Original Research Article

Effect of Thermo-mechanical Cyclic Stresses on Flexural Strength of Two Aesthetic Restoratives with Modified Filler Systems

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Abstract

Background: Numerous restorations have been used in dentistry to substitute the lost natural tooth structure. In attempts to improve the mechanical properties of glass ionomer, a nano-zirconia reinforced glass ionomer was introduced. The aim of the study was to compare the impact of cyclic stresses on flexural strength of two aesthetics restorative materials. *Materials and methods:* A nano-hybrid composite resin and a nano-zirconia reinforced glass ionomer were used in this study. A total of 80 specimens, 40 for each restorative were fabricated. In each group, 10 specimens were assigned for each aging condition as following; control, thermo-cyclic stresses, mechanical-cyclic stresses and combined thermo-mechanical cyclic stresses. Then the specimens were submitted to 3-point loading test using the Universal Testing Machine. *Results:* In all test conditions, nano-hybrid composite resin showed statistically significant higher mean flexural strength than nano-zirconia reinforced glass ionomer (61.1MPa SD ±17.3, 24.2 MPa SD ± 9.9 P-value <0.001). The control condition showed the highest statistically significant mean flexural strength (59.4 MPa SD± 26.8, P-value<0.001), while the combined condition showed the lowest statistically significant mean of flexural strength (29.7 MPa, SD±15.9, P-value <0.001). *Conclusions***:** Nano-hybrid composite showed superior performance than zirconia reinforced glass ionomer when subjected to cyclic stresses; either thermal, mechanical or combined.
Keywords: Composite resin, dental material, glass ionomer, mechanical properties, nanofillers, dental restorations.

INTRODUCTION

The ultimate target of the restorative materials is to substitute the lost natural tooth structure biologically, physically, mechanically, and functionally. Such replacement is compromised by the complex stresses generated from dynamic thermal and masticatory forces in the oral cavity resulting in deformation, distortion or even fracture of the restoration by time. [1] Nowadays composite resin and glass ionomer symbolize the most popular direct restorative materials, because each separately fulfils a wide range of the ideal requirements. However, both materials lend themselves for progressive developments to improve their shortcomings [2-4].

Nanotechnology, the science of matter at billionth of meter in size, is one of the recent contributors in their advancements that have revolutionized the filler system incorporated in composite resin and glass ionomer [5].

In the current composite resins, high filler loading is attained by blending micro and nano-sized particles, thus improving the mechanical strength, minimizing polymerization shrinkage and providing better aesthetic characteristics [6, 7]. All these improvements are guaranteed by the complete spherical shaped filler particles that allow the maximum packing density as in "Ceram X universal". The latter gives a new dimension to the composite restorations by its Sphere-Tec, the latest technology in the filler system [8, 9].

On the other hand, the incorporation of nano-sized zirconia fillers, renowned by its reinforcing behaviour, to the conventional glass ionomer powder has helped to overcome its low flexural strength and

fracture toughness; enhance material translucency; and finally provide superior handling [10-12]. This new member "Zirconomer Improved" to the glass ionomer family is also claimed by the manufacturer as new improved eco-friendly white amalgam, because it combines the benefits of both dental amalgam and conventional glass ionomer [13].

As both restorative materials are featured by their advanced filler systems, their mechanical sustainability in the aggressive oral environment will be one of the fundamental concerns regarding their performance.

Flexural strength is a significant mechanical property to evaluate the strength of brittle materials, as the investigated restoratives. Flexural strength, also known as transverse strength or modulus of rapture, is one the important mechanical properties that determines resistance to failure under complex stresses. Such condition reflects the nature of the oral stresses [1].

Laboratory mock-ups of clinical service are often conducted during testing of the physico-

mechanical properties of the restoratives to elucidate the aging effect of thermal, mechanical and chemical fluctuations besides the sorption and desorption characteristics on their performance, because clinical trials are usually costly and time consuming [14, 15].

