

FRACTURE RESISTANCE IN PREMOLARS RESTORED WITH DIRECT RESTORATIVE MATERIALS CONTAINING NANO- ZIRCONIA FILLERS: AN INVITRO COMPARATIVE STUDY

Amira Mohammad Samy*^{ID}, Nagy Abdulsamee**^{ID}, Passant Nagi***^{ID}
and Shimaa Mahmoud Ameen****^{ID}

ABSTRACT

Background: Zirconia is a crystalline dioxide of zirconium (ZrO_2) white powder used in restorative esthetic dentistry as a superior additive to enhance mechanical properties. It is considered an intervention based on esthetic and functional properties to achieve the goal of restorative dentistry that blends art and science.

Objectives: This invitro study was conducted to assess and compare the fracture resistance of nano-zirconia-containing glass ionomer restorative materials and nano-zirconia-containing resin composite.

Methods: Maxillary premolars restored with two zirconia containing direct restorative materials (zirconfill composite and zirconomer improved glass ionomer) were evaluated for fracture resistance. Forty freshly extracted maxillary intact premolars were collected and stored. After occluso-mesial standardized cavity preparation, according to the used material teeth were randomly divided into two main groups ($n=20$). Each group is then divided into two subgroups ($n=10$) to be tested at different time intervals (24 hours and one week). Data were collected, tabulated and statistically analyzed.

Results: Fracture resistance test showed a significant difference between zirconfill composite and zirconomer improved at day one-time interval (zirconfill showed better fracture resistance), while there was no significant difference on day 7.

Conclusion: Nano zirconia modification in glass-ionomer powder could enhance its dental application as a restorative material. Zirconia has a strengthening effect on the physico-mechanical properties of resin composite.

Clinical significance: For clinical success, clinicians must be aware of the properties of restorative materials and smart selection with accurate manipulation.

KEYWORDS: Composite, Fracture Resistance, Glass Ionomer and Zirconia Fillers.

* Lecturer of Operative Dentistry, Faculty of Oral and Denal Medicine, Badr University.

** Ex Professor of Dental Biomaterials, Faculty of Dentistry, Misr University for Science and Technology

*** Lecturer of Pediatric Dentistry, Faculty of Dentistry, Cairo University.

**** Lecturer of Operative Dentistry, Faculty of Dental Medicine for Girls, Al Azhar University

INTRODUCTION

Removal of tooth structure through cavity preparation has weakened the teeth and increased their susceptibility to fracture⁽¹⁾. Restorative materials are used to restore hard dental tissue loss from dental caries and any other non-carious defects. So, the restorative material's ability to withstand masticatory forces is one of the significant properties to be considered in restorative practice.

Diversity of restorative materials that is commercially available to withstand occlusal forces and restore the lost tooth structure to esthetic harmony. Zirconfill is a nano-hybrid light-curable composite resin; its inorganic filler zirconia/silica mixed compound is presented as nanoparticles and nano-agglomerates.

Glass ionomer restorative materials are indispensable in restorative applications due to their distinct characteristics, such as their ability to chemically adhere to moist tooth substrate, anti-cariogenic properties, and biocompatibility⁽²⁾. Despite its advantages, glass ionomer restorations have limitations, including weak mechanical properties such as brittleness, lack of strength, and hardness. Therefore, modifications have been made to these materials to enhance their mechanical properties, which are crucial for clinical functionality⁽²⁾.

Zirconia particles are added to the glass component to strengthen further the material's structural integrity for long-lasting durability and excellent occlusal load tolerance to overcome the strength drawbacks of conventional glass ionomers⁽³⁾. Due to their high mechanical properties, biocompatibility, and aesthetics, Zirconia particles have an interest as a reinforcement material in restorative dentistry. Zirconomer Improved® is a recently introduced variant of glass ionomer reinforced by addition of Zirconia fillers, increased strength ceramic fillers and outstanding esthetic properties⁽⁴⁾.

Therefore, it is a must to assess their strength properties as one of the objectives for fulfilling mechanical properties to meet the demands of load-bearing areas.

The durability of a restorative system is predominantly determined by the fracture resistance of the restorative material, which can bear the intraoral forces generated during both functional and parafunctional activities⁽⁵⁾. The fracture of restorative materials refers to a partial or complete breakage of the material caused by excessive force. The ability to resist fracture is a crucial property that directly correlates with the material's susceptibility to cracking. Both experimental and theoretical endeavors have been pursued to establish a relationship between a material's strength and its ability to resist fracture⁽⁶⁾. Therefore, this invitro study aimed to assess the fracture resistance of nano-zirconia-containing glass ionomer restorative materials compared to nano-zirconia-containing resin composite. The null hypothesis assumed that there is no significant difference between the two groups zirconfill composite and zirconomer improved glass ionomer at any time interval.

