



## A COMPARATIVE EVALUATION OF MICROLEAKAGE, COMPRESSIVE STRENGTH, FLEXURAL STRENGTH AND FLUORIDE RELEASE OF ZIRCONOMER AND KETAC SILVER: AN IN VITRO STUDY.

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### Abstract

**Background:** A successful restorative material forms a better adhesion, resist the microleakage and releases fluoride. However, existing glass ionomer cements cannot be used as a posterior restorative material in stress bearing areas. A new ionomer called Zirconomer, zirconia reinforced glass ionomer claims to exhibit high physical and biological properties.

**Aim:** To assess and compare the microleakage, compressive strength, flexural strength and fluoride release from zirconomer with ketac silver.

**Materials & Methods:** On twenty caries free premolar teeth (10 per each group), a class v cavity was restored with zirconomer and ketac silver. The microleakage was assessed using dye penetration test and stereomicroscope. The compressive and flexural strengths of these materials were measured using Instron Universal testing machine. The amount of fluoride released from the modified ionomers at pH 5 and pH 7 was estimated using Orion fluoride specific electrode. The obtained data was then subjected to statistical analysis.

**Results:** Data was analysed using paired t-test for intergroup comparisons and unpaired t-test for intragroup comparisons. The overall microleakage ( $0.5 \pm 0.48$ ) of zirconomer was significantly less ( $p=0.000$ ) compared to ketac silver ( $1.9 \pm 0.83$ ). Zirconomer demonstrated a significant higher compressive strength ( $330.25 \pm 60.14$ ), flexural strength ( $33.058 \pm 2.36$ ) than ketac silver ( $p=0.000$ ). Zirconomer demonstrated high fluoride release from day 1 to day 7 at both pH 5 and pH 7.

**Conclusion:** Zirconomer demonstrated better physical and biological properties compared with ketac silver.

### Introduction

A restoration which is durable, aesthetic, biocompatible and functional is considered to be the effective replacement of the natural tooth structure.<sup>[1]</sup> The conventional glass ionomer cements (GIC) even though chemically bond to enamel and dentine with several beneficial properties, their sensitivity to moisture, low mechanical strength and wear resistance made them less durable as a restorative material.<sup>[2]</sup>

Exponential increase of GICs clinical application resulted from improvements in formulation, simplification of bonding techniques, increased aesthetic demands and decline in the amalgam popularity.<sup>[3]</sup> Numerous modified GICs developed were metal reinforced GICs, resin modified glass ionomer cement (RMGICs), poly acid modified composites or compomers, nano filled resin modified photo-polymerisable GICs each having respective advantages and disadvantages.<sup>[4]</sup>

Continuous quest by manufacturers and researchers to overcome the drawbacks and to improve the formulation led to a new generation of GIC named Zirconomer (SHOFU, Japan). By incorporating zirconia particles, material claims to exhibit high bond and compressive strengths, fluoride

release with less microleakage and improved aesthetics, as well as less occlusal wear and fast setting reaction.<sup>[5]</sup>

Microleakage is the property on which the integrity of restoration interface and the longevity of restoration can be estimated.<sup>[6]</sup> Furthermore, the triumph of restorative material depends upon the compressive strength and flexural strength since these are the forces which resist the masticatory forces and other parafunctional forces.<sup>[7]</sup> The amount of fluoride released by the restorative materials is responsible for the arresting of demineralisation which produce a cariostatic and anti-bacterial action.<sup>[8]</sup>

Little much of published research is available comparing the mechanical and biological properties of zirconomer to other modified GICs. Hence, the present invitro study was aimed at testing and comparing the properties like micro leakage, compressive strength, flexural strength and fluoride release of the zirconomer (zirconium reinforced glass ionomer) with ketac silver (metal reinforced glass ionomer).

### Methods

**Materials used in the study:** Two commercially available GICs, zirconomer and ketac silver grouped and investigated in the present study were listed in the Table 1. Both the

materials were manipulated according to manufacturer's instructions for all the specimens.

