Comparison of Microhardness of Indirect Composite and Zirconia With and Without Ageing: an Invitro Study

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**Abstract:** A fixed dental prosthesis (FDP) often consists of a high-strength metal core with an esthetic ceramic or composite resin veneer. Ceramic veneers are widely used due to their biocompatibility, color stability, and abrasion resistance. However, indirect composites have been introduced as an alternative due to their ease of fabrication and repairability. This study aims to compare the microhardness of indirect composite and zirconia before and after an aging process. Rectangular blocks of indirect composite and zirconia were digitally designed and milled. Microhardness was measured using a Vickers microhardness tester before and after thermocycling (1000 cycles). Statistical analysis was performed using Student’s t-test. A statistically significant reduction in microhardness was observed in both materials after thermocycling (p<0.05). Zirconia exhibited higher microhardness values compared to the indirect composite, both before and after aging. Aging reduced the microhardness of both materials, with zirconia demonstrating superior hardness retention compared to the indirect composite. These findings highlight the need for further research on the long-term performance of indirect composites in clinical settings.

# INTRODUCTION

Dental restorative materials play a crucial role in modern prosthodontics, providing both functional and esthetic solutions for tooth replacement. Among the commonly used materials, ceramics such as zirconia and indirect composite resins have gained popularity. Zirconia is widely used for crowns, bridges, and implant-supported restorations due to its excellent mechanical properties, biocompatibility, and superior esthetics. It has a high fracture toughness, making it suitable for load-bearing posterior restorations. Indirect composite resins, on the other hand, offer advantages such as ease of repair, lower brittleness compared to ceramics, and reduced wear on opposing dentition[(Fidan & Dereli, 2022)](https://paperpile.com/c/xERqoK/1Mq2)[(Kukiattrakoon & Kosago, 2021)](https://paperpile.com/c/xERqoK/037H). These materials are frequently employed in inlays, onlays, veneers, and crown restorations, particularly in cases where conservative tooth preparation is preferred[(Tulsani et al., 2021)](https://paperpile.com/c/xERqoK/3lMn). Additionally, indirect composites allow for better customization and shade matching compared to ceramics, improving esthetic outcomes for anterior restorations.

The mechanical properties of these materials have been extensively studied. Research has shown that zirconia has significantly higher flexural strength, hardness, and fracture resistance than composite resins, making it the material of choice for high-stress areas[(Chokkattu et al., 2022; Marya et al., 2022; Schmitt et al., 2009)](https://paperpile.com/c/xERqoK/AXf7+HOVn+Jyfe). Indirect composite resins, however, provide advantages in terms of shock absorption, reducing the impact on the opposing dentition. Previous studies have also explored the influence of thermocycling and aging on these materials, demonstrating that prolonged exposure to moisture and temperature fluctuations can lead to material degradation. Composite resins tend to absorb water over time, leading to polymer matrix breakdown, whereas zirconia undergoes phase transformation, affecting its long-term stability. Understanding these behaviors is crucial for selecting the appropriate restorative material for different clinical applications. The comparison between zirconia and indirect composite resins is particularly significant because both materials are widely used in fixed prosthodontics but exhibit distinct mechanical behaviors[(Pires-de-Souza et al., 2009)](https://paperpile.com/c/xERqoK/pGCb). While zirconia is known for its excellent wear resistance and fracture toughness, indirect composite resins offer better shock absorption and ease of repairability[(Goyal et al., 2008; Kannan et al., 2022)](https://paperpile.com/c/xERqoK/ol4g+pF6q). By analyzing their microhardness before and after aging, this study provides insight into their durability, guiding clinicians in material selection for long-term restorations.

Microhardness is a critical parameter that reflects a material’s resistance to wear and surface degradation[(Glantz et al., 2002)](https://paperpile.com/c/xERqoK/m4LK). Since restorative materials are subjected to varying forces and environmental conditions in the oral cavity, understanding how these factors influence their longevity is essential. Additionally, thermocycling simulates the aging process by exposing materials to temperature fluctuations and moisture, mimicking the intraoral environment. Studying the effect of thermocycling on microhardness helps in predicting the long-term clinical performance of these materials.The need for this study arises from the increasing use of both zirconia and indirect composite resins in fixed prosthodontics, necessitating a comprehensive evaluation of their durability over time. By comparing their microhardness before and after an aging process, this study aims to determine their relative suitability for long-term prosthetic applications. The null hypothesis was formulated as There is no difference in the microhardness of indirect composite and zirconia before and after aging.

