

## Comparative Evaluation of the Effect of 5% Sodium Hypochlorite on the Microhardness of Posterior Bulkfill and Nanohybrid Composite Resin

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### **Abstract:**

**Aim :** The aim of this study was to evaluate the effect of 5% Sodium hypochlorite (NaOCl) on the microhardness of Posterior Bulkfill and Nanohybrid composite resin.

**Methods:** 34 disc-shaped composite samples were prepared using Teflon moulds of 10 mm diameter and 2 mm depth and divided into Group 1 and Group 2, with 17 samples each of Bulkfill composite and Nanohybrid composite, respectively. The samples were immersed in 5ml of 5% NaOCl for 40 minutes and the solution was replenished after every 5 minutes. Pre and post immersion Vickers hardness testing was performed for each group using a 100-gram load and dwell time of 10 seconds on top surface. Statistical analysis was done.

**Results:** Mean initial microhardness was  $68.09 \pm 5.73$  and  $61.05 \pm 5.13$  for Bulkfill and nanohybrid composite discs, respectively. After immersion, the microhardness reduced to  $63.14 \pm 4.68$  for Bulkfill and  $57.05 \pm 4.92$  for Nanohybrid composite, respectively. This difference was statistically significant. ( $p < 0.05$ )

**Conclusion:** Bulkfill and Nanohybrid composite resins showed decrease in microhardness on exposure to 5% NaOCl used as endodontic irrigant thus necessitating replacement of pre-endodontic build-up before final post endodontic restoration.

**Keywords:** hardness, immersion, nanocomposites, resin, sodium hypochlorite.

## Introduction

Successful endodontic treatment depends on the complete elimination of micro-organisms from the root canal space. Various microorganisms are involved in diseases of pulp and periapex, which lead to endodontic infection of the root canal system.<sup>[1]</sup> The aim and primary goal of endodontic treatment is the reduction in microbial irritants from inside the canal system of an infected tooth and ensuring a completely microbe-free canal space. Complete mechanical and chemical debridement of the root canal space will ensure successful endodontic treatment. Cleaning, and shaping followed by irrigation is essential for cleaning difficult anatomic structures in the teeth such as fins, cul-de-sac, ramifications, lateral and accessory canals, and other irregularities that might be overlooked during instrumentation<sup>[2,3]</sup> Thus, during endodontic treatment, under all aseptic conditions it is mandatory to remove the pulp tissue completely, enlarge the canal, irrigate and chemically disinfect the canal and finally achieve clean space for 3-dimensional obturation.<sup>[1]</sup>

Teeth that can be saved by endodontic treatment to preserve their health and function are generally structurally weak and not intact, which makes the pre-endodontic restoration essential in achieving successful endodontic treatment. There has been increasing attention on pre-endodontic restoration as a strategy to boost the effectiveness and durability of endodontic procedures while also enhancing the overall structural strength of teeth.<sup>[4]</sup> The pre-endodontic buildup or rewalling procedure is a valuable step for rebuilding lost walls. This step provides various benefits such as protecting and stabilizing the tooth during endodontic treatment, minimizing marginal leakage, reducing the risk of tooth structure fracture, facilitating instrument introduction and irrigant reservoir, maintaining the coronal seal, and preventing the growth of gingival overhangs into access, makes rubber dam isolation easy, creates space and holds the irrigating solution, prevents bacterial microleakage, fracture resistance for remaining weak structures, etc. Additionally, this process also serves to improve functionality and aesthetics, while offering stable reference points that make the treatment more precise and predictable. Overall, the pre-endodontic buildup procedure is a simple yet essential step that can greatly enhance the outcome of endodontic treatment<sup>[5,6]</sup>.