#### Specimen preparation and testing for microleakage:

With a straight fissure bur (ISO size no.109/013) and inverted cone bur (ISO size no.010/013), a standard class V cavity (size 2×3×3mm) with coronal margin in the enamel and the cervical margin in the cementum was made on the buccal surface of randomised two groups of ten caries free premolar teeth each (n=10). Teeth were restored with respective ionomer cements, finished, polished and then thermocycled for 300 cycles between the temperatures of 5±2°C and 55±5°C with an immersion time of 15sec and transfer time of 5 sec. Two coats of nail varnish were then applied on all the tooth surfaces, except 1 mm around the restoration and root apices were sealed with yellow sticky wax. After drying, teeth were immersed in 2% methylene blue dye (Spectrum Reagents and Chemicals Pvt. Ltd.,) solution for 24 hours. The nail varnish was removed with scalpel and the teeth were sectioned longitudinally in a bucco-lingual direction using a diamond disk and then examined under stereo microscope (VWR Vista Vision) at 10x magnification to assess dye penetration at tooth restoration interface. Degree of dye penetration is assessed based on the criteria given by Khera and Chan (1978).<sup>[9]</sup> Data obtained was then subjected to statistical analyses.

#### Specimen preparation and testing for compressive strength:

Ten specimens (n=10) measuring 6mm height and 4mm diameter of each cement type were prepared in silicone-lubricated stainless-steel split moulds. After setting, removed specimens were sequentially fine-sanded with 320-, 400-, and 600- grit silicon carbide paper to ensure no visible surface defects. Specimens were later cleansed and stored in distilled water at 37±2°C until testing. Specimens were mounted vertically between the platens of a universal testing machine (Instron Corp, Canton, Mass.) which has a crosshead speed of 0.5 cm/min. The maximum load applied in long axis to fracture the specimens was recorded and compressive strength (MPa) is calculated using the formula.  $C=4P/\pi D^2$  Where P is the maximum applied load (N), D is the measured diameter of the sample (mm). The obtained data was subjected to statistical analyses.

#### Specimen preparation and testing for flexural strength:

Ten bar shaped specimens(n=10) of 25×2×2mm size were prepared using preformed stainless-steel split moulds. Flexural strength testing was carried out using universal testing machine (Instron Corp, Canton, Mass.). The samples were subjected to three-point bending test on the machine with a 25mm distance between the supports at a crosshead speed of 0.5 cm/min. The flexural strength, F

(MPa) is calculated using the formula.  $FS (MPa) = 3Fm/2bd^2$  Where Fm is the maximum load before rupture, l is the distance between the two supports, b is the breadth and d the depth of the specimens. The obtained data were subjected to statistical analyses.

#### Specimen preparation and testing for fluoride release:

Ten specimens of each material were prepared in cylindrical plastic tubes, each of 5mm internal diameter and 2mm thickness. 5 specimens of each group were dipped in 15ml of de ionised distilled water at pH 7 and pH 5 (adjusted by adding 1N HCL and 1N KOH using a standardised digital pH meter) in plastic containers before fluoride release and stored in an incubator at 37°C for 24 hours. After 24hrs, the specimens were removed, washed with respective elutant, dried with absorbant paper and transferred to new containers. This procedure was repeated consecutively for 7 days. Before measurement, 2.7 ml of each sample solution was pipetted into a clean plastic test tube, and 0.3 ml of TISAB III (Total ionic strength adjustment buffer) concentrate with CDTA (1,2-cyclohexylenedinitrotetraacetic acid) was added to each solution. Fluoride release was measured in ppm using a fluoride specific electrode (Orion 9609BN, Orion Research Inc., USA) with a combination of ion analyser (Orion EA 940, Orion Research Inc., USA).

Data obtained was computerised and analysed using Statistical Package for Social Sciences (SPSS) version 23.0 (IBM SPSS, Chicago). Unpaired and paired t tests were used for inter and intragroup comparisons of all experiments. A p ≤ 0.05 was set for statistical significance and a value of ≤ 0.001 represents a highly significant relation.

#### Results:

For the results of microleakage, mean values of both the groups were analysed and it was observed that zirconomer demonstrated a highly significant (p=0.00) less overall microleakage (0.5±0.48) as well as at both enamel (0.8±1.04) and cemental margins (0.9±0.94) when compared to ketac silver (1.9±0.83),(1.0±0.44), (1.9±0.81) respectively. Microleakage at enamel margin is significantly lower (p=0.00) than at cemental margin for both the modified ionomer cements. (Table 2)

Upon intergroup comparison, compressive strength (330.25 ± 60.14) and flexural strength (33.05 ± 2.06) of zirconomer was found to be significantly higher (p=0.00) than ketac silver (171.66 ± 61.79 for compressive strength and 24.06 ± 5.30 for flexural strength). (Table 3)

