# PROPERTIES OF COMPOSITE RESIN ALKASIT AND ZIRCONIA-REINFORCED GLASS-IONOMER CEMENT IN DIFFERENT STORAGE

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## ABSTRACT

**Background**: The temperature and salivary pH in a person's mouth are highly dynamic (e.g., before, during, and after eating) and so restorations in a cavity must be resilient to these variable conditions. Temperature and immersion conditions affect the mechanical properties of a restoration. This study aimed to determine the effect of environmental conditions on diametral tensile strength (DTS) and surface microhardness of a resin composite with alkaline fillers or zirconia–reinforced glass ionomer cement (Zr-reinforced GIC).

**Method**: Thirty specimens of a resin composite with alkaline fillers (Cention-N, Ivoclar-Vivadent, Lichtenstein) and 30 specimens with zirconia-reinforced GIC (Zirconomer, Shofu, Japan) were stored at different conditions (23°C and 37°C; with and without immersion in water) for 24 hours. DTS was tested with a Universal Testing Machine (AGS-X series, Shimadzu, Japan) and surface microhardness was tested with a Vickers Microhardness tester (HMV-G Series Micro Vickers Microhardness Tester, Shimadzu, Japan). Data were analyzed statistically using a one-way ANOVA test (and Shapiro-Wilk test.

**Result**: The values of microhardness and DTS increased significantly both for the composite resin alkasite and zirconia-reinforced GIC with increasing temperature in the groups without immersion. However, there was a significant decrease in microhardness and DTS after immersion in distilled water at 37°C for both the composite resin alkasite and zirconia-reinforced GIC.

**Conclusion**: It can be concluded that storage conditions affect the microhardness and DTS of resin composite Alkasite and Zirconia-reinforced GIC

## INTRODUCTION

A new technology of restorative materials to replace amalgam has been carried out. An alkasite composite resin and a Zirconia-reinforced glass ionomer cement (zirconia-reinforced GIC) have been developed to replace amalgam. Alkasite composite resin is a new category of composite restorative materials with a urethane dimethacrylate

(UDMA) basis (Cention-N, Ivoclar-Vivadent, Liechtenstein) and zirconia-reinforced GIC is a modification of the glass ionomer cement (GIC) with an additional zirconia composition (Zirconomer Improved, Shofu, Japan) to improve the mechanical properties of this material. Both restorative materials have been suggested as replacements for amalgam.[1,2] Alkasite composite resins have a compressive strength of approximately 300 MPa, while amalgam has compression strength of 340 MPa.[1,3] Compared to amalgam, alkasite composite resins have a closer color matching to teeth, low shrinkage rate, and good biocompatibility.[1] Modification of GIC with the addition of zirconia has been done to improve the mechanical properties of GIC. The microhardness of conventional GIC is approximately between 40-46 VHN, while by reinforcing with zirconia, the microhardness values become approximately between 48-54 VHN. This increase occurs without reducing its superiority to release fluoride and more affordable restorative materials.[3,4,5]

In the oral cavity, restoration material will be exposed to various chemicals, temperature changes, and mechanical forces. These conditions can cause material deformation and, consequently, restorative materials should have good mechanical properties such as microhardness and diametral tensile strength (DTS).[6,7] The microhardness properties affect the resistance of restorative materials to scratches when finished and polished.[3] The DTS test can be used to measure the brittleness and compression of a restorative material through the simulation of mastication in the mouth.[6] Previous studies have tested the DTS of alkasite composite resin and zirconia-reinforced GIC under immersion storage at 37°C, but there has been no comparison of these materials under different conditions.[7,8] The mechanical properties of restorative material are also influenced by the polymerization and hardening processes, which is influenced by both intrinsic (e.g., material composition) and extrinsic (e.g., temperature and moisture) factors.[4,9] In the clinical situation, the use of rubber dam can cause changes in the temperature and humidity during filling, however, temperature and saliva in the oral cavity can also affect the restorative materials after the rubber dam is released. The difference of this condition is thought to affect the mechanical properties of the restorative material because some materials are sensitive to environmental changes.[9] However, information is needed on the effects of environmental changes during the initial 24 hours in different storage on the mechanical properties of alkasite restorative materials and zirconia-reinforced GIC.

The purpose of this study was to determine the effect of temperature and humidity changes on the mechanical properties of alkasite composite resin and zirconia-reinforced GIC at 23°C and 37°C in dry conditions and in 100% humidity of distilled water during 24 hours.