The materials used for pre-endodontic restoration and core buildup are flowable composites, restorative composite resins, packable composite resins, silver amalgam, or glass ionomer cement. By bonding these materials to the remaining tooth structure, dentists can reinforce weakened areas and prevent further damage or fracture.<sup>[4]</sup> Tooth coloured restorative resins have developed and modified throughout the years to achieve enough longevity of the restoration to improve the mechanical properties and to meet the increasing esthetic demand.<sup>[7]</sup> Improvements in dental composite properties arise from filler particle reinforcement and are also influenced by the concentration and size of the filler particles.. Recent research embeds nanoparticles into polymeric matrices, yielding a new generation of composites with smaller particles and higher filler content. These advancements offer superior aesthetics, enhanced wear resistance, increased fracture toughness, and reduced brittleness.<sup>[8]</sup>

The use of fillers can alter the properties of the recycled material. When compared to micro-filled traditional composites, nanofilled composites exhibit superior mechanical properties. Therefore, these composites offer burnishing properties similar to those of micro-filled composites, and the wear resistance is comparable to that of hybrid composite resins.<sup>[9]</sup> Nanocomposite or nanohybrid composite with a filler content of 82% by weight and surface-modified silica/zirconia shows good aesthetics and compressive, tensile and flexural strength.<sup>[10]</sup> Bulk fill composite resins developed as an alternative material for posterior teeth to simplify the technique, minimize the chances of failure, and reduce clinical time by allowing a single increment of 4-5mm thick.<sup>[11]</sup> The deeper polymerization achieved with bulk-fill composites with thicker increments arises from advancements in both the translucency of the composites and the formulation enhancements in filler components and organic matrix.<sup>[7]</sup>

In the oral cavity, resin composites are subjected to various types of chemical and physical degradation, including wear and erosion caused by chewing, food, and microbes. The breakdown of restorative materials in the mouth is a multifaceted process that involves their disintegration and dissolution, leading to changes in the properties of the materials. According to several studies, resin restorations can fail due to significantly differing intraoral environments and constant exposure to masticatory forces and certain food chemicals. Low-pH liquids like acids have also shown deleterious effects on the strength of composites.<sup>[12,13]</sup> It is important to note that the degradation of composite surfaces can also occur due to immersion in water, thermocycling, and citric acid exposure<sup>[13]</sup>

To ensure the durability of restorative materials in teeth, it's crucial to assess their mechanical properties, which are affected by both compressive and flexural forces. Laboratory tests are commonly used to evaluate the performance of biomaterials. Microhardness tests are a widely utilized method for studying both material properties and tooth hardness. This technique offers speed and simplicity, requiring only a small specimen surface area for evaluation. It involves impressing diamond indenters (either Knoop's or Vickers) onto specimen surfaces under controlled loads and durations.<sup>[14,15]</sup> Composites used in endodontic treatment are constantly exposed to various irrigating solutions of different pH.<sup>[14-16]</sup>

An irrigant of choice, Sodium hypochlorite (NaOCl) is most used in the concentration of 0.5-5.25% and pH ranging between 11 and 12.<sup>[17]</sup> Use of 0.5% to 5.25% NaOCl and 17% Ethylenediaminetetraacetic acid (EDTA) in a sequential manner is a recommended irrigation method. NaOCl has excellent antimicrobial capacities and great efficacy in dissolving vital or necrotic tissues.<sup>[18]</sup> The solution of NaOCl generates hypochlorous acid that functions as a solvent and releases chlorine. Chlorine released from NaOCl reacts with the amino acids from the proteins to form chloramines, which disrupt the cell metabolism and membrane, whereas hypochlorous acid and hypochlorite ions degrade amino acids. Chlorine's strong oxidizing properties inhibit bacterial enzymes and irreversibly oxidize the sulphur-containing groups, making it a strong antimicrobial agent.<sup>[16,19]</sup>

The aim of the present study was to evaluate the effect of 5% NaOCl on the microhardness of Posterior Bulkfill and Nanohybrid composite resin. Thus, the null hypothesis tested was there is no effect of 5% NaOCl on the microhardness of bulkfill and nanohybrid posterior restorative composite resins.

### Materials and Methods:

This experimental in-vitro study was carried out at the Department of Conservative Dentistry and Endodontics after obtaining the Institutional Ethics Committee approval.

Ethical clearance number: MGM/DCH/IRRC/194/2023

Inclusion criteria: Bulk fill [Tetric-N-Ceram] and Nanohybrid posterior restorative composite resin [3MTM Filtek Z250, St. Paul, MN, USA] were included in the study to prepare the study samples.

Exclusion criteria: Prepared Samples that had any defects, errors during preparation or those damaged during finishing and polishing were excluded from the study.