MATERIALS AND METHODS

Fracture Resistance Test:

Sample size calculation

Using the power analysis procedure, sample size calculation was conducted. Depending on a continuous response variable from independent control and experimental subjects, with one control per practical subject. In a previous study⁽¹⁾, the response within each subject group was normally distributed with a standard deviation of 1.9. Suppose the actual difference in the experimental and control means is 3.2. In that case, we must study 7 experimental and 7 control subjects in each time interval. The null hypothesis that the population means of the experimental and control groups are equivalent can be rejected with a probability (power) of 0.8 while maintaining a Type I error probability of 0.05 for the test. The calculation was increased by 25% to compensate for dropped-out samples resulting in processing failures.

TABLE (1) Materials utilized for this investigation.

Materials	Materials' specification	Composition	Manufacturer	Batch number
Zirconfill composite https://www.eurotechnew.pt/index_htm_files/ZIRCONFILL%20ENG.pdf	Nano-hybrid universal restorative composite	Fillers: Inorganic fillers of zirconia and silica (80 wt %). Matrix: a mixture of monomers (Bis-GMA, Bis-EMA, TEGDMA and UDMA). Dentin shade.	TECHnew, Brazil.	16002
Zirconomer Improved https://www.shofu.de/en/produkt/zirconomer-improved-uk/	Zirconia-reinforced restorative glass ionomer	Powder: aluminosilicate glass, zirconium oxide and pigments. Liquid: Polyacrylic acid solution and tartaric acid. Universal shade.	Shofu, Japan	3130
37% Phosphoric Acid Etching Gel in Syringe https://multimedia.3m.com/mws/media/2768680/adper-single-bond-2-technical-profile.pdf	Scotchbond Universal	35% phosphoric acid by weight. The phosphoric acid gel etchant is thickened to a high viscosity with fumed silica and a water soluble surfactant	3M Espe, St.Paul, MN, USA.	41263
Adper™ Single Bond 2 Adhesive https://multimedia.3m.com/mws/media/2768680/adper-single-bond-2-technical-profile.pdf	A total etch, visible-light activated dental bonding agent. (Two steps-etch and rinse adhesive).	BisGMA, HEMA, dimethacrylates, ethanol, water, a novel photoinitiator system and a methacrylate functional copolymer of polyacrylic and polyitaconic acids. 10 percent by weight of 5nm diameter silica filler.	3M Espe, St.Paul, MN, USA.	N716057

Specimens' preparation

Teeth collection:

A total of 40 maxillary premolars extracted from patients aged 20-30 years, who were seeking orthodontic treatment at the Oral Surgery Clinic, Faculty of Dental Medicine, Al-Azhar University, Girls Branch, were collected under a research protocol that had been approved by the ethics committee (REC-PD-22-16). Subsequently, the teeth were thoroughly cleaned. Inclusion criteria were applied to ensure that only healthy teeth without any caries lesions, cracks, previous restorations, or other anomalies were included in this study. Following extraction, teeth were stored in a buffered saline containing 0.1% thymol until used

for the analysis⁽⁷⁾. Teeth that were similar in size, morphology, and shape were selected to minimize the potential effect of shape and size variations on the study results. The buccolingual and mesiodistal dimensions of each tooth were measured in millimeters at the most prominent points of the crown using a digital caliper, with a precision of ± 0.5 mm. The measurements showed that the buccolingual dimensions were approximately 9mm (± 0.5 mm), while the mesiodistal widths were about 7mm (± 0.5 mm)⁽⁸⁾.

Periodontium simulation and Mounting of the teeth

To simulate the periodontium, roots of teeth were coated with melted set-up wax (Cavex, Holland) up to 2 mm depth away from the cemento-enamel

junction, using a periodontal probe to create a uniform coating of approximately 0.3 mm around the roots. Teeth were then mounted on acrylic resin blocks using a cylindrical stainless-steel split mold, which had a diameter of 2 cm and a height of 2.5 cm. The level of the acrylic resin was adjusted to 2 mm below the cemento-enamel junction of each tooth to ensure an accurate representation of the periodontium⁽⁹⁾. A parallelogram device (Paraflex, BEGO-Germany) allowed accurate vertical centralization of the tooth in the acrylic mold⁽¹⁰⁾.

