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Evaluation of the microleakage and compressive strength of conventional GIC and Nanoparticles Incorporated GIC.

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# Abstract

Glass Ionomers cements (GIC) are being used widely as restorative materials. The incorporation of nanoparticles into glass powder resulted in higher mechanical values by occupying the empty spaces between the Glass ionomers particles. Aim of the study is to evaluate the microleakage and compressive strength of conventional GIC and nanoparticles incorporated GIC. Materials and methods include 40 freshly extracted human deciduous teeth. Group I Conventional GIC, Group II Zirconomer cement, Group III Conventional GIC with 8% by weight alumina nanoparticles, Group IV Conventional GIC with 8% by weight Nanohydroxyapatite. Order of compressive strength is Group II > Group IV  $\geq$  Group III > Group I. Order of microleakage is Group II  $\geq$  Group IV  $\geq$  Group I  $\geq$  Group III. Order of microleakage is Group II > Group III  $\geq$  Group I  $\geq$  Group IV. Order of microleakage is Group II  $\geq$  Group III  $\geq$  Group IV  $\geq$ Group I. Restoration in primary teeth differs from the permanent teeth. The ideal requisites for restorative

material are good color stability, coefficient of thermal expansion similar to that of tooth structure, excellent marginal seal, and the ability to adhere chemically to tooth. It is important to reduce the marginal leakage which is the precursor of tooth discoloration, staining of restoration, secondary caries, marginal deterioration, postoperative sensitivity and pulpal pathology. The sealing ability was highest for Conventional GIC and poor for Zirconomer. None of the GIC materials evaluated completely prevented microleakage. The compressive strength was highest for Zirconomer and least for the Conventional GIC. Nanoparticles improve the mechanical properties.

**Keywords:** Glass Ionomers Cement, Nanoparticles, Zirconomer, Compressive Strength and Microleakage.

## Introduction

Nanotechnology is the production of materials in the range of 0.1 to 100 nanometers.<sup>1</sup> Nanotechnology has revolutionized restorative dentistry by providing nanofillers. These filler particles are very minute, higher

proportions can be achieved, and result indistinctive physical, mechanical, and optical properties.<sup>2</sup>

Glass Ionomer cements(GIC) are being used widely as restorative materials because of its excellent properties such as chemical bonding, biocompatibility and fluoride release. The shortcomings are poor aesthetic, prolonged setting reaction, compromised mechanical properties and weaker bond strength; it is highly sensitive to moisture and has fast dehydration. This led to limitation of its applications in the non-stress bearing regions. The compressive strength of a material is an important factor to be considered in relation to masticatory forces. The addition of cellulose fibers, hydroxyapatite and fluoroapatite and nanoparticles have been introduced to overcome these shortcomings.<sup>3</sup>

Hydroxyapatite nanoparticles have similar composition with teeth and bone, making them biocompatible. It is a natural calcium phosphate ceramic, predominant in 97% enamel as the building blocks.<sup>4</sup> The incorporation of nanoparticles into glass powder of GIC resulted in higher mechanical values by occupying the empty spaces between the Glass ionomer particles and reinforce GIC. <sup>5</sup> Microleakage is the common cause of failure of restoration. Materials show varying degrees of marginal leakage because of dimensional changes and a lack of adaptability to cavity walls.<sup>6</sup>

The purpose of the present study was to evaluate compressive strength and microleakage of different types of nanoparticles incorporated into conventional GIC.

## Aim of The Study

To evaluate the microleakage and compressive strength of conventional GIC and Nanoparticles incorporated GIC.

OBJECTIVES: To improve mechanical properties of conventional glass ionomers cement. To compare

sealing ability of conventional and nano particle incorporated Glass Ionomers cement.

#### **Materials and Methods**

40 Freshly extracted human deciduous teeth were collected. Ethical consideration is not applied as it is an invite study.

#### Methodology

#### 1) Microleakage

SAMPLE PREPARATION: Forty extracted teeth were collected and stored in 0.2% sodium azide. Surface debridement was performed with ultrasonic scaling. After that standardized class V cavities 3mm long, 2mm width and 1.5mm depth were prepared. Forty samples were randomly divided into four equal groups, Group I – IV, consisting of 10 samples of each group,

Group I Conventional GIC (Type IX GIC),

Group II Zirconomer cement,

Group III Conventional GIC with 8% by weight alumina nanoparticles,

Group IV Conventional GIC with 8% by weight nano Hydroxyapatite.

