RESEARCH ARTICLE

# Comparative Evaluation of Water Sorption, Solubility, and Microhardness of Zirconia-reinforced Glass Ionomer, Resin-modified Glass Ionomer, and Type IX Glass Ionomer Restorative Materials: An *In Vitro* Study

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## Abstract

The challenge that practicing dentists face every day is to decide which dental material is best suited for each dental treatment. New glass-ionomer cement (GIC) formulations have been introduced in order to overcome the drawbacks of conventional ones thereby catering to the needs of the pediatric population.

**Aim and objective:** The study aimed to evaluate and compare water sorption, solubility, and microhardness of zirconia-reinforced glass ionomer, resin-modified glass ionomer, type IX glass ionomer cements.

**Materials and methods:** 90 specimens were prepared in total of which 45 cylindrical specimens with dimensions of (6 × 4) mm and 45 disks with (10 × 2) mm were prepared from Zirconomer, RMGIC, and Type IX GIC restorative materials, each material having 30 specimens (15 disks, 15 cylinders). After taking the initial weight (W1), the 45 cylinders (15 of each material) were immersed in artificial saliva at 37°C for 28 days after which the weights W2 and W3 were weighed. The other 45 disks (15 of each material) were subjected to microhardness test under microhardness tester. Results were subjected to ANOVA and Tuckey's post hoc test.

**Results:** Zirconomer showed the maximum resistance to water sorption and solubility followed by RMGIC and type IX GIC with a significant $p$ value of < 0.001 difference. For microhardness, Zirconomer showed the highest value with a significant $p$ value of < 0.001 difference. But, there was no significant difference between RMGIC and Type IX GIC depicting almost equal strength.

**Conclusion:** Water sorption, solubility, and microhardness of Zirconomer were significantly high in comparison to the other groups and it can be used as a posterior restorative material for stress-bearing areas.

**Clinical significance:** As pediatric dentistry demands restorations to be completed frequently in less than ideal conditions, Zirconomer has shown to be better than RMGIC and conventional GIC probably because of the improvisation in the GIC properties.

**Keywords:** Artificial saliva, Microhardness, Type IX glass ionomer cement, Vitremer, Water sorption, Water solubility, Zirconomer.

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## Introduction

One of the most important factors for the disease burden in both adults and children worldwide is dental caries. So, in order to treat this disease properly, the search for an ideal restorative material with good physical and chemical properties exists. The most widely used cements in this field are the glass ionomers. Because of the ease of their material handling properties, biocompatibility, and also for extended fluoride release, they are the most preferred materials in pediatric dentistry.

Novel materials such as Zirconomer and Zirconomer Improved (white amalgams) do overcome the drawbacks of previously used amalgam as well as the conventional GICs to a great extent as they exhibit the strength of amalgam and, at the same time, maintain the fluoride-releasing capacity of GICs.

Also, another set of glass ionomers called the resin-modified GICs are available now which are the amalgamation of the original GIC component and a small quantity of polymerizable resin. They have the advantages of conventional GICs and also the benefit of fracture and wear resistance that is imparted because of the resin component.

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The manufacturing of such type of materials will be a big boon to the practicing pediatric dentists worldwide.

Hence, the main aim of this in vitro study is to evaluate and compare the water sorption, solubility, and microhardness of

Zirconomer, RMGIC, and Type IX GIC restorative materials so as to evaluate the material of choice for stress-bearing areas.