Cavity preparation

Standardized mesio-occlusal cavity (MO) was carried out using five-axis CNC machines (CINCINNATI Milacron VT440-41) with water coolant to avoid the effect of heat generation during the teeth preparation and cracking; mesio-occlusal cavity design was done using AutoCAD software, and then transferred to the control panel of the CNC machine (Figure 1) to start the milling.



Fig. (1) CNC machine

The occlusal part in the box-shaped preparation was 2 mm wide (buccolingual), 4mm length (mesiodistally), and 2 mm depth, and the proximal box extended 2 mm apical to the isthmus with 1.5 mm axial depth and gingival seat buccolingual was 2 mm width⁽¹¹⁾. Dimensions were confirmed in the occlusal portion (in relation to the cusp tips buccolingually) and gingival seat with a digital caliper.

Depth was confirmed using periodontal probe.

Specimens grouping

Following the completion of cavities preparation for all specimens, the teeth were randomly assigned to two groups (n=20 per group) based on the restorative material used according to allocation ratio (1:1) blindly using closed envelopes for the authors. For the restoration of each group, Zirconfill® light-curable composite resin was used for one group (n=20). Meanwhile, Zirconomer Improved® was used to restore the other group (n=20). For restoration of teeth in the two main groups, a circumferential metal matrix Automatrix® (Dentsply, Milford, DE, USA) was securely placed around each tooth's prepared mesio-occlusal (MO) surface. The cavities were cleaned using a gentle air-water spray to remove any remaining debris.

To restore the teeth in the zirconfill group, a two-step etch-and-rinse dentin bonding agent was used following the manufacturer's instructions to simulate the clinical protocol. The process involved the application of a 37% phosphoric acid etching gel for 15 seconds then rinsed thoroughly for 15 seconds, followed by 2 consecutive coats of Adper™ Single Bond 2 Adhesive with gentle agitation. The adhesive was then light cured using a standard 1200 mW/cm² actual irradiation output and a wavelength of 440–490 nm for 20 seconds. Resin composite was applied in increments and adapted to the cavity walls using a composite applicator. Each increment was then polymerized for 20 seconds.

For restoration of the specimens of Zirconomer Improved® group, Following the manufacturer's instructions, two scoops of powder and one drop of liquid were dispensed onto a mixing pad. The powder was then divided into two equal parts, and the first half was mixed with the liquid for 5-10 seconds using a cement spatula. The remaining half was mixed until a thick putty-like consistency was achieved, which took 30 seconds. The mixture

was filled with a small condenser. The material was applied to the proximal wall from the level of the gingival seat up to the pulpal floor. Matrix band is removed after initial setting of the material when it becomes opaque and rigid; surface of the restoration was painted with petroleum jelly.

Fracture Resistance Test procedure:

Each group of restored teeth is then divided into two subgroups (n=10) to be tested at different time intervals (24 hours and one week). Each specimen was mounted separately onto a computer-controlled materials testing machine (Instron, Model 3345 Universal Testing Machine; Norwood, MA, USA) equipped with a 5KN load cell. The data was recorded using computer software (Bluehill Lite from Instron®), and the samples were fastened onto the lower fixed compartment of the testing machine using screws. To conduct the fracture test, a metallic rod with a spherical tip (diameter of 3.6 mm) was affixed to the upper movable compartment of the testing machine. The load was applied occlusally in compressive mode, with the rod travelling at a crosshead speed of 0.5 mm/minute. The load at which the samples failed was indicated by an audible cracking sound and verified by a sudden and noticeable drop in the load-deflection curve, which was recorded using computer software (Bluehill Lite from Instron®). The load required to cause fracture was measured in Newtons.⁽¹²⁾

Statistical Analysis:

All data were presented as means and standard deviations. Statistical analysis of the given data was performed using IBM SPSS software package version 24.0. (Armonk, NY: IBM Corp, US) and Graph Pad Prism (GraphPad Software, Inc, US). Shapiro-Wilk and Kolmogorov-Smirnov tests were used to examine the given data for normality,

and the results showed that the significant level (P-value) was insignificant because the P-value >0.05, which meant that all the data came from a normal distribution (parametric data) that resembled a normal Bell curve. As a result, Paired t-test was used to evaluate the effect of time on both groups, and the One Way ANOVA test was used to compare two intervals, followed by Tukey's Post Hoc test for multiple comparisons.