Sample size calculation: Reference for formula: Bernard Rosner. Fundamentals of Biostatistics (5th edition). (Based on equation 8.27). Using G\*Power software, sample size was calculated with effect size 1.24 and power 80%.<sup>[20]</sup>

Materials used in the study:

The composition of different Composite materials used in the study. (Table No.1)

**Table no:1 Composition of different Composite materials used in the study**

Sr. No.	Material Name	Type	Manufacturer	Organic matrix

1.	Tetric-N Ceram	Bulkfill	Ivoclar Vivadent	Bis-GMA, UDMA, TEGDMA
2.	Filtek Z250 XT	Nanohybrid	3M ESPE	Bis-GMA, TEGDMA, UDMA, Bis-EDMA

### Preparation of Composite Resin Samples:

Samples were crafted utilizing Teflon moulds measuring 10 mm in diameter and 2 mm in height. The moulds were positioned on Mylar strips, which were placed on a glass slab, and subsequently filled with nanohybrid posterior restorative composite resin [3MTM Filtek Z250, St. Paul, MN, USA] and bulk-fill posterior restorative composite [Tetric N Ceram] in accordance with the manufacturer's guidelines. A Mylar strip was then positioned on the upper surface of the mould, and the material was levelled using a microscope glass slide to achieve uniform surface finishing and eliminate any surplus material. Following the removal of excess material, the glass microscope slide was withdrawn, leaving behind the Mylar strip. The samples were then subjected to curing using a Light Emitting Diode (LED) curing light for 40 seconds at an intensity of 1000 mw/cm<sup>2</sup>, maintaining 1 mm from the tip. Before curing each sample, the intensity of the light curing unit was verified using an external handheld radiometer. Upon completion of curing, the cured samples were disengaged from the moulds. Subsequently, the samples underwent polishing using the Shofu Composite polishing kit (San Marcos, California, USA) with a low-speed hand piece and coolant, following the manufacturer's instructions. To ensure thorough polymerization, the samples were incubated at 37°C, 100% humidity in a lightproof container for 24 hours. A total of 34 samples were prepared and randomly allocated into two groups of 17 samples each (n=17) as follows: Group 1 - Samples comprising bulk-fill composite, Group 2 - Samples composed of nanohybrid composite.

### Microhardness Evaluation:

17 Disc-shaped samples, each of bulk fill and nanohybrid resin, were prepared as mentioned above and were subjected to evaluation of micro hardness testing. After finishing and polishing of the samples, microhardness testing was measured through the top surface for each sample before and after immersion into the test solution i.e. 5% NaOCl at a load of 100gm and a dwell time of 10 s using microhardness tester with specifications as: Microhardness tester, Reichert Austria Make, Sr.No.363798, Reference Standard: ISO 6507. For every sample, three indentations were made not closer than 1mm to each other, and accordingly, the mean Vickers hardness number (VHN) values were calculated for every sample before and after immersion into NaOCl. Hardness values were expressed as where  $1 \text{ VHN} = 1.854 \text{ P/d}^2$ , with P being the indentation load and d being the diagonal length.<sup>[12]</sup>

### Immersion into the test Solution:

The specimens used for microhardness tests were immersed into 5ml of 5% NaOCl for 40 minutes, and the solution was replenished after every 5 minutes to mimic the clinical scenario.<sup>[2,21]</sup> Post immersion hardness testing was done on the same top surface used during baseline or pre immersion hardness testing.

### Statistical analysis:

Statistical analysis was done of the obtained data with Statistical Package for Social Sciences (IBM SPSS Statistics for Windows, version 21.0, Armonk, NY: IBM Corp.) at 95% CI and 80% power for the study. Kolmogorov-Smirnov and Shapiro-Wilk test was done to check for normal distribution of the data. Descriptive statistics were performed in terms of mean, standard deviation. An independent sample t-test was applied to compare microhardness between bulkfill and nanohybrid composite, respectively. A paired t-test was applied to compare between initial and after immersion in sodium hypochlorite.