**Materials and Methods**

Specimens of 60 cylindrical shape of alkasite composite resin (Cention-N, Ivoclar-Vivadent, Liechtenstein) were divided into 2 test groups for DTS testing (n=30; diameter 6 mm, height 3 mm) and for Vickers microhardness testing (n=30; diameter 6 mm, height 2 mm). Other specimens of 60 cylindrical shape of zirconia-reinforced GIC (Zirconomer Improved, Shofu, Japan) were also divided into 2 test groups for DTS testing (n=30; diameter 6 mm, height 3 mm) and for Vickers microhardness testing (n=30; diameter 6 mm, height 2 mm). All specimens were made and manipulated according to the manufacture's instructions. The specimen were allowed to harden for 10 minutes in the mold before being treated without light curing. Each specimen group was then divided into 3 subgroups (n=10), and stored at 23°C in dry condition, 37°C in dry condition, and 37 °C in distilled water immersed for 24 hours respectively. The diametral tensile strength test was then performed using the Universal Testing Machine with a load of 250 kgf dan crosshead speed of 0.5 mm/minute (AGS-X Series, Shimadzu, Japan) until fracture and microhardness testing using Vickers Microhardness Tester with a load of 50gr in 15 seconds with 5 indentations in each specimen (HMV-G Series Vickers Microhardness Tester, Shimadzu, Japan). The data were analyzed using the Shapiro-Wilk test and One-Way ANOVA.

## Results

Table 1 and Table 2 showed that there was a significant difference in the mean value of microhardness and DTS between all treatment groups with both the alkasite composite resin and zirconia-reinforced GIC. The mean value of surface microhardness and DTS for the two materials with different storage treatments showed a significant difference between each group (Figure 1 and Figure 2).

**Table 1.** Mean Values of Surface Microhardness of Alkasite Composite Resin Material and Zirconia-Reinforced Glass Cement Ionomers After Different Storage

| 24-Hour Storage | Mean Surface Microhardness ± SD (VHN) | |
|---|---|---|
| | Alkasite Resin Composite (Cention-N) | Zirconia-Reinforced GIC (Zirconomer Improved) |
| Dry, 23°C | 47.54 ± 0.40[a] | 114.76 ± 1.38[d] |
| Dry, 37°C | 63.54 ± 2.40[b] | 121.52 ± 0.48[e] |
| Water immersed, 37°C | 55.05 ± 0.41[c] | 60.48 ± 0.84[f] |

- Different letters (a, b, c, d, e or f) above the columns indicate a significant difference ($p<0.05$). Data are means ± SD.

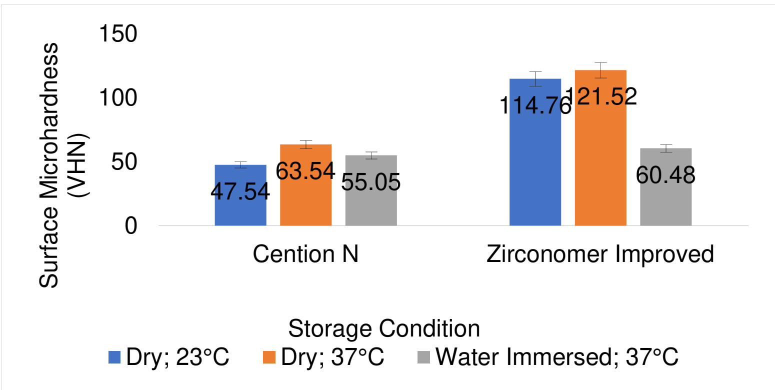


**Figure 1**. Mean Values of Surface Microhardness of Alkasite Composite Resin and Zirconia-reinforced GIC.

**Table 2.** The Mean Value of The Diametral Tensile Strength of Alkasite Composite Resin and Zirconia-Reinforced Glass Ionomer Cement After Different Storage

| 24-Hour Storage | Mean Diametral Tensile Strength ± SD (MPa) | |
|---|---|---|
| | Alkasite Resin Composite (Cention N) | Zirconia-Reinforced Glass Ionomer Cement (Zirconomer Improved) |
| Dry, 23 °C | 33.93 ± 1.07[a] | 17.49 ± 0.49[d] |
| Dry, 37 °C | 43.94 ± 1.29[b] | 24.76 ± 0.97[e] |
| Water immersed, 37 °C | 42.20 ± 0.62[c] | 7.43 ± 0.27[f] |

* Different letters (a, b, c, d, e or f) above the columns indicate a significant difference ($p<0.05$). Data are means ± SD.