RESULTS

Means and standard deviations between the compared groups are shown in (Table 2) and represented in the bar chart (Figure 2).

Fracture resistance test, the mean \pm standard deviation of the Zirconfill group at one-day and seven-day time intervals were (630.72 \pm 84.34) and (1049.82 \pm 163.96) respectively. While for the Zirconomer group, the mean \pm standard deviation of fracture resistance at one day and seven days were (750.91 \pm 93.14) and (957.42 \pm 118.44) respectively, as listed in Table (3), as shown in Figure (1). Performed Paired t-test for significance evaluation of storage time effect for both groups separately revealed a significant difference at P-value \leq 0.05, listed in (Table 3).

On the other hand, One Way Analysis of Variance (ANOVA) was used to compare the significance between different time intervals of Zirconfill and Zirconomer groups, revealing highly significant differences as P-value <0.0001. The significance was followed by multiple comparisons using Tukey Post hoc test, which showed significant differences between different time intervals for each group separately while revealing insignificant differences between both groups for each time interval individually, as listed in Table (3) and shown in Figure (2).

TABLE (2) Intragroup Comparison of Fracture Resistance between Different Time Intervals using Paired t-test:

Material	Time interval	M	SD	SEM	95% CI		t	df	P-value
					Upper	Lower			
Zirconfill	One Day	630.72	84.34	26.67					
	Seven Days	1049.82	163.96	51.85	-556.83	-271.37	-6.417	9	0.00 *
Zirconomer	One Day	750.91	93.14	29.45					
	Seven Days	957.42	118.44	37.45	-346.86	-66.16	-3.328	9	0.009*

M; Mean, SD; Standard Deviation, SEM; Standard Error of Mean, CI; Confidence Interval, df; Degree of Freedom, P; Probability Level.

** Significant Difference at P-value ≤ 0.05 using Paired T-test*

TABLE (3): Intergroup Comparison of Fracture Resistance between Different Time Intervals of Different Materials using One Way ANOVA:

Material	Time interval	M	SD	SEM	Tukey`s Post Hoc	t	df	P-value
Zirconfill	One Day	630.72	84.34	26.67	A	-6.417	3	<0.0001*
	Seven Days	1049.82	163.96	51.85	B			
Zirconomer	One Day	750.91	93.14	29.45	A	-3.328		
	Seven Days	957.42	118.44	37.45	B			

M; Mean, SD; Standard Deviation, SEM; Standard Error of Mean, df; Degree of Freedom, P; Probability Level

**; Significant Difference at P-value ≤ 0.05 using Paired T-test*

The same letters in the column revealed insignificant differences using Tukey`s post hoc test for multiple comparisons. Different letters in the same column revealed significant differences using Tukey`s post hoc test for multiple comparisons.

**; Significant Difference at P-value ≤ 0.05 using One Way ANOVA*

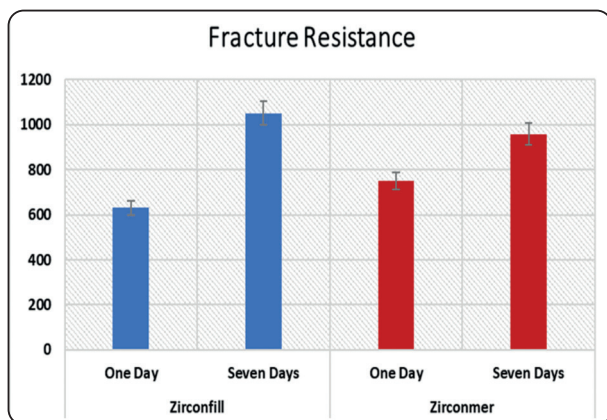


Fig. (2): Bar Chart revealing Comparison of Fracture Resistance between Different Time Intervals of Different Materials

DISCUSSION

This study evaluated the fracture resistance of two nano-zirconia-reinforced direct restorative materials. Mechanical strength evaluation indicates the clinical success of restorations. The ability of dental composites to resist crack propagation from an initial flaw is crucial, as the bulk fracture is one of the primary causes for the shorter lifespan of resin composites compared to durable glass ionomers. Literature reported that a micro-crack initiated in a resin composite restoration with zirconia/silica nano-fibers could significantly improve the mechanical

properties as the crack opening resisted the bridging fibers and the resin matrix was reinforced ⁽¹³⁾.