## Materials and Methods

Three types of GIC were used in the study:

1. **Zirconia-reinforced glass ionomer cement (zirconomer-improved, SHOFU, INC, Japan)**
2. **Resin-modified glass ionomer cement (Vitremer-3M ESPE, USA)**
3. High Strength posterior restorative cement (GC Fuji IX - GC Corporation, Tokyo, Japan)

### Specimen Preparation

A total of 90 specimens of which 45 cylindrical test specimens with dimensions of 4 mm diameter × 6 mm height were prepared from a custom-made stainless steel mold (Fig. 1) to assess water sorption and solubility and 45 circular disk test specimens of dimensions 10 mm diameter and 2 mm height to assess microhardness. Of the 45 cylindrical specimens, 15 of them were prepared from conventional GIC (GC Fuji IX), which was the control group; 15 of them from RMGIC and the other remaining 15 from Zirconomer restorative materials as the experimental groups. Similarly, of the 45 disk specimens, 15 of them were prepared from conventional GIC, which served as the control group; 15 of them from RMGIC and the remaining 15 from Zirconomer restorative materials, respectively. A thin layer of petroleum jelly was coated on the lateral walls of the mold to prevent material adhesion. The powder and liquid of the conventional GIC, Zirconomer, and RMGIC were mixed according to the manufacturer's instructions and placed in the molds. The mixed cement was placed into the custom-made stainless steel mold by slightly overfilling them and was placed between two glass plates with the Mylar strips placed between steel mold and the glass plate to prevent the adhesion of GIC to glass plate. The glass plates were held firmly during setting to avoid the presence of air bubbles and to obtain a smooth surface. RMGIC specimens were cured for 40 s using visible light-cure unit. After setting, the pellets were removed from the mold, and the excess was trimmed using a Bard-Parker blade. The specimens were randomly grouped into their respective groups.

Zirconomer: 30 (15 cylinders, 15 disks), RMGIC: 30 (15 cylinders, 15 disks), Type IX GIC: 30 (15 cylinders, 15 disks) (Fig. 2).

## Preparation of Artificial Saliva

In 1 liter of distilled water, the chemicals namely 0.4 g sodium chloride, 1.21 g potassium chloride, 0.78 g sodium dihydrogen phosphate dihydrate ($NaH_2PO_4$ $2H_2O$), 0.005 g hydrated sodium sulfide (Na2S $9H_2O$), 1 g urea $CO(NH_2)_2$ were added and 10 N sodium hydroxide was added to this mixture until the pH value was measured to be as 6.75 ± 0.15. To prepare 10 N NaOH - 40 g of NaOH was dissolved in 100 mL distilled water. It was made sure that solid was added to water and does not reverse as it produces heat during mixing.[4]

## Assessment of Water Sorption, Solubility

The thermocycling test was done on the 45 specimens by subjecting them to 500 cycles in water between 5° and 55°C for 30 seconds to simulate the oral environment in a Thermocycling Unit. Immediately, they were weighed with digital weighing machine and the initial weight was termed as W1 (μg). Then, they were immersed in artificial saliva and stored at 37°C for 28 days (Fig. 3).

After removing the samples from the artificial saliva, they were washed with water, dried with an absorbent paper, dried in air for 15 seconds, and weighed again. This weight was termed as W2 (μg). After this, they were dehydrated in an oven at 37°C for 24 hours and weighed again for third time and this weight was termed W3 (μg). Diameter and thickness of each sample were measured by taking the means of two measurements at right angles to each other by using Vernier calipers.

V (volume) of each specimen was calculated by using the mean thickness and diameter as:

$$V = \pi r^2 h$$

Where r is sample radius in mm and h is sample thickness in mm.

Water sorption is calculated by **Wsp** = (w2 – w1)/v, Water solubility is calculated by **Wsol** = (w1 – w3)/v, Where V is the volume of sample in $mm^3$.

The net values for water sorption and solubility were evaluated and calculated for the results.