### Results

The bulkfill pre-immersion values had the highest mean microhardness values of  $68.09 \pm 5.73$ . Mean microhardness of bulkfill composite resin after the immersion in NaOCl was reduced to  $63.14 \pm 4.68$ . This difference in the microhardness before and after was statistically highly significant. ( $p < 0.001$ ). The mean microhardness of the nanohybrid composite was  $61.05 \pm 5.13$  initially, and after immersion in NaOCl it was reduced to  $57.05 \pm 4.92$ , respectively. This difference in microhardness was statistically highly significant. ( $p < 0.001$ ) (Table no.2)

**Table no. 2: Comparison of microhardness in bulkfill and nanohybrid composite before and after immersion in sodium hypochlorite**

					95% CI		
		Mean	N	Std. Deviation	p value	Lower	Upper
Bulkfill	Before	68.0294	17	5.73811	<0.001*	3.78454	5.98017
	After	63.1471	17	4.68296			
Nanohybrid	Before	61.0529	17	5.13080	<0.001*	3.50718	4.49282
	After	57.0529	17	4.92571			

Mean microhardness in bulkfill composite resin was  $68.09 \pm 5.73$ , and nanohybrid composite was  $61.05 \pm 5.13$  initially, respectively. This difference in microhardness between bulkfill and nanohybrid composite was statistically significant. ( $p < 0.05$ ). After immersion, the microhardness was  $63.14 \pm 4.68$  for bulkfill and  $57.05 \pm 4.92$  for nanohybrid composite, respectively. This difference was statistically significant. ( $p < 0.05$ ). (Table no.3)

**Table No: 3 Comparison of microhardness before and after immersion in sodium hypochlorite between bulkfill and nanohybrid composites**

				95 % CI		
		Mean	Std. Deviation	p value	Lower	Upper
Before	Bulkfill	68.0294	5.73811	0.001*	3.17370	10.77925
	Nanohybrid	61.0529	5.13080			
After	Bulkfill	63.1471	4.68296	0.001*	2.73644	9.45180
	Nanohybrid	57.0529	4.92571			

Irrespective of the composite material used, there was a statistically significant reduction in the microhardness of composite resin after immersion in 5% NaOCl. After immersion, the mean VHN reduced to  $63.14 \pm 4.68$  for bulkfill and  $56.05 \pm 4.92$  for nanohybrid composite, respectively. This difference was statistically significant. ( $p < 0.05$ ).

## Discussion

According to ENDODONTICS: Colleagues for Excellence, 2002, the Major Goals of RCT are to - Remove irritants from the root canal system and fill or obturate the cleaned and shaped system to prevent future recontamination of the sealed root canals. Recontamination can be prevented by a good apical and coronal seal.<sup>[22]</sup> Safavi et al suggested that a perfect post-endodontic restoration should be performed for higher success. A permanent restoration showed better results than teeth with provisional restorations.<sup>[23]</sup>

But the clinical lifespan of composite resin used can vary due to various factors related to the tooth, risks associated with the patient, chosen restorative techniques, and the materials implemented. The properties of materials like fracture toughness, microhardness, surface roughness, compressive strength, etc, are influenced by their composition and the environment to which they are exposed.<sup>[13]</sup> The surface hardness of composite resins in posterior stress-bearing areas is an important mechanical property influenced by the effectiveness of polymerization and bonding between monomers. Hardness is influenced by many properties such as the size, shape, and fraction of fillers in the inorganic phase. Hardness is found to increase with higher filler amount and the specific composition and structure of the organic matrix.<sup>[24]</sup>

Hardness, a key mechanical property, signifies a material's ability to withstand indentation or penetration, a characteristic shaped by both filler attributes (like size, weight, volume) and the resin's chemical composition. The relationship between filler volume and mechanical traits, such as hardness and elastic modulus, is well-documented. Typically, the hardness of resin composites is gauged through methods like Vickers or Knoop's hardness testing, where a diamond tip applies a predetermined force and duration for indentation. The resulting hardness is calculated by dividing the applied load by the indentation area, observed microscopically, and adjusted by a coefficient. This technique stands out for its simplicity, reproducibility, and non-destructive nature, providing valuable insights into material performance.<sup>[12]</sup>

Thus, in this study, microhardness was measured on the top cleaned and polished surface of the composite disc-shaped specimens. To obtain the microhardness values, Vickers hardness testing was performed in this study using 100g load with a dwell time of 15 s, because it is an appropriate and practical method for evaluating changes on the surface and in deeper hard tissue structures. Furthermore, this test is widely accepted due to its highly accurate readings and as it uses only one type of indentation for all the surface treatments<sup>[2,12]</sup>

