dispersed in the glass phase respond by sintering and decreasing in size. Eventually, ZLS reveals a significant shrinkage after firing, which was found to be clinically relevant and affects the accuracy. [10] However, as described by CHO et al 2012[11] and Bajaj 2013[12] there was a significant change in marginal integrity during the characterization and glaze firing steps of pressable ceramic core crowns. All systems suffered continued marginal discrepancy through all firing cycles. On the contrary, Miura et al 2014[13] and Vojdani et al 2015[14] revealed that there were no significant differences in any marginal design before and after firing the porcelain with different cervical margin designs.

Numerous researchers have examined and documented effects of heating temperature and holding period, but few studies investigated tempering temperatures effect. Questions are raised, whether subjecting the crowns to the tempering cycle suggested by the manufacturer will show any improvement on edge stability in comparison to crowns unexposed to tempering, and it is also unclear if tempering technique can be applied to lithium disilicate and other zirconia reinforced lithium silicate materials. The null hypotheses were that neither

different monolithic ceramic materials, different thermal temperatures nor the combination of both materials and tempering temperatures would affect restorations' marginal fit.

MATERIAL AND METHODS

A power analysis was designed to have adequate power to apply a statistical test of the null hypothesis that there is no difference between tested groups. By adopting an alpha (α) level of (0.05), a beta (β) of (0.2) (i.e. power=80%), and an effect size (f) of (0.544) calculated based on the results of a previous study[11]; the minimum required sample size (n) was found to be (28) samples (i.e. 14 samples per group and 7 samples per subgroup). Sample size calculation was performed using G*Power version 3.1.9.7.

A lower first molar prototype was prepared to receive an all-ceramic crown following manufacturers recommendations with 1mm rounded shoulder finish line. The prepared model was scanned, and the virtual wax patterns were designed using CAD/CAM system software (Exocad GmbH, Darmstadt, Germany) and milled in wax (YAMAHACHI DENTAL MFG, GAMAGORI, Japan) by a 5-axis CAD/CAM milling machine (CAM 5-S1 IMPRESSION VHF, Germany).

The parameters of wax patterns design were, an even 1.5 mm occlusal thickness, 1 mm axial wall thickness, and 40 μm cement thickness. The wax patterns were separated from the sprues and finished then examined for their fit over the die with 2.5X magnification.

A total of 28 wax patterns (N=28) were milled and randomly allocated to two groups fourteen patterns each (n=14) for lithium disilicate: Group (E) (IPS Emax Press; Ivoclar, Liechtenstein, Germany), and Zirconia reinforced lithium silicate as Group (C) (Celtra Press, Dentsply, Hanau, Germany). The patterns were further subdivided into two subgroups according to thermal tempering temperature (subgroup T1) 9% and (subgroup T2) 5% below pressing temperature.

The wax crowns were sprued to the thickest portion of the occlusal surface of the non functional cusp and invested in IPS PressVEST Premium (Ivoclar Vivadent) for, while for ZLS Celtra Press investment (Sirona Dentsply) for group (C). After wax elimination, all restorations were pressed following each manufacturer's recommendations temperatures in pressing furnace (Programat, EP 3000, Ivoclar Vivadent, Schaan, Liechtenstein).

After cooling down to room temperature, devesting and reaction layer removal was performed following manufacturer's instructions. Thermal tempering was performed to all groups, subgroup (T1) specimens were subjected to a temperature that is 9% below the recommended pressing temperature while in the other subgroup (T2) were subjected to experimental temperature 5% below the recommended pressing temperature (Table. 1).

TABLE (1) Pressing and Tempering temperatures for each material.

Material	Pressing Temperature (T0)	Tempering Temperature (T1)	Tempering Temperature (T2)
IPS e.max Press, group (E)	917°C	835°C	872°C
Celtra Press, group (C)	870°C	790°C	826°C

Vertical marginal gap distance measurements

The vertical marginal gap was evaluated by stereomicroscope (STEREOSCOPE VE-S, VELAB, Texas, USA) using an image analysis system (Image J 1.53t, National Institute of Health, USA). For each crown, the area of interest was captured with fixed magnification of 40X and camera settings (12MP, IOS 50,1/250s). The vertical margin gap distance between the cervical margin of the crowns and the finish line was assessed at the same marked points

in all groups. These readings were calculated using the image analysis software. The data obtained was collected, tabulated, and then subjected to statistical analysis.