**Fig. 1:** Stainless steel molds with the prepared specimens


**Fig. 2:** Total prepared 90 samples of zirconomer, Vitremer, and Type IX GIC

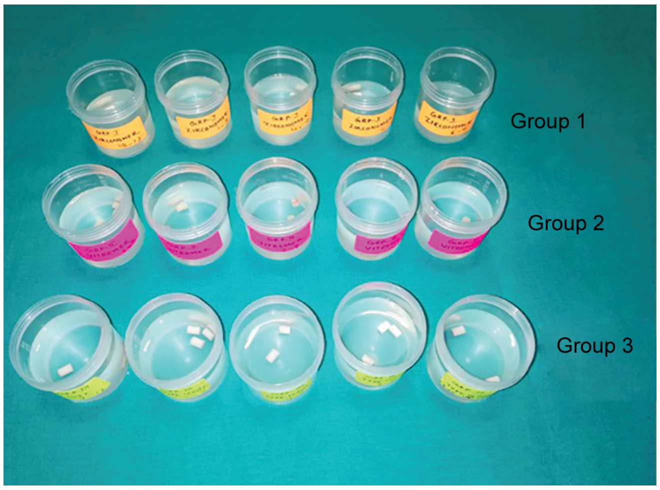


**Fig. 3:** Samples stored in artificial saliva for water sorption and solubility

**Fig. 4:** Sample tested under microhardness tester

## Assessment of Microhardness

In this study, Vicker's Microhardness Tester Reichert Austria Make with Sr No. 363798 was used. In this test, the specimen was held under microhardness tester and a load of 50 g was applied for 30 seconds (Fig. 4). The notches that were formed were then measured after releasing the load. Two notches were made on each sample with the Vickers microhardness indentor and the values were calculated accordingly depending on the depth of notch. Measuring the diagonals of the notch, an average value for each sample was taken. Then the Vickers hardness value was recorded for each sample accordingly from the chart in Vickers Hardness Number (VHN).

## Results

The results were tabulated and statistically analyzed using the one way ANOVA followed by the Tuckey's post hoc test. $p = 0.05$ or less was considered statistically significant.

In Table 1 which consists of three groups, Zirconomer (Group 1) showed an average of 34.57 (µg/mm$^3$) for water sorption and 5.78 (µg/mm$^3$) for solubility. Group 2 depicts that RMGIC showed an average of 59.68 (µg/mm$^3$) for water sorption and 12.11 (µg/mm$^3$) for solubility. Group 3 depicts that Type IX GIC showed an average of 84.08 (µg/mm$^3$) for water sorption and 27.23 (µg/mm$^3$) for solubility. Comparing the results, Zirconomer showed the least values in terms of water sorption and solubility among all the three groups followed by RMGIC and Type IX GIC with a *p* value of $< 0.001$ between the three groups which was statistically significant. Similarly, Table 2 depicts that zirconomer showed an average of 75.60 (HV) for microhardness followed by RMGIC showing 70.04 (HV) and Type IX GIC 65.36 (HV). Comparing the results, Zirconomer showed the highest values in terms of microhardness among all the three groups followed by RMGIC and Type IX GIC with a *p* value of $< 0.001$ between the three groups which was statistically significant.

However, in this study, no significant difference was found between resin-modified GIC and Type IX GIC by further using Tukey's post hoc analysis.

## Discussion

There are various restorative materials available in the market today. The primary requisite for any good material should be degradation resistant, biocompatible, and non-irritating to the surrounding tissue. Apart from being esthetically pleasing, they also need to withstand the arduous conditions of the oral cavity soon after placement.[5] Gold Label 9 GIC includes the addition of Srontium glass, which provides good radiopacity and SnapSet characteristics. Apart from the advantages such as being esthetic, tooth colored, chemical binding with tooth, it has got less-desirable physical and mechanical properties such as poor polishability, susceptibility to dehydration and moisture contamination during initial setting along with low fracture toughness and flexural strength, the reason for which we are studying the newer materials.[6]

We chose Zirconia Reinforced GIC (Zirconomer) in this study as it has high strength, has been reinforced with Zirconia fillers, and has been widely used in dentistry.[7]

Resin Modified GIC (Vitremer) was also part of this study because besides having the practical advantage of on command initial light-hardening in less than a minute, it is biocompatible, has low shrinkage during the hardening reactions, and has improved physical strength when compared to conventional GICs.[8]