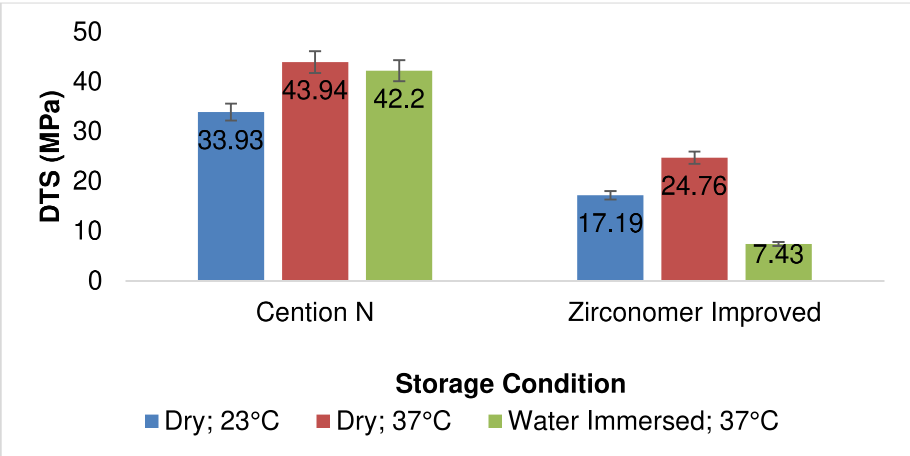


**Figure 2.** Mean Values of Diametral Tensile Strength (DTS) of Alkasite Composite Resin and Zirconia-Reinforced Glass Ionomer Cement.

**DISKUSI**

In this study, the mechanical properties of the alkasite composite resin and zirconia-reinforced GIC were tested by storing specimens for 24 hours in three different treatment conditions: dry at a room temperature of 23°C; dry at 37°C; or immersed in distilled water at 37°C. The results showed an increase in the value of microhardness and DTS after being kept dry at a temperature of 23°C compared to a storage temperature of 37°C, but there was a decrease in DTS after being stored in distilled water at 37°C, both for the alkasite composite resins and zirconia-reinforced GIC. The mean values of the surface microhardness and DTS of the two materials stored in dry conditions increased with higher temperature.

The improved degree of polymerization was demonstrated by a significant increase in the microhardness value of the alkasite composite resin with dry storage at 23 °C (47.54 ± 0.40 VHN) compared to the dry storage at 37°C (63.54 ± 2.40 VHN). The results of this study are consistent with the results of previous studies that showed the microhardness value of other commercial composite resins stored dry at 23°C which increased from 30.49–90.39 VHN to 32.33–92.40 VHN after being stored at 37°C.[10] In the wet storage treatment (100% humidity in distilled water at 37°C), there was a significant decrease in the microhardness value of alkasite composite resins after immersion in distilled water at 37°C for 24 hours. The decrease in the value of microhardness for the alkasite composite resin from dry to wet conditions was 13.5%. Nonetheless, the microhardness value of alkasite composite resin stored in distilled water at 37°C (55.05 ± 0.41 VHN) is still under the range of microhardness values found in commercial composite resins with light polymerization (45.55–90.39 VHN).[11] The presence of different filler particles and matrix structures can also result in different microhardness of materials.[12] The filler amount of alkasite composite resin is 78.4% wt., which can reduce water absorption by the matrix. Water absorption can result in a decrease in microhardness of the alkasite composite resin. The decrease of microhardness value by 13.5% could be caused by the hydrophobicity of urethane dimethacrylate (UDMA), and therefore, reduces water absorption.[13] UDMA was also reported to increase material flexibility and improve mechanical properties, especially in DTS.[14] This supports the results of this study, particularly regarding the DTS of the alkasite composite resin, which had an insignificant decrease of 3% after being immersed in distilled water. The quality and stability of the silane coupling agent in this material also plays an essential role in minimizing the deterioration of the bond between the filler and the polymer matrix and the amount of water absorbed.[11]

One study was conducted to determine the effects of heat applications at 80 ± 2°C placed on the top surface of a GIC, showed a significant increase in surface microhardness compared to a surface not receiving heat treatment.[15] In this study, it was found that dry storage at 37°C treatment resulted in an increase in surface microhardness and also the DTS of the zirconia-reinforced GIC that was stored at 23°C. Higher temperatures during the hardening of GIC in the first 24 hours tend to make the polyalkenoate acid in zirconia-reinforced GIC more active in breaking down the glass filler particles so that more polyalkenoate acid matrix bonds with filler metal ions.[15] This can affect the overall GIC hardening reaction because it is accelerated by higher temperatures and resulted in a faster formation of the calcium polyalkenoate matrix.[15]

From the results of this study, it can be seen that microhardness properties of restorative materials can be influenced by temperature and humidity conditions. Although claimed as a substitute for amalgam, the microhardness value of alkasite composite resins is still below that of amalgam (90 VHN), while the zirconia-reinforced GIC gives a fairly high microhardness value under dry 37 °C conditions, but its microhardness decreases below that of amalgam, after storage in 100% distilled water conditions similar to those of the oral cavity.[16]