Scanning electron microscopy (SEM)

The microstructure of the glass-ceramic was determined by scanning electron microscopy (SEM) (DSM 962 device, Zeiss, Germany) at magnification of 20.000X. For the SEM images, the specimens were examined three times: after pressing (T0), (T1) and (T2) thermal tempering. They were etched with an aqueous -5% hydrofluoric acid (HF) for 40 sec. After the chemical etching, the specimens were washed using acetone and distilled water. Next, they were placed in an ultrasonic bath at room temperature for 10 min. Then, they were imaged under SEM after being sputter-coated with gold.

X-ray diffraction analysis (XRD) and Energy Dispersive x-ray analysis (EDAX)

For each group, the crown was submitted to XRD (Xpert pro, USA) to determine the crystalline phases. Samples were placed on the holder of the diffractometer and scanned using Cu K α x-ray angle from 4-80 degrees, 2θ with a step size of 0.04 degrees and 5 seconds-step intervals. EDAX was carried out to quantify elements by x-ray microanalysis (FEI Czech SEM-USA).

RESULTS

Mean and standard deviation values for marginal gap distance of the two ceramic types with different interactions of variables are shown in Fig. 1. Three-way ANOVA showed that the interaction between the three variables (Ceramic material, tempering process, tempering temperature) had a statistically significant effect on mean marginal gap distance (*P*-value <0.001).

Before tempering, there was no statistically significant difference between ceramic types in mean marginal gap distance. Also, there was no statistically significant change in marginal gap

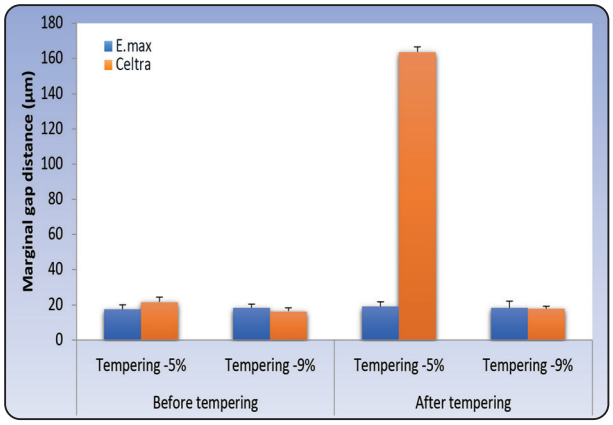

Fig. (1): Bar chart representing mean and standard deviation values for marginal gap distance of the two ceramic types with different interaction of variables.

distances of E.max ceramics after tempering procedures, But for Celtra there was a statistically significant increase in marginal gap distance after tempering procedure (P-value <0.001).

After (T1) tempering, there was no statistically significant difference between ceramic types (P-value = 0.577). On the other hand, after (T2) tempering, there was a statistically significant difference between ceramic types (P-value <0.001). Pair-wise comparisons between ceramic types revealed that Celtra showed statistically significant higher mean marginal gap distance compared to E.max.

There was no statistically significant change in marginal gap distances for E.max before tempering, -9% (T1) and –5% (T2) groups (P-value = 0.548). On the other hand, for Celtra with (T2) tempering temperature there was a statistically significant increase in marginal gap distance after tempering procedure (P-value <0.001) compared to (T1) and before tempering.

Scanning electron microscopy (SEM)

a. **Group (E) after pressing (T0):** Elongated rod-shaped crystals, sharp and pointed edges forming an interlocking microstructure. The length measured of LDS crystals was 3.293 μm, while the width measured was 687.5 nm. (Fig. 2)

b. **Group (E) subgroup (T1) samples:** Longer crystals forming an interlocking microstructure. The length measured of LDS crystals was 5.213 μm, while the width measured was 685.5 nm. (Fig. 2)

c. **Group (E) subgroup (T2) samples:** Crystals with more rounded edges, ill-defined edges and more fused together. The length measured of LDS crystals was 4.145μm, while the width measured was 871.2 nm. (Fig. 2)

d. **Group (C) after pressing (T0):** Elongated rod-shaped crystals were randomly oriented with highly interlocking microstructure. The length of crystals measured was 3.672 μm, while the width measured was 519.1 nm. (Fig. 3)

e. **Group (C) subgroup (T1) samples:** Elongated rod-shaped crystals were randomly oriented with highly interlocking microstructure. The length of crystals measured was 1.933 μm, while the width measured was 773.9 nm. (Fig. 3)

f. **Group (C) subgroup (T2) samples:** The SEM image shows ill-defined edges and more fused together compared to samples subjected to tempering (T1) and after pressing samples. The length of crystals measured was 1.758 μm, while the width measured was 640.4 nm. (Fig.3)

X-ray diffraction analysis (XRD):

The X-ray analysis (XRD) detected diffraction peaks that correspond to crystalline phases present indicating that the material is predominantly crystalline structure; LDS was identified to be the main crystalline phase for IPS Emax press samples, while lithium silicate was identified to be the main crystalline phase for Celtra press samples.