# MATERIALS AND METHODS

This in vitro study was approved by the institutional ethical committee. The materials evaluated included:

* Indirect composite ( SR Nexco, Ivoclar Vivadent, Liechtenstein)
* Zirconia ( BruxZir, Glidewell, USA)

**Sample Preparation:** A sample size of 24 was calculated using GPower software. Rectangular bars (20 x 5 x 2.5 mm) were designed in Blender software, and STL files were generated for milling zirconia samples. Silicon molds were created from the zirconia samples to fabricate indirect composite specimens. The composite samples were light-cured for 60 seconds to ensure polymerization.



Fig 1. Image showing designed STL file.



Fig 2. Samples of zirconia and indirect composite.

**Thermocycling:** All samples were subjected to 1000 thermocycles between 5°C and 55°C, with an immersion time of 30 seconds in each bath and a transition time of 10 seconds[(Akmal & Duraisamy, 2020; Duraisamy, 2021; Poornima et al., 2021)](https://paperpile.com/c/xERqoK/ugWT+zj7l+t4go). This process was carried out to simulate intraoral aging conditions. Thermocycling is a widely accepted method for simulating the aging process of dental materials in laboratory settings. This procedure involves subjecting samples to repeated temperature changes, mimicking the variations they would experience in the oral environment. The thermal cycling process consists of immersing samples in two water baths maintained at different temperatures, typically 5°C (cold) and 55°C (hot)[(Fischer et al., 2008; Sailer et al., 2007)](https://paperpile.com/c/xERqoK/eo7Z+fW8g). Each cycle consists of submerging the samples in each bath for a fixed duration, usually 30 seconds, followed by a transition period of 10 seconds. This cycling process induces mechanical stress and promotes water absorption, which can degrade the material over time. In this study, all samples were subjected to 1000 thermocycles to replicate long-term intraoral conditions. The number of cycles was selected based on previous literature, where it has been suggested that 1000 thermocycles approximately correspond to one year of clinical use[(Duraisamy & Senior Lecturer, Department of Prosthodontics and Implantology, S Poornima et al., 2021)](https://paperpile.com/c/xERqoK/ugWT+zj7l).

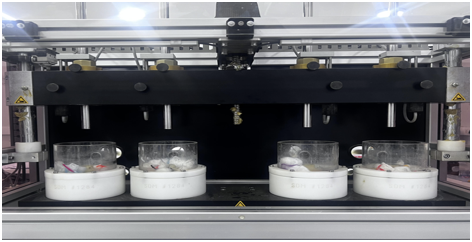


Fig 3. Image showing thermocycling.

**Microhardness Testing:** Vickers microhardness testing was performed to evaluate the surface hardness of indirect composite and zirconia before and after thermocycling[(Pjetursson et al., 2007)](https://paperpile.com/c/xERqoK/UroM). A Shimadzu HMV G microhardness tester was used at magnifications of 10x and 40x. A load of 2.942N was applied for 15 seconds. Five separate indentations were made on each sample, and the mean value was calculated. Post-aging microhardness values were recorded to assess the impact of thermocycling on material properties. Microhardness testing is a crucial method for assessing the mechanical properties of restorative materials([(Adel et al., 2023; Aparna et al., 2021; Chokkattu et al., 2022, 2023; Ganapathy s; Ganapathy, 2021; Jain & Verma, 2022; Laghari et al., 2023; Marya et al., 2022; Merchant et al., 2022; Muthuswamy Pandian et al., 2022; Pandiyan et al., 2022; Poornima et al., 2021; Ramakrishnan et al., 2023; Ramamurthy et al., 2022; Solanki et al., 2023; Sreevarun et al., 2023; Subramanian & Harikrishnan, 2023; Verma & Muthuswamy Pandian, 2021; Wadhwani et al., 2022)](https://paperpile.com/c/xERqoK/rRjG+sEF2+M1je+lh4P+g7Nt+ps3R+zV0Y+MVSi+XAHH+utex+Jyfe+pUgj+HOVn+iJWn+pbJM+zj7l+qxNg+3Luv+k3mu+TIcf)). In this study, Vickers microhardness testing was employed to evaluate the surface hardness of indirect composite and zirconia before and after thermocycling. The Vickers hardness test involves applying a standardized force to the sample surface using a diamond-shaped indenter, which forms an indentation(Chehelgerdi et al., 2023). The size of the indentation is then measured under a microscope to determine the material’s hardness.