With respect to fluoride release, at pH5 and pH7, zirconomer had a significantly higher fluoride release than ketac silver (p=0.00). Both the groups showed a maximum fluoride release on day 1 and least on day 7 and fluoride release pattern showed a gradual decrease from day 1 to day 7 at both pH5 and pH7. (Figure 1)

**Table 1:** Materials used in the study

Group	Material	Manufacturer	Type	Formulation
I (ZN)	Zirconomer (ZN)	Shofu INC, Kyoto, Japan	Zirconia reinforced GIC	Powder-Liquid
II(KS)	Ketac Silver (KS)	3M ESPE, St. Paul, MN, USA	Silver reinforced GIC	Powder-Liquid

**Table 2:** Inter and intragroup comparison of microleakage of both the test groups

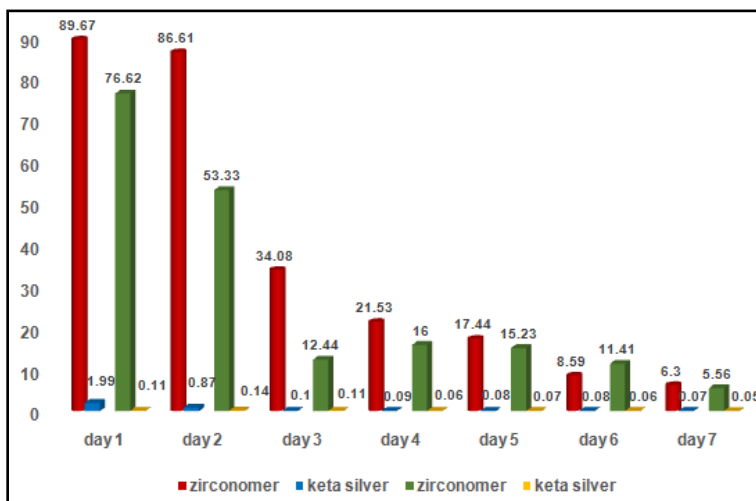
Group	Sample (n)	Overall Microleakage (Mean±SD)	p value	At enamel margin (Mean±SD)	p value	At cemental margin (Mean±SD)	p value	Enamel margin Vs Cemental margin
I (ZN)	10	0.5±0.48	0.00* HS	0.8±1.04	0.00* HS	0.9±0.94	0.00* HS	p = 0.00*
II(KS)	10	1.9±0.83		1.0±0.44		1.9±0.81		p = 0.00*

\*HS = High statistical significance, SD = standard deviation

**Table 3:** Intergroup comparison of compressive and flexural strengths

Group	Sample (n)	Compressive strength (Mean±SD)	p value	Flexural strength (Mean±SD)	p value
I (ZN)	10	330.25 ± 60.14	0.00* HS	33.05 ± 2.06	0.00* HS
II(KS)	10	171.66 ± 61.79		24.06 ± 5.30	

\*HS = High statistical significance, SD = standard deviation

**Figure 1:** Pattern of fluoride release (ppm) of both ionomers at pH5 and pH7 (day 1 to day 7)

### Discussion:

Remarkable developments occurred in the basic composition of glass ionomer technology by reinforcing them with disperse glass phases, fibres, metals, sintered metal and glass particles and resins to improve the physical and mechanical properties.<sup>[10]</sup> This incessant thirst of the material properties is to develop a material for clinical purposes which would adhere optimally to the tooth structure and which can withstand the masticatory forces.<sup>[11]</sup>

Microleakage testing verdicts the clinical performance of a restorative material in turn influencing the longevity of the

restorations.<sup>[6]</sup> Furthermore, degree of chemical adhesion of GICs to both enamel and dentin varies.<sup>[12]</sup> Therefore, microleakage was assessed at both enamel and cemental margins.