The nanoparticles were mixed with the GIC cement by spatulation, the powder and liquid were mixed according to the manufacturer's instructions, and the cavity was restored. Samples were stored in distilled water for 24 hrs. The teeth were then subjected to thermo cycling procedure at 500 thermocycles, at a temperature range 4-55°C and dried after thermocycling. The specimens were coated with 2 layers of nail varnish, a moist cotton pellet was placed over the restoration to prevent desiccation and root apices were closed with sticky wax. Samples were immersed in 2% Methylene blue dye for 24hrs at 37°C. After that brushed thoroughly under tap water for 30 sec and the sticky wax was removed with Wax knife and nail varnish was removed with BP blade. The teeth were sectioned longitudinally in a bucco-lingual

direction through the centre of the restorations using water cooled low speed diamond disc. The micro gaps were assessed under scanning electron microscope at 50X magnification and the readings were recorded in micrometers. The data collected was subjected to statistical analysis.

#### 2) Compressive Strength:

Sample Preparation: The sixty samples were randomly divided into four groups. Group I Fuji IX Extra GC, Group II Zirconomer (Shofu), Group III Conventional GIC cement with 8% by weight alumina nanoparticles, Group IV Conventional GIC cement with 8% by weight Nanohydroxyapatite. The nanoparticles were mixed with the GIC by spatulation on a ceramic tile to obtain uniform distribution. The freshly mixed cement was placed in a cylindrical metal mold (4X6mm) covered on both sides with slides, and left in an incubator at 37° C for 1hr to allow setting. The specimens were removed from the molds and placed in distilled water at 37 °C for 24 hours. The compressive strength was then tested using universal testing machine at a crosshead speed of 1 mm/min and the readings were recorded in megapascals. The data collected was subjected to statistical analysis.

## Results

The present study is aimed at evaluating the microleakage and compressive strength of conventional GIC, Zirconomer, alumina incorporated GIC and hydroxyapatite incorporated GICs. The study consisted of two parts in which estimation of microleakage and compressive strength of respective cements was done. Microleakage estimated by measuring microgap between restoration and teeth, which comprised of 40 sample size and compressive strength includes 60 sample size which were divided into 4 equal groups of conventional GIC, GIC Zirconomer, Alumina infused and Nanohydroxyapatite infused GIC (Table 1-5).

Order of compressive strength is Group II > Group IV  $\geq$ Group III > Group I. Order of microleakage (Coronal, Middle) Group II  $\geq$  Group IV  $\geq$  Group I  $\geq$  Group III. Order of microleakage (Apical) Group II > Group III  $\geq$ Group I  $\geq$  Group IV. Order of microleakage Group II  $\geq$ Group III  $\geq$  Group IV  $\geq$  Group I.

## Discussion

The quest to search for an ideal restorative material has been a challenge in restorative dentistry. Glass ionomers are a class of biomaterials in widespread use in modern dentistry.<sup>7</sup> Restoration in primary teeth differs from the permanent teeth because of limited lifespan of teeth, different morphology, and their susceptibility to caries, lower biting force in children. The ideal requisites for restorative material are that it should have good color stability, coefficient of thermal expansion similar to that of tooth structure, excellent marginal seal, and the ability to adhere chemically to tooth.<sup>8</sup> It is important to reduce the marginal leakage which is the precursor of tooth discoloration, staining of restoration, secondary caries, marginal deterioration, postoperative sensitivity, and pulpal pathology.<sup>8</sup>

Nanotechnology is the art and science of material engineering in a scale of less than 100nm.<sup>9</sup> Nanotechnology is of great interest for the development of dental materials. Nanotechnology has been used in composites, ceramics to produce low shrinkage dental materials with high wear resistance, biocompatibility.<sup>10</sup> Recently, application consisting of a posterior restorative GIC combined with a novel nanofilled coating material, the compounded nanofillers protect against the abrasive wear and the coating acts as a glaze, enhancing its esthetic properties.<sup>11,12</sup>