Therefore, the aim of this research was to explore the impact of the various stresses: thermal, mechanical or combined on the flexural strength of the Ceram X universal and Zirconomer Improved. The subsequent null hypotheses were tested: 1) No difference exists in the flexural strength values of the nano-hybrid composite resin and the zirconia reinforced glass ionomer. 2) The different aging conditions do not affect the flexural strength of the nano-hybrid composite resin and the zirconia reinforced glass ionomer.

MATERIALS AND METHODS
Materials
Two nano-restoratives were designated for this research. (Table1)

Table-1: Detailed description of the designated nano-restoratives.

Restorative material	Code	Type	Composition	Manufacturer	Lot number
Ceram X	CX	Nano-hybrid composite resin	Filler type and particle size: Blend of pre-polymerized Sphere TEC™ fillers (Methacrylate functionalised silicon dioxide nano filler), non-agglomerated barium glass (0.6 μm) and ytterbium fluoride (0.6 μm) Filler loading: 77- 79 by weight% (59-61% by volume) Matrix: Methacrylate modified poly-siloxane Di-methacrylate resin	Dentsply, DeTrey GmbH, Konstanz, Germany	1609000376
Zirconomer Improved	ZI	Zirconia reinforced glass ionomer cement	Powder: Fluoro-aluminosilicate glass, Zirconium oxide, pigments and others Liquid: Polyacrylic acid solution and Tartaric acid	Shofu Inc. Kyoto, Japan	12150285

Methods
Preparation of nano-hybrid composite resin (CX) specimens

In accordance to ISO 4049 specifications [16], forty bar shaped flexural strength specimens were prepared at room temperature of 23 ± 1°C and in a relative humidity of 50±10 % in customized split Teflon mold, measuring 2 mm height × 2 mm width × 25 mm length. The composite resin was injected directly from its respective syringe into the mold which was squeezed between two plastic bands and two microscopic slides to express the excess and ensure proper surface finish. The specimens were then polymerized from upper and lower sides through the plastic bands, to avoid the occurrence of oxygen-inhibited layer on the surface of the specimen, by means of a light emitting diode light

source (Elipar free light, 3M, ESPEAG, Dental Products, St. Paul, Seefeld Germany) with 8 mm curing tip in five overlapping exposures, 20 s duration each, at an intensity of 600 m W/cm^2; verified between specimens using a radiometer. The overlapping irradiations are required, as the exit window of the light-cure unit is smaller than 25 mm. After irradiation, the specimens were removed from the mold. The excess flash was removed by abrading with P150 abrasive paper without touching the upper and lower surfaces. Then the specimens were immediately stored in distilled water at 37°C for 24h before testing.

Preparation of nano-zirconia reinforced glass ionomer (ZI) specimens

In accordance to ISO 4049 specifications [16], forty bar shaped flexural strength specimens were prepared at room temperature of 23 ± 2°C and in a relative humidity of 50 ±10 % in customized split Teflon mold, measuring 2 mm height × 2 mm width × 25 mm length. The nano-zirconia reinforced glass ionomer is a hand-mixed version. Therefore, mixing the powder/ liquid ratio according to manufacturer's instruction was done with plastic spatula on impermeable mixing pad; then the thick putty-like mix was immediately condensed in excess into the mold which is squeezed between two plastic bands and two microscopic slides to express the excess and ensure smooth surface finish. The set specimens were then removed from the mold. The excess flash was abraded with P150 abrasive paper without touching the upper and lower surfaces. Finally, the specimens were painted with two coats of Cocoa Butter for protection against desiccation. Then the specimens were immediately stored in distilled water at 37°C for 24h before testing.

Grouping of specimens

The specimens were allotted to one of the following groups:

Group I: Control group (C) =Baseline

Ten specimens of each investigated restorative material, stored in distilled water at 37°C for 24h, were blotted dry with filter paper and then the 3-point loading test was conducted to measure the flexural strength.

Group II: Thermo-cyclic group (TC)

Ten specimens of each investigated restorative material in a mesh bag were subjected to 10,000 thermal cycles between two water chambers representing the maximum and minimum temperatures in the oral cavity: 55±2°C and 5±2°C. (Thermo-scientific Precision Water Baths Model 2854; Clarkson Laboratory & Supply Inc., Chula Vista, CA). The immersion period in each water chamber was 30 s and the travelling time between two chambers was 15 s. [15]

Once this aging process was complete, the specimens were blotted dry with filter paper, and then the 3-point loading test was conducted to measure the flexural strength.