In the present study, class V cavities were selected because of its configuration or "C" factor. The "C" factor of class V restoration is 5 which corresponds to the ratio between the number of bonded to unbonded surfaces which is responsible for the internal bond disruption as well as marginal gaps around the restorations and cavity walls.<sup>[13]</sup>

The restored teeth were thermocycled to eliminate the dimensional changes occurring due to difference in

coefficient of thermal expansion between the tooth and the restorative material when subjected to varying oral temperatures.<sup>[14]</sup> 2% methylene blue dye is one of the most effective method used to test the microleakage and its low molecular weight facilitates diffusion easily, easily detectable and it is not absorbed by dentinal matrix hydroxyapatite crystals as suggested by Pasricha.<sup>[15]</sup>

The present study demonstrated a microleakage with both the tested groups but a significant less value was registered with zirconomer. This might be due to the chemical structure of zirconomer which comprises zirconia particles as fillers which could cause interference in the chelating reaction between the carboxylic group (-COOH) of poly-acrylic acid and the calcium ions (Ca<sup>2+</sup>) of tooth apatite.<sup>[12]</sup> Greater microleakage with Ketac silver could be attributed to the disruption of polyacrylate matrix in the cement as well as poor handling properties.<sup>[10]</sup> Result agrees with the findings obtained from study conducted by Rawan Albeshti et al.<sup>[10]</sup>

Though compressive strength evaluates the strength of restorative materials frequently and considered to be an indicator of success in restorative dentistry, the utmost apposite measure of the strength of GIC's is obtained with only a flexural test since GIC would only fracture at the atomic level by tensile or shear failure.<sup>[16]</sup> Significant difference in the compressive and flexural strength was observed for the two tested glass ionomer cements in present study. However, the higher strengths with zirconomer might be credited to zirconium oxide filler particles. The glass component in zirconomer is subjected to finely controlled micronization to achieve optimum homogenous particle size and further reinforces the durability and the strength of the material to withstand occlusal load.<sup>[17]</sup> Results with zirconomer in present study were in good agreement with the studies conducted by Chitharanjan Shetty et al.<sup>[11]</sup> and Vemina et al.<sup>[3]</sup>

Fluoride release is tested in the present study as it reflects the anticariogenic property and antibacterial activity of the materials.<sup>[8]</sup> Among various methods employed, the ion selective method in conjunction with total ionic strength adjustment buffer (TISAB) was opted in this study as fluoride ions estimation will be as accurate as possible.<sup>[18]</sup> Deionised distilled water is used as an eluting solution instead of artificial saliva because it is suggested that components from saliva form a pellicle on the surface of the restorative material that impede the ion release. Furthermore, the deionised distilled water devoid of chemicals makes fluoride measurement easier and accurate.<sup>[19]</sup>

Maintenance of samples was done that 37<sup>o</sup>c prior to fluoride release to simulate the oral temperature and respecting the fact that temperature and fluoride release are directly proportional to each other.<sup>[20]</sup> Literature from earlier studies demonstrated a constant fluoride release

from GICs after 7th day hence fluoride release pattern was observed for 7 days in the current study.<sup>[20]</sup> Results of present study showed the continuous release of fluoride was from both the tested glass ionomer cements throughout the study period. Also, fluoride release was high on the first 24 hours for both the cements. Study by De Moor RJ et al.,<sup>[21]</sup> have demonstrated similar fluoride release pattern. An initial high release over the first 24 hours was likely due to surface wash off effect/burst effect. During the acid dissolution of powder particle surfaces, a large amount of fluoride diffuses quickly from the reaction matrix exposed on the material surface and is slowly replaced by fluoride diffusing from the matrix below the surface.<sup>[8]</sup> Later the fluoride release reduced gradually and was almost constant on the 6th and 7th days. After the initial burst, fluoride release decreased and maintained constantly as the glass dissolves in the acidified water of the hydrogel matrix.<sup>[8]</sup>

Fluoride release was experimented at two different pH's (pH 5 and pH 7) as the previous studies suggest that the amount of fluoride release at low pH was considerably greater than at higher pH.<sup>[20]</sup> In the present study also, zirconomer and ketac silver showed a high fluoride release at pH5 when compared to pH7. The increase in the amount of fluoride in acidic media could be explained by the fact that a decrease in pH increases the dissolution of the material leading to a higher fluoride level in the acidic immersion.<sup>[20]</sup> Also, in the present study, zirconomer showed a maximum fluoride release at both pH5 and pH7 when compared to ketac silver and these findings were statistically significant. The higher fluoride released by zirconomer may due to its inherent composition that may contribute to a major acid base mechanism and thus more fluoride release during setting reaction.<sup>[8]</sup>

The present study findings conclude that zirconomer demonstrated a significant higher compressive strength, flexural strength and high fluoride release whereas less micro leakage compared with Ketac silver. However, results obtained may not be correlated with the clinical conditions as the oral environment is dynamic and necessitates further long-term in vivo studies with large sample size.