Sorption by definition means weight loss per area or volume due to dissolution or decomposition of material within a time period at specific temperature in saliva or oral fluids.[9] Sorption can increase the volume of the material as it can act as a plasticizer and cause deterioration of the matrix structure of the material due to which dimensional changes such as break in margin contours and staining are observed ultimately leading to restoration failure.[10] Solubility is another important factor as it influences the rate of degradation and biocompatibility, loss of marginal integrity leading to reduced longevity, and survival of restorations.[11] Therefore, water sorption and solubility of cements lead to dimensional changes which ultimately lead to failure of the restoration.[12]

In this study, the results showed that zirconomer (Table 1) has better sorption and solubility resistance followed by RMGIC and Conventional GIC. This is probably because the uniform inclusion of Zirconia particles in the glass component further reinforces the material for lasting durability and high tolerance to occlusal load. The powders also have different grain sizes and different additives, such as yttrium oxide and alumina, which are distributed homogeneously throughout the whole material.[13] This is important as the grain-size variety affects the resulting porosity as well as the translucency of the cement. The polyalkenoic acid and the glass

**Table 1:** Comparison of water sorption, solubility of three groups with mean SD using ANOVA test

| *Materials* | *Group 1: Zirconia-reinforced GIC* | | *Group 2: Resin-modified GIC* | | *Group 3: Type IX GIC* | |
|---|---|---|---|---|---|---|
| *Sample No.* | *Water sorption (μg/mm³)* | *Water solubility (μg/mm³)* | *Water sorption (μg/mm³ )* | *Water solubility (μg/mm³)* | *Water sorption (μg/mm³ )* | *Water solubility (μg/mm³)* |
| No. 1 | 23.87 | 10.61 | 58.35 | 11.94 | 88.86 | 37.13 |
| No. 2 | 38.46 | 5.3 | 58.35 | 7.96 | 71.61 | 38.46 |
| No. 3 | 37.13 | 3.98 | 51.72 | 11.94 | 59.68 | 35.8 |
| No. 4 | 35.8 | 5.30. | 67.64 | 15.91 | 59.68 | 15.91 |
| No. 5 | 33.16 | 5.3 | 63.66 | 11.94 | 110.07 | 18.57 |
| No. 6 | 30.5 | 3.98 | 58.35 | 14.59 | 70.29 | 27.85 |
| No. 7 | 42.44 | 3.98 | 67.63 | 11.94 | 128.64 | 22.55 |
| No. 8 | 41.11 | 3.98 | 55.7 | 13.26 | 79.57 | 27.85 |
| No. 9 | 33.16 | 2.65 | 67.63 | 9.28 | 87.53 | 17.24 |
| No. 10 | 38.46 | 3.98 | 59.68 | 17.24 | 80.9 | 23.87 |
| No. 11 | 34.48 | 5.3 | 54.38 | 11.94 | 87.53 | 25.2 |
| No. 12 | 25.2 | 9.28 | 55.7 | 11.94 | 79.57 | 22.55 |
| No. 13 | 34.48 | 5.3 | 63.66 | 9.28 | 88.86 | 41.11 |
| No. 14 | 31.83 | 9.28 | 55.7 | 13.26 | 92.84 | 27.85 |
| No. 15 | 38.46 | 7.96 | 57.03 | 9.28 | 75.6 | 26.52 |
| Average | 34.57 | 5.78 | 59.68 | 12.11 | 84.08 | 27.23 |
| Mean | 34.5693 | 5.7453 | 59.6787 | 12.1133 | 84.082 | 27.2307 |
| Standard deviation | 5. 26798 | 2. 38615 | 5. 15994 | 2. 54916 | 17. 86137 | 7.8104 |
| N (No. of samples) | 15 | 15 | 15 | 15 | 15 | 15 |
| F value | 73.864 | 74.873 | 73.864 | 74.873 | 73.864 | 74.873 |
| *p* value | < 0.001** | < 0.001** | < 0.001** | < 0.001** | < 0.001** | < 0.001** |

**non-significant; (SD, standard deviation; μg/mm³, microgram/millimeter)

components have been specially processed to impart superior mechanical and handling qualities.[14] However, complete elimination of water sorption and solubility of this material were not observed in this study.