The microhardness testing in this study was performed using a Shimadzu HMV G microhardness tester at magnifications of 10x and 40x. A load of 2.942N was applied for 15 seconds to ensure accurate readings[(Krajangta et al., 2022; Wachtman et al., 2009)](https://paperpile.com/c/xERqoK/brMy+cXaT). Five separate indentations were made on each sample, and the mean value was calculated.

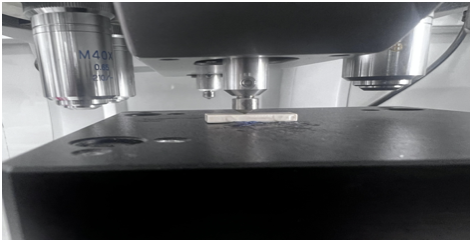
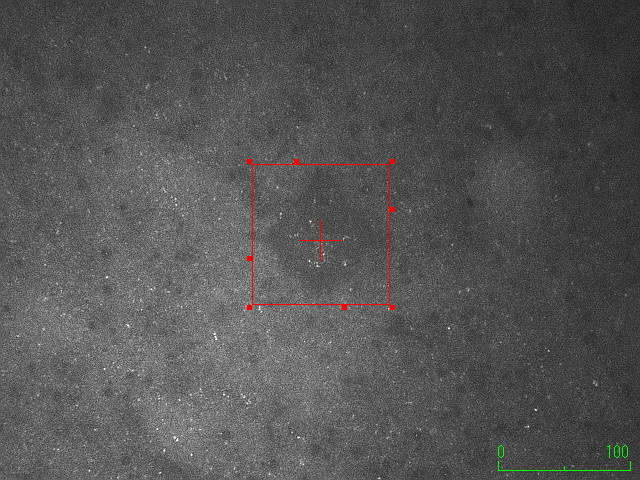
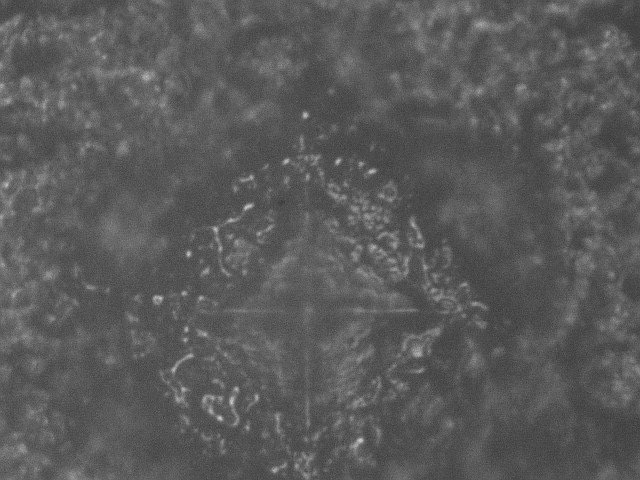
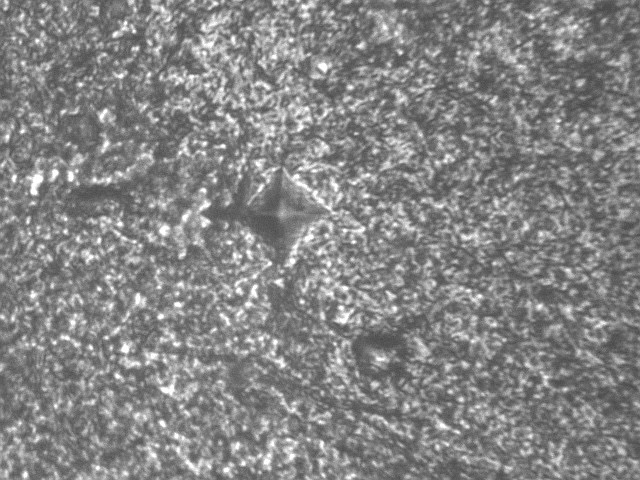
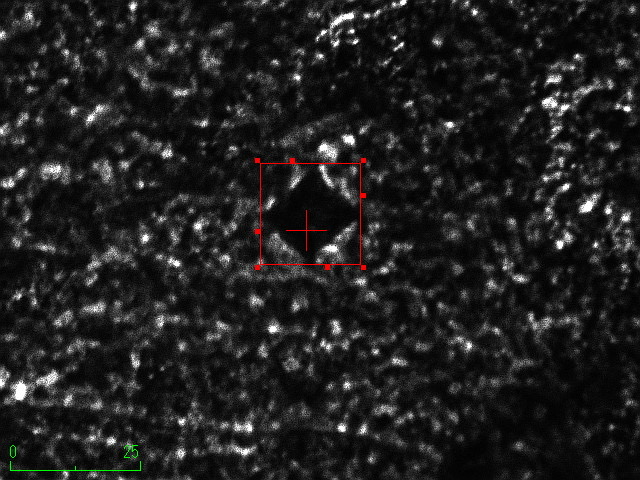
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Fig 4. Image showing Vickers hardness testing of the sample