## References

1. Hegde MN, Hegde P, Bhandary S, Deepika K. An evaluation of compressive strength of newer nanocomposite: An in vitro study. *J Conserv Dent* 2011; 14:36-39.
2. Cho SY, Cheng AC. A review of glass ionomer restorations in the primary dentition. *J Can Dent Assoc.* 1999; 65:491-495.
3. Chalissery VP, Marwah N, Almuhaiza M, AlZailai AM, Chalisserry EP, Bhandi SH, et al. Study of the mechanical properties of the novel zirconia reinforced glass ionomer cement. *J Contemp Dent Pract* 2016; 17:394-8.
4. Nicholson JW. Chemistry of glass-ionomer cements: a review. *Biomaterials* 1998; 19:484-494.
5. Zirconomer [Package Insert]. Japan: Shofu Inc.; 2012
6. Gupta KV, Verma P, Trivedi A. Evaluation of microleakage of various restorative materials: An in vitro study. *J Life Sci* 2011; 3:29-33

7. Xie D, Brantley WA, Culbertson BM, Wang G. Mechanical properties and microstructures of glass-ionomer cements. *J Dent Mater* 2000; 16:129–38
8. Tiwari S, Kenchappa M, Bhayya D, Gupta S, Saxena S, Satyarth S, Singh A, Gupta M. Antibacterial activity and fluoride release of glass-ionomer cement, compomer and zirconia reinforced glass-ionomer cement. *J Clin Diagn Res* 2016; 10: 90-93.
9. Khera S, Chan K. Microleakage and enamel finish. *J Prosthet Dent*. 1978; 39:414–9.
10. Albeshti R, Shahid S. Evaluation of microleakage of Zirconomer: a zirconia reinforced glass ionomer cement. *Acta Stomatol Croat* 2018; 52(2):97–101.
11. Shetty C, Sadananda V, Hegde MN, Lagiseti AK, Shetty A, Mathew T, et al. Comparative Evaluation of Compressive Strength of Ketac Molar, Zirconomer, and Zirconomer Improved. *Sch J Dent Sci*. 2017;4(6):259-261.
12. Salman KM, Naik SB, Kumar NK, Merwade S, Brigit B, Jalan R. Comparative evaluation of microleakage in Class V cavities restored with giomer, resin-modified glass ionomer, zirconomer and nano-ionomer: An in vitro study. *J Int Clin Dent Res Organ* 2019; 11:20-5
13. Walia R, Jasuja P, Verma KG, Juneja S, Mathur A, Ahuja L. A comparative evaluation of microleakage and compressive strength of Ketac Molar, Giomer, Zirconomer, and Ceram-x: An in vitro study. *J Indian Soc Pedod Prev Dent* 2016; 34:280-4.
14. Doerr CL, Hilton TJ, Hermes CB. Effect of thermocycling on the microleakage of conventional and resin-modified glass ionomers. *Am J Dent*. 1996; 9(1):19-21.
15. Pasricha S. Comparative evaluation of microleakage of tooth coloured restorative materials after exposure to 33% hydrogen peroxide –An in vitro study. *Int J Contemp Dent* 2011; 2:28-37
16. Bayindir F, Yilmaz CB. Comparison of diametral tensile, flexural and compressive strengths of five core build-up materials. *J Oral Rehabil* 2007; 17(1): 18-23.
17. Safy, R., Abd Elmohsen H. Assessment of the fracture resistance of novel zirconia reinforced glass ionomer in comparison to nano hybrid resin composite restorations. *Egyptian Dental Journal* 2019; 65(4): 3735-3744
18. Glasspoole EA, Erickson RL, Davidson CL. (2001). A fluoride-releasing composite for dental applications. *Dent Mater* 17:127-133
19. Gandolfi MG, Chersoni S, Acquaviva GL, Piana G, Prati C, Mongiorgi R. Fluoride release and absorption at different pH from glass-ionomer cements. *Dent Mater* 2006;22(5): 441–9
20. Yan Z, Sidhu SK, Mahmoud GA, Carrick TE, McCabe JF. Effects of temperature on the fluoride release and recharging ability of glass ionomers. *Oper Dent* 2007; 32: 138– 143. De Moor RJ, Verbeeck RM, De Maeyer EA. Fluoride release profiles of restorative GIC formulations. *Dent Mater*. 1996; 12:88-95.