GIC (GIC) seems to meet most of these requirements along with certain drawbacks for use in primary molar due to its low physical properties and poor long-term

performance. To overcome this, a high strength restorative material which has been reinforced with ceramic and zirconia filler known as Zirconomer (white amalgam) has been introduced in dentistry.<sup>8</sup> Fuji IX was selected as the GIC material because it is one of the strongest commercially available conventional restorative GIC, and is recommended for pediatric purpose. GIC glass is mainly composed of SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, CaF<sub>2</sub>, Na<sub>3</sub>AlF<sub>6</sub>, and AlPO<sub>4</sub>. However, Strontium can be substituted for Calcium with little change in the GIC structure in order to increase the opacity of GIC to Xrays, because Strontium and Calcium have similar ionic radii. Fuji IX contains Strontium instead of Calcium.<sup>13</sup> Fuji IX GIC is considered as a gold standard, it could be efficiently used for the assessment of CS of the modified cement.7

Efforts have been made to improve the physical and mechanical properties by addition of a variety of fillers like amalgam alloys and stainless steel powders, carbon and aluminosilicate fibers, and hydroxyapatite powders of various compositions and nanoparticles such as titanium dioxide (TiO2) nanotubes, Nanohydroxyapatite, and nanofluoroapatite.<sup>14</sup>

The present study involves two nanoparticles, namely Al<sub>2</sub>O<sub>3</sub>, ZrO<sub>2</sub>. They were selected because they exhibit relatively low toxicity when present in other dental restorative formulations.<sup>14</sup> Incorporation of alumina fibres into the glass powder of GIC helps in improving the flexural strength. This technology is called Polymeric Rigid Inorganic Matrix Material. It is a light-cured GIC. It involves the incorporation of a continuous network / scaffold of alumina and SiO<sub>2</sub> ceramic fibres into the powder. This increases the depth of cure, reduces the polymerization shrinkage, improves wear resistance and increases the flexural strength.<sup>15</sup>

Hydroxyapatite, the main mineral component of the tooth structure and bone, is a bioceramic containing calcium and phosphorus. The Hydroxyapatite particles were added to Glass Ionomer powder due to their biocompatibility and similar composition to apatite in human dental and skeletal systems. The present study was conducted to compare evaluate the microleakage and CS of conventional GIC, Zirconomer, Conventional GIC incorporated with Nanohydroxyapatite, and alumina nanoparticles.<sup>16</sup>

Microleakage is a phenomenon resulting from diffusion of bacteria, fluids, food debris, other ions and molecules along the tooth-restoration interfaces. It causes recurrent caries, discoloration, restorative failure and pulpal pathology. Methods for investigation of microleakage include dye penetration, fluid filtration, electrical conductivity, neutron activation method, radioisotope method and so on. The most commonly used method however, is by using colored dye agents or chemical traces which are able to penetrate easily into the micro gaps between the tooth-restoration interfaces.<sup>17</sup>

In the present study, human extracted primary teeth were selected. Reason for the selection of primary teeth is that restoration differs from permanent teeth due to the limited life span of teeth, different morphology of primary molars, lower biting forces in children, and their susceptibility to caries. Also, the permanent teeth contain more inorganic content as compared with the primary teeth, leading to the strong bond, which in turn might have led to the decrease in microleakage. According to Hirayama<sup>18</sup> who revealed that peritubular dentin of permanent teeth, with thicker peritubular dentin, there is relatively less intertubular dentin. And since intertubular dentin is the major area where bond occurs,

primary teeth provide lesser bonding as compared with the permanent teeth leading to increase in microleakage.<sup>8</sup> One cause of microleakage is the difference between coefficients of thermal expansion of dentin and the restorative material. Thermal stress in the oral environment can cause periods of expansion and contraction in the restorative material and dentin. When coefficients of thermal expansion are different the stresses lead to gap formation. These coefficients are similar for GIC and dentin. Thermocycling is the only method for simulation of thermal stresses in the oral environment.<sup>19</sup> Here 500 thermocycles were used to simulate long-term clinical use of the restoration.

Cervical lesions due to caries, erosion, or abrasion present a special challenge to any restorative dentist because in such cavities, the restorative material is usually required to adhere to different types of tooth tissues. The occlusal stress generated in the cervical region during normal function and parafunction may increase microleakage or deteriorate the margins of Class V restorations.<sup>20</sup>