(a) (b)

Fig 5. Figure showing indentation of indirect composite prior to simulated aging(a) Figure showing indentation of indirect composite post simulated aging(b)

1. (b)

Fig 6. Figure showing indentation of zirconia prior to simulated ageing(a) Figure showing indentation of zirconia post simulated ageing(b)

# STATISTICAL ANALYSIS

Data were analyzed using IBM SPSS Statistics 20. Normality testing was performed using the Kolmogorov-Smirnov test. Paired Student’s t-tests were used to compare pre- and post-aging values, with significance set at p<0.05. A masked researcher performed the statistical analysis to minimize bias. Data were analyzed using IBM SPSS Statistics 20. Normality testing was performed using the Kolmogorov-Smirnov test. Paired Student’s t-tests were conducted to compare pre- and post-aging values, with significance set at p<0.05.

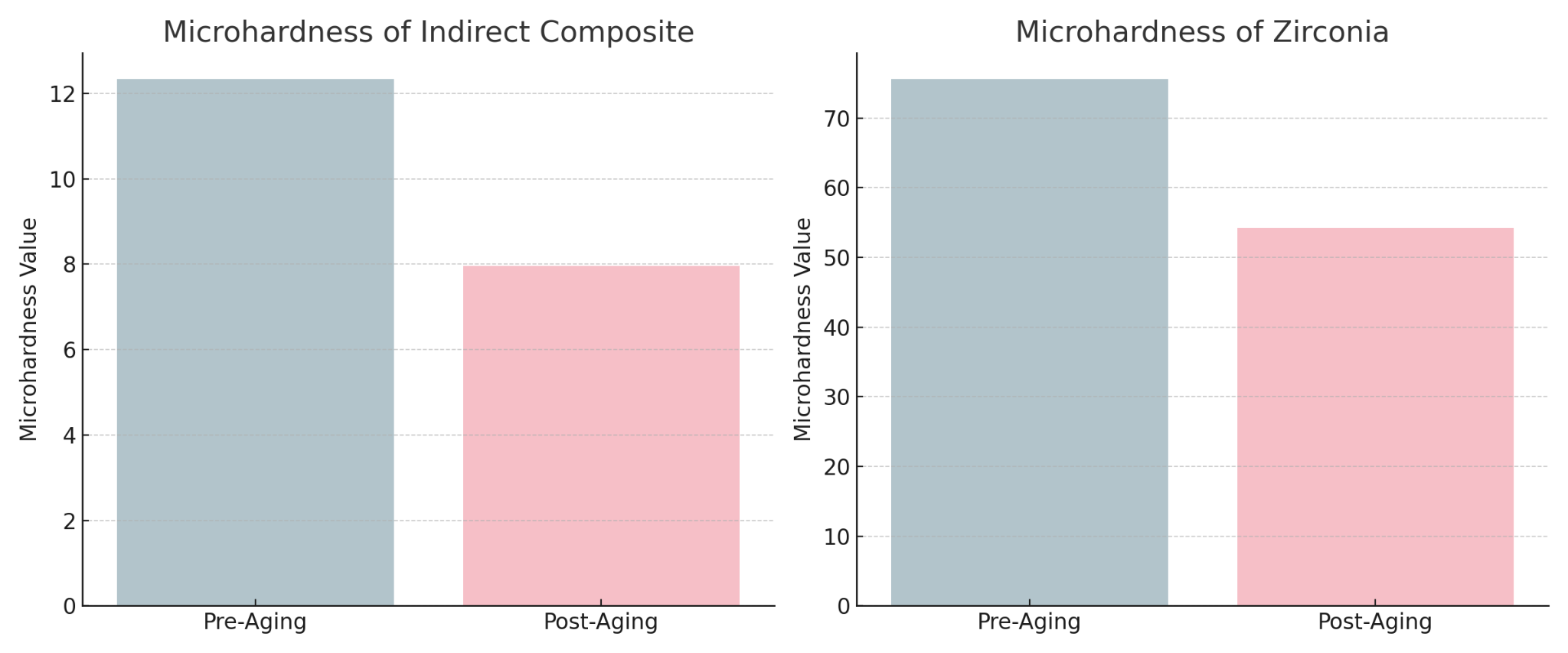
# RESULTS

In this experimental and comparative in vitro study, all data showed normal distribution detected by Kolmogorov-Smirnov test. The results of the paired t-test demonstrated a statistically significant reduction in microhardness for both indirect composite and zirconia after aging. For indirect composite, post-aging, the microhardness has decreased (12.333 ± 4.376, p < 0.01). The statistically significant decrease suggests that thermocycling had a notable impact on the material's surface hardness, likely due to polymer degradation and water absorption. For zirconia, post-aging, the microhardness has decreased significantly (75.583 ± 21.403, p < 0.01). Despite this reduction, zirconia maintained a substantially higher overall microhardness compared to indirect composite both before and after aging.