The increased water sorption and solubility values of RMGIC (Table 1, Group 2) when compared to Zirconomer can be explained by the filler content, size, and surface area of filler particles and coupling agents which affect the cement's solubility of this cement.[15] According to Interaction Theory, from an atomic point of view, the polymers of RMGIC have a variable degree of water sorption based on their microscopic and molecular structure. For instance, the absorption of water by this material is important to complete the acid base reaction and is assisted by the presence of hydrophilic constituents such as hydroxyethyl methacrylate (HEMA) which can increase the water sorption ability.[2]

But, when compared to the conventional GIC, RMGIC also showed good sorption and solubility resistance because of the modification of powder by resin filler particles and large number of carboxylic acid groups in the liquid of cements; namely, tartaric acid which is a dicarboxylic acid. Hence, large number of cross-linking is established between the polymer chains, reducing the empty spaces and thus, the water inflow into the material is reduced thereby improving its water sorption resistance.[16]

(Table 1, Group 3) depicts conventional GICs being the control having the highest water sorption and solubility values. The early dissolution of glass ionomer cements in water can be attributed to two causes:[1] They contain free calcium and aluminum ions that are present in the fresh cement which can be removed during chemical reactions and secondly sodium that forms water-soluble salts with the matrix. In addition, aluminum ions react rather slowly with the matrix-forming anions and, before they are bound, are vulnerable to early water leaching.

The above results were similar with the study done by Bhatia HP, in 2017, et al.[4] who compared the sorption, solubility, and compressive strength of three different glass ionomer cements in artificial saliva-type IX GIC, silver-reinforced GIC with zirconomer, and concluded that the sorption and solubility values in artificial saliva were highest for GIC type IX–Extra followed by Zirconomer, and the least value was seen for Miracle Mix.

However, there are contradictory results by Heshmat H et al., 2019[11] who in their study in vitro evaluation of water sorption and solubility of G-Cem and FujiCem in water and acid concluded that the two understudy cements had no significant difference in water or acid solubility but FujiCem, an RMGIC showed greater water sorption than G-Cem self-adhesive resin cement. However, this result which is different to our results could be due to different GIC brands.

Microhardness is one of the most important physical characteristics for the comparative study of dental materials as it emphasizes on the mechanical properties of a material. Hardness is the resistance of a material to plastic deformity typically measured under an indentation load. The indentation produced by the machine on the material is useful to calculate the hardness of the material.

Zirconomer showed highest microhardness (Table 2, Group 1). This could be explained according to an article by YW Gu et al. in his SEM studies may be due to the good mix of both the large- and small-sized particles in the matrix of Zirconomer which suggest that the bonding between the particles and the hydrogel salt