A crucial property directly related to cracking is fracture resistance, giving information about tooth structural integrity, even though invitro studies are un-actual in reproducing typical chewing strokes ⁽⁸⁾. The inclination of premolar cusps and their position in the dental arch make them prone to cusp fracture when subjected to occlusal load. Occluso-proximal cavities were designed to simulate a common clinical scenario ⁽¹⁴⁾. Maxillary premolars were known to show the least anatomical variations and have the same potential for fracture as molar teeth ⁽¹⁵⁾. Therefore, they are appropriate for evaluating the effectiveness of materials in enhancing their fracture resistance ⁽¹⁶⁾. Cavity standardization using a CNC milling machine to avoid human variations and deviations by any other means as free hand cavity preparations.

Zirconia has gained significant attention as a reinforcing material in dentistry owing to its excellent mechanical properties and a broad range of applications as a biomaterial, particularly in dental restorations. Also, it can enhance esthetic properties of glass ionomers by reducing the opaqueness of it ⁽⁴⁾. As claimed by the manufacturer, the nanoclusters are agglomerates of nano-particles that work as a single unit allowing a higher loading percentage of inorganic fillers to be incorporated into the resin matrix, improving physical and mechanical properties. This agrees with **Al-Jeaidi** stating that incorporating zirconia nano-particles in bulk-fill composite materials could improve fracture resistance ⁽¹⁷⁾. Composite resins have a similar elastic modulus as dentin, so it is a recommended material for ensuring fracture resistance of the tooth ⁽¹⁸⁾. In addition, zirconia's transformational toughening property prevents the development of cracks and endows composites with robust mechanical properties ⁽⁴⁾.

Zirconia particles incorporated into the glass powder of zirconomer improved are subjected to controlled micro-ionization for optimum grain size that has exclusive characteristics of zirconia called transformation toughness that gives higher strength and higher tolerance to occlusal load due to particle size homogeneity, which explains the higher fracture resistance results of this comparative study at seven days' time interval that does not significantly differ statistically with zirconfill composite group ⁽²⁾. This may also be due to Ytria-stabilized zirconia particles that contribute to increasing strength, elastic modulus and material durability ⁽¹⁾. It was found that the micro-sized YSZ-GIC powders exhibited a bimodal particle distribution, which ensured that the glass ionomer cement achieved a high packing density, resulting in superior mechanical properties for Zirconomer. ⁽¹⁴⁾. The researchers approved the homogenous incorporation of micro-ionized ZrO₂ particles in glass-ionomer material to enhance the performance of new restorative materials in load-bearing areas due to the robust bonds between the ceramic glass matrix and zirconium oxide particles. ⁽⁴⁾. Structural microscopic analysis of zirconomer improved samples observed fine submicron grain structures of glass particles with nano-sized zirconia ceramic crystals, glassy-depleted zirconia ceramics based on polycrystalline zirconia powder that is considered a revolution in modern restorative dentistry due to its advanced physico-mechanical properties thus termed 'ceramic steel' ⁽¹⁹⁾. Also, the literature proved that the mechanical properties increase by adding nano-ZrO₂ particles due to increased surface area and better particle distribution ⁽²⁰⁾. It was stated that glass powder and polyalkenoic acid were also specially processed to convey these high glass ionomer strength properties ⁽²⁾. Some studies reported that using a low elastic modulus material as glass-ionomer compared to dentin presents the advantage of resiliency and higher deformation under occlusal forces, reducing the risk of fracture ⁽¹⁸⁾.

CONCLUSIONS

Within this study's limitation, zirconia-reinforced glass-ionomer can be used in high-stress bearing areas and as a structural base and core material as an effective alternative to resin composite to gain the benefits of anti-cariogenic properties and chemical adhesion with healthy and caries affected tooth structure.

Recommendations:

- 1- Conducting long-term clinical studies to validate the invitro results is recommended. These studies would reflect the dynamic intraoral conditions and chemical, thermal, and physical stresses that restorative materials undergo over time.
- 2- Further studies on mesio-occluso-distal cavities should be done.
- 3- An innovation of a resin-modified batch of zirconomer is recommended to be proportioned and encapsulated as pre-weighted capsules, as mechanical mixing is more accurate than hand proportioning and mixing.