The mean difference, standard deviation, and statistical significance (p-value) are reported, along with the 95% confidence interval (CI) for each material. A p-value < 0.01 indicates a statistically significant reduction in microhardness post-aging.

Table 1: Paired t-test results comparing pre-aging and post-aging microhardness values of indirect composite and zirconia

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| **Group** | **Mean ± SD** | **Std. Error Mean** | **95% confidence interval** | | **t-value** | **df** | **p-value** |
| **Lower CI** | **Upper CI** |
| **Indirect Composite** | 12.333 ± 4.376 | 1.263 | 9.553 | 15.114 | 9.763 | 11 | 0.000 |
| **Zirconia** | 75.583 ± 21.403 | 6.178 | 61.985 | 89.182 | 12.233 | 11 | 0.000 |

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1. **(b)**

Fig.7: (a) (b) Bar Graphs of Microhardness Before and After Aging

This figure presents separate bar graphs for the pre- and post-aging microhardness values of indirect composite and zirconia (Saadh et al., 2024). The decrease in microhardness after aging is evident in both materials.

# DISCUSSION

When it comes to assessing the microhardness of dental composite resins and ceramics, the impact of environmental conditions and aging processes must be considered. Several investigations have revealed that parameters such as storage conditions and thermocycling application can alter the microhardness of these materials[(Edelhoff et al., 2008)](https://paperpile.com/c/xERqoK/ZYav).