Group III: Mechanical cyclic group (MC)

Ten specimens of each investigated restorative material were subjected to mechanical cyclic loading in a chewing simulator machine (Multi-functional Chewing Simulator CS-4.2; SD Mechatronik, Feldkirchen-Westerham, Germany). In this study, an intermittent axial chewing force of 68.6 N was delivered to the specimen 10,000 times with a frequency of 1.2Hz, because clinically the mean physiological chewing force is expected to range from 49 to 150 N with a frequency range of 1.2 to 1.6 Hz [17, 18]. Once this aging process was complete, the 3-point loading test was conducted to measure the flexural strength.

Group IV: Mechanical and thermal cyclic group (MTC)

Ten specimens of each investigated restorative material were subjected first to mechanical cycling then thermal cycling as discussed before. Once this aging process was complete, the 3-point loading test was conducted to measure the flexural strength,

The 3-point loading test

Following each aging test condition, the specimen dimensions (Length, width, and height) were verified at their centres to an accuracy of 0.01 mm using a digital micrometre. Afterwards, each specimen was positioned in a 3-point loading test set up on 2 parallel supports 20 mm apart from each other, and then loaded until fracture using a universal testing machine with a 5KN load cell at a crosshead speed of 1.0 mm/min. The load-to-failure of each specimen was recorded in Newton. The flexural strength in MPa was automatically calculated using the computerized programmed based on the formula mentioned below.

Flexural strength (FS)= $3Fl / 2wh^2$

Where:

F - Load exerted at failure in Newton.
l - Span between the parallel supports in milli-meter (20.0 mm).
w - Specimen width in milli-meter.
h - Specimen height in milli-meter.

STATISTICAL ANALYSIS

Numerical data were explored for normality by checking the distribution of data and using tests of normality (Kolmogorov-Smirnov and Shapiro-Wilk tests). Data showed normal (parametric) distribution. Data were presented as mean, standard deviation (SD) and 95% confidence interval (95% CI) for the mean values. Two-way Analysis of Variance (ANOVA) was used to study the effect of material, condition and their interaction on mean flexural strength concentration. Bonferroni's post-hoc test was used for pair-wise comparisons when ANOVA test is significant. The significance level was set at P≤ 0.05. Statistical analysis was performed with IBM® SPSS® Statistics Version 20 for corrections [19, 20].

RESULTS
Two-way ANOVA results

The results showed in (Table 2) that the restorative type had a statistically significant effect on the mean flexural strength (*P*-value <0.001, *Effect size=* 0.851) regardless of the effect of aging condition. The latter had also a statistically significant effect on the mean flexural strength (*P*-value < 0.001, *Effect size=*

0.656) regardless of the effect of the restorative type. The interaction between the two variables had a statistically significant effect on the mean flexural strength (*P*-value < 0.001, *Effect size*= 0.252). Since the interaction between the variables is statistically significant, so the variables were dependent upon each other.

Effect of different interactions
Comparison between restoratives within each aging condition

CX showed statistically significantly higher mean flexural strength than ZI in all test regimens: control condition (*P*-value <0.001, *Effect size*= 0.723), thermo-cycled condition; (*P*-value <0.001, *Effect size*= 0.461), mechanical-cycled condition: (*P*-value <0.001, *Effect size*= 0.632), and finally the combined thermo- and mechanical-cycled condition: (*P*-value <0.001, *Effect size*= 0.461). (Table 2, Figure1)

Comparison between different aging conditions within each material

Pair-wise comparisons between the aging conditions of CX revealed that control showed the statistically significantly highest mean flexural strength. There was no statistically significant difference between thermo-cycled and mechanical-cycled specimens; both showed lower mean flexural strength values. The statistically significantly lowest mean flexural strength was observed with the combination of both thermo- and mechanical-cycled condition. (Table 2, Figure1).