REFERENCES

1. Swathi UB, Ramesh S, Pradeep S. Fracture Resistance of Mesio-Occlusal-Distal Cavities Restored with Composite, Resin Modified GlassIonomer Cement and Zirconomer - An In Vitro Study. *Int. J. Dent. Oral Sci.* 2020; S10: 44-9.
2. Chalissery V, Marwah N, Almuhaiza M, et al. Study of the Mechanical Properties of Novel Zirconia-reinforced Glass Ionomer Cement. *J. Contemp. Dent. Pract.* 2016, 17(5):394-8.
3. Wan Bakar Z, Sajjad A, Mohamad D, et al. Various recent reinforcement phase incorporations and modifications in glass ionomer powder compositions: A comprehensive review. *J. Int. Oral Health.* 2018; 10(4):161-7.
4. Melo T, Oliveira I, Brandim A et al. Properties of zirconia-containing glass-ionomer cement. *Ceramica.* 2019; 65: 394-9.
5. Safy R and Abd Elmohsen H. Assessment of the Fracture Resistance of Novel Zirconia Reinforced Glass Ionomer Compared to Nano Hybrid Resin Composite Restorations. 2019. *Egypt. Dent. J.* 2019; (65); 3735- 44.
6. Ali Alrahlah. Diametral Tensile Strength, Flexural Strength, and Surface Microhardness of Bioactive Bulk Fill Restorative. *J. Contemp. Dent. Pract.* 2018; 19 (1):13-9.
7. Dere M, Özcan M, Göhring N. Marginal quality and fracture strength of root-canal treated mandibular molars with overlay restorations after thermo-cycling and mechanical loading. *J Adhes Dent.* 2010; 12: 287-94.
8. Hamoudaa I and Shehatab S: Fracture resistance of posterior teeth restored with modern restorative materials. *J. Biomed. Res.* 2011; 25(6): 418–24.
9. Mosallam R and Haridy M. Fracture Strength of Endodontically Treated Teeth Restored with Endocrown Restorations with /without Resin Composite Base Materials – An In Vitro Study. *Egypt. Dent. J.* 2019; 65:2837-2848.
10. Petronijevic B, Markovic D, Sarcev I, et al. Fracture resistance of restored maxillary premolars. *Contemp. Mater.* 2012; 2(3):219-25.
11. Barata T, Bresciani E, Fafundes T, et al. Fracture resistance of Class II glass-ionomer cement restorations. *Am. J. Dent.* 2008; 21(3):163-7.
12. Guray Efes B, Yaman B, Gumustas B, et al. The effects of glass ionomer and flowable composite liners on open-sandwich class II fracture resistance restorations. *Dent. Mater. J.* 2013; 32(6):877-82.
13. Guo G, Fan Y, Zhang J, et al. Novel dental composites reinforced with zirconia–silica ceramic nanofibers. *Dent. Mater.* 2012;28(4):360-8.
14. Kamath U and Salam A: Fracture resistance of maxillary premolars with mod cavities restored with Zirconomer: An in vitro comparative study. *Int. J. App. Dent. Sci.* 2016; 2(2):77-80.
15. Sud N, Gupta A, Sharma V et al. Comparative evaluation of the fracture resistance of maxillary premolars with Mesio-occlusal distal cavities restored with Zirconomer, Amalgam, Composite and GIC: An in vitro study. *Int. J Comm. Health. Med. Res.* 2019;5(1):52-5.
16. Mincik J, Urban D, Timkova S et al. Fracture Resistance of Endodontically Treated Maxillary Premolars Restored by Various Direct Filling Materials: An In Vitro Study. *Int. J. Biomater.* 2016;1-5.
17. Al Jaedi Z: Fracture resistance of endodontically treated teeth restored with Zirconia filler containing composite core material and fibre posts. *Pak. J. Med. Sci.* 2016; 32(6):1474-8.
18. Ciavoi G, Mărgărit R, Todor L, et al. Base Materials' Influence on Fracture Resistance of Molars with MOD Cavities. *Materials.* 2021; 14(18):5242-51.
19. Kupka T, Karolus M, Fryc M: The newest clinical version of glass-polyalkenoate restorative biomaterial infused with 3Y-TZP nanocrystals. *J. App. Biotech. Bioengin.* 2018; 5(6):338-40.
20. Moheet I, Luddin N, Rahman I, et al. Modifications of Glass Ionomer Cement Powder by Addition of Recently Fabricated Nano-Fillers and Their Effect on the Properties: A Review. *Eur. J. Dent.* 2019; 13(3):470-7.