Tuncer et al., found that thermocycling had a substantial effect on the microhardness of composite resins[(Tuncer et al., 2013)](https://paperpile.com/c/xERqoK/jEOl). After 10,000 thermocycles, the microhardness values of the composite resins were discovered to decline. This suggests that thermocycling may have a negative impact on the microhardness of dental materials. Furthermore, Gale and Darvell's work emphasized the necessity of thermocycling in replicating oral circumstances[(Gale & Darvell, 1999)](https://paperpile.com/c/xERqoK/3b7R). Thermocycling, which involves subjecting materials to cyclic thermal loads and immersion in water, can simulate temperature variations and moisture levels in the mouth cavity that are typically experienced during ordinary activities such as eating and drinking. Furthermore, thermocycling has been shown to significantly affect the binding strength between zirconia ceramic and resin composite. Additionally, after 500 thermocycles, the surface microhardness of composite restorations treated with different dental adhesive systems was evaluated . The findings revealed that thermocycling had a substantial impact on the surface microhardness of the composite restorations. This shows that thermocycling can reduce the microhardness of dental materials, which could affect their overall durability and long-term stability. Finally, thermocycling is critical in evaluating the microhardness of dental materials such as indirect composite and zirconia([(Adel et al., 2023; Aparna et al., 2021; Chokkattu et al., 2022, 2023; Ganapathy, 2021; Ganapathy 2021; Jain & Verma, 2022; Laghari et al., 2023; Marya et al., 2022; Merchant et al., 2022; Muthuswamy Pandian et al., 2022; Pandiyan et al., 2022; Poornima et al., 2021; Ramakrishnan et al., 2023; Ramamurthy et al., 2022; Solanki et al., 2023; Sreevarun et al., 2023; Subramanian & Harikrishnan, 2023; Verma & Muthuswamy Pandian, 2021; Wadhwani et al., 2022)](https://paperpile.com/c/xERqoK/rRjG+sEF2+M1je+lh4P+g7Nt+ps3R+zV0Y+MVSi+XAHH+utex+Jyfe+pUgj+HOVn+iJWn+pbJM+zj7l+qxNg+3Luv+k3mu+TIcf)). Several investigations have been undertaken to acquire a better knowledge of the comparative microhardness of zirconia and indirect composite materials. In one investigation, the microhardness values of zirconia and hybrid composite resins were examined. The findings revealed that zirconia has greater microhardness values than hybrid composite resins. This shows that zirconia has a higher hardness, making it more resistant to wear and surface flaws.

Another study looked at how different particles in resin composites affected their microhardness[(Adel et al., 2023; Aparna et al., 2021; Chokkattu et al., 2022, 2023; Ganapathy; Jain & Verma, 2022; Laghari et al., 2023; Marya et al., 2022; Merchant et al., 2022; Muthuswamy Pandian et al., 2022; Pandiyan et al., 2022; Poornima et al., 2021; Ramakrishnan et al., 2023; Ramamurthy et al., 2022; Solanki et al., 2023; Sreevarun et al., 2023; Subramanian & Harikrishnan, 2023; Verma & Muthuswamy Pandian, 2021; Wadhwani et al., 2022)](https://paperpile.com/c/xERqoK/rRjG+sEF2+M1je+lh4P+g7Nt+ps3R+zV0Y+MVSi+XAHH+utex+Jyfe+pUgj+HOVn+iJWn+pbJM+zj7l+qxNg+3Luv+k3mu+TIcf)). The researchers discovered that resin composites containing silica and zirconia particles had greater microhardness values than other resin composites .

This suggests that the addition of zirconia particles in resin composites can boost microhardness. However, when compared to zirconia and amalgam materials, the microhardness values of indirect composite resins are often lower. The effect of aging on the microhardness of zirconia and indirect composite materials varies. Aging has the ability to reduce the microhardness of zirconia by causing phase transitions and structural deterioration. This shows that zirconia materials may become less resistant to wear and surface flaws over time. The effect of aging on the microhardness of indirect composite materials, on the other hand, is less evident. More research is required to identify the specific effect of aging on the microhardness of indirect composite materials. Temperature fluctuations, moisture, and chemical interactions can all cause structural changes or degradation during the aging process. In the case of dental materials, the oral environment, which is prone to continuous temperature changes, chemical exposure from saliva, and mechanical forces during mastication, can contribute to the observed drop in microhardness.

# CONCLUSION

Within the limitations of this study, it can be concluded that the parameters such as composition, particle content, and age can all affect the microhardness of zirconia and indirect composite materials. After aging, zirconia has been demonstrated to have greater microhardness values than indirect composite materials and the microhardness values of both indirect composite and zirconia dropped.

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