Pair-wise comparisons between the aging conditions of ZI revealed that control showed the statistically significantly highest mean flexural strength. There was no statistically significant difference between thermo-cycled and mechanical-cycled specimens; both showed lower mean flexural strength values. The statistically lowest mean flexural strength was observed with the combination of both thermo- and mechanical-cycled condition. Combination condition showed statistically significantly lower mean flexural strength than control and thermo-cycled condition and non-statistically significant difference from mechanical-cycled condition. (Table 2, Figure1)

Table-2: The mean flexural strength (MPa), standard deviation (SD), 95% confidence interval (95% CI) values and results of two-way ANOVA test for comparison between flexural strength of the different interactions

Condition	Ceram X		Zirconomer Improved		Partial Eta Squared (*Effect size*)	*P*-value(*Between materials*)
	Mean FS (MPa) (SD)	95% CI	Mean FS (MPa) (SD)	95% CI		
Control	84.3 (8.0) [A]	79.2 – 89.4	34.4 (8.5) [A]	29.3 – 39.5	0.723	<0.001*
Thermo-cycled	54.7 (8.1) [B]	49.6 – 59.9	26.2 (9.5) [B]	21.1 – 31.4	0.461	<0.001*
Mechanical cycled	61.2 (11.8) [B]	56.1 – 66.3	20.8 (4.3) [BC]	15.7 – 25.9	0.632	<0.001*
Both	44.0 (7.4) [C]	38.9 – 49.1	15.4 (4.9) [C]	10.3 – 20.6	0.461	<0.001*
Partial Eta Squared (*Effect size*)	0.647		0.293			
P-value (*Between conditions*)	<0.001*		<0.001*			

: Significant at P ≤ 0.05, Different superscripts in the same column are statistically significantly different
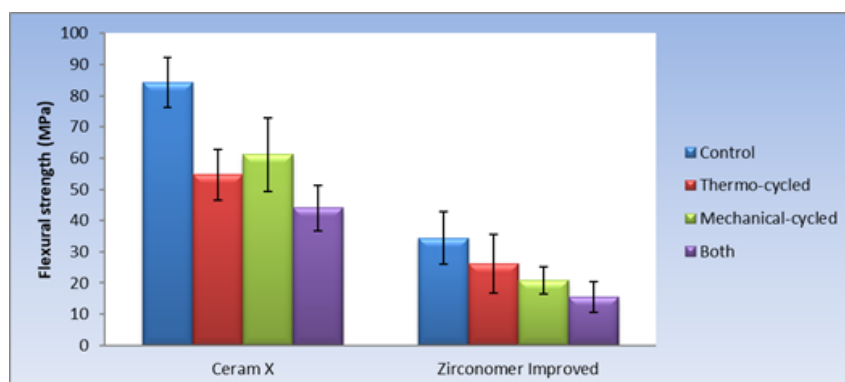

Fig-1: Bar chart representing mean flexural strength (MPa) and standard deviation values for flexural strength of the different interactions

DISCUSSION

CX is described as a nano-hybrid composite comprising organically modified ceramic nanoparticles of poly-siloxane backbone. The latter can be best designated as inorganic – organic hybrid particles where the inorganic siloxane section gives strength and the organic methacrylic share grants the particles polymerization with the polymeric matrix [6-9].

On the other hand, ZI is developed by the incorporation of novel nano-sized zirconia fillers into conventional glass ionomer to enhance material's strength, handling and translucency with the aim of displaying durability similar to amalgam [21].

When reviewing the reasons for replacement of dental restorations, their bulk fracture during service seems to be among the most relevant causes following the recurrent caries [22].

Therefore, understanding of the mechanical behaviour of these two nano-restoratives in test conditions imitating the destructive oral environment is valuable in assessing their functional survival rates [23]. This will lead certainly to more proper selection of the restorative to the appropriate application.

As a rule of thumb, stronger materials allow for more equal stress distributions, and better deformation tolerance, thus reducing the risk of failure and increasing the long-term clinical service. The three-point loading test for flexure strength has been chosen by the International Standards Organization as an efficient indicator for a resin-based filling's sustainability under complex masticatory stresses [24]. Also, lately, the three-point loading test for flexure strength has been also strongly recommended as a substitutive for compressive fracture strength test described in the International Organisation for Standardisation (ISO 9917-1: 2007) [25], as the lone distinctive performance testing of glass ionomer restoratives [26- 28].