**Table 2:** Comparison of microhardness of three groups with mean SD using ANOVA test

| | *Group 1: Zirconia-reinforced GIC* | | | *Group 2: Resin-modified GIC* | | | *Group 3: Type IX GIC* | | |
|---|---|---|---|---|---|---|---|---|---|
| *Materials* | *Microhardness in HV* | | | *Microhardness in HV* | | | *Microhardness in HV* | | |
| Sample # | Reading 1 | Reading 2 | Average | Reading 1 | Reading 2 | Average | Reading 1 | Reading 2 | Average |
| No. 1 | 89.6 | 91.2 | 90.4 | 74.78 | 75 | 74.89 | 65.2 | 61.8 | 63.5 |
| No. 2 | 81.2 | 83 | 82.1 | 65.4 | 65.2 | 65.3 | 69.05 | 70.2 | 69.62 |
| No. 3 | 70.5 | 74.1 | 72.3 | 72.34 | 72.4 | 72.37 | 67.22 | 71.5 | 69.36 |
| No. 4 | 80.2 | 82.2 | 81 | 67.73 | 67.21 | 67.47 | 61.18 | 62 | 61.59 |
| No. 5 | 74.83 | 75.81 | 75.32 | 66.96 | 69.3 | 68.13 | 67.73 | 66.6 | 67.16 |
| No. 6 | 76.56 | 76 | 76.28 | 66.96 | 69.3 | 68.13 | 65.14 | 65 | 65.07 |
| No. 7 | 74.8 | 75.6 | 75.2 | 80.03 | 82.11 | 81.07 | 62.9 | 60.11 | 61.5 |
| No. 8 | 70.2 | 72 | 71.1 | 84.82 | 81.2 | 83.01 | 66.22 | 63.4 | 64.81 |
| No. 9 | 69.11 | 69 | 69.05 | 63.9 | 61.42 | 62.66 | 64.5 | 66.2 | 60.35 |
| No. 10 | 80 | 82 | 81 | 65.2 | 63.8 | 64.5 | 70.2 | 67.22 | 68.78 |
| No. 11 | 73 | 75 | 74 | 70.33 | 70 | 70.16 | 60.4 | 61.5 | 60.95 |
| No. 12 | 65.2 | 67.8 | 66.5 | 69 | 71.63 | 70.31 | 68.9 | 70.11 | 69.5 |
| No. 13 | 70.11 | 71 | 70.55 | 73.11 | 70.7 | 71.9 | 66 | 66.4 | 66.2 |
| No. 14 | 68.8 | 69.72 | 69.26 | 68.3 | 68 | 68.15 | 62.11 | 60.8 | 61.45 |
| No. 15 | 79 | 81 | 80 | 61.2 | 63.9 | 62.55 | 71.33 | 69.8 | 70.56 |
| Mean HV | | | 75.604 | | | 70.04 | | | 65.36 |
| Standard deviation | | | 6.3515 | | | 5.99748 | | | 3.65244 |
| F value | | | 13.201 | | | 13.201 | | | 13.201 |
| *p* value | | | < 0.001** | | | < 0.001** | | | < 0.001** |

**non-significant; (SD, standard deviation; HV, Vicker's hardness value)

matrix is relatively strong.[17] Also, an exclusive characteristic of zirconia called transformation toughening gives it higher strength, toughness, hardness, and corrosion resistance. Importantly, the manufacturing process gives high strength to Zirconomer in which cold isostatic pressing for shaping Y-TZP (Yttrium Tetragonal Zirconia Particles) which produces stable, chalk-like nonsintered green-stage objects with a very high primary density. This is followed by additional compression with hot isostatic post compaction (HIP) performed at 50°C below the sintering temperature.[18] This procedure removes residual porosity and produces dense, fully-sintered-type oxide-ceramic blanks.[19]

The probable reason for decreased microhardness of RMGIC (Group 2) compared to Zirconomer (Group 1) in this study may be because of its composition being close to conventional GICs which also exhibit an acid-base reaction. However, the RMGIC light activation promotes a rapid polymer network formation that strongly reduces the salt formation rate on acid base reaction.

So, it is supposed that the photoactivation reaction may be responsible for the low microhardness data observed with this material. Another explanation is that RMGIC releases additional fluoride after immersion in acidic environments which results in the dissolution of matrix-forming constituents within the restorative material.[20] Least microhardness of Type IX GIC (Table 2, Group 3) might be because glass ionomer being hydrophilic, the early contamination of glass ionomer resulted in the binding of water molecules by polyacrylic acid (PAA) and ion-leachable glass. In this way, the chemical setting was disrupted and the decrease in hardness occurred as a result of the absorption of water as the initial phase of degradation.

Moreover, the presence of excess water during the growth of the hydrated silicate phase might have resulted also in a weaker material.[21] Also, as Type -IX GIC used in this study was not light-cured; therefore, it seems the presence of voids on the top surface can account for its lower microhardness.

The results of this study were identical with the study done by Lokhande P et al. in 2015[22] who evaluated the microhardness of type II GIC (restorative) and zirconia-based GIC and concluded that zirconia-based GICs showed better microhardness values as compared to type II GIC probably because of the addition of the zirconia particles to the conventional GIC in order to improve the physical properties.