Major peaks for LDS ($Li_2Si_2O_5$) were observed at 2θ values of 24.7 degrees, 24.2 degrees, and 40 degrees. Traces of lithium metasilicate (Li_2SiO_3) and lithium phosphate (Li_3PO_4) were also detected. The XRD data showed, in Group (E) the highest peak intensity was in subgroup (T1) while equal intensity was in both after pressing and in subgroup (T2). Group (C) the highest peak intensity was in subgroup (T2) and lowest after pressing. (Fig. 4)

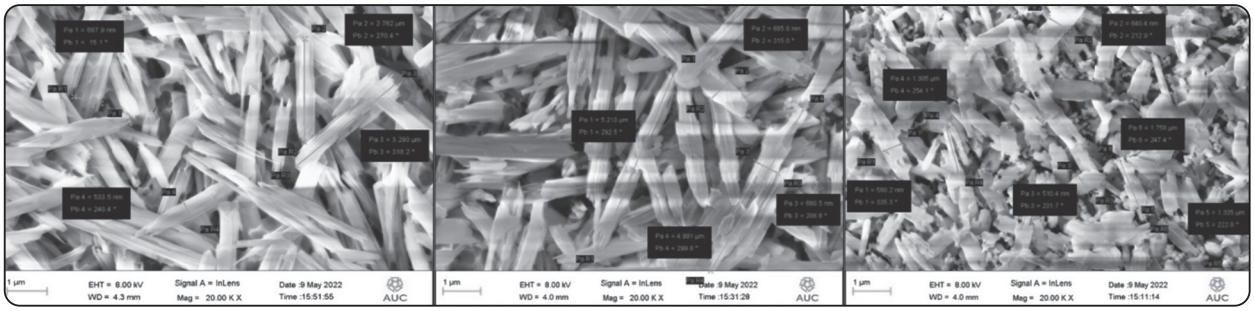

Fig. (2): SEM images (20.000x) of IPS E.max press specimens, from T0 to T2.

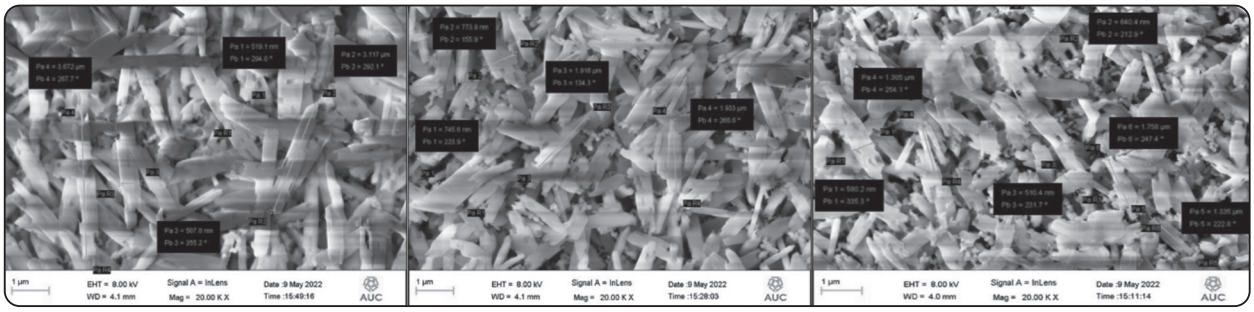

Fig. (3) SEM images (20.000x) of IPS Initial Celtra press specimens from T0 to T2.