In the present study, Class V cavities were selected because of its configuration or "C" factor. For the dye penetration test, the root apices were sealed with yellow sticky wax. This procedure was carried out in accordance with the study conducted by Owen B.M and Johnson W.W. Two coats of nail varnish were applied leaving 1mm wide margin around the restoration to avoid any dye penetration from invisible cracks, areas devoid of enamel or cementum etc as supported by Alavi AA and Kianimanesh N. 2% methylene blue dye was selected as a measure of microleakage because of its low cost, ease of manipulation, convenience, and also the low molecular weight of the dye is smaller than bacteria that could detect leakage where bacteria could not penetrate. Results showed that microleakage was least for conventional GIC followed by Nano Hydroxyapatite incorporated GIC, then Alumina incorporated GIC and highest for Zirconomer. The compressive strength(CS) is an important property in restorative materials, particularly in the process of mastication. This test is more suitable to compare brittle materials, which show relatively low result when subject to tension. To test CS of a material, two axial sets of force are applied to a sample in an opposite direction, in order to approximate the molecular structure of the material. According to ISO 991711, cylindrical shaped specimens are tested, so cylindrical molds were used in this study. Distilled water is used as storage media.

In the present study, results showed that the CS was highest Zirconomer. followed Nano for bv Hydroxyapatite incorporated GIC, and then Alumina incorporated GIC and least for Conventional GIC. The superior mechanical property is ascribed to the inclusion of zirconia fillers. The glass component in Zirconomer is subjected to finely controlled micronization to achieve optimum homogenous particle size and further leading to enhanced mechanical property such as higher strength. Homogeneity of the glass particles further reinforces the durability of the material and the strength to withstand occlusal load. Zirconomer Improved which exhibited better CS than Ketac Molar as per manufacturer's claims has the durability of amalgam. The material reinforced with nano-zirconia fillers is responsible for imparting enhanced mechanical properties especially making it suitable for posterior load bearing areas as per various studies.

## Conclusion

The sealing ability was highest for Conventional GIC and poor for Zirconomer among four groups. None of the GIC materials evaluated completely prevented

microleakage, which was greatest at the gingival margins. The compressive strength was highest for Zirconomer and least for the Conventional GIC. Nanoparticles improves the mechanical properties of the Conventional GICs.

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## Legends Tables

Group	N	Minimum	Maximum	Mean	Std. Deviation	F value	P value
Group I	15	100.4	114.4	106.027	4.8773	336.016	<0.001**
Group II	15	145.4	163.3	155.507	5.0024		
Group III	15	110.2	124.2	116.560	4.0265		
Group IV	15	112.2	128.6	120.573	4.1444		

Table 1: Intergroup comparison of mean compressive strength (One way anova)

\*\*-highly significant (p<0.001)

Inference: There is statistically significant difference present in the mean compressive strength in various groups. One way anova signifies overall comparison, to know individual comparisons post hoc tukey test should be done

Group	Ν	Minimum	Maximum	Mean	Std. Deviation	F value	P value
Group I	10	1.40	32.11	11.1825	10.16220	2.172	0.108 NS
Group II	10	2.43	40.46	20.1196	13.09449		
Group III	10	4.96	18.96	10.2433	4.29928		
Group IV	10	3.06	29.16	12.2618	9.12017		

NS- Not significant (p>0.05)

Inference: There is no statistically significant difference present in the mean microleakage values at coronal

Table 3:	Intergroup	comparison	of microleakage	(Middle)
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Group	N	Minimum	Maximum	Mean	Std. Deviation	F value	P value
Group I	10	1.76	53.37	13.2029	15.42540	0.282	0.838 NS
Group II	10	7.38	42.63	17.6480	13.03958		
Group III	10	4.67	26.89	13.9143	6.78115	]	
Group IV	10	2.55	34.34	15.5755	10.04198	]	

# NS- Not significant (p>0.05)

Inference: There is no statistically significant difference present in the mean microleakage values at middle

Group	N	Minimum	Maximum	Mean	Std. Deviation	F value	P value
Group I	10	4.20	61.63	23.09	16.73	4.457	0.009*
Group II	10	26.49	75.88	44.81	15.04		
Group III	10	11.44	67.38	35.20	18.83		
Group IV	10	5.63	50.01	21.77	14.27		

Table 4: Intergroup comparison of mean microleakage (Apical)

\*-Significant (p<0.05)

Inference: There is statistically significant difference present in the mean microleakage in various groups.

Table 5: Intergroup comparison of microleakage

Group	N	Minimum	Maximum	Mean	Std.	F value	P value
					Deviation		
Group I	15	0.00	49.04	10.5508	13.14104	1.15	0.337 NS
Group II	15	0.00	44.83	18.3500	15.90605		
Group III	15	0.00	32.50	13.1911	11.31897		
Group IV	15	0.00	36.61	11.0248	10.55814	]	

NS- Not significant (p>0.05)