Increase in microhardness and DTS of GIC can be influenced by its composition. In this study, microhardness values of zirconia-reinforced GIC (121.52 ± 0.48 VHN) were higher than those found in other studies on resin-modified GIC (approximately 100 VHN) using the same storage method.[17] Zirconia-reinforced GIC showed a significant increase in DTS between the 23°C dry storage (17.49 MPa) and 37°C dry storage (24.76 MPa). This means that zirconia filler reinforced to GIC has been shown to improve the mechanical properties of the material. The presence of zirconia particles in the Zirconomer Improved provides a superior characteristic called transformation toughening. These characteristics provide higher strength, toughness, microhardness, and corrosion resistance so that zirconia particles can increase material strength, durability, and tolerance to occlusal load.[1]

However, there was a significant decrease in the microhardness values in both the alkasite composite resin and zirconia-reinforced GIC in wet storage (100% humidity in distilled water at 37°C for 24 hours). The decrease in the microhardness value of zirconia-reinforced GIC was found to be almost 50% after wet storage. GIC material is a water-based cement that hardens due to the reaction of water-soluble ions; consequently, this material is very susceptible to water before it hardens completely.[6,17] This makes the strength of GIC very sensitive to changes in wet conditions. The GIC hardening reaction is an acid-base reaction between glass particles and polyalkenoate acid. The reaction initially occurs rapidly, but most GIC remains susceptible to water absorption for at least one hour after restoration.[3] Other studies have reported a decrease in mechanical strength in resin-modified GIC after immersion in water. Water plays a role in dissolving GIC components and making the material soften, and the decrease in microhardness has been associated with the presence of a polyacid matrix and inorganic components (glass filler), which dissolves and then increases the hydrolytic degradation in the matrix-filler bond.[18,19] Although strengthened by zirconia, the nature of GIC in this study will remain the same as that of conventional GIC. Despite a microhardness decrease of 50% after immersion in distilled water, the microhardness of zirconia-reinforced GIC is still within the range of other commercially available conventional GICs (40.74–65.10 VHN).[20]

The DTS of the alkasite composite resin after dry storage at 37°C (43.94 ± 1.29 MPa) decreased insignificantly when compared to the DTS after immersion in 37°C in distilled water (42.20 ± 0.62 MPa). There was a significant difference in the value of DTS between the alkasite composite resin after dry storage at 23°C (33.93 MPa) and dry storage at 37°C (43.94 MPa). Higher temperatures, especially in the first 24 hours of the polymerization process, affect the kinetic energy of polymerization of composite resins (i.e., increasing the movement of molecules at the propagation stage so that more polymer chains are formed). The more crosslinking polymer bond is formed, polymerization will completely occur.[10,21] Adequate polymerization of composite resin is needed to achieve higher mechanical properties.[3,6]

The role of water molecules in the polymer resin composite structure is described as a plasticizing effect. Degradation of the material begins with the absorption of water in the matrix-filler interfacial bonds, and the water will split into $H^+$ and $OH^-$ ions (hydrolysis). Then the $H^+$ ion will bind to Si-O- from the inorganic filler component so that it breaks the siloxane matrix-filler bond.[22] This statement supports the results of this study, which showed a decrease in microhardness and DTS of alkasite composite resins after immersion in distilled water due to the release of siloxane matrix-filler bonds, which reduces the mechanical properties of composite resins.

If water contamination occurs, there will be an increase in the solubility of GIC, which can decrease the compressive strength because water can break the acid-base bond formed in the GIC.[23,24] In this study, the DTS results for the zirconia-reinforced GIC in dry storage at 37°C, had DTS value of 24.76 ± 0.97 MPa, but after storage at 100% distilled water at the same temperature, the DTS values decreased to 7.43 ± 0.27 MPa. This value is below the mean DTS of resin-modified GIC (17–22 MPa) discussed earlier.[25] Similar results were observed with the Zirconomer Improved (zirconia-reinforced GIC) specimens, which has DTS of approximately 7.4 MPa. This is in accordance with previous research, which resulted DTS value at approximately 7.1 MPa.[13]

In clinical applications, it is important to identify the indications use of the two restorative materials according to the clinical cases, especially those requiring resistance to occlusal loads under conditions of temperature change and humidity in the oral cavity.

## Conclusion

Various storage methods affect the microhardness and DTS of alkasite composite resin and zirconia-reinforced GIC. Under dry conditions with higher storage temperatures, from 23°C to 37°C, there was a significant increase of microhardness and DTS in both the alkasite composite resin and zirconia-reinforced GIC. However, when stored in 100% distilled water at 37°C, a decrease in microhardness occurs in both materials, with a significant decrease in DTS of zirconia-reinforced GIC.

## Acknowledgement

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**Conflict of Interest :** None

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