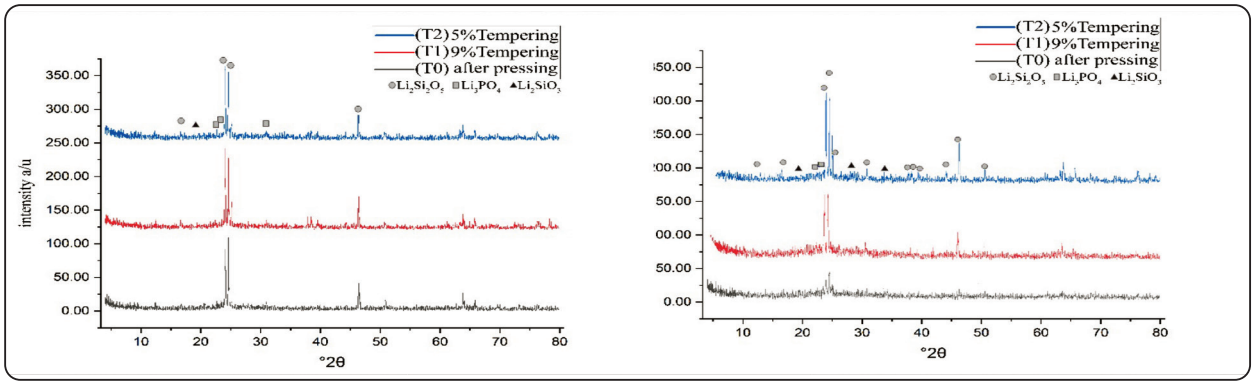

Fig. 4: X-ray diffraction patterns of E group (left) and C group (right)

Energy Dispersive x-ray analysis (EDAX)

Energy Dispersive X-Ray Analysis (EDAX) was used to identify the elemental composition of materials. EDXA crystalline composition of both heat pressing and thermal tempering, samples were not different.

DISCUSSION

Defective margins can lead to food and bacterial plaque accumulation. As the majority of dental luting agents are soluble, periodontal inflammation, caries, and stress concentration may arise. [11] There are numerous commercially available ceramic systems, making it challenging to identify the best option for each clinical circumstance.

In this current study, two different heat-pressable lithium disilicate ceramic systems were selected including pressable lithium disilicate glass ceramics like the IPS e.max Press, which is the gold standard of LDS materials. [15] To sufficiently enhance strength to withstand occlusal force and edge stability, zirconia-reinforced lithium silicate glass-ceramics have been developed by adding 10% zirconia to the glassy content like Celtra press.[16] The manufacturers recommend tempering Power firing cycle for ZLS to increase mechanical properties, as it allows further crystallization and reinforces the material. The recommended was a 9% below heat-pressed temperature, while a 5% below heat-pressed temperature was selected as the experimental tempering temperature in the current study.

Wang et al. 2015 [17] reported that ceramic materials' characteristics were intimately correlated with their microstructure and method of preparation which is affected by temperature. Moreover, marginal distortion in ceramic restorations can also be caused by shrinkage of ceramic during firing, and framework design. Previous studies have reported that the high temperature has an impact on the marginal adaptation of all-ceramic restorations.[11] Therefore, this study was designed to evaluate the effect of different thermal tempering on the marginal accuracy of crowns fabricated from different types of heat-pressed LDS ceramics.

Null hypothesis was disproved as this study results showed statistically significant differences in the marginal fit of various monolithic materials at various tempering temperatures. The results of this present study showed that there was no statistically significant difference with E group crown systems after heat pressing procedure unlike C group which showed a statistically significant increase in marginal gap distance after tempering procedure. Higher mean marginal discrepancy values after tempering procedure could be attributed to the pyroplastic flow of the ceramic material at high temperature and changing crystals size and phases. [5, 11, 16, 18]

(T2) tempering showed statistically significantly higher mean marginal gap values 91.4 ±43.7μm, than (T1) tempering mean values 18.1±1.8μm. This might be due the high temperature of (T2) tempering that is relatively close to the point at which pressable ceramic begins to soften with lack of support during tempering cycle.[19] While following the tempering firing (T1), neither of the two crown systems under examination showed any significant marginal discrepancy values. This indicated that the accuracy of the marginal fit was unaffected by the lower tempering firing cycle (T1).[5, 11]

According to Hallmann et al 2019, [5] Differential Thermal Analysis (DTA) and XRD analysis revealed that the transformation of the lithium silicate (LS) phase to the LDS phase was completed for IPS e.max ingots while for Celtra Press ingots it was not. The glass-ceramic was produced at a lower temperature resulting in an incomplete transformation of lithium metasilicate into lithium disilicate. Lithium metasilicate in the Celtra Press specimens started to react with the glass matrix at 820˚C. The peak intensity for Celtra press at 833˚C–847˚C was the highest and attributed to the crystallization temperature of the LDS phase. The LDS phase is responsible for the final properties of the ceramic material. [5]

Moreover, as more tempering applied more transformation and shrinkage occurred, as described by Schweitzer et al 2020 [10] the ceramic microstructure is compacting and refining during the firing process of ZLS, because the ZrO_2 is dispersed into the glass phase and decreasing in size which revealed a significant shrinkage after firing. It is possible that all of these factors may have contributed to the significant marginal disturbance observed in C group.