The flexural strength is described as the ultimate stress that a material can bear before failure when subjected to bending. It is considered a simultaneous intermingling of compressive stresses at the upper face of the specimen, tensile stresses at its lower face, and shear stresses parallel to the applied loading. Therefore, it is the most proper representative of the strength as a material could only fail by either separation or slipping of atomic planes (i.e. tensile or shear failures respectively) [1].

Moreover, ISO 4049 categorises dental resinous restoratives into two types: Type I comprises filling materials appropriate for restorations of stress bearing surfaces, and Type II includes filling materials for other indications. Thus, a minimum of 80 MPa is required for flexural strength for Type I and 50 MPa for Type II [16, 29].

Regarding the mean values of flexural strength for both evaluated nano-restoratives, the first null hypothesis of the study was rejected, where CX showed higher flexural strength than ZI throughout the experimental study in all tested groups. CX in the control group was in the range of the threshold value set by the ISO standard for flexural strength in posterior restorations, while ZI displayed unfortunately lower value than the suggested one. Taking this into the account, CX could be used safely for stress bearing restorations, while ZX employment in paediatric or geriatric restorations would be more appropriate, for the quite low masticatory stresses applied to these restorations and their limited service period in the oral cavity.

The difference in the mechanical behaviour of both restoratives could be directly related to the integrity of their structural architecture. This corroborates to the findings in previous studies Geirsson *et al*. 2004 and Rodrigues *et al*. 2015. [30, 31]. The inclusion of air voids in the final set structure is considered structural flaws acting as a source of stress concentration increasing both the heterogeneity and naturally the brittleness of the restorative material, and finally, rendering the material more prone to fracture under flexure [1].

CX, as nanohybrid composite set by addition polymerization at room temperature giving rise to a continuous structure consisting of three elemental components: a highly crosslinked polymer matrix, a high-volume fraction of inorganic filler of Sphere TEC technology with reduced interparticle matrix and a coupling agent added to ensure matrix-filler adhesion [1]. Thus, the presence of the gradient sized fillers guarantees the least void percentage as reported previously by Elbishari *et al*. 2012. [32] On the other hand, the microstructure of the ZI is tailored as a sequence of the acid-base setting reaction. Thus, the included water that primarily exists as a solvent, together with the water delivered by the reaction, may accumulate into ultrafine globules during and after setting and eventually diffuse through the set cement leaving behind pores [33-36].

Moreover, the inclusion of such air voids is likely to be a consequence of the manipulative process of the investigated restorative materials during specimen preparation. CX was supplied as single paste in syringe form. The single paste is directly packed into the Teflon mold without mixing and minimum handling, accordingly no opportunity for air incorporation. This could be also extrapolated from earlier studies, where the incorporation of air during spatulation and insertion of hand-mixed chemically cured materials contributed significantly to higher level of porosity in comparison to the minimum handling of

the single-paste light-activated materials during specimen preparation [37- 40].

On the contrary, ZI is supplied as two components in powder liquid version, where manual mixing is required for 30 s with the inevitable air bubbles incorporation due to the viscous nature of the liquid. This finding agrees with several studies, which showed that the hand mixed version of glass ionomer, when compared to encapsulated type, displayed greater operator variability regarding proportioning of the powder and liquid and mixing which might ultimately end by higher intrinsic porosity, reduced strength and higher failure rate [41 -44].

Another important fact, which could be attributed to the different mechanical behaviour of the investigated nano-restoratives, originates principally from the reinforcing filler system, which resists the stresses on loading rather than their weak matrices [1].

The higher flexural strength of CX could be explained by the specific features of their filler particles. First, they prevent upon loading any mechanical stress concentration in the composite structure by their spherical shape without any sharp angles or protuberances. Secondly, they allow more balanced denser filler particle distribution by the gradient particle size in the nanohybrid composite structure (Table1) [7-9].