However, Patil KM et al., in 2016,[23] studied to measure and compare the mechanical properties of five commercially available dental materials in order to ascertain proper materials for clinical treatment came to the conclusion that among Composite Z350, Giomer, Ketac Molar, Zirconomer and Compoglass F; Ketac Molar (RMGIC) showed better microhardness than Zirconomer which is contradicting to our study.

The limitations of the tests in this study when relating to water sorption and solubility include the weight gain in the samples represent the water gain when in reality, it is the difference between the gain in water and dissolution of low-molecular weight organics. Also, the constant handling of the specimens may have also caused minute wear of the surface. So, as water sorption of three different materials from different manufacturers are evaluated in this study and as there are several factors influencing water uptake, numerical comparisons are not always possible. One more drawback of the present study is that it assessed only the water sorption, solubility, and microhardness of the materials studied. However, other physical properties such as fracture toughness, shear strength, and elastic modulus of core materials have been reported to correlate the physical as well as handling properties of

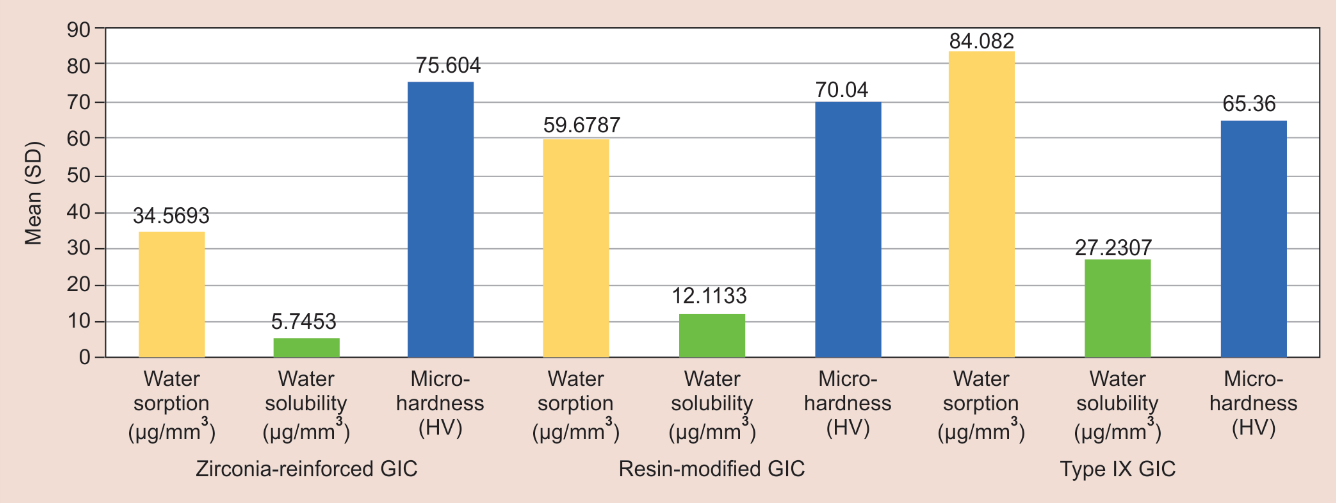


**Fig. 5:** Comparison of means of water sorption, solubility, and microhardness of zirconia-reinforced GIC, resin-modified GIC, type IX GIC

materials.[24] Nevertheless, future studies which determine all the physical properties of these materials are recommended before drawing any clinical conclusions.

## Conclusion

Ultimately, an ideal restorative material must be able to withstand the forces of mastication and be durable in the oral environment for a long time. This is the desired property which leads the clinician to favor one material over another. However, many studies published to date are mostly regarding compressive strength only; much work is lacking in area of Microhardness of Zirconia. So, our study adds the knowledge to this lacunae as it is an important parameter to assess the clinical durability of a material. In this context, this study proves that Zirconomer showed comparatively better physical and mechanical properties compared to resin-modified and conventional GIC in terms of sorption, solubility, and microhardness (Fig. 5). Hence, it can be suggested for use as a posterior restorative material in pediatric dentistry.


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