The results of this study showed that, after tempering procedure, the crystalline structure of ZLS glass ceramic group decreased as shown in SEM, while C group crystals showed a length of 3.672 μm after pressing, but a shorter crystal size was measured at T1 1.933μm. Furthermore, smaller fused crystals appeared (clusters-like) in T2 measuring 1.758μm, which could be due to ZrO_2 which hindered crystal growth. [5, 20, 21]

Although, SEM data revealed that average length of the LDS glass-ceramic (E group) crystals increased after being exposed to (T1) tempering. The (E group) rod-shaped crystals in the pressed specimens measured 3.293μm in length, whereas after (T1) tempering were 5.213μm showing longer more interlocking crystals. Increasing crystals size agreed with XRD results as the highest intensity of LDS was with (T1) tempering for (E group).

After a (T2) tempering cycle, all the groups showed decreased crystals size in comparison to (T1) tempering, this could be explained by high temperatures and short tempering cycles which are not adequate for crystals growth as revealed in (T2) tempering. [22]

However, after the heat-pressing process SEM results showed that the (E group) rod-shaped crystals, sharp and pointed edges formed an interlocking microstructure. The length of the crystals measured was 3.293 μm. It showed longer crystals after tempering (T1) with 5.213μm, while after (T2) tempering was 4.145μm. This difference may be explained by Ostwald ripening (describes the change of an inhomogeneous structure over time), in which the larger crystals grow at the expense of the smaller ones. [9, 23, 24, 25] The growth of the grain size indicates that the crystallization process continues during tempering procedure and more lithium disilicate crystals are precipitated, [20] which coincide with XRD results showing LDS peak intensity in T1 tempering.

The significant difference and clinically unacceptable measurements after (T2) tempering for (C group) samples compared to LDS after (T2) tempering temperature not only could be explained by the zirconia content increasing the viscosity of the glass ceramic and limiting the flow as mentioned earlier [20, 26] But also the low thermal stability of (C group) might have had an effect as maximum

pressing temperature should be below 880°C. LS phase starts crystallization at high temperature around 833°C. Crystallization continues as the thermal temperature increases which means the lithium silicate crystals didn't complete crystallization after pressing. [5] The (T2) tempering temperature (826°C) is 7°C below crystallization temperature. These coincide with the XRD results revealing that the highest LDS intensity was in T2 which is the highest tempering temperature.

The peripheral crystals melting shown in SEM results augments that assumption as it might be attributed to late-stage crystallization of LDS starting at 833°C. [5, 18, 27] Therefore, the marginal deformity of the (C group) is a result of both uncomplete crystallization and the high tempering temperature applied. Balkaya et al[28] and CHO 2012[11] et al also declared a significant different marginal integrity change during further firing stage which agrees with the results of these study.

In this present study the EDXA crystalline compositions of both heat pressing and thermal tempering, samples were not different. LDS glass-ceramics can be reprocessed while preserving good mechanical qualities and without appreciably changing the crystalline composition, according to El-Etreby et al 2017. [29, 30]

Fixing the tempering temperature on all materials for standardization purposes was mandatory although some manufacturers recommend different tempering temperatures which may not be compatible with other materials is among the limitations of this study. Further investigations for the effect of different tempering temperatures on the strength, colour stability, and other mechanical properties with different types of restoration, different thicknesses, and experimenting different holding times is necessary.

CONCLUSIONS

Within the limitations of this in vitro study, the following conclusions were drawn:

1. Mean marginal gap distance for lithium disilicate was not affected after tempering.
2. ZLS after -5% tempering showed the highest mean marginal gap distance.
3. All tested groups showed clinically acceptable marginal adaptation except ZLS with -5% tempering which showed clinically unacceptable marginal adaptation >120 μm.

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