With respect to the filler system in ZI, certain limitations restricted its reputable reinforcing effect. Influential information, regarding the percentage of the incorporated nano-zirconia or the particle size distribution of the different filler particles, was not disclosed by the manufacturer. Therefore, the lower flexural strength of ZI might be referred to the non-uniform and non-homogenous distribution of nanoparticles in glass ionomer powder. Also, the greater surface area of the reduced sized ZrO_2 particles compared to the reactive glass may interfere with the acid base setting reaction, creating fewer polyacrylate crosslinks leading to poor interfacial bonding between ZrO_2/glass and the polyacrylate matrix, resulting in an overall decline the mechanical strength. This assumption is in alignment with the findings of other studies, which found by scanning electron microscopy that the cracks propagate around the glass and ZrO_2particles rather than propagate through the particles. This suggested that the interfaces between the ZrO_2/glass particles and the ionomer matrix might represent the weak sites in the cement [45-47].

The second null hypothesis must be also rejected for both evaluated nano-restoratives, each of which showed a significant reduction in flexural strength after thermal cycling, mechanical cycling and mechanical cycling followed by thermal cycling.

In laboratory studies, thermal cycling (TC) is a frequently used aging procedure for matching temperature fluctuations that occur in the oral cavity during eating, drinking, and breathing. The thermal cycling regimen differs significantly among experimental studies regarding number of cycles, the temperature of water baths, dwelling time in each bath and transfer time. The adopted thermal cycling protocol in this experimental study correspond to one -year in vivo clinical service as postulated by several authors [14, 48]. The observed significant decline in the flexural strength of both nano-restoratives matched the results of several previous investigations [49-51]. This reduction could be attributed to the repetitive contraction expansion stresses resulting from mismatching in the coefficients of both filler and matrix components of each nano-restorative. Additionally, the developing thermal stresses might create fissures in the material that could allow continuous ingress of fluids during immersion in the different water baths. Thus, thermal cycling combines both thermal and hydrolytic degradation by repeated sudden changes in temperature.

Moreover, the degradation by thermocycling was significantly higher in ZI could be related to its possible higher percentage of porosity than that of CX, and according to previous study by Aws *et al*. 2018 [52] porosity will act as a reservoir for water within the structure of material in which water can be retained and transported through the restorative structure.

Also, mechanical cycling (MC) of materials has also gained increased importance in laboratory studies for simulating oral chewing forces. The current study was conducted following the same protocol proposed by previous studies to achieve a constant occlusal load distributed during chewing [17, 18, 53]. The mechanical cycling, like the thermal cycling, reduced significantly the flexural strength of both nano-restoratives. Although this falloff in their flexural strength is higher than that observed due to thermal cycling, but the difference was insignificant. The reduction can be referred to the possibility of localized build-up of higher stresses around their structural defects such as pre-existing voids introduced during material processing, imperfect interfaces, and residual stresses, thus leading to increased susceptibility to crack initiation and growth under repetitive loading application ending up into catastrophic failure [54].

The use of the mechanical cycling combined with thermo-cycling (MTC) raised significantly the detrimental effect on the flexural strength of both nano-restorative materials. This problem has been also addressed by Drummond & Babna 2003 who reported that the reduction in the flexural strength of fiber reinforced resin materials due to thermo-mechanical cycling indicated that their use in the oral environment reduced their mechanical performance thus threatening their clinical service [55].

CONCLUSIONS

The following points could be concluded: 1). The flexural strength of the nanohybrid composite resin material is higher than the nano-zirconia reinforced glass ionomer. 2) The nanohybrid composite resin material showed better mechanical performance than the nano-zirconia reinforced glass ionomer under the different aging test conditions. 3) The aging condition either thermal cycling or mechanical cycling caused comparable detrimental impact on the flexural strength of both nano-restorative materials. 4) The exposure to the combined effect of aging conditions was the most challenging for both nano-restoratives materials.

Clinical implication: This experimental study could help the clinician to highlight feasible mechanical aspect of the restorative's short- and long-term behaviour.

Data Availability

The data concerning the findings of this investigation are available at the corresponding author upon request.

Conflict of Interest

No conflicts of interest regarding the publication of this paper.

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