A Study by Reham et al concluded that incrementally placed nanohybrid resin composite showed greater reduction in compressive strength and surface hardness than bulkfill one, as compared to bulkfill composite when immersed in different beverages.<sup>[8]</sup> Studies have found that acidic media significantly soften and roughen dental composite biomaterial, which can be highly damaging to dental health.<sup>[14]</sup>

During endodontic treatment, dental composites are exposed intermittently or continuously to different chemical agents, which could cause chemical degradation and loss of many dental properties. To standardize the procedure, all the samples were immersed for 40 minutes in the selected irrigating solution i.e 5% NaOCl, and to mimic the clinical scenario, the solution was replenished every 5 minutes.<sup>[2,25]</sup>

In the study, the samples were not exposed to any mechanical forces so any observed change in hardness would be related to chemical dissolution. Restorative materials are exposed to varying environments inside the oral cavity. Materials need to be able to withstand changes in temperature and acidic-base conditions that may arise from food and drinks. Hence, they should exhibit minimal effects or remain resistant in such circumstances.<sup>[13]</sup>

In this study, Bulkfill composites showed less reduction in the mean VHN values as compared to nanohybrid composite resin post immersion in 5% NaOCl. Changes in the constituents or the composition of the restorative substance could be accountable for the reduction in microhardness. Hypochlorite and hypochlorous acid, the reactive chlorine derivatives found in NaOCl, possess notable oxidizing power. It is seen that substrates with oxidative capabilities may break polymer chains, leading to the decomposition of resin-based materials, thus affecting their properties. Oxidative substrates might also influence the hardness of composites by potentially causing debonding within the filler and matrix system.<sup>[2,16]</sup>

The high hardness value of bulkfill composite resin could be found because of the variance in the chemical composition of the resin matrix and viscosity of monomers, and also the structural flexibility, which affects the curing depth of composite resins.<sup>[26]</sup> The filler content has been associated with different properties like depth of polymerization, colour stability, hardness, compressive strength and stiffness. Filler/matrix debonding or cracking and degradation caused by hydrolysis of the filler surface may alter the mechanical properties of composites.<sup>[27]</sup>

The introduction of nanofibers significantly increases the number of groups containing carbon-carbon double bonds in composites. However, this could potentially decrease the degree of conversion in an acceptable range. This is because while the total number of reacted double bonds from fillers and the matrix may increase, the percentage of reacted bonds could be lower due to their initially high abundance.<sup>[27]</sup>

Composite degradation when subjected to external substances, like test solutions, typically occurs through two primary mechanisms: diffusion into the polymer network and hydrolysis reactions. Diffusion entails the infiltration of the test liquid into the polymer matrix, inducing resin swelling and monomer release. Conversely, hydrolysis targets siloxane bonds, particularly those linking the silane coupling agent and silanol groups on the silica surface, leading to filler detachment. These processes collectively result in the degradation and softening of composite resins, consequently diminishing key physical and mechanical properties such as compressive strength and hardness<sup>[9]</sup>

According to the present study and within its limitations, it necessitates the use of bulk fill restorative composite resin to be used as a pre-endodontic buildup material as compared to nanohybrid posterior restorative composite resin, as it has shown less reduction in the microhardness values. However, before the final prosthetic restoration, it appears that alteration of the composite brought on by contact with NaOCl necessitates a total replacement of the pre-endodontic build-up by a permanent restoration to achieve an adequate coronal seal.

## CONCLUSION(S):

Within the limitations of the study, it can be said that contact of NaOCl with bulkfill and nanohybrid restorative composite resin causes a reduction in microhardness. It was found that the mean microhardness value for nanohybrid resin changes more as compared to bulkfill composite resin. The ability to resist masticatory forces and fractures can be reduced due to an alteration in the mechanical properties of the composite resin, especially directed towards an endodontically treated tooth. Therefore, it is necessary to replace the pre-endodontic restoration before initiating the final restoration for a better clinical outcome and long-term success of treatment. Further investigating studies with different irrigating solutions with different core build-up materials and various material properties such as water absorption, solubility, and micro leakage